APRIL 1965 ANNUAL REVIEWS

ANALYTICAL CHEMISTRY

ANALYTICAL REVIEWS

1965

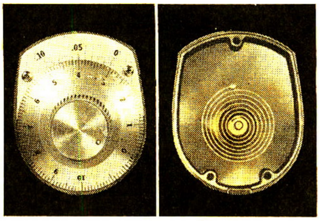
APPLICATIONS

WHAT'S NEWs . . . . from OHAUS

At OHAUS, constant research and engineering developments bring new solutions to old problems.

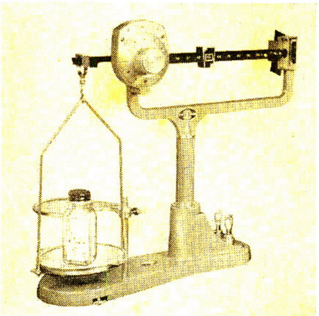
Each year the OHAUS product line reflects new conveniences and speed of operation by the inclusion of these developments.

The most recent of these has been the introduction of the "EASY TURN DIAL."

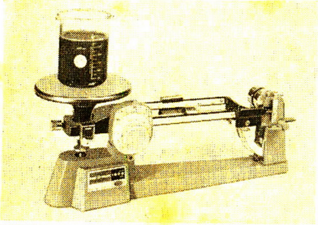


NEW DIAL BALANCES

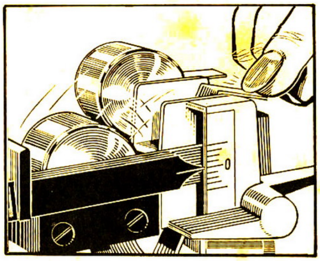
This dial actuates a special alloy temperature-compensated coil spring for true precision and repeatability over long periods of time. The dial operates under low stress thereby enhancing low hysteresis and drift characteristics for a virtually limitless life. Readings are made directly with vernier to 1 part in 1000.



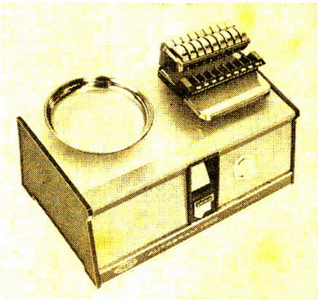
FIRST ON THE DIAL-O-GRAM 310
To the superlative features of the CENT-O-GRAM we have added new speed, accuracy and convenience with the inclusion of this new dial.



NEXT . . . THE DIAL-O-GRAM 1600
Addition of the dial to the completely new OHAUS Low Form Single Pan Balance opened up a whole new world of operational ease.



"TOUCH-N-WEIGH" UNIQUE EXTERNAL WEIGHT LOADING system is also very much a part of the completely new DIAL-O-GRAM 1600. A touch of the finger drops one or both of the 500 gram weights directly and securely into their beam pocket. When finished weighing, both are returned to their storage rack at a touch of the return key.

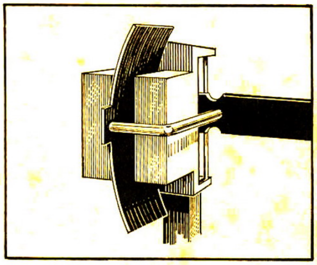


The **AUTOGRAM 1000** was the first all new balance to feature this new external weigh loading through the use of 9-100 gram weights. Also featured is the direct reading optical scale graduated to 100 grams with readability to 0.1 gram by means of a vernier.

SHARP CLEAR IMAGE is obtained by the use of high-quality optics and first-surface coated mirrors.

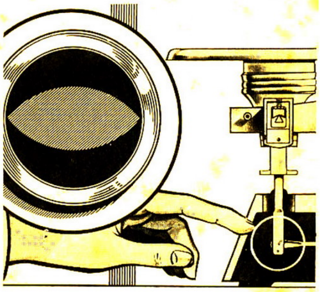
BUILT-IN TARE to 200 GRAM CAPACITY is provided by a convenient front panel control knob, "up front" where you need and want it. Additional tare is available through use of weights on the right platform. No time consuming leveling is required on this completely portable balance as the AUTOGRAM 1000 is not sensitive to out-of-level conditions.

A FRONTAL ASSAULT was also made by OHAUS engineering on the age old messy and generally impractical use of oil filled dash pots as well as other objection ridden damping systems. Again the modern clean and simple solution was found in:



MAGNETIC DAMPING

Only magnetic damping is friction-free, maintenance-free and uniform over the life of the balance. The simple use of a metal vane which is an integral part of the oscillating beam passing through a magnetic field does the trick. For your speed and convenience magnetic damping is standard equipment on the DIAL-O-GRAM 310, DIAL-O-GRAM 1600 and the AUTOGRAM 1000.



PATENTED ELIPTICAL CHECK PINS
Each check pin functions as a miniature knife edge, reducing friction and increasing accuracy.

You can depend on OHAUS ENGINEERING to continue to lead the field in the coming year so as to assure you that **"When Quality is in the Balance, that balance will be OHAUS!"**

OHAUS®

OHAUS SCALE CORPORATION
1050 COMMERCE AVE., UNION, N. J.
Dept. ZA

ANALYTICAL CHEMISTRY

April 1965, Vol. 37, No. 5
Copyright 1965 by the American Chemical Society
Issued April 12, 1965

1965 ANALYTICAL REVIEWS
APPLICATIONS

REVIEWS

AUTHORS' BIOGRAPHIES

THE AUTHORS

The analytical chemists who have contributed to this year's Review of Analytical Applications include representatives from government, industry, and universities. Many of them have written reviews for ANALYTICAL CHEMISTRY in earlier years


ALTSHULLER


SWANN


KINGSLEY


ADAMS

Air Pollution

A. P. Altshuller is the Chief, Chemical Research and Development Section, Laboratory of Engineering and Physical Sciences, Division of Air Pollution, Public Health Service at the Robert A. Taft Sanitary Engineering Center in Cincinnati, Ohio. He received his B.S. at the University of Chicago in 1948 and his M.S. and Ph.D. degrees in 1950 and 1951 from the University of Cincinnati. From 1951 to 1955 he was an aeronautical reseach scientist engaged in fuels research with what is now the Lewis Research Center of NASA. Since 1955 he has held various research assignments in the air pollution program at the Taft Center. He assumed his present position in 1961. He has published over 80 papers related to spectrophotometric analysis, gas chromatography, coulometric analysis, infrared spectrophotometry, photochemistry and kinetics, solution thermodynamics and statistical thermodynamics, thermochemistry, and various aspects of atmospheric chemistry. He is Chairman of the ACS Committee on Air Pollution and a member of the ASTM Committee on Air Pollution.

Clinical Chemistry

George R. Kingsley, Chief Biochemist, Veterans Administration Center and Clinical Professor of Physiological Chemistry, University of California School of Medicine, Los Angeles, was formerly biochemist at the Philadelphia General Hospital (1931–42) and with the U. S. Army (1942–46). He obtained his A.B. at Tusculum College, M.S. at the University of Kentucky, and went on to a fellowship at Yale University. He has published over 70 papers on the development of analytical methods in clinical chemistry and medical research. He is a member of the ACS and the American Association of Clinical Chemistry.

Coatings

Melvin H. Swann, born in Easton, Md., attended Asbury College in Kentucky, and did graduate work at the Universities of Maryland and Virginia. He has been chief of the analytical section, Coating and Chemical Laboratory, Aberdeen Proving Ground, for the past 19 years. He is responsible for quality control analysis of paints and related materials, and for research and development in the application and analysis of coating materials. His fields of interest include paints, plastics, resins, phosphate coatings, metal conditioners, and coating materials. He has approximately 43 publications to his credit.

Martha L. Adams, originally from Springfield, Ill., did her undergraduate work at the College of William and Mary, then received her M.S. from the University of Maryland. She taught general chemistry at the St. Helena Extension of the College of William and Mary before moving into her present position as analytical chemist in the Coating and Chemical Laboratory at Aberdeen Proving Ground. Her work involves chemical analysis of coatings and related materials with particular emphasis on the applications of spectrophotometric analysis to coating resins. She is coauthor of 16 papers.


ESPOSITO


GUENTHER


ROGERS


GILBERTSON

George G. Esposito, research analytical chemist at Aberdeen Proving Ground, has been working with resinous coating materials for the past 14 years. His main field of endeavor is in research and development of analytical procedures for military specifications, specializing in chromatography, colorimetry, and analytical physical separations. He received his education at Loyola College and the University of Maryland. He has had 26 papers published.

Essential Oils and Related Products

Ernest Guenther, born in Munich, Germany, attended the Technische Hochschule there as well as the University of Munich. He took his Ph.D. at the University of Zurich in 1920. Associated with Fritzsche Brothers, Inc., since 1924, he is now Senior Vice President of the company. An expert in the field of essential oils, he has published approximately 150 papers in various scientific journals and is author of six volumes of "The Essential Oils." His ACS activities include numerous lectures before local chapters throughout the United States and Canada. He expects to continue making field investigations in various parts of the world, taking new motion pictures for subsequent lectures.

James A. Rogers, Jr. is currently Director of Laboratories of Fritzsche Brothers, Inc. He received his undergraduate degree at St. Johns University, Brooklyn, N. Y. After serving three years in the U. S. Navy, he joined Fritzsche Brothers, in 1946, in the Production-Research Laboratory at the Clifton, N. J., factory. He received a M.S. degree from Stevens Institute of Technology in Hoboken, N. J. He set up and directed the company's Instrumental Laboratory in 1952, and applied instrumental analysis to all phases of flavor, perfume, and essential oil research and control. In 1962 he was appointed Director of Laboratories. His field of interest involves identification and structural determinations of essential oil constituents and related products by absorption spectrophotometric and gas chromatographic methods. He is a member of the Society for Applied Spectroscopy, Coblentz Society, and Chairman of the Instrumental Methods Group of the Essential Oil Association.

Gilbert Gilbertson is Coordinator of Laboratories at Fritzsche Brothers, Inc., and is concerned primarily with research and development, patent applications, biological testing, and the channeling of technical reports. He is a member of the Fritzsche Quarter-Century Club and has in the past held various positions with the Company in production, the Flavor Laboratories, the Analytical Laboratories, and research and development. Serving in the U. S. Navy, he studied elec-

USSARY

KOENIG

GEHRKE

SLOMAN

tronics at the U. S. Naval Research Station in Washington, D. C. He studied chemistry at Pratt Institute, Brooklyn, New York, and received his B.S. from Seton Hall University, South Orange, N. J.

Roman T. Koenig has been in charge of Technical Information and the Libraries at Fritzsche Brothers, Inc., since 1959. He is also the editor of the Fritzsche Library Bulletin, a monthly publication comprising current technical articles which services the essential oil, perfume, and flavor industries all over the world. Previously he was a member of the staff of the Technical Information Division of Esso Research and Engineering Co., and prior to that he had been engaged in physico-chemical research in industry, and taught at universities both in the United States and England. Born in Poland, he obtained his Ph.D. in chemistry in 1946 at the Imperial College of Science and Technology of the University of London. He is a member of the ACS.

Fertilizers

Charles W. Gehrke is Professor of agricultural chemistry and Supervisor of the Experiment Station Chemical Laboratories at the University of Missouri. In addition to teaching and research in the field of analytical biochemistry, his duties include those of State Chemist for the Missouri Fertilizer Control Law. He received his B.A. degree from Ohio State University in 1939, a B.Sc. degree in Education in 1941, and a M.Sc. degree in 1941. From 1941 to 1945 he was Head of the Department and Professor of chemistry at Missouri Valley College. He returned to Ohio State in 1946 as instructor in agricultural biochemistry and received his Ph.D. in 1947. He assumed his present position at the University of Missouri in 1949. He is active in the ACS, AOAC, and IFT. His research interests include the development of quantitative gas chromatographic methods for amino acids, nucleosides, fatty acids, and residues; characterization and interactions of proteins; and automation of analytical methods for biological molecules. He is author of at least 75 publications in analytical and biochemistry.

James P. Ussary is Instructor of agricultural chemistry and Chemist in the Experiment Station Chemical Laboratories at the University of Missouri. His duties include supervision within the state fertilizer control laboratory, and development of automated analytical methods. He received his B.S. degree from the University of Missouri in 1960. Following two years in industry, he returned to the University of Missouri where he received his M.S. in 1964 and joined the University staff at this time. His current interests include the development of automatic analytical methods for K_2O and P_2O_5 in fertilizers, and biochemical molecules in plant and animal materials.

Food

Katherine G. Sloman, Research Specialist, Analytical Chemistry,

BORKER

BEEGHLY

PASZTOR

at General Foods Technical Center, (B.A., Smith College and M.A., Columbia University) has specialized in the applications of analytical procedures to foods. She has had wide experience with the standard methods of food analysis, and with the problems encountered both in method development for specific analytical problems, and for the needs of quality control. Recently she has worked on special methods required for the determination of food additives, and for other trace components in foods.

Emanuel Borker, Group Leader, Analytical Services, General Foods Technical Center received his bachelor's degree in 1941 from Brooklyn College. After holding positions as an analytical chemist with Columbia Cheese Company and Calvert Distilling Corp., he joined General Foods in 1946 and has been with the Corporate Research Department and Maxwell House Division. Publications cover primarily the determination of minor and trace constituents of food products. He is an Associate Referee of the Association of Official Agricultural Chemists and a member of ACS, AAAS, Institute of Food Technologists, and ASTM Committee E-15.

Ferrous Metallurgy

Hugh F. Beeghly has made numerous contributions to analytical chemistry and metallurgy since joining the Jones & Laughlin Steel Corp. upon graduation from West Virginia University (B.S., '36). He is now research associate in nuclear technology with the company, has published more than 40 papers, and contributed chapters to several books. He was the recipient of a special merit award of the Pittsburgh Junior Chamber of Commerce for "outstanding service to chemistry in Pittsburgh during 1949." He has been active in his local section of the ACS since 1940 as Councilor, Editor of *The Crucible,* and Chairman of the Pittsburgh Section. Other professional groups with which he has worked include AIChE, AIME, ANS, AISI, ASTM, Spectroscopy Society of Pittsburgh, and the Pittsburgh Diffraction Society. Currently he is secretary of the Pittsburgh Section ANS and of the Pittsburgh Section's ACS Affairs Committee.

Laszlo C. Pasztor, Research Supervisor at the Jones & Laughlin Steel Corp., Pittsburgh, Pa., was born in Hungary. He was graduated (1943) and received his M.S. (1954) from the University of Science of Budapest and currently is doing graduate work at the University of Pittsburgh. His work at Jones & Laughlin Steel Corp., Graham Research Laboratory, over the last eight years has involved research and development of analytical procedures for the steel industry. His publications are mainly in the field of solvent extraction separations and spectrophotometric methods. He is a member of the ACS, the Spectroscopy Society of Pittsburgh, the SAS, and an active member of the Society for Analytical Chemists of Pittsburgh.

WILL

BACHMAN

BANKS

Nonferrous Metallurgy. I. Light Metals

Fritz Will, III, was born in Richmond, Virginia. After service in the U. S. Army during World War II, part of which was spent at the University of Maryland and North Carolina State College, he received a B.S. degree in Chemistry (1949), his M.S. (1951), and his Ph.D. in Analytical Chemistry (1953) from the University of Virginia. While at the University of Virginia he was a member of Alpha Chi Sigma, Sigma Phi Epsilon, Sigma Xi, and the recipient of the American Institute of Chemists Medal and a scholarship award from the Virginia Section of the ACS. He was a John L. Pratt Research Fellow in Analytical Chemistry (1951-53). In 1953 he joined the Analytical Chemistry Division of the Alcoa Research Laboratories in New Kensington, Pennsylvania. His research interests include absorption, reflectance, and fluorometric spectroscopy, countercurrent distribution, adsorption chromatography, trace analysis, and gas analysis, from which he has published several articles. He is a member of ACS and its Division of Analytical Chemistry, the Pittsburgh Chemists Club, and RESA. He was Secretary and Chairman of the Society for Analytical Chemists of Pittsburgh and was Chairman of the 1962 Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy.

Nonferrous Metallurgy. II. Zirconium, Hafnium, Vanadium, Niobium, Tantalum, Chromium, Molybdenum, and Tungsten

Robert Z. Bachman is an assistant chemist with the Ames Laboratory of the U. S. Atomic Energy Commission. He received the B.S. degree in chemistry from Iowa State University in 1951 and has been employed by the Ames Laboratory since that time. The past nine years he has supervised the established methods laboratory of the analytical chemistry group in which capacity he has been in charge of the wet chemical service analyses. In this position his principal interest is the analytical chemistry of those metals covered by this review along with titanium, thorium, uranium, and the rare earths. He and Dr. Banks are coauthors of a review of the wet chemical methods for the separation and determination of titanium, zirconium, hafnium, vanadium, niobium, tantalum, chromium, molybdenum, and tungsten for "Progress in Nuclear Energy, Series IX, Analytical Chemistry" and a chapter discussing wet chemical methods for the separation and determination of thorium in "Analysis of Essential Nuclear Reactor Materials." He is a member of the ACS.

Charles V. Banks is Professor of chemistry at Iowa State University and senior chemist with the Ames Laboratory of the Atomic Energy Commission. His research interests include fundamental studies of the factors responsible for the selectivity of organic analytical reagents; solution chemistry of metal ions; and separation techniques. He is active in the determination of gases and other trace impurities in metals and metal salts and in the

GOWARD

COOK

WILLIAMS

general analytical chemistry of such elements as U, Th, rare earths, Ti, V, Cr, Zr, Nb, Mo, Hf, Ta, and W. He has published over 100 research papers over the past 18 years. Dr. Banks is a member of several honorary and scientific societies including the ACS and AAAS.

Vacuum Fusion and Vacuum Extraction Analysis for Nitrogen in Metals. Basic Principles and Current Status

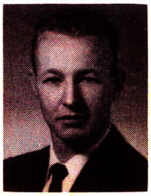
G. W. Goward is Technical Supervisor of Oxidation and Coatings at the Advanced Materials Research and Development Laboratory, Pratt & Whitney Aircraft, North Haven, Conn. Dr. Goward received his B.S. from the University of Alberta in 1951 and his Ph.D. in analytical chemistry from Princeton University in 1954. He came to his present place of employment after spending seven years at Westinghouse where his work involved the analytical chemistry of reactor materials. His research interests include analysis for gases in metals and high temperature oxidation and coating protection of gas turbine engine materials. Dr. Goward is a member of ACS, Sigma Xi, and ASTM.

Pesticide Residues

J. William Cook is Deputy Director of the Division of Food Chemistry, Bureau of Scientific Research, Food and Drug Administration, Washington, D. C. He received his B.S. degree in 1935 and his M.S. degree in 1938 from Oregon State College. He worked in the field of poultry nutrition at Washington Agricultural Experiment Station before becoming a member of the Food and Drug Administration in 1939. Since then he has served in various capacities in FDA, the past 13 years in pesticide chemicals. For a number of years he was responsible for the evaluation of the chemistry, methods of analysis, and residue data presented in petitions submitted for tolerances for pesticide chemicals under the Miller amendment. He has published approximately 30 scientific papers primarily on the chemistry and metabolism of pesticide chemicals. He is a member of American Chemical Society, Institute of Food Technology, and Phi Lambda Upsilon. He is a member of the advisory board of the series of books entitled "Residue Reviews." He has served as United States delegate to an FAO Conference on the use of pesticide chemicals in agriculture and is a member of the FAO working party on methodology and residue data for pesticide chemicals. He has been appointed a titular member of the Pesticide Section of the Division of Applied Chemistry, International Union of Pure and Applied Chemistry.

Sidney Williams is Assistant to the Chief, Pesticides Branch, Division of Food Chemistry, U. S. Food and Drug Administration, Washington, D. C. Mr. Williams received his B.S. and M.S. degrees in chemistry from the University of Massachusetts. Since joining the Food and Drug Administration in 1941, he has served in a number of capacities at various locations throughout the United States. Much of this work was concerned with the analysis of pesticide residues in many different

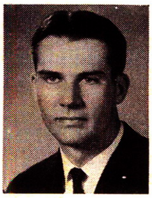
WIMER



PAPENDICK

GAMBRILL

THEIVAGT



WADELIN

food products. For a number of years he was group leader of a team analyzing for pesticide residues. In his present position he assists in the supervision of the development of methods of analysis for pesticide residues and evaluation of data developed from those methods. He is a member of the American Chemical Society and the American Association for Advancement of Science.

Petroleum

Charles M. Gambrill, Analytical Coordinator, Technical Service, Petroleum Chemicals Division, Ethyl Corporation, has been active in analytical work since obtaining his bachelor's degree from the University of Maryland in 1924. He was employed by the Hagan Corporation and the Mathieson Alkali Works, Inc., prior to joining Ethyl in 1928. He is the author of several papers in the field of petroleum analysis. In technical societies he has been active in the ACS, the ACS Analytical Division and the local Section: in ASTM, particularly in its Committee D-2 on Petroleum Products, receiving in 1960 the ASTM Award of Merit in recognition of his work; in API on the Committee on Analysis; in CRC on analysis of engine exhausts; and the Association of Analytical Chemists (Detroit) receiving the Anachem Analytical Award in 1958 on his contribution to analytical chemistry. The Petroleum Reviews have been coordinated by Gambrill, and they present the contributions of twenty specialists in the field of petroleum analyses.

Pharmaceuticals and Related Drugs

D. C. Wimer is a senior chemist in the Analytical Research Department of Abbott Laboratories. He received his B.S. from the University of Illinois in 1951. He is the author of several papers in the field of nonaqueous titrimetry. His interests lie in the areas of functional group analysis, acid–base interactions, and nonaqueous solvent chemistry. He is a member of the ACS and Sigma Xi.

J. G. Theivagt is a senior chemist in the Analytical Research Department of Abbott Laboratories. He received his B.S. in 1952 from the University of Illinois. He is the author of several papers in the field of pharmaceutical analysis, and his major interests are chromatographic techniques and functional group methods as applied to formulation quality control and stability. He is a member of the Analytical Chemistry Division of the ACS, AAAS, and Sigma Xi.

V. E. Papendick is a chemist in the Analytical Research Department of Abbott Laboratories. He received his B.S. from Michigan State University in 1958. His research interests include spectrophotometric, colorimetric, polarographic, and functional group methods of analysis. He has coauthored several papers using ultraviolet spectrophotometry. He is a member of ACS.

Rubber

Coe W. Wadelin is a graduate of Mt. Union College (B.S., 1950) and Purdue University (M.S., 1951; Ph. D., 1953). Since 1953 he has

ABERNETHY

WALTERS

SKOUGSTAD

FISHMAN

been a chemist in the Research Division of the Goodyear Tire and Rubber Co. in Akron, Ohio, where he is concerned with the analysis of rubber and rubber chemicals. He is a member of the ACS and ASTM.

Solid and Gaseous Fuels

Roy F. Abernethy, Chemist-in-Charge, Tests Methods, Coal Utilization, Pittsburgh Coal Research Center, Bureau of Mines, came to Pittsburgh in 1930. He received the B.S. and M.S. degrees in chemistry from his home state University of North Carolina. Most of his work has been in the development of methods for and analysis of coal, coke, and related materials, having more than 60 papers published. He has been a member of ACS since 1935, attended ISO meetings in England on coal analysis in 1953 and 1958, and in Germany in 1964 and is Secretary of ASTM Committee D-5 on Coal and Coke.

Jack G. Walters, Research Chemist, Coal Utilization and Preparation, Pittsburgh Coal Research Center, Bureau of Mines, joined the staff in 1948. Undergraduate work was taken at Juniata College (B.S., '48) and graduate work at the University of Pittsburgh. As a research chemist he has been concerned with the mechanism and kinetics of the thermal decomposition of coal, coal plasticity, development of analytical methods for liquid and gaseous products of carbonization, and surveying the carbonization characteristics of American coals. He is a member of ACS and AAAS.

Water Analysis

Marvin W. Skougstad, born in Beloit, Wis., has been employed by the Water Resources Division, U. S. Geological Survey, Denver Colo., since 1958. He received his B.S. in chemistry (1939) and Ph.D. in chemistry and metallurgy (1949) from the University of Wisconsin. After receiving his B.S. degree, he became a chemist with the Washburn Co., Rockford, Ill. Two years later he joined the staff of Fairbanks-Morse and Co., Beloit, Wis. as a chemist. He served there from 1941 to 1946. After receiving his Ph.D., he became an associate professor of chemistry at St. Olaf College, Northfield, Minn., and held this position until he joined the U. S. Geological Survey. His research interests are centered on occurrance and distribution of minor elements in water, development of methods for water analysis, and spectrographic methods for determining minor elements in water. He is a member of the ACS and the Society for Applied Spectroscopy.

Marvin J. Fishman, born in Denver, Colo., received his B.A. degree (1954) and M.S. degree (1956) from the University of Colorado. He has been employed by the Water Resources Division, U. S. Geological Survey, Denver, since 1956. His research interests are centered on development of methods for water analysis, including atomic absorption, absorption of trace elements on clay minerals, and chemistry and behavior of manganese and iron in natural water. He is a member of the ACS and the Society for Applied Spectroscopy.

April 1965, Vol. 37, No. 5
ANALYTICAL CHEMISTRY

Washington, D. C. 20036
1155 Sixteenth St., N. W.

Phone: 202-737-3337 Teletype WA 23

Editor, LAWRENCE T. HALLETT

Assistant Editors: John K. Crum, Patricia A. Morgan, Josephine Pechan, Virginia E. Stewart

Contributing Editor: R. H. Müller

Production Staff

Art Director: Joseph Jacobs

Associate Editor: Charlotte C. Sayre

Assistant Editor: Elizabeth R. Rufe

AMERICAN CHEMICAL SOCIETY PUBLICATIONS

Director of Publications, Richard L. Kenyon

Assistant Director of Publications and Director of Research Journals, Richard H. Belknap

Director of Business Operations, Joseph H. Kuney

Executive Assistant to the Director of Publications, Rodney N. Hader

Assistant to the Director of Publications William Q. Hull

REGIONAL EDITORIAL BUREAUS

NEW YORK, N. Y. 10017
733 Third Ave.

PHILADELPHIA, Pa. 19103
1725 John F. Kennedy Blvd.

PITTSBURGH, Pa. 15230
530 William Penn Place

CHICAGO, Ill. 60603
36 South Wabash Ave.

CLEVELAND, Ohio 44114
1367 East Sixth St.

SAN FRANCISCO, Calif. 94104
57 Post St.

LOS ANGELES, Calif. 90005
422 South Western Ave.

HOUSTON, Tex. 77002
1121 Walker St.

FRANKFURT/MAIN, West Germany
32 Grosse Bockenheimerstrasse

LONDON, W. 1, England
77 South Audley St.

TOKYO, Japan
Apt. 306, 47 Dai-machi, Akasaka, Minato-ku

WASHINGTON, D. C. 20036
1155 Sixteenth St., N. W.

Advertising Management

REINHOLD PUBLISHING CORP.

ANALYTICAL REVIEWS

1965

APPLICATIONS

1965 Review of Analytical Applications

THIS IS THE 17TH ANNUAL REVIEW ISSUE. Our policy of publishing applications reviews and fundamental reviews on an alternate year basis is still being followed. The objective of the 1965 review issue is to present the significant developments in analytical applications during the past two years. This issue contains 260 pages and consists of 16 reviews. The Buyers' Guide, which in previous years was mailed with the review issue, will be mailed with the regular July issue this year.

The petroleum review merits special comment because 20 specialists and a coordinator were required to prepare the review in this complex field. A special review, "Vacuum Fusion and Vacuum Extraction Analysis for Nitrogen in Metals. Basic Principles and Current Status," deals with a rather specific topic which the editors feel is important but which does not represent a continuing review.

As in the past, the reviewers have attempted to be critical and selective rather than all-inclusive in their coverage of developments in the last two years. We thank the authors for their efforts in preparing these reviews. The work involved in collecting the source material, assembling it, and putting it in logical sequence results in a major contribution to the field of analytical chemistry.

L. T. Hallett

MANUSCRIPT REQUIREMENTS published in December 1964 issue, page 2559, outlines scope and copy requirements to be observed in preparing manuscripts for consideration. Manuscript (3 copies) should be submitted to the Editor, 1155 Sixteenth St., N.W., Washington, D. C. 20036.

1965 Subscription Rates

	1 yr.	2 yr.	3 yr.
Members, domestic and foreign	$ 4.00	$ 7.00	$10.00
Nonmembers, domestic and Canada	5.00	9.00	12.00
Nonmembers, foreign except Canada	15.00	27.50	40.00

Postage: Canada, and Pan-American Union, $2.50; foreign, $3.50. Single copies: current, $1.50. Postage: Canada and Pan-American Union, $0.15; foreign, $0.20. Rates for back issues and volumes are available from Special Issues Sales Dept., 1155 Sixteenth St., N.W., Washington, D. C. 20036. Claims for missing numbers will not be allowed if received more than 60 days from date of mailing plus time normally required for postal delivery of journal and claim. No claims allowed because of failure to notify the Subscription Service Department of a change of address, or because copy is "missing from files."

Published monthly with review issue added in April by the American Chemical Society, from 20th and Northampton Sts., Easton, Pa., 18043. Executive offices, Editorial Headquarters, and Subscription Service Department, 1155 Sixteenth St., N.W., Washington, D. C. 20036. Advertising Office, 430 Park Ave., New York, N. Y. 10022. News Bureaus: 733 Third Ave., New York, N. Y. 10017; 1725 John F. Kennedy Blvd., Philadelphia, Pa. 19103; 530 William Penn Place, Pittsburgh, Pa. 15230; 36 South Wabash Ave., Chicago, Ill. 60603; 1367 East Sixth St., Cleveland, Ohio 44114; 57 Post St., San Francisco, Calif. 94104; 422 South Western Ave., Los Angeles, Calif. 90005; 1121 Walker St., Houston, Tex. 77002; 1155 Sixteenth St., N.W., Washington, D. C. 20036; Grosse Bockenheimerstrasse 32, Frankfort/Main, West Germany; 77 South Audley St., London, W. 1, England.

Second class postage paid at Easton, Pa.

CHANGE OF ADDRESS: Notify Subscription Service Department, American Chemical Society, 1155 Sixteenth St., N.W., Washington D. C. 20036. Such notification should include both old and new addresses, with ZIP code numbers, and be accompanied by mailing label from a recent issue. Allow four weeks for change to become effective.

Air Pollution

A. P. Altshuller

Laboratory of Engineering and Physical Sciences, Division of Air Pollution, Robert A. Taft Sanitary Engineering Center, Public Health Service, U. S. Department of Health, Education, and Welfare, Cincinnati, Ohio 45226

Most of the work covered in this review was published or presented between October 1962 and October 1964. Some references are included even though they were published before October 1962 because the studies appeared in journals that are not available on a current basis. Conversely, references from readily available journals are included to December 1964. The previous review (*8*) included papers published or presented between January 1961 and October 1962, with a few more recent references.

Methods of analysis for inorganic and organic pollutants in urban atmospheres are of about equal interest, as indicated by the number of publications. The greatest amount of work has been directed toward analyses for inorganic sulfur compounds, oxidants, C_1 to C_{10} hydrocarbons, and polycyclic hydrocarbons. About half of the papers reporting analyses for organic gases and vapors entailed gas chromatographic techniques. Entirely new methods have been developed for the analyses of aza heterocyclic compounds and of peroxyacyl nitrates. A number of detailed analytical studies were made of hydrocarbons, oxygenated organic substances, and lead in automobile exhaust.

Symposia and general sessions on air pollution were held at the spring and the fall meetings of the American Chemical Society in 1963 and 1964. The majority of presentations at these meetings dealt with analytical chemistry and closely related topics. Papers reporting varied applications of analytical techniques to air pollution were presented at the 1963 and 1964 meetings of the Air Pollution Control Association and the American Industrial Hygiene Conference. The California State Department of Public Health sponsored conferences on analytical methods in air pollution studied in 1963 and 1964. Proceedings of the National Conference on Air Pollution held in Washington, D. C., on December 10–12, 1962, have been published (*210*). One session was concerned with the application of measuring and monitoring results.

A monograph volume on atmospheric chemistry by Junge contains a chapter on polluted atmospheres (*136*). Methods of analysis used in the United States for polluted atmospheres have been reviewed by Lodge and others (*175*). Another volume of the U.S.S.R. literature on air pollution is available (*164*).

The place of data-gathering techniques and monitoring equipment in various air monitoring programs has been discussed (*258*). A compilation of chemical indicator systems is available (*46*). ASTM Committee D-22 late in 1962 prepared a new edition of ASTM standards for sampling and analysis in air pollution.

BIOLOGICAL INDICATORS

A wide variety of biological substrates are being used to detect air pollutants in the laboratory. Plants, particularly varieties of tobacco wrapper, petunia, and pinto bean, are used in both laboratory and field. Apparent photosynthesis, morphological or biochemical changes in bacterial cultures, immobilization of paramecium, inhibition of cell growth in vitro, and human eye irritation all have been used as indicators of various pollutants.

Concern about obtaining quantitative results in plant-damage studies has stimulated investigators to study the environmental factors influencing reproducibility. Damage to pinto bean caused by ozone and peroxyacetyl nitrate has been investigated to determine the effects of length of exposure to light before, during, and after fumigation; intensity of light; and age of plants (*73*). The influence of drought conditions, nutrient, soil, and dark conditioning on degree of damage to tobacco wrapper, pinto bean, and petunia exposed to irradiated auto exhaust has been investigated (*245*). Damage to tobacco wrapper varieties caused by ozone was measured as a function of light, temperature, ozone concentration, and leaf maturation (*183, 184*). A variety of factors including reactant concentrations; light before, during, and after fumigation; length of previous dark period; and temperature were used to study damage to cotton, pinto bean, and endive by the products of irradiated propylene–nitrogen dioxide mixtures (*105*).

The more subtle effects of pollutants on plant growth can be investigated by measurements of the rate of fixation of carbon dioxide (*72*). Both ozone and peroxyacetyl nitrate adversely effect photosynthesis and carbon dioxide exchange (*270*).

Oxidant (PAN) type damage along with several other patterns of damage were observed when tobacco wrapper, pinto bean, and petunia were exposed to the products from irradiation of 1-butene–nitric oxide, 1,3,5-trimethylbenzene–nitric oxide, and propionaldehyde–nitric oxide mixtures (*146*). At the same concentrations and conditions of exposure, these same plant varieties showed no damage when fumigated with the products from ethylene–nitric oxide, 1,3-butadiene–nitric oxide, toluene–nitric oxide, formaldehyde–nitric oxide, or paraffinic hydrocarbon–nitric oxide mixtures (*146*).

Damage to petunia in the field has been attributed to atmospheric aldehydes (*34, 35*). Laboratory irradiations with propionaldehyde in air and propionaldehyde–nitric oxide mixtures also produced injury to petunia, but the pattern of injury was not the same as in the field (*115*). Different varieties of petunia were used in the two investigations.

The leaf injury to several varieties of tobacco wrapper has been measured with ozone, ozonated hexene-1, and ambient air systems (*106*). Differences in susceptibility to ozone damage were observed in plants grown in the eastern and western United States.

Injury to tobacco wrapper, pinto bean, and petunia has been used to evaluate results from irradiations of various automobile exhaust mixtures (*149*). Damage usually decreased with decreasing hydrocarbon concentration and decreasing ratio of hydrocarbon to nitrogen oxide; no damage could be detected at ratios of 3:1.

Several groups of investigators have used bacteria for bioassaying pollutants. The reduction in maximum luminescence and changes in cell viability in *Photobacter sp.* has been used to study reactant variations in the irradiated *cis*-2-butene–nitric oxide system (*246*). Inhibition of growth of *E. coli* has been measured after exposure to the products of irradiated 1-butene–nitrogen dioxide mixtures (*83*). Morphological and biochemical changes in bacterial cultures have been used in bioassaying polycyclic hydrocarbons (*275*).

The immobilization of *Paramecium candatum* when exposed to long-wavelength ultraviolet radiation also can be used to bioassay polycyclic hydrocarbons (*80, 81*). Only the polycyclic hydrocarbons with carcinogenic properties tend to immobilize the paramecium.

The inhibition of growth of cells in vitro has been used for bioassay (*277*). The effects on inhibition of growth were measured for sulfur dioxide, sodium sulfate, sodium sulfite, and sodium bisulfite.

Measurement of eye irritation of human subjects continues as an important technique for evaluating the biological effects of various photochemical systems. Measurements both with panels and individual were obtained to evaluate eye irritation as a function of formaldehyde concentration formed from the irradiation of ethylene and propylene–nitrogen oxide mixtures (*243*). Nonlinear responses were reported at concentrations below 0.3 p.p.m. of formaldehyde. Blink rate appeared to be related to intensity of eye irritation at low irritant concentrations. In another study at low concentrations of irradiated auto exhaust, eye irritation continually decreased with decreases in hydrocarbon and formaldehyde concentrations (*149*). In this same study eye irritation peaked at about 1 p.p.m. of nitrogen oxides.

SAMPLING AND CALIBRATION METHODS

Large amounts of particulate collected in air filtration plants provide useful samples for subsequent detailed analysis (*170, 171*). Methods for collecting atmospheric sulfates have been discussed (*215*). The sampling of both particulate and gaseous fluorides on glass-fiber filters has been investigated (*205*). Collection on glass-fiber filters gave results in good agreement with those obtained by impinger collection. An aluminum-tube technique was used to remove hydrogen fluoride; membrane filters removed particulate but not gaseous fluoride (*205*).

Huygen has published a series of papers on use of impregnated filter paper techniques for sampling sulfate, sulfur dioxide, hydrogen fluoride, and hydrogen sulfide (*125–127*). This work included evaluations of the collection efficiencies and capacities of filters impregnated with various solutions, and of the stabilities of collected samples with variations in concentration, temperature, and humidity. The results obtained with impregnated filters compared favorably with those obtained with impingers (*125, 126*). The use of such treated filter papers was discussed in conjunction with automatic tape samplers and automatic analysis techniques (*125*). The suitability of impregnated membrane filters for collecting gases has been considered by Lodge and coworkers (*174*). The collection efficiency and capacity of impregnated membrane filters have been determined for traces of sulfur dioxide in air (*207*).

The adsorption of high-molecular-weight hydrocarbons in the inlet sampling system of flame ionization analyzers results in long response times (*122*). Increasing sample system temperatures to 150° C. decreases the response time to samples such as auto exhaust appreciably, but some adsorption still occurs at 150° C.

Silica gel continues to find considerable use as an adsorbent, particularly for aromatic hydrocarbons (*45, 61, 77, 294*), other aromatic substances (*45*), and chlorinated hydrocarbons (*82, 151*). Silica gel impregnated with various reagents and dispersed in an air stream has been used for the collection and analysis of sulfur dioxide, arsine, and aniline (*166*). Desorption of substances such as *n*-butane, dichlorodifluoromethane, and atmospheric gases from charcoal has been enhanced by use of carbon tetrachloride (*191*). Methods for preparing brominated charcoal have been discussed by Turk and coworkers (*192*), who also have developed a gas chromatographic technique for determining the efficiency of brominated charcoal for removal of olefins from gas streams (*75*). The utility of a trap containing dimethyl sulfolane on C-22 firebrick maintained at ice-water temperature has been evaluated for collecting lower hydrocarbons (*247*). This same substrate and other types of gas liquid partition substrates have received considerable attention in the past for the same application (*9*). Sampling of large polar molecules such as nitrobenzene, benzaldehyde, and *n*-butanol has been done with good efficiencies by use of collecting tubes containing silicone elastomer or polyethylene glycol on celite (*61*). In another study silicone grease on C-22 firebrick in glass tubes was used in concentrating furfurol, nitrobenzene, phenol, etc., with subsequent quantitative analysis (*15*).

Flexible containers fabricated from plastic films are being used for sampling (*10, 52, 59, 280*) and for calibration (*138, 156*). The stability of sulfur dioxide, nitrogen dioxide, ozone, and hydrocarbons was measured in two types of plastic containers (*59*).

Aliphatic hydrocarbons have been collected and stored satisfactorily for up to 10 days in plastic containers (*52*). A variety of flexible containers, including rubber bladders, can be used for collecting and storing atmospheric methane (*10*). Gas mixtures in plastic containers are satisfactory for the calibration of monitoring devices and other instrumentation (*138, 156*). Mixtures of sulfur dioxide in plastic bags were used in evaluation of membrane filter techniques (*207*). Samples of chlorinated hydrocarbons collected in silica gel were desorbed into plastic bags and then transferred to an infrared cell for analysis (*82*).

Several dynamic calibration methods for sulfur dioxide and nitrogen dioxide have been described (*70, 153, 217, 222*). A multiple flowmeter dilution system (*70*) and motor-driven syringe techniques (*153, 217*) have been used. Metering of an analyzed cylinder mixture of nitrogen dioxide in air by an asbestos flowmeter through a compact flow dilution system provided precise control over the final mixtures in the parts per million range (*222*).

Most calibration systems for gases ultimately involve use of a sample from a gas cylinder. Nitric oxide has been generated into a gas stream by electrolysis (*113*). Methods for the rapid conversion of nitric oxide to nitrogen dioxide also were considered.

PARTICULATES AND AEROSOLS

Inorganic Particulates. Methods of collection and analysis of lead have been reviewed (*49*). A detailed discussion has been given of the double extract–mixed color dithizone procedure for lead (*142*). The dithizone procedure for lead has been employed for the rapid analysis of particulate lead and organolead collected on a membrane filter followed by an iodine scrubber (*85*). The x-ray spectrographic analysis of lead collected on millipore filters was reported (*273*). Lead has been determined both colorimetrically and polarographically after collection on celluloid filters (*200*). Microchemical techniques with millipore filters were employed to identify and estimate size of lead oxide particles (*91*). A spectrographic technique is available for beryllium in air and other types of samples (*143*). Selenium has been determined by a ring-over procedure applicable to airborne particulates (*293*). Activation analysis has been used for the qualitative analysis of a number of metals in particulate material (*206*). Polarographic techniques have been developed for determining a number of heavy metals in air samples (*68*) and for cyanide (*185*).

Sulfuric acid droplets have been studied by optical and electron microscopy (*286, 287*). Sulfuric acid was identified by reaction with thymol blue coated slides in a cascade impactor and on impregnated filter paper following the impactor (*286*). Size distribution also was determined. A titration procedure for sulfuric acid in atmospheric samples has been described and possible interference by acidic and basic substances has been discussed (*58*). The content of sulfuric acid in the presence of metal sulfates has been reported (*285*). An improved millipore filter technique has been developed to identify and size individual sulfate particles (*173*). Both atmospheric sulfur dioxide and sulfuric acid plus sulfate particles have been determined by a ring-oven technique (*128*). Although the results for sulfur dioxide were in reasonable agreement with those obtained by the pararosaniline method, the results for sulfuric acid and particulate sulfate did not agree with those obtained by the turbidimetric method. Sulfur trioxide and sulfate in the micromole range have been analyzed by a modification of the barium–rhodizonate method (*261*).

Aerosols. Much of the laboratory work on photochemical aerosols related to auto exhaust up to 1963 has been reviewed (*67*). Several recent investigations indicate that the principal constituent of the aerosol formed by irradiating mixtures of sulfur dioxide with lower molecular weight olefins and nitrogen dioxide is sulfuric acid aerosol (*79*, *212*). It has been known for many years, however, that aersols that must have organic content are formed when certain higher-molecular-weight hydrocarbons are irradiated with nitrogen oxide in the absence of sulfur dioxide. More detailed studies of the formation of such aerosols produced when higher-molecular-weight acyclic and cyclic olefins or aromatic hydrocarbons are irradiated in the presence of nitrogen oxides have been made recently (*96*, *146*, *212*, *259*). It has been suggested that sulfuric acid aerosol does not play a major role in the visibility reduction experienced during high levels of the photochemical type of air pollution (*103*).

Organic Particulates. Caffeine has been determined at atmospheric concentrations by the ring-oven technique (*203*). The paper chromatographic isolation of nuclides in air has been described (*292*).

A review was made of the reactivity, concentration ratio of polynuclear aromatic hydrocarbons, and other aspects of analyses of the organic particulate fraction (*141*). The separation and analysis of polynuclear aromatic hydrocarbons present in urban atmospheres and various sources of pollution have been discussed (*226*).

Additional studies have been made of the separation of polynuclear aromatic hydrocarbons by paper and column chromatography (*50*, *109*, *271*). Combinations of paper or column chromatographic and spectrophotometric techniques continue to be investigated for the separation and analysis of benzo[*a*]pyrene and other polynuclear aromatic hydrocarbons (*189*, *295*, *300*). Selective fluorescent quenching has been investigated as a technique for the identification of various polycyclic hydrocarbons after paper chromatographic separation (*110*). Thin-layer separations and various analytical techniques including fluorescent quenching have been applied in the separation and analysis of benzo[*a*]pyrene and other polynuclear aromatic hydrocarbons (*228*, *235*, *241*). A technique is now available for direct spectrophotofluorometric and spectrophosphorimetric analysis of polycyclic hydrocarbons as well as aza heterocyclics on thin-layer chromatograms (*232*, *238*). Gas liquid partition chromatography has been applied following partition procedures to the separation of polynuclear aromatic hydrocarbons in coal tar (*167*, *168*) and air particulate samples (*165*). Benzo[*a*]pyrene is not separated from benzo[*e*]pyrene on the substrates used.

Selective quenching in various solvent systems has been studied systematically by Sawicki and coworkers for various classes of polynuclear compounds (*234*, *237*). The term quenchofluorometric was proposed for this type of analysis. Low-temperature fluorescence and phosphorescence spectra can be used to characterize a wide variety of polycyclic aromatic and heterocyclic compounds (*229*).

Methods for the separation and analysis of carbazoles, acridines, and quinolines have been developed by Sawicki and coworkers. Some members of these classes of substances, such as some of the polynuclear aza heterocyclic hydrocarbons, are known to be carcinogenic to animals. Thin-layer chromatography can be used for the separation of these types of compounds (*230*, *233*, *240*). Spectrophotofluorometry and spectrophosphorimetry have been used in the subsequent analyses (*25*, *26*, *232*). Quenchofluorometric methods allow the characterization and analysis of aza heterocyclics, polynuclear amines, imino heterocyclics, or polynuclear hydrocarbons in the presence of the other classes of compounds (*237*). Thin-layer chromatography has been used with spectrophotofluorometric analysis to determine polynuclear quinolines and acridines in coal-tar pitch and in air near tarring operations (*233*). Analyses have been made for carbazoles in urban air and coal-tar fumes (*26*).

INORGANIC GASES

Considerable work continues on the analyses of inorganic gases, particularly sulfur oxides and oxidants. A number of portable instruments have been developed for analyses of inorganic gases. Automation of colorimetric chemical procedures in the laboratory has been discussed (*299*).

Interference problems have been considered in various methods for continuous air analyses (*194*). A condensation nuclei instrument (*248*) can be used with appropriate reactions to convert gases to airborne particles for analyses of a variety of gases, but nitrogen dioxide and ammonia received particular consideration (*282*). Argon ionization detectors using helium carrier gas can be applied to analyze for inorganic gases such as hydrogen, carbon monoxide, and carbon dioxide in the part per million range (*30*, *218*). Gas-solid chromatography with thermal conductivity detection has been used to analyze the carbon monoxide, carbon dioxide, hydrogen chloride, chloride, and phosgene produced in burning chlorinated fire-retardant building paper (*88*). Narrow-band absorption infrared, ionic-amperometric, and radioactive clathrate detectors have been discussed as possibilities for use in trace analysis (*66*).

Carbon Monoxide and Carbon Dioxide. Two new colorimetric procedures are available for determining carbon monoxide in the range of 5 to 1500 or 1800 parts per million (*40*, *163*). A portable conductometric apparatus for atmospheric carbon monoxide has been developed (*284*). Simultaneous determinations of carbon monoxide and carbon dioxide can be made by use of infrared spectrophotometry (*44*).

Sulfur Dioxide and Sulfur Trioxide. A review of analytical methods for sulfur dioxide is available (*119*). A new spectrophotometric method for sulfur dioxide based on its reaction with ferric ion and 1,10-phenanthroline (*256*) has been developed. A number of investigations have been made of various aspects of the determination of sulfur dioxide by the West-Gaeke pararosaniline procedure. The mechanism of reaction was investigated (*123*). The elimination of nitrogen dioxide interference can be best achieved by addition of *o*-toluidine or sulfamic acid during analysis (*208*, *305*). The interference of heavy metals, particularly iron, was eliminated by use of EDTA (*305*). An interlaboratory comparison of the West-Gaeke analytical procedure has been conducted by use of standardized solutions of sodium bisulfite (*145*). This procedure was compared with the peroxide candle method (*250*). Improved procedures have been proposed for use in the lead peroxide method for determining sulfur oxides (*31*, *201*). A number of portable and simplified instruments have been developed for sulfur dioxide analysis (*111*, *118*, *155*, *197*). Impedance measurements for determining sulfuric acid concentrations after oxidation of sulfur dioxide were used as the basis for an instrument for measurement of emission levels of sulfur dioxide (*162*). Modifications of existing instrumentation have been described (*121*, *196*). The response characteristics of the Thomas Autometer were studied (*223*).

A sensitive method for determination of atmospheric sulfur trioxide as sulfate is available (*304*). Two instruments have been developed for determination of sulfur trioxide in flue gases (*118, 159*).

Hydrogen Sulfide. The molybdenum blue method for hydrogen sulfide continues to find use in atmospheric analysis (*38*). Hydrogen sulfide also has been determined by collection on membrane filters impregnated with lead acetate followed by spectrophotometric analysis of the colloidal lead sulfide (*202*).

Nitrogen oxides. Nitrites and nitrogen oxides have been determined colorimetrically by reaction with *p*-hydroxyazobenzene sulfonamide (*209*). Nitrates or nitrite plus nitrate can be analyzed by use of 1-aminopyrene (*231*). A number of autocatalytic spectrophotometric methods have been developed for the analysis of nitrites (*242*). Nitrogen dioxide was analyzed in nitrogen and oxygen in the p.p.m.-range by electron-capture gas chromatography (*190*). Higher nitrogen oxide concentrations, as in flue gases, can be measured acidimetrically after oxidation of nitrogen oxide to nitric acid with hydrogen peroxide (*161*). Various methods have been tested to develop a procedure for analyses of nitric oxide, nitrogen dioxide, and nitric acid in air and in emissions (*266, 267*).

Design parameters and performance data have been given for a miniaturized colorimetric recording air analyzer for analysis of nitrogen dioxide (*220*). An instrument for continuous photometric analysis of nitrogen dioxide, apparently similar to instruments in use in the United States, is discussed in the Japanese literature (*296*). Fiber-glass filter paper impregnated with acidified potassium permanganate can oxidize nitric oxide to nitrogen dioxide (*78*).

Fluorides. An atmospheric analyzer has been developed and tested for analyses of hydrogen fluoride in the part-per-billion range (*132*). Spectrographic procedures for determining airborne fluoride as SrF_2 or SiF_4 were studied, but the sensitivities achieved proved insufficient for atmospheric analyses (*42, 43*). A silica gel tube procedure for analysis of hydrogen fluoride in the field has been described (*262*). Fluorine survey papers treated with lime acetate and sodium acetate can be analyzed by use of the thorium chloranilate procedure (*6*). Several methods for determining fluorine in plant tissues have been reported (*1, 290, 291*).

Ozone and Oxidants. A new spectrophotometric method for ozone has been developed that is more specific than existing colorimetric procedures (*33*). Phenolphthalin reagent responded not only to ozone but also to nitrogen dioxide and hydrogen cyanide, and the nitrogen dioxide interference was difficult to eliminate (*268*). The accuracy of the phenolphthalin method was improved by sampling and handling the sample at 0° C. (*176*).

The coulometric-type oxidant sensor has been evaluated for air pollution applications (*288*), and continuous recording instrumentation has been developed (*180*). A galvanic monitoring instrument for oxidant also is available (*112*). The nitrogen dioxide equivalent method has been incorporated in a continuous monitoring instrument for both ozone and oxides of nitrogen (*276*). Regener developed an instrument based on the chemiluminescent reaction of rhodamine B with ozone; the sensitivity of the instrument to ozone was about 0.001 p.p.m., with negligible interference from nitrogen dioxide, sulfur dioxide, or peroxyacylnitrate (*213*). A feasibility study was made of a detector for ozone based on the oxidation of a polyelectrolyte film with subsequent measurement of conductivity or potential (*117*). Both phenolphthalin and sodium diphenylamine sulfonate have been used in a colorimetric analyzer for oxidants (*3*). Responses to oxidants were evaluated for an instrument that incorporates neutral potassium iodide reagent, a recording ultraviolet photometer, and coulometric analyzers (*37*). The interference of sulfur dioxide in determination of oxidant by instrumentation based on iodometric or coulometric techniques was eliminated by use of U-tubes packed with glass-fiber strips treated with chromium trioxide and sulfuric acid (*221*).

Hydrogen peroxide has been determined in air on silica gel indicator tubes treated with ethanolic titanous sulfate (*188*).

ORGANIC GASES AND VAPORS

Much research effort continues on the application of gas chromatography to the analyses of hydrocarbons, organic nitrates, and halogenated organic substances. The status of research on the application of gas chromatography to air pollution up to 1963 has been reviewed (*9*). A monograph and a review are available on polarographic methods for the determination of organic substances in sanitary chemistry (*147, 148*).

Hydrocarbons. An important recent advance was the development of larger-diameter open tubular columns (*84, 133*). Such columns often provide better resolution than packed columns, while accepting the larger sample sizes needed in trace analyses. McEwen has used temperature-programmed smaller-diameter open tubular columns to rapidly separate C_5 to C_8 aliphatic hydrocarbons in dilute gas mixtures (*182*). A silver salt ethylene glycol retarding trap for unsaturates has been used with a larger-diameter tubular column for analyzing trace quantities of paraffins in the C_2 to C_8 range and olefins in the C_2 to C_5 range (*23*). Chemical adsorption with temperature-programmed packed columns has been used for trace analysis of C_2 to C_8 aliphatic hydrocarbons and some aromatic hydrocarbons (*181*). Trace analyses for C_2 to C_4 hydrocarbons on silica gel and C_4 to C_7 aliphatic hydrocarbons on alkalized alumina were obtained in less than 5 minutes by the use of very short columns and by differential analysis involving mercuric sulfate-sulfuric acid adsorption (*129*).

Columns consisting of alumina coated with liquid substrates have been used for the separation of C_1 to C_4 hydrocarbons at ambient or subambient temperatures (*134, 154*). The liquid 1,2,3 - tris(2 - cyanoethoxy)propane has been used as the stationary phase in the gas chromatographic analysis of aromatic hydrocarbons in blow-by gases, automobile exhaust, and irradiated automobile exhaust (*13, 53, 150*). The flame ionization detector was superior to the argon ionization detector in sensitivity as well as other performance characteristics for analyses of light hydrocarbons (*154*). Flame ionization detectors are routinely used by almost all investigators now doing trace hydrocarbon analyses.

The C_2 to C_6 hydrocarbons and sulfur dioxide in a variety of atmospheric and source emission samples were analyzed by gas chromatography after concentration on firebrick at liquid oxygen temperature (*16*). Methane and carbon monoxide were oxidized to carbon dioxide, trapped, and analyzed as carbon dioxide.

Several discussions of the use of flame ionization analyzers for atmospheric monitoring are available (*69, 265*). An atmospheric flame ionization methane analyzer has been developed by use of a solid adsorbent to retain all organics except methane (*204*). Differential chemical adsorption techniques were used with flame ionization detection to develop group analyses for olefins and for unsaturated hydrocarbons (*130*). An atmospheric olefin analyzer that depends on the vapor-phase bromination of olefins has been built (*17*).

Despite the advent of gas chromatography, analyses for aromatic hydrocarbons continue to be made by ultraviolet spectrophotometry (*7, 64, 77*). Analyses usually follow collection in an ultraviolet transparent solvent (*7, 64*) or in a silica gel sampling tube (*77*).

Halogenated Substances. A variety of analytical techniques have been applied to the trace analysis of halogenated compounds. Gas chromatography with thermal conductivity, flame ionization, argon ionization, and electron capture detectors and infrared spectrophotometry have been applied to the analysis of chlorinated hydrocarbons in air (*27*). The optimum operating parameters and relative responses in electron capture gas chromatography have been reported for a wide variety of organic and inorganic substances containing fluorine, chlorine, bromine, or iodine atoms (*51*). Infrared spectrophotometry, using a long-path infrared cell, has been applied to the analysis of carbon tetrachloride and chloroform in the 0.01- to 0.1-p.p.m. range (*82*). The samples were collected and concentrated on silica gel, flushed with nitrogen into a Saran plastic bag and then transferred to the infrared cell. Coulometric titration for bromide has been used to determine 1,2-dibromoethane after its absorption in methanolic sodium hydroxide (*74*). The feasibility of using polarographic techniques was investigated for the analysis of several low-molecular-weight chlorinated hydrocarbons in air (*71*). Spectrophotometric and autocatalytic methods were developed for use in the analysis of iodoalkanes and, with considerably decreased sensitivity, of bromo- and chloroalkanes (*227*). A colorimetric technique has been described for analyzing chloromethane in air in the presence of other chlorinated hydrocarbons (*151*).

Aldehydes. A modification of the Schiff test has been developed into a spectrophotometric method for formaldehyde (*178*). A spectrophotofluorometric method is available for formaldehyde and acrolein based on their reactions with J-acid (*239*). The applicability of modifications of the chromotropic acid method for formaldehyde were discussed for air and source sampling (*14*). A modification of the 3-methyl-2-benzothiazolone hydrazone method for aliphatic aldehydes has been developed to provide increased sensitivity (*104*).

Organic Nitrogen Compounds. Atmospheric analysis of peroxyacyl nitrates is now possible by the use of electron-capture gas chromatography (*65, 255*). Since peroxyacyl nitrates readily decompose, appropriate inlet and column conditions are essential. Similar techniques can be used to analyze for peroxyacyl nitrates, alkyl nitrates, and other electron-capture substances in irradiation chamber studies (*24, 160*). Infrared absorptivities for several peroxyacyl nitrates have been reported (*254*). A colorimetric technique is available for 2-nitropropane in air (*135*).

Miscellaneous Compounds. The operating conditions necessary for gas chromatographic determination of mercaptans in air have been studied (*87*). Alcohols and esters were determined in air by reaction with *p*-dimethylaminobenzaldehyde (*172*). Ethylene oxide in air has been analyzed colorimetrically after conversion to formaldehyde (*28, 152*). Tetramethyl lead and tetraethyl lead can be determined in air by the iodine procedure with portable sampling equipment (*169*). Airborne phenols and amines have been determined colorimetrically with stabilized diazonium salts as reagents (*177*).

ATMOSPHERIC REACTIONS

Two reviews of photochemical and kinetic studies relevant to air pollution treat the subject from somewhat different viewpoints (*11, 289*). These types of investigations are strongly dependent on the availability of adequate methods of trace analysis. Investigations at realistic atmospheric concentration levels (0.01–1 p.p.m.) impose much more stringent analytical requirements than does work even in the range of 1 to 100 p.p.m. of reactants. Long-path infrared cells can be used as irradiation chambers so that reactions can be followed in situ. A series of investigations by the long-path infrared technique have been reported concerning the thermal oxidation of nitric oxide alone (*94*) and in the presence of dienes (*93*) and the photooxidation of hydrocarbons in the presence of nitric oxide (*95*). Infrared spectrophotometry was used with colorimetric analysis of nitrogen dioxide in a study of the photooxidation of olefin nitric oxide systems (*253*). Gas chromatography and infrared spectrometry have been combined in an investigation of the photooxidation of diethylketone to provide more adequate analyses of products that can be provided by either technique alone (*139, 140*). Gas chromatography, particularly gas chromatography combined with colorimetric analyses, provides another approach to detailed analyses in investigations of the kinetics of hydrocarbon–ozone reactions (*39*) and in photooxidation studies involving the ethylene–nitric oxide system (*12*); the photooxidation of various paraffins, olefins, aromatics, and aldehydes with nitric oxide (*146*); and the irradiation of automobile exhaust (*13, 160*). A time-of-flight mass spectrometer has been used to analyze the products in the photooxidation of acetaldehyde at partial pressures at and above 1 mm. (*107*).

In a radiotracer study C^{14} was used to label ethylene photooxidized with nitrogen oxide. The yields of chemical and radiochemical formaldehyde were determined in the absence and presence of added organic substances (*198, 199*). The products formed from the oxidation of various polycyclic aromatic hydrocarbons on particulate matter have been separated by use of thin-layer chromatography (*131*). The atmospheric oxidation of sulfur dioxide was followed in laboratory and in plumes by various analytical procedures for sulfur dioxide and sulfates (*90*).

Materials of construction, pigment films, and dyed fabrics all can be used as physical indicators of pollutants. Subjects of reported studies include the reaction of sulfur dioxide with building materials (*92*); of hydrogen sulfide with lead pigment films (*99*); and of ozone, nitrogen dioxide, and irradiated auto exhaust with various fabrics treated with a number of dyes (*219*).

ANALYSES OF SOURCES OF POLLUTION

A number of catalytic devices, a direct-flame afterburner, oxidation by addition of air to the exhaust manifold, and adjustments of carburetor and ignition have all received much attention for the reduction of hydrocarbons in automobile exhaust. Since this work is all of a proprietary nature, detailed-composition studies have not been reported. Although much work continues on the chemical composition of automobile exhaust and blow-by gases, considerable effort also has been directed toward analyses of diesel exhaust, emissions from stationary combustion sources, and industrial waste gases. A number of investigations have involved methods of analyses and control of nitrogen oxides from both vehicles and stationary sources. Analyses for benzo[*a*]pyrene and other polycyclic aromatic hydrocarbons in a wide variety of emissions from combustion processes and in waste gases have been reported.

Automobile Exhaust and Blow-by Emissions. The chemical nature of the lead in automobile exhaust gases has been discussed (*116*). The concentrations of lead and of fine particles in auto exhaust were measured with variations in car speed and anti-knock additives (*193*). The concentration of lead in and immediately about various test vehicles was determined in a comparison of the amounts formed when tetramethyl lead or tetraethyl lead was used in the fuel (*36*). A direct relation was reported between variations in benzo[*a*]pyrene and carbon monoxide in automotive emissions and variations in speed and carburetor adjustment (*97*).

There is much interest in the development of a nitrogen oxide monitoring instrument for automobile exhaust. After oxidation of nitric oxide to nitrogen dioxide in the presence of a twofold excess of oxygen, nitrogen oxides have been analyzed in a double-beam colorimeter (*264*). The total response time for the analyzer was in the range of 20 to 30 seconds. In another investigation a double-beam colorimeter also was used but oxidation was accomplished with glass filter paper impregnated with sodium dichromate–sulfuric acid solution (*214*). Although this oxidizer system provided faster response, the lifetime of this chemical oxidizer was short. Analyses for hydrogen, carbon monoxide, carbon dioxide, oxides of nitrogen, formaldehyde, total aldehydes, and total hydrocarbons are available for different modes of operation of the automobile engine (*124*). Paper chromatography has been used to identify a number of carbonyl compounds in automobile exhaust (*20*). Phenolic compounds in auto exhaust have been analyzed by spectrophotometric and chromatographic methods (*21, 252*). Analyses were made of several vehicles operated on liquefied petroleum gas for carbon monoxide, nitrogen oxides, and hydrocarbons (*263*). Only four-carbon and low-molecular-weight hydrocarbons were found in the exhaust, usually with very low concentrations of butenes.

Analyses for one- to five-carbon hydrocarbons have been made in blow-by and exhaust (*257*). In another investigation one- to eight-carbon paraffins, two- to five-carbon olefins, and six- to ten-carbon aromatics were measured in blow-by and exhaust gases (*150*).

Catalytic reduction of nitrogen oxides in automobile exhaust has been investigated (*18, 19*). Chromium and depleted uranium catalysts were used to oxidize hydrocarbons in automobile exhaust (*86, 278*).

Other Sources of Emissions. A comparison was made of polycyclic aromatic hydrocarbons produced by diesel and gasoline-powered vehicles under road conditions (*260*). The production of polycyclic aromatic hydrocarbons in diffusion flames and diesel engine operation has been investigated (*211*). With careful attention to sampling of diesel engine exhaust, it has been shown that nitrogen dioxide makes up a smaller fraction of the total nitrogen oxides than previously reported and the nitrogen dioxide levels approach equilibria values (*102*). Air samples from idling diesels in a railway roundhouse were analyzed for nitrogen oxides, sulfur dioxide, formaldehyde, acrolein, total aldehydes, and light hydrocarbons (*22*). Aldehyde concentrations inside an automobile following various diesel buses were obtained (*244*).

The utility of gas chromatography in analyzing various fuel gases for inorganic gases and for hydrocarbons has been demonstrated (*56*). Sampling and analysis techniques were discussed for metals, polynuclear hydrocarbons, sulfur dioxide, nitrogen dioxide, total hydrocarbons, aldehydes, and organic acids in power plant emissions (*62*).

A large number of studies of polycyclic aromatic hydrocarbons have been made in combustion and industrial processes. The effects of changes in fuel composition (*272*) and operating conditions (*29*) on concentrations of polynuclear hydrocarbons have been investigated by use of laboratory combustion devices. Benzene and some alkylbenzenes also were produced in a laboratory combustion chamber with aliphatic hydrocarbons as fuels (*195*). Polynuclear hydrocarbons and other pollutants were measured in various heat-generation and incineration facilities (*100*). Analyses were obtained for benzo[*a*]pyrene and other polycyclic aromatic hydrocarbons in waste gases from coke plants (*297, 298*), in stack gases from pulp mills (*108*), in rubber manufacturing (*301*), in the glass industry (*303*), and in molding sand mixtures and foundary dust (*302*).

The sulfur dioxide–sulfur trioxide equilibrium has been considered in flue gases (*137*). A flue gas sampler for sulfur dioxide and sulfur trioxide has been evaluated by application to industrial flue-gas emissions (*269*). Analyses for sulfur dioxide and other pollutants were made on flue gases from iron furnaces (*274*). The formation of sulfur trioxide in the combustion of petroleum fuel oils has been discussed (*179*).

Techniques for sampling and analysis by gas chromatography have been described for sulfur dioxide, hydrogen sulfide, and organic sulfur compounds in gaseous effluents from kraft pulp processes (*2, 47*). A combination of spectrophotometric and titration methods for these effluents also has been developed and applied in several kraft pulp mills (*101*). Emissions of oxides of nitrogen were measured in a wide variety of stationary sources of combustion (*186*).

FIELD STUDIES

Only a selection of all of the field work done in the period under review is included. Many studies involving routine analytical methods have been excluded.

The occurrence of iodine-129 in nature and its possible utility as an atmospheric tracer substance in radioactive activation techniques for analysis has been discussed (*76*). Methods of electron-capture gas chromatography provide extremely high sensitivities for a number of inorganic and organic halogenated gases and volatile liquids (*51*). Such substances were suggested as tracer substances for large-scale studies. Sulfur hexafluoride and difluorodichloromethane already have been used successfully in small-scale atmospheric tracer measurements with analysis by electron-capture gas chromatography (*55*). Low-level vertical tracer experiments can be done with methane as a tracer substance and analysis by flame-ionization gas chromatography (*10*).

Iron and germanium concentrations have been determined in airborne particulates collected in air filtration plants (*41, 171*). High levels of arsenic were found in sedimented dusts collected in the city of Rome (*279*). Arsenic levels also were measured on particulate matter collected in Prague (*281*). The diurnal total lead levels at various locations in a number of U. S. cities have been reported (*48, 144*). The size distribution of sulfate-containing particulates was measured for samples collected in Pittsburgh (*60*) and Cincinnati (*215*). Analyses in a vehicular road tunnel have been made for a variety of substances including metals, sulfates, nitrates, polycyclic aromatic hydrocarbons, sulfur dioxide, nitrogen oxides, carbon monoxide, and aliphatic aldehydes (*63*).

The results of the piperonal test for aromatic compounds correlate satisfactorily with the usual spectrophotometric method for polycyclic aromatic hydrocarbons in analysis of samples from a large number of U. S. cities (*236*). Analyses for benzo[*a*]pyrene and benz[*a*]anthracene were made at several locations along with analyses for lead and carbon monoxide as indicators of dispersion of vehicle emissions; these analyses were performed to calculate the automotive contribution to atmospheric levels of polycyclic hydrocarbons (*57*). Benzo[*a*]pyrene and, in some studies, other polycyclic aromatic hydrocarbons have been analyzed in London (*170*), Liege (*216*), Stockholm (*89*), Hamburg (*114*), Prague (*249*), Budapest (*224*), and Kiev (*283*). Samples collected by aircraft over Northern Kyushu also were analyzed for polynuclear hydrocarbons (*225*).

In two studies reported from the United States and Japan, mobile laboratories were used for analysis of sulfur dioxide, nitrogen oxides, oxidants, carbon monoxide, and carbon dioxide; in one of the studies analyses also were made for total hydrocarbons and aldehydes (*120, 187*). Ground-level concentrations of carbon monoxide, methane, and nitrous oxide were obtained by very-long-path infrared measurements in Columbus, Ohio (*32*). The very low carbon monoxide levels in London during the Suez crisis when gasoline was rationed have been compared with the carbon monoxide levels during periods of normal vehicular traffic (*158*). During normal traffic the maximum daily levels of carbon monoxide at some locations in London can exceed 100 p.p.m. and occasionally reach several hundred parts per million.

A formula has been developed to relate the concentration of atmospheric sulfur compounds to mean external wind speed and the sulphation of lead dioxide within a cylindrical louvred screen (*157*). Orientation and exposure to wind of both cubical and cylindrical instruments were discussed. Results of analyses of atmospheric sulfur dioxide in samples obtained with prefiltered and with unfiltered sampling systems have been compared (*251*). Continuous sulfur dioxide levels in the inner town of Hamburg were compared during the period 1958–63 and related to sulfur dioxide emissions from an oil refinery in Hamburg (*98*).

Methane concentrations at various locations in several U. S. cities were measured by flame-ionization gas chromatography (*10*). Tetraalkyl lead concentrations have been determined at traffic sites, gas stations, and refineries (*48*, *144*). The tetraalkyl lead constituted 1 to 5% of the total lead concentration. Lead analyses of samples from the atmosphere near a factory producing tetraethyl lead in Italy have been reported (*54*). Methods have been discussed for 24-hour sampling of di- and trichlorophenoxy acetic acid derivatives (2,4-D) as combined aerosol and gas, with analysis by electron-capture techniques (*4*, *5*). Analyses were made in the vicinity of spraying operations in the State of Washington during the summer of 1963; concentrations ranged from undeterminable to 3.1 micrograms per cubic meter.

LITERATURE CITED

(1) Adams, D. F., Air Pollution Symposium, Division of Water and Waste Chemistry, 145th Meeting, ACS, New York City, N. Y., Sept. 8–13, 1963.
(2) Adams, D. F., 56th Annual Meeting, Air Pollution Control Association, Detroit, Mich., June 9–13, 1963.
(3) Adams, D. F., *J. Air Pollution Control Assoc.* **13,** 88 (1963).
(4) Adams, D. F., Jackson, C. M., Bamesberger, W. L., Air Pollution Symposium, Division of Water and Waste Chemistry, ACS, 147th Meeting, Philadelphia, Pa., April 5–10, 1964.
(5) Adams, D. F., Jackson, C. M., Bamesberger, W. L., *Weeds* **12,** 280 (1964).
(6) Adams, D. F., Sullivan, D. C., Air Pollution Symposium, Division of Water and Waste Chemistry, 145th Meeting, ACS, New York City, N. Y., Sept. 8–13, 1963.
(7) Alekseeva, M. V., Krylova, N. A., Khrustaleva, V. A., *Gigiena i Sanit.* **28,** 31 (1963).
(8) Altshuller, A. P., ANAL. CHEM. **35,** 3R (1963).
(9) Altshuller, A. P., *J. Gas Chromatog.* **1,** 6 (1963).
(10) Altshuller, A. P., Bellar, T. A., Clemons, C. A., Vander Zanden, E., *Intern. J. Air Water Pollution* **8,** 29 (1964).
(11) Altshuller, A. P., Bufalini, J. J., *Photochem. Photobiol.* Vol. IV, No. 2, March 1965.
(12) Altshuller, A. P., Cohen, I. R., *Intern. J. Air Water Pollution* **8,** 611 (1964).
(13) Altshuller, A. P., Leach, P. W., *Ibid.*, **8,** 37 (1964).
(14) Altshuller, A. P., Leng, L. J., Wartburg, A. F., *Ibid.*, **6,** 381 (1962).
(15) Araki, S., Kato, T., *Japan Analyst* **12,** 1027 (1963).
(16) Araki, S., Katoh, T., *Ibid.*, **11,** 533 (1962).
(17) Austin, R. R., *ISA Trans.* **1,** 211 (1962).
(18) Baker, R. A., Doerr, R. C., Air Pollution Symposium, Division of Water and Waste Chemistry, 147th Meeting, ACS, Philadelphia, Pa., April 5–10, 1964.
(19) Baker, R. A., Doerr, R., *J. Air Pollution Control Assoc.* **14,** 409 (1964).
(20) Barber, E. D., Lodge, J. P., ANAL. CHEM. **35,** 348 (1963).
(21) Barber, E. D., Sawicki, E., McPherson, S., *Ibid.*, **36,** 2442 (1964).
(22) Battigell, M. C., *J. Occupational Med.* **5,** 54 (1963).
(23) Bellar, T. A., Bellar, M. L., Sigsby, J. E., Jr., Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Pittsburgh, Pa., March 2–6, 1964.
(24) Bellar, T. A., Sigsby, J. E., Jr., Air Pollution Symposium, Division of Water and Waste Chemistry, 144th Meeting, ACS, Los Angeles, Calif., March 30–April 5, 1963.
(25) Bender, D. F., Sawicki, E., Wilson, R. M., Jr., ANAL. CHEM. **36,** 1011 (1964).
(26) Bender, D. F., Sawicki, E., Wilson, R. M., Jr., *Intern. J. Air Water Pollution* **8,** 633 (1964).
(27) Boettner, E. A., Dallos, F. C., Stewart, R. D., American Industrial Hygiene Conference, Philadelphia, Pa., April, 1964.
(28) Bolton, N. E., Ketcham, N. H., *Arch. Environ. Health* **8,** 711 (1964).
(29) Boubel, R. W., Ripperton, L. A., *J. Air Pollution Control Assoc.* **13,** 553 (1963).
(30) Bourke, P. J., Dawson, R. W., Denton, W. H., *J. Chromatog.* **14,** 387 (1964).
(31) Bowden, S. R., *Intern. J. Air Water Pollution* **8,** 101 (1964).
(32) Bowman, R. L., Shaw, J. H., *Appl. Opt.* **2,** 176 (1963).
(33) Bravo, H. A., Lodge, J. P., ANAL. CHEM. **36,** 671 (1964).
(34) Brennan, E., Leone, I. A., Daines, R. H., Air Pollution Symposium, Division of Water, Air, and Waste Chemistry, 148th Meeting, ACS, Chicago, Ill., Aug. 30–Sept. 4, 1964.
(35) Brennan, E. G., Leone, I. A., Daines, R. H., *Science* **143,** 818 (1964).
(36) Brief, R. S., *Arch. Environ. Health* **5,** 527 (1962).
(37) Bryan, R. J., Cherniac, I., 57th Annual Meeting, APCA, Houston, Texas, June 21–25, 1964.
(38) Buck, M., Stratmann, H., *Staub* **24,** 241 (1964).
(39) Bufalini, J. J., Altshuller, A. P., Air Pollution Symposium, Division of Water and Waste Chemistry, 145th Meeting, ACS, New York City, N. Y., Sept. 8–13, 1963.
(40) Burianec, Z., Burianova, *Collection Czech. Chem. Commun.* **28,** 2895 (1963).
(41) Burrows, I. E., Lindsey, A. J., *Intern. J. Air Water Pollution* **6,** 439 (1962).
(42) Burrows, K. M., Horwood, J. F., *Spectrochim. Acta* **19,** 1 (1963).
(43) *Ibid.*, p. 17.
(44) Cameroni, R., Albasini, A., *Farmaco (Pavia), Ed. Prat.* **19,** 1227 (1964).
(45) Campbell, E. E., Ide, H. M., American Industrial Hygiene Conference, Philadelphia, Pa., April 1964.
(46) Campbell, E. E., Miller, H. M., Chemical Detectors, Los Alamos Sci. Lab., Univ. of California, Los Alamos, N. M., 1961, 1964. **LAMS-2378,** Vols. I and II, 1961, 1964.
(47) Cave, G. C. B., *Tappi* **46,** 1, 5, 11 (1963).
(48) Cholak, J., *Arch. Environ. Health* **8,** 126 (1964).
(49) *Ibid.*, p. 222.
(50) Cleary, G. J., *J. Chromatog.* **9,** 204 (1962).
(51) Clemons, C. A., Altshuller, A. P., Air Pollution Symposium, Division of Water, Air, and Waste Chemistry, 148th Meeting, ACS, Chicago, Ill., Aug. 30–Sept. 4, 1964.
(52) Clemons, C. A., Altshuller, A. P., *J. Air Pollution Control Assoc.* **14,** 407 (1964).
(53) Clemons, C. A., Leach, P. W., Altshuller, A. P., ANAL. CHEM. **35,** 1546 (1963).
(54) Coghi, L., Bellelli, E., *Nuovi Ann. Igiane Microbiol.* **14,** 207 (1963).
(55) Collins, G. F., Bartlett, F. E., Turk, A., Edmonds, S. M., Mark, H. L., *J. Air Pollution Control Assoc.* **15,** in press (1965).
(56) Colson, E. R., Brown, G. M., *J. Inst. Fuels* **35,** 399 (1962).
(57) Colucci, J. M., Begeman, C. R., 57th Annual Meeting, APCA, Houston, Texas, June 21–25, 1964.
(58) Commins, T., *Analyst* **88,** 364 (1963).
(59) Conner, W. D., Nader, J. S., *Am. Ind. Hyg. Assoc. J.* **25,** 291 (1964).
(60) Corn, M., De Maio, L., *Science* **143,** 803 (1964).
(61) Cropper, F. R., Kamisky, S., ANAL. CHEM. **35,** 735 (1963).
(62) Cuffe, S. T., *Arch. Environ. Health* **6,** 422 (1963).
(63) Cummins, R. L., Konopinsk, V. J., American Industrial Hygiene Conference, Philadelphia, Pa., April 1964.
(64) Dambranskas, T., Cook, W. A., *Am. Ind. Hyg. Assoc. J.* **24,** 568 (1963).
(65) Darley, E. F., Kettner, K. A., Stephens, E. R., ANAL. CHEM. **35,** 589 (1963).
(66) Diamond, P., *Am. Ind. Hyg. Assoc. J.* **24,** 399 (1963).
(67) Doyle, G. J., Jones, J. L., *J. Air Poll. Control Assoc.* **13,** 365 (1963).
(68) Dubois, L., Monkman, J. L., *Am. Ind. Hyg. Assoc. J.* **25,** 485 (1964).
(69) Dubois, L., Reynolds, R. G., Teichman, T., Monkman, J. L., *J. Air Pollution Control Assoc.* **14,** 295 (1964).
(70) Duckworth, S., Levaggi, D., *Ibid.*, **13,** 429 (1963).
(71) Duffy, T. L., Argonne National Laboratory *Rept.* **TID-7616** 211 (1960).
(72) Dugger, W. M., Koukol, J., Reed, W. D., Palmer, R. L., *Plant Physiol.* **38,** 468 (1963).
(73) Dugger, W. M., Taylor, O. C., Thompson, E. C., Cardiff, E., *J. Air Pollution Control Assoc.* **13,** 423 (1963).
(74) Dumas, T., *J. Agr. Food Chem.* **10,** 476 (1962).
(75) Edmonds, S. M., Turk, A., Morrow, J., Dill, A., Davis, S., Air Pollution Symposium, Division of Water and Waste Chemistry, 144th Meeting, ACS, Los Angeles, Calif., March 30–April 5, 1963.
(76) Edwards, R. R., *Science* **137,** 851 (1962).
(77) Elkins, H. B., Pagnotto, L. D., Comproni, E. M., ANAL. CHEM. **34,** 1797 (1962).
(78) Ellis, C. F., *Intern. J. Air Water Pollution* **8,** 297 (1964).
(79) Endow, N., Doyle, G. J., Jones, J. L., *J. Air Pollution Control Assoc.* **13,** 141 (1963).
(80) Epstein, S. S., Small, M., Koplan, J., Mantel, N., Falk, H. L., Sawicki, E., *Arch. Environ. Health* **7,** 531 (1963).
(81) Epstein, S. S., Small, M., Koplan, J., Mantel, N., Hutner, S. H., *J. Nat. Cancer Inst.* **31,** 163 (1963).
(82) Erley, D. S., *Am. Ind. Hyg. Assoc. J.* **23,** 388 (1962).

(83) Estes, F. L., Duncan, R. C., Pan, Chao-Han, Air Pollution Symposium, Division of Water and Waste Chemistry, 144th Meeting, ACS, Los Angeles, Calif., March 30–April 5, 1963.

(84) Ettre, L. S., Cieplinski, E. W., Averill, W., *J. Gas Chromatog.* **1**, 2, 7 (1963).

(85) Eye, M. G., MacPhee, R. D., Air Pollution Symposium, Division of Water and Waste Chemistry, 144th Meeting, ACS, Los Angeles, Calif., March 30–April 5, 1963.

(86) Feenan, J. J., Anderson, R. B., Swan, H. W., Hofer, L. J. E., *J. Air Pollution Control Assoc.* **14,** 113 (1964).

(87) Feldstein, M., Balestrieri, S., Levaggi, D. A., 6th Conference on Methods in Air Pollution Studies, Sponsored by California Dept. of Public Health, Berkeley, Calif., Jan. 6–7, 1964.

(88) Fish, A., Franklin, N. H., Polland, R. T., *J. Appl. Chem.* **13,** 506 (1963).

(89) Frank, A., Gerhardson, G., *Nord. Hyg. Tidskr.* **43,** 42 (1962).

(90) Gartell, F. E., Thomas, F. W., Carpenter, S. B., *Am. Ind. Hyg. Assoc. J.* **24,** 113 (1963).

(91) Gernet, E. V., *Novoe v Oblasti Sanit-Khim. Analiza* (Raboty po Prom-Sanit Khim.) 25, 1962.

(92) Gilardi, E. F., Manganell, R. M., American Industrial Hygiene Conference, Philadelphia, Pa., April 1964.

(93) Glasson, W. A., Tuesday, C. S., Air Pollution Symposium, Division of Water and Waste Chemistry, 147th Meeting, ACS, Philadelphia, Pa., April 5–10, 1964.

(94) Glasson, W. A., Tuesday, C. S., *J. Am. Chem. Soc.* **85,** 2901 (1963).

(95) Glasson, W. A., Tuesday, C. S., Symposium on Air Pollution, Division of Water, Air, and Waste Chemistry, 148th Meeting, ACS, Chicago, Ill., Aug. 30–Sept. 4, 1964.

(96) Goetz, A., *J. Air Pollution Control Assoc.* **14,** 213 (1964).

(97) Gofmekler, V. A., Manita, M. D., Manusadzhants, I., Stepanov, L. L., *Gigiena i Sanit* **28,** 3 (1963).

(98) Grafe, K., *Staub* **23,** 557 (1963).

(99) Gregory, C. J., Manganell, R. M., Air Pollution Symposium, Division of Water, Air, and Waste Chemistry, 148th Meeting, ACS, Chicago, Ill., Aug. 30–Sept. 4, 1964.

(100) Hangebrauck, R. P., Von Lehmden, D. J., Meeker, J. E., *J. Air Pollution Control Assoc.* **14,** 267 (1964).

(101) Harding, C. I., Hendrickson, E. R., Sundaresan, B. B., Walker, C. V., Air Pollution Symposium, Division of Water, Air, and Waste Chemistry, 148th Meeting, ACS, Chicago, Ill., Aug. 30–Sept. 4, 1964.

(102) Harkins, J., Goodwine, J. K., *J. Air Pollution Control Assoc.* **14,** 34 (1964).

(103) Harkins, J., Nicksic, S. W., Air Pollution Symposium, Division of Water, Air, and Waste Chemistry, 148th Meeting, ACS, Chicago, Ill., Aug. 30–Sept. 4, 1964.

(104) Hauser, T. R., Cummins, R. L., ANAL. CHEM. **36,** 679 (1964).

(105) Heck, W. W., *J. Air Pollution Control Assoc.* **14,** 255 (1964).

(106) Heggestad, H. E., Burleson, F. R., Middleton, J. T., Darley, E. F., *Intern. J. Air Water Pollution* **8,** 1 (1964).

(107) Heicklen, J. P., Johnston, H. S., Air Pollution Symposium, Division of Water and Waste Chemistry, 145th Meeting, ACS, New York City, N. Y., Sept. 8–13, 1964.

(108) Hendrickson, E. R., Walker, C. G., Chapnerkar, V. D., *Am. Ind. Hyg. Assoc. J.* **24,** 121 (1963).

(109) Héros, M., *Rev. Pollution Atm.* **5,** 205 (1963).

(110) Héros, M., Amy, L., *Compt. Rend.* **255,** 695 (1962).

(111) Herrmann, G., *Chem. Tech.* **15,** 342 (1963).

(112) Hersch, P., Deuringer, R., ANAL. CHEM. **35,** 897 (1963).

(113) Hersch, P., Deuringer, R., *J. Air Pollution Control Assoc.* **13,** 538 (1963).

(114) Hettche, H. O., *Intern. J. Air Water Pollution* **8,** 185 (1964).

(115) Hindawi, I. J., Altshuller, A. P., *Science* **146,** 540 (1964).

(116) Hirschler, D. A., Gilbert, L. F., *Arch. Environ. Health* **3,** 297 (1964).

(117) Hirshou, J. M., "A Detection System for Ozone Employing Polyelectrolyte Films," WADC Tech. Rept. **58-50,** May 1958, **OTS 140792,** U. S. Dept. of Commerce, Washington 25, D. C.

(118) Hissink, M., *J. Inst. Fuels* **36,** 372 (1963).

(119) Hocheiser, S., "Methods of Measuring and Monitoring Atmospheric Sulfur Dioxide," Public Health Service Publication No. **999-AP-6,** 1964.

(120) Hochheiser, S., Storlazzi, M., Basbagill, W. J., American Industrial Hygiene Conference, Philadelphia, Pa., April 1964.

(121) Hoschele, K., *Staub* **24,** 140 (1964).

(122) Houser, E. A., Chapman, R. L., *J. Air Pollution Control Assoc.* **13,** 535 (1963).

(123) Huitt, H. A., Lodge, J. P., Jr., ANAL. CHEM. **36,** 1305 (1964).

(124) Hunn, R. W., *Arch. Environ. Health* **5,** 592 (1962).

(125) Huygen, C., *Anal. Chim. Acta* **28,** 349 (1963).

(126) *Ibid.,* **29,** 448 (1963).

(127) *Ibid.,* **30,** 556 (1964).

(128) Huygen, C., *Mikrochim Acta* **1,** 6 (1963).

(129) Innes, W. B., Bambrick, W. E., *J. Gas Chromatog.* **2,** 309 (1964).

(130) Innes, W. B., Bambrick, W. E., Andreatch, A. J., ANAL. CHEM. **35,** 1198 (1963).

(131) Inscoe, M. N., Symposium on Air Pollution, Division of Water, Air, and Waste Chemistry, 148th Meeting, ACS, Chicago, Ill., Aug. 30–Sept. 4, 1964.

(132) Ivie, J. O., Zielensk, L. F., Thomas, M. D., Thompson, C. R., 57th Annual Meeting, APCA, Houston, Texas, June 21–25, 1964.

(133) Jentzch, D., Hovermann, W., *J. Chromatog.* **11,** 440 (1963).

(134) Jeung, E., Helwig, H. L., Air Pollution Symposium, Division of Water and Waste Chemistry, 144th Meeting, ACS, Los Angeles, Calif., March 31–April 5, 1963.

(135) Jones, L. R., *Am. Ind. Hyg. Assoc. J.* **24,** 11 (1963).

(136) Junge, C. E., "Air Chemistry and Radioactivity," Academic Press, New York, 1963.

(137) Juntgen, H., *Erdoel Kohle* **16,** 119 (1963).

(138) Jutze, G. A., Lewis, R. J., 57th Annual Meeting, APCA, Houston, Texas, June 21–25, 1964.

(139) Kallend, A. S., Pitts, J. N., Jr., Air Pollution Symposium, Division of Water and Waste Chemistry, 144th Meeting, ACS, Los Angeles, Calif., March 30–April 5, 1963.

(140) Kallend, A. S., Schuck, E. A., Pitts, J. N., Air Pollution Symposium, Division of Water and Waste Chemistry, 145th Meeting, ACS, New York City, N. Y., Sept. 2–13, 1964.

(141) Katz, M., Monkman, J. L., *Occupational Health Rev.* **16,** 3 (1964).

(142) Keenan, R. G., Byers, D. H., Saltzman, B. E., Hyslop, F. L., *Am. Ind. Hyg. Assoc. J.* **24,** 481 (1963).

(143) Keenan, R. C., Holtz, J. L., *Am. Ind. Hyg. Assoc. J.* **25,** 254 (1964).

(144) Kehoe, R. A., Cholak, J., McIlhinny, J. G., Lofquist, G. A., Sterling, T. D., *Arch. Environ. Health* **6,** 255 (1963).

(145) Kelso, F. S., Hartlage, T. A., Sleva, S. F., Air Pollution Symposium, Division of Water and Waste Chemistry, 147th Meeting, ACS, Philadelphia, Pa., April 5–10, 1964.

(146) Klosterman, D. L., Leach, P. W., Sigsby, J. E., Altshuller, A. P., Air Pollution Symposium, Division of Water and Waste Chemistry, 147th Meeting, ACS, Philadelphia, Pa., April 5–10, 1964.

(147) Kogan, I. B., *Novoe v Oblasti Sanit-Khim. Analiza* (Raboty po Prom-Sanit Khim.) 1962, 64.

(148) Kogan, I. B., "Polarographic Analysis in Industrial Sanitary Chemistry," Moscow, 1961.

(149) Korth, M. W., Rose, A. H., Stahman, R. C., *J. Air Pollution Control Assoc.* **14,** 168 (1964).

(150) Korth, M. W., Sigsby, J. E., Jr., 57th Annual Meeting, APCA, Houston, Texas, June 21–25, 1964.

(151) Krivoruchko, F. D., *Gigiena Truda i Prof. Zaboleraniya* **6,** 51 (1962).

(152) Krylova, N. A., *Gigiena i Sanit.* **26,** 48 (1961).

(153) Kuczynski, E. R., *J. Air Pollution Control Assoc.* **13,** 435 (1963).

(154) Kuley, C. J., ANAL. CHEM. **35,** 1472 (1963).

(155) Kundig, S., Hogger, D., *Staub* **24,** 408 (1964).

(156) Lawrence, E. F., Cook, W. A., Boettner, E. A., American Industrial Hygiene Conference, Philadelphia, Pa., April 1964.

(157) Lawrence, E. N., *Intern. J. Air Water Pollution* **8,** 381 (1964).

(158) Lawther, P. J., Cummins, B. T., Henderson, M., *Ann. Occupational Hyg.* **5,** 241 (1962).

(159) Laxton, J. W., Jackson, P. J., *J. Inst. Fuels* **37,** 12 (1964).

(160) Leach, P. W., Leng, L. J., Bellar, T. A., Sigsby, J. E., Altshuller, A. P., *J. Air Pollution Control Assoc.* **14,** 176 (1964).

(161) Leithe, W., *Mikrochim. Acta* **1-2,** 166 (1962).

(162) Leong, K. J., Doow, G. F., MacFarland, H. N., *Am. Ind. Hyg. Assoc. J.* **25,** 309 (1964).

(163) Levaggi, D. A., Feldstein, M., *Ibid.,* p. 64.

(164) Levine, B. S., (transl.) USSR Literature on Air Pollution and Related Occupational Diseases, Vol. 9; Official Technical Services, U. S. Dept. Commerce, Washington 25, D. C.

(165) Liberti, A., Cartoni, G. P., Cantuti, V., *J. Chromatog.* **15,** 141 (1964).

(166) Lidzey, R. G., Longmaid, F. M., *Chem. Ind.* **1964,** 150.

(167) Lijinsky, W., Domsky, I., Mason, G., Ramahi, H. Y., Safari, T., ANAL. CHEM. **35,** 952 (1963).

(168) Lijinsky, W., Mason, G., *J. Gas Chromatog.* **1,** 9, 12 (1963).

(169) Linch, A. L., Davis, R. B., Stalzer, R. F., Anzilotti, W. F., *Am. Ind. Hyg. Assoc. J.* **25,** 81 (1964).

(170) Lindsey, A. J., Stanbury, J. R., *Intern. J. Air Water Pollution* **6,** 387 (1962).

(171) *Ibid.,* **7,** 1 (1963).

(172) Lipina, T. G., *Novoe v Oblasti Sanit-Khim. Analiza* (Raboty po Prom-Sanit. Khim.) 150 (1962).

(173) Lodge, J. P., Parbahakar, K. J., *Anal. Chim. Acta* **29,** 372 (1963).

(174) Lodge, J. P., Jr., Pate, J. B., Huitt, H. A., *Am. Ind. Hyg. Assoc. J.* **24,** 380 (1963).

(175) Lodge, J. P., Jr., Xinteras, C., Pate, J. B., Altshuller, A. P., *Intern. J. Air and Water Pollution* **7,** 79 (1963).

(176) Louw, C. W., Halliday, E. C., *Ibid.*, p. 1033.

(177) Lugg, A., ANAL. CHEM. **35,** 899 (1963).

(178) Lyles, G. R., Blanchard, V. J., 57th Annual Meeting, APCA, Houston, Texas, June 21–25, 1964.

(179) MacFarlane, J. J., *J. Inst. Fuels* **35,** 502 (1962).

(180) Mast, G. M., Saunders, H. E., *ISA Trans.* **1,** 325 (1962).

(181) Mayrsohn, H., O'Neal, Q., Air Pollution Symposium, Division of Water, Air, and Waste Chemistry, 148th Meeting, ACS, Chicago, Ill., Aug. 30–Sept. 4, 1964.

(182) McEwen, D. J., ANAL. CHEM. **35,** 1636 (1963).

(183) Menser, H. A., Heggestad, H. E., Street, O. E., *Phytopathology* **53,** 1304 (1963).

(184) Menser, H. A., Heggestad, H. E., Street, O. E., Jeffrey, R. N., *Plant Physiol.* **38,** 605 (1963).

(185) Miller, G. W., Long, L. E., George, G. M., Sikes, W. L., ANAL. CHEM. **36,** 980 (1964).

(186) Mills, J. L., Leudtke, K. D., Woolrich, P., Perry, L., "Emission of Oxides of Nitrogen from Stationary Sources in LA County," Rept. No. 2, 3, 4, Los Angeles County Air Pollution Control District, Calif.

(187) Miuva, T., Kimura, K., Tada, O., *J. Sci. Labour* **38,** 623 (1962).

(188) Mokhov, L. A., Mareva, N. S., *Zh. Prikl. Khim.* **35,** 2573 (1962).

(189) Monkman, J. L., Moore, G. E., Katz, M., *Am. Ind. Hyg. Assoc. J.* **23,** 487 (1962).

(190) Morrison, M. E., Rinker, R. G., Corcoran, W. H., ANAL. CHEM. **36,** 2256 (1964).

(191) Morrow, J. I., Turk, A., Baecht, W., Air Pollution Symposium, Division of Water and Waste Chemistry, 144th Meeting, ACS, Los Angeles, Calif., March 30–April 5, 1963.

(192) Morrow, J. I., Turk, A., Davis, S., Air Pollution Symposium, Division of Water and Waste Chemistry, 144th Meeting, ACS, Los Angeles, Calif., March 30–April 5, 1963.

(193) Mueller, P. K., Helwig, H. L., Alcocer, A. E., Gong, W. K., Jones, E. E., *Am. Soc. Testing Mater., Spec. Tech. Publication No.* **352,** 601 (1962).

(194) Mueller, P. K., Terraglio, F. P., Air Pollution Symposium, Division of Water, Air, and Waste Chemistry, 148th Meeting, ACS, Chicago, Ill., Aug. 30–Sept. 4, 1964.

(195) Mukai, M., Thomas, J. F., Tebbens, B. D., Air Pollution Symposium, Division of Water, Air, and Waste Chemistry, 148th Meeting, ACS, Chicago, Ill., Aug. 30–Sept. 4, 1964.

(196) Nader, J. S., Coffey, V. L., *Am. Ind. Hyg. Assoc. J.* **24,** 563 (1963).

(197) Nash, T., *Intern. J. Air Water Pollution* **8,** 121 (1964).

(198) Nicksic, S. W., Harkins, J., Fries, B. A., *J. Air Pollution Control Assoc.* **14,** 158 (1964).

(199) *Ibid.*, p. 224.

(200) Odescalchi, C. P., Straneo, G., *Rass. Med. Ind.* **29,** 356 (1960).

(201) Okita, T., *Kokuritsu Koshu Eiseiin.*, Bull. Inst. Public Health **12,** 127 (1963).

(202) Okita, T., Lodge, J. P., Jr., Air Pollution Symposium, Division of Water, Air, and Waste Chemistry, 148th Meeting, ACS, Chicago, Ill., Aug. 30–Sept. 4, 1964.

(203) Ordoveza, F., West, P. W., *Anal. Chim. Acta* **30,** 227 (1964).

(204) Ortman, G. C., Air Pollution Symposium, Division of Water, Air, and Waste Chemistry, 148th Meeting, ACS, Chicago, Ill., Aug. 30–Sept. 4, 1964.

(205) Pack, M. R., Hill, A. C., Benedict, H. M., *J. Air Pollution Control Assoc.* **13,** 374 (1963).

(206) Parkinson, T. F., Grant, L. G., *Nature* **197,** 479 (1963).

(207) Pate, J. B., Lodge, J. P., Jr., Neary, N. P., *Anal. Chim. Acta* **28,** 341 (1963).

(208) Pate, J. B., Lodge, J. P., Jr., Swanson, G. A., Ammons, B. E., Air Pollution Symposium, Division of Water, Air, and Waste Chemistry, 148th Meeting, ACS, Chicago, Ill., Aug. 30–Sept. 4, 1964.

(209) Popa, I., Stan, T., *Farmacia* **10,** 745 (1962).

(210) Proceedings National Conference on Air Pollution, U. S. Dept. Health, Education, and Welfare, *Public Health Service*, 1963.

(211) Ray, S. K., Long, R., *Combustion Flame* **8,** 139 (1964).

(212) Reckner, L. R., Scott, W. E., Air Pollution Symposium, Division of Water and Waste Chemistry, 145th Meeting, ACS, New York City, N. Y., Sept. 8–13, 1963.

(213) Regener, V. H., *J. Geophys. Res.* **69,** 3795 (1964).

(214) Ripley, D. L., Clingenpeel, J. M., Hurn, R. W., *Intern. J. Air Water Pollution* **8,** 455 (1964).

(215) Roesler, J. F., Stevenson, H. J. R., Nader, J. S., 57th Annual Meeting, APCA, Houston, Texas, June 21–25, 1964.

(216) Rondia, D., *Arch. Med. Sociale Hyg. Travail Med. Legale* **21,** 578 (1963).

(217) Rossano, A. T., Jr., Cooper, H. B. H., *J. Air Pollution Control Assoc.* **13,** 518 (1963).

(218) Russell, S., *Am. Ind. Hyg. Assoc. J.* **25,** 359 (1964).

(219) Salvin, V. S., *J. Air Pollution Control Assoc.* **13,** 416 (1963).

(220) Saltzman, B. E., Mendenhall, A. L., Jr., ANAL. CHEM. **36,** 1300 (1964).

(221) Saltzman, B. E., Wartburg, A. F., Air Pollution Symposium, Division of Water and Waste Chemistry, 145th Meeting, ACS, New York City, N. Y., September 1963.

(222) Saltzman, B. E., Wartburg, A. F., Air Pollution Symposium, Division of Water, Air, and Waste Chemistry, 148th Meeting, ACS, Chicago, Ill., Aug. 30–Sept. 4, 1964.

(223) Sanderson, H. P., Penner, P. E., Katz, M., ANAL. CHEM. **36,** 2296 (1964).

(224) Saringer, K., *Egeszsegtudomany* **7,** 25 (1963).

(225) Saruta, N., Ishinishi, N., Kodama, Y., Kunitake, E., *Fuknoka Acta Med.* **54,** 273 (1963).

(226) Sawicki, E., *Chemist-Analyst* **53,** 24, 56, 88 (1964).

(227) Sawicki, E., Bender, D. F., Hauser, T. R., Wilson, R. M., Jr., Meeker, J. E., ANAL. CHEM. **35,** 1479 (1963).

(228) Sawicki, E., Elbert, W. C., Stanley, T. W., *J. Chromatog.* **15,** in press (1965).

(229) Sawicki, E., Johnson, H., *Microchem. J.* **8,** 85–101 (1964).

(230) Sawicki, E., Johnson, H., *Mikrochim. Acta* (2–4), 435 (1964).

(231) Sawicki, E., Johnson, H., Stanley, T. W., ANAL. CHEM. **35,** 1934 (1963).

(232) Sawicki, E., Pfaff, J. D., Air Pollution Symposium, Division of Water, Air, and Waste Chemistry, 148th Meeting, ACS, Chicago, Ill., Aug. 30–Sept. 4, 1964.

(233) Sawicki, E., Stanley, T. W., Elbert, W. C., *Occupational Health Rev.* **16,** 3, 8 (1964).

(234) Sawicki, E., Stanley, T. W., Elbert, W. C., *Talanta* **11,** 1433–41 (1964).

(235) Sawicki, E., Stanley, T. W., Elbert, W. C., Pfaff, J. D., ANAL. CHEM. **36,** 407 (1964).

(236) Sawicki, E., Stanley, T. W., Hanser, T. R., Johnson, H., Elbert, W., *Intern. J. Air Water Pollution* **7,** 57 (1963).

(237) Sawicki, E., Stanley, T. W., Johnson, H., Air Pollution Symposium, Division of Water, Air, and Waste Chemistry, 148th Meeting, ACS, Chicago, Ill., Aug. 30–Sept. 4, 1964.

(238) Sawicki, E., Stanley, T. W., Johnson, H., *Microchem. J.* **8,** 257 (1964).

(239) Sawicki, E., Stanley, T. W., Pfaff, J., *Anal. Chim. Acta* **28,** 156 (1963).

(240) Sawicki, E., Stanley, T. W., Pfaff, J. D., Elbert, W. C., *Anal. Chim. Acta* **31,** 359 (1964).

(241) Sawicki, E., Stanley, T. W., Pfaff, J. D., Elbert, W. C., *Chemist Analyst* **53,** 6 (1964).

(242) Sawicki, E., Stanley, T. W., Pfaff, J. D., Johnson, H., ANAL. CHEM. **35,** 2183 (1963).

(243) Schuck, E. A., Cranz, F. W., Endow, N., Eldridge, R. W., Air Pollution Symposium, Division of Water and Waste Chemistry, 144th Meeting, ACS, Los Angeles, Calif., March 31–April 5, 1963.

(244) Schumann, C. E., Gruber, C. W., *J. Air Pollution Control Assoc.* **14,** 53 (1964).

(245) Seidman, G., Hindawi, I. J., Heck, W. W., 6th Conference on Methods in Air Pollution Studies, sponsored by California State Dept. of Public Health, Berkeley, Calif., Jan. 6–7, 1964.

(246) Serati, W. F., Mueller, P. K., Budinger, F. E., Air Pollution Symposium, Division of Water, Air, and Waste Chemistry, 148th Meeting, ACS, Chicago, Ill., Aug. 30–Sept. 4, 1964.

(247) Shoji, H., Yamamoto, T., Nishida, K., *Trans. Japan Soc. Civil Engrs.* **91,** 25 (1963).

(248) Skala, G. F., ANAL. CHEM. **35,** 702 (1963).

(249) Skramovsky, V., *Acta Unio Intern. Contra Cancrum* **19,** 733 (1963).

(250) Stalker, W. W., Dickerson, R. C., Kramer, G. D., *Am. Ind. Hyg. Assoc. J.* **24,** 69 (1963).

(251) Stalker, W. W., Meadows, F. L., American Industrial Hygiene Conference, Philadelphia, Pa., April 1964.

(252) Stanley, T. W., Sawicki, E., Johnson, H., Pfaff, J. D., *Mikrochim. Acta* **1965,** 384–91.

(253) Stephens, E. R., Air Pollution Symposium, Division of Water and Waste Chemistry, 145th Meeting, ACS, New York City, N. Y., Sept. 8–13, 1963.

(254) Stephens, E. R., ANAL. CHEM. **36,** 928 (1964).

(255) Stephens, E. R., Darley, E. F., 6th Conference on Methods in Air Pollution Studies, sponsored by California State Dept. of Public Health, Berkeley, Calif., Jan. 6–7, 1964.

(256) Stephens, B. G., Lindstrom, F., ANAL. CHEM. **36,** 1308 (1964).

(257) Stephens, E. R., Pattison, J. N., Air Pollution Symposium, Division of Water and Waste Chemistry, 144th Meeting, ACS, Los Angeles, Calif., March 30–April 5, 1963.

(258) Stern, A. C., *Am. J. Public Health* **53,** 1972 (1963).

(259) Stevenson, H. J. R., Sanderson, D. E., Altshuller, A. P., American Industrial Hygiene Assoc. Conference, Philadelphia, Pa., April 1964.

(260) Sullivan, J. L., Cleary, G. J., *Brit. J. Ind. Med.* **21,** 117 (1964).
(261) Sullivan, J. O., Warneck, P., *Mikrochem. J.* **8,** 241 (1964).
(262) Suworowa, S. N., Worobew, A. M., Rabowski, G. W., *Gigiena i Sanit* **28,** (1963).
(263) Swartz, D. J., Wilson, K. W., King, W. J., *J. Air Pollution Control Assoc.* **13,** 154 (1963).
(264) Sweeney, M. P., Swartz, D. J., Rost, G. A., MacPhee, R., Chao, J., *J. Air Pollution Control Assoc.* **14,** 247 (1964).
(265) Tabor, E. C., 5th Conference on Methods in Air Pollution Studies, sponsored by California State Dept. of Public Health, Los Angeles, Calif., Jan. 31–Feb. 1, 1963.
(266) Tada, O., *J. Sci. Labour* **40,** 185 (1964).
(267) Tada, O., *Reports Inst. Sci. Labour* **60,** 7 (1962).
(268) Tada, O., Nakaaki, K., *J. Sci. Labour* **39,** 595 (1963).
(269) Taga, T., *Heat and Management* (Tokyo) **12,** 1 (1963).
(270) Taylor, O. C., Cardiff, E. A., Mersereau, J. D., 6th Conference on Methods in Air Pollution Studies, sponsored by California State Dept. of Public Health, Berkeley, Calif., Jan. 6–7, 1964.
(271) Tebbens, B. D., Thomas, J. F., Mukai, M., Air Pollution Symposium, Division of Water and Waste Chemistry, 144th Meeting, ACS, Los Angeles, Calif., March 30–April 5, 1963.
(272) Tebbens, B. D., Thomas, J. F., Mukai, M., *Am. Soc. Testing Mater., Spec. Tech. Publication No.* **352,** 3–31 (1962).
(273) Thielen, V. C., Alcoce, A. E., Helwig, H. L., Air Pollution Symposium, Division of Water and Waste Chemistry, 144th Meeting, ACS, Los Angeles, Calif., March 30–April 5, 1963.
(274) Thieme, W., *Staub* **24,** 165 (1964).
(275) Thomas, J. F., Kato, T., Air Pollution Symposium, Division of Water and Waste Chemistry, 145th Meeting, ACS, New York City, N. Y., Sept. 8–13, 1963.
(276) Thomas, M. D., Ivie, J. O., Air Pollution Symposium, Division of Water and Waste Chemistry, 144th Meeting, ACS, Los Angeles, Calif., March 30–April 5, 1963.
(277) Thompson, J. R., Pace, D. M., *Can. J. Biochem. Physiol.* **40,** 207 (1962).
(278) *U. S. Atomic Energy Comm. Bureau of Mines*, **TID-8213,** OTS, Dept. of Commerce, Washington 25, D. C., January 1963, "Development of Depleted-Uranium Catalysts for Destruction of Air Pollutants in Automobile Exhaust. I. Development of Catalysts Containing Depleted Uranium, Rept. of Research Program."
(279) Valori, P., Alasia, A. M., Gualdi, G., *Nuovi. Ann. Igiane Microbiol.* **14,** 32 (1963).
(280) Vander Kolk, A. L., Van Farowe, D. E., American Industrial Hygiene Conference, Philadelphia, Pa., April 1964.
(281) Vandvacek, V., *Cesk. Hyg.* **8,** 333 (1963).
(282) Van Luik, F. W., Jr., Rippere, R. E., Anal. Chem. **34,** 1617 (1962).
(283) Veselaia, I. V., *Gigiena i Sanit.* **26,** 76 (1961).
(284) Voliberg, N. S., *Novoe v Oblasti Sanit-Khim. Analiza* (Raboty po Prom-Sanit Khim.) **1965,** 1962.
(285) Voznesenskii, D. N., Dolgopolova, V. Z., *Sb. Tr. Fiz-Khim. Lab. Vses Nauchn-Issled Inst. Okhanny Truda* **50** (1962).
(286) Waller, R. E., *Intern. J. Air Water Pollution* **7,** 773 (1963).
(287) Waller, R. E., Brooks, A. G. F., Cartwright, J., *Ibid.*, p. 779.
(288) Wartburg, A. F., Brewer, A. W., Lodge, J. P., *Ibid.*, **8,** 21 (1964).
(289) Wayne, L. G., "The Chemistry of Urban Atmospheres, Technical Progress Report" Vol. III, Los Angeles County Air Pollution Control District, December 1962.
(290) Weinstein, L. H., Mandl, R. H., McCune, D. C., Jacobson, J. S., Hitchcock, A. E., *Contrib. Boyce Thompson Inst.* **22,** 207 (1963).
(291) Weinstein, L. H., Mandl, R. H., McCune, D. C., Jacobson, J. S., Hitchcock, A. E., 57th Annual Meeting, APCA, Houston, Texas, June 21–25, 1964.
(292) Welford, G. A., Chiotis, E. L., *Anal. Chim. Acta* **31,** 376 (1964).
(293) West, P. W., Cimerman, Ch., Anal. Chem. **36,** 2013 (1964).
(294) Whitman, N. E., Johnston, A. E., *Am. Ind. Hyg. Assoc. J.* **25,** 485 (1964).
(295) Wyszynska, H., Zwolinski, J., *Roczniki Zakladu Hyg.* **13,** 503 (1962).
(296) Yanagisawa, S., Mitsuzawa, S., Hirose, A., Arai, M., *Japan Analyst* **12,** 1037 (1963).
(297) Yanysheva, N. Ia., Andrienko, L. G., Balenko, N. V., Kiveeva, I. S., *Gigiena i Sanit.* **29,** 7 (1964).
(298) Yanysheva, N. Ia., Kiveeva, I. S., Serzhautova, N. N., *Ibid.*, **27,** 3 (1962).
(299) Zaleiko, N. S., *J. Air Pollution Control Assoc.* **13,** 531 (1963).
(300) Zdrazil, J., Picha, F., *Cesk. Hyg.* **7,** 482 (1962).
(301) Zdrazil, J., Picha, F., *Pracovni Lekar* **15,** 62 (1963).
(302) *Ibid.*, p. 207.
(303) Zdrazil, J., Picha, F., *Ibid.*, **16,** 74 (1964).
(304) Zurio, N., *Med. Lavoro* **53,** 325 (1962).
(305) Zurlo, N., Griffini, A. M., *Ibid.*, p. 330.

Clinical Chemistry

G. R. Kingsley

Clinical Biochemistry Laboratory, Veterans Administration Center and Department of Biological Chemistry, School of Medicine, University of California, Los Angeles 25, Calif.

This Review is a continuation of the author's last review of publications of significant developments in clinical chemistry (*15A*) and covers the period from December 1963 to December 1964.

REVIEWS AND NEW BOOKS

Increasing interest of clinical chemists in their role in the paramedical field is illustrated in an ACS committee report by Mason (*21A*) which was devoted principally to a survey of legislative and judicial developments affecting chemists who provide laboratory services related to health, and whether or not they should direct clinical chemistry laboratories (*4A*). New developments in clinical analyses were reviewed by Van Kampen (*28A*); Schoen (*23A*) discussed clinical analysis, its functions and problems, and Bodansky (*1A*) made an interesting report on diagnostic biochemistry and clinical medicine. Whitby (*31A*) in an inaugural lecture at the University of Edinburgh, "Clinical Chemistry, Time for Investment," discussed two of the major problems facing clinical chemistry, "expanding work loads and the need to maintain and improve standards of accuracy." Reviews of specific subjects of interest to clinical chemists were made by: Korenman (*16A*) on biological methods of chemical analysis, Hamilton (*8A*) on biochemical analysis, Tomas and Heer (*25A*) and Escriu and Heer (*6A*) on biochemical ultramicroanalysis, Cocks (*5A*) on chemical microscopy, Heftmann (*9A*) on chromatography, Strickland (*24A*) on electrophoresis, Morrison (*22A*) on extraction, Williams and Bridges (*33A*) on fluorescence of solutions, White (*32A*) on fluorometric analysis, Hobbs (*12A*) on gas analysis, Horning and Van den Heuvel (*13A*) and Juvet (*14A*) on gas chromatography, Evans (*7A*) on infrared spectrometry, West (*30A*) on inorganic microchemistry, Kunin and McGarvey (*17A*) on ion exchange, Walton (*29A*) on ion exchange chromatography, Boltz (*2A*) on light absorption spectrometry, Ma and Gutterson (*19A*) on organic microchemistry, Mangold *et al.* (*20A*) on thin layer chromatography, Hirt (*11A*) on ultraviolet spectrometry, and Cambell and Brown (*3A*) on x-ray absorption and emission.

Thompson and King (*26A*) added over 200 pages of new material to their 2nd edition of "Biochemical Disorders in Human Disease" in which the disorders are presented in terms of the diseases of the various organs. Lynch *et al.* (*18A*) made an interesting presentation of methodology and clinical interpretation in the chemistry section of "Medical Laboratory Technology."

"Fluorescence Assay in Biology and Medicine," published by Udenfriend (*27A*) should be of interest to all clinical chemists. Henry's (*10A*) "Clinical Chemistry, Principles and Techniques" was quickly recognized as the most comprehensive available reference work in clinical chemistry. Chapters are presented on photometry and spectrophotometry, flame photometry, gasometric techniques, paper electrophoresis, micro and ultramicro technique and equipment, and accuracy, precision, and control charts. One or two methods are described for each specific analysis and most of the usual chemistry determinations are covered. However, some specialty determinations of interest to the clinical chemist are not covered, such as steroids, adrenaline, catecholamines, and toxicological assays, etc. Each method is presented under the following divisions: Principle, Reagents, Procedure, Notes, Accuracy and Precision, and Normal Values. The extensive bibliography presented in this book makes it invaluable in clinical laboratory work, as a reference book, and is the best since the publication of Peters and Van Slyke's classic "Quantitative Clinical Chemistry."

The procedures presented in the fourth volume of "Standard Methods of Clinical Chemistry" are referred to in the appropriate sections of this review.

APPARATUS AND EQUIPMENT

Progress in new and significant instrumentation for the chemist was reviewed by Mueller (*21B*). Boling (*3B*) constructed a flame photometer with simultaneous digital read-out for sodium and potassium. Stevenson *et al.* (*29B*) patented an apparatus for blood acid-base balance determination. A new manometric apparatus and technique for semimicro serum CO_2 and blood urea determination was developed by Afonso (*1B*). Spurr *et al.* (*28B*) constructed an inexpensive oxygen electrode to measure O during enzyme reactions. Natelson and Stellate (*22B*) designed an instrument to extract gases from large volumes of solvent for injection into the gas chromatograph. Taylor (*30B*) patented an apparatus to separate blood gases on chromatographic columns for thermal conductivity measurement. Lapaeva (*14B*) constructed an electrodialyzer for the simultaneous dialysis of two different protein solutions. New synthetic copolyetherester membranes for dialysis were described by Lyman *et al.* (*18B*). Houtsmuller (*13B*) constructed a simple spring knife for applying serum to viscous media. A simplified and improved apparatus for preparative column electrophoresis was described by Hochstrasser *et al.* (*11B*). Winsten *et al.* (*34B*) devised a continuous flow electrophoresis apparatus utilizing siliconized borosilicate glass microbeads. Lewis and Clark (*16B*) designed a preparative method for disk electrophoresis and a preparative device for collecting protein components from an electrophoretic column. Pasieka (*24B*) reported further improvements on high voltage paper electrophoresic apparatus.

Reports were published on specialized and advanced electronic equipment for direct or potential application to methodology by the clinical chemist by: Williams *et al.* (*33B*) who described a broad range recording micro-spectro-photo-densitometer, West and Hume (*32B*) who used radiofrequency plasma emission spectrophotometer, Schwarz and Shain (*26B*) who described generalized circuits for electroanalytical instrumentation, Morrison (*20B*) who described a generalized analog instrument synthesizer, Lauer (*15B*) who devised a multipurpose instrumental system for control of current or potential in electrochemical studies, Ewing and Brayden (*8B*) who studied the Heath analog computer as a versatile analytical tool, and Booman and Holbrook (*4B*) who used electroanalytical controlled potential instrumentation.

Draper and Mencken (*7B*) described an automatic pipetting machine for fluid volumes in the range from 0.01 to 1.09 ml. Holmes (*12B*) designed a transfer pipet for solvent extraction. Chen and Myers (*5B*) applied a new inexpensive automatic pipette to many procedures in clinical chemistry to measure volumes from 0.02 to 0.5 ml. Aikawa *et al.* (*2B*) described an automatic diluting buret made from a disposable infusion kit, buret, and a fish-tank aerator. Adaptations and modifications of the versatile Beckman DU spectrophotometer were made by; Zak *et al.* (*35B*) who converted to automatic recording-integrated densitometry, Underwood and Robertson (*31B*) who adapted components to make automatic recording photometric titrations, Rockenmacher and Farr (*25B*) who modified the fluorescence attachment for easy changing of all filters and by Mansell and Hunemorder (*19B*) who adapted the spectrophotometer to measure submicrogram amounts of mercury based on the absorption of Hg resonance line at 253.7 mμ. Danzer and Cohn (*6B*) constructed a Lucite cuvette to determine hemoglobin saturations with the Beckman DB spectrophotometer. Hansen and Horton (*10B*) designed spectrometer cells for single and multiple internal reflection studies in ultraviolet, visible, near infrared, and infrared regions. Onishi *et al.* (*23B*) devised an improved double-beam spectrophotometer in which the vibrating mirro-chopper system was replaced by a secter-gate system. Ludvigsen (*17B*) described a steel block homogenizer for instantaneous homogenization. A rotating Plexiglas microscope stage for urine sediments was presented by Faulkner and O'Mara (*9B*). Shipotofsky (*27B*) developed a simple, sensitive, versatile Geiger counter for scanning paper chromatograms.

AUTOMATION

A special report in *Chemical & Engineering News* (*2C*) called attention to the big swing to automation in analytical methods used in clinical laboratories. Another report (*1C*) cited the use of automatic monitoring of the human body's physiological functions in an elaborate electronic instrument system at the surgical wing of the National Institute of Health's clinical center at Bethesda, Md. A news report (*4C*) was made of a fully automatic system for mass analyses proposed by Drs. Gunnar and Junger which when fully utilized will perform 30 to 40 million analyses per year. This system will have 40 analytical channels with a capacity of 400–500 analyses per hour. Thiers and Oglesby (*80C*) analyzed the precision, accuracy, and inherent errors of the Technicon AutoAnalyzer by using two parameters; interaction between samples and instrumental drift, and applied correction techniques.

Thiers (*78C*) modified the AutoAnalyzer sample module to take samples directly from the vacutainer tube. A multichannel (10) analyzer was fabricated by Thiers (*79C*), for simultaneous across-the-board chemical analyses, to determine its economic feasibility and clinical value. McNair (*56C*) studied the effect of sample size on the accuracy and precision of the AutoAnalyzer. Grossman (*33C*) modified the AutoAnalyzer by the addition of a variable chart drive and toggle switch to permit rapid recordings. Everard and Seymour (*25C*) modified the sampler module of the AutoAnalyzer to permit the analysis of two sets of specimens simultaneously. Skeggs (*69C–71C*) constructed a multiple flow system for automatic sequential analysis which permits the delivery of several colored final streams to a colorimeter having multiple flow cells but a single optical path. Results of eight determinations; albumin, total protein, chloride, carbon dioxide, sodium, potassium, glucose, and urea N were steady-state recorded on precalibrated charts in readable form. McKay *et al.* (*55C*) devised a photometric system which can be adjusted to perform calculations automatically and provide direct readout of concentration. Lowell *et al.* (*50C*) described a simple, inexpensive transport apparatus for sequential colorimetric analysis which required automatic sample imput, colorimetric digital read-out and indexing. Kabara *et al.*

(*38C*) described a new instrument for evaluating electrophoretograms on various media.

The enzymatic alcohol dehydrogenation method for blood alcohol determination was adapted to the AutoAnalyzer by Leithoff (*47C*). Semiautomatic analysis of gluscosamine and galactosamine in protein hydrolysates were described by Walborg *et al.* (*82C*) and applied to four different chromatographic systems with the Spinco amino acid analyzer. Malmstadt and Hadjiioannou (*51C*) used an automatic spectrophotometric reaction rate method for the selective enzymatic determination of some alpha amino acids. Krampitz and Wieneke (*45C*) described the construction and operating procedures for a recording automatic amino acid analyzer. Kirkpatrick and Anderson (*43C*) described a method for resolving 11 basic amino acids from serum, urine, and protein hydrolysates with the Spinco analyzer. The manual amino acid and peptide colorimetric method utilizing trinitrobenzene–sulfonic acid has been adapted to the AutoAnalyzer by Hochstrasser (*36C*). Bonnafe (*9C*) described an automated analyzer for the chromatographic separation and determination of amino acids, peptides, and proteins. Carstairs (*13C*) carried out a continuous colorimetric analysis of amino acids in column effluents by means of the reaction with ninhydrin in the presence of hydrindantin. King (*42C*) used the amino acid AutoAnalyzer to measure the ninhydrin positive component of urine. Kolbezen and Eckert (*44C*) used a sampling mechanism in conjunction with a Beckman DK-2 spectrophotometer to collect gas samples from 10 separate atmospheres and record the ammonia concentration of each.

Golub (*31C*) adapted the direct and total bilirubin method of Rand and DiPasqua, and Gambino (*28C*) the bilirubin method of Jendrassik and Grof to the AutoAnalyzer. Wieme and van Raepenbusch (*86C*) adapted the Kingsley–Robnett Cornith CA colorimetric calcium method to the AutoAnalyzer. Malmstadt and Hadjiioannou (*52C*) titrated micro amounts of serum calcium by an automatic procedure in 10 seconds. Summers *et al.* (*77C*) described a procedure for the automated simultaneous determination of copper and magnesium using a pressurized dialysis system to increase sensitivity. A completely automated system for simultaneous determination of calcium and phosphorus with the AutoAnalyzer was reported by Kessler and Wolfman (*41C*). Jones and McGuckin (*37C*) described a semiautomatic procedure for complexometric titration of both calcium and magnesium using Eriochrome blue SE as indicator. Hill (*34C*) adapted Schachters serum magnesium method to the AutoAnalyzer for fluorometric measurement of magnesium. Nevius and Lanchantin (*61C*) adapted the AutoAnalyze flame photometer to the use of natural gas instead of propane. Merrills (*57C*) described a semiautomatic method for the estimation of catecholamines in blood and tissues. Etienne *et al.* (*24C*), Cooper *et al.* (*17C*) and Boy (*10C*) have described and studied methods for determining cholesterol with the AutoAnalyzer. Kenny and Jamieson (*40C*) constructed a special glass unit which eliminates preliminary solvent extraction in the determination of cholesterol with the AutoAnalyzer. Zak and Epstein (*89C*) modified the Zimmerman color reaction to permit the determination of 17-keto steroids with the AutoAnalyzer. Automated techniques were developed by Antonis (*3C*) for the determination of cholesterol, fatty acids, and phospholipids. Automated digestion for the determination of serum phospholipids with the Technicon Kjeldahl Digestor was carried out by Whitley and Alburn (*84C*). Lofland (*49C*) devised a method for the determination of triglycerides in serum using isopropanol and the AutoAnalyzer.

Griffiths (*32C*) adapted the diacetyl–naphthol reaction for the estimation of creatine to the AutoAnalyzer. Modification of the glucose manifold of the AutoAnalyzer was made by Glenn (*30C*) for the determination of amylase in pancreatic juice.

Schwartz and Bodansky (*73C*) reviewed the automated methods for the determination of enzyme activity. Automated methods were described: by Brooks (*11C*), Strandjord and Clayson (*76C*), and Hochella and Weinhouse (*35C*) for the determination of lactic dehydrogenase and lactic acid, by Schaffert *et al.* (*72C*) for the determination of serum glutamic oxalactic and glutamic pyruvic transaminases, by Epstein *et al.* (*23C*), Baginski *et al.* (*5C*), Tietz and Green (*81C*), Morganstern *et al.* (*59C*), Keay and Trew (*39C*), and Bell *et. al.* (*6C*) for the determination of phosphorus and alkaline phosphatase. Pardue *et. al.* (*63C*) described a simple automatic potentiometric method for the assay of glucose oxidase in blood and plasma. Similar procedures were used by Frings and Pardue (*27C*) for the enzymatic determination of galactose, and by Malmstadt and Pardue (*54C*) and Pardue (*62C*) for the determination of glucose. A direct automated enzymatic method for the determination of glucose was reported by Getchell *et al.* (*29C*). Discombe (*21C*), Burns *et al.* (*12C*), and Crofford and Lacy (*18C*) also described automated blood glucose methods. Fingerhut *et al.* (*26C*) applied the ferrocyanide-phosphomolybdate reaction to an automated serum glucose method and Russell *et al.* (*68C*) used the *O*-toluidine reagent. Procedures for the simultaneous automated determinations of glucose and urea *N* were described by Natho and Frankel (*60C*), Winsten (*87C*), and Sobolewski (*74C*). Chen and Sharton (*16C*) evaluated the automated serum dye–albumin method by means of 2 (4-hydroxyazobenzene) benzoic acid. Catravas (*15C*) developed an automatic continuous method for the alkaline hydrolysis and detection of peptides. Catanzaro (*14C*) studied the automated digestion techniques of the Technicon Kjeldahl Analyzer. Curtain (*20C*) described an automatic multiple column apparatus for analytical zone electrophoresis of serum proteins.

Partially automated methods for the determination of serum protein–bound iodine were reported by Widdowson and Northam (*85C*), Stevens and Levandoski (*75C*), Malmstadt and Hadjiioannou (*53C*), Leonards and Davoren (*48C*), and Benotti and Benotti (*7C*). Fully automatic techniques for the determination of iodoamino acids in protein digests were described by Block and Mandl (*8C*) and for protein-bound iodine by Riley and Gochman (*66C*). Meschia (*58C*) adapted Brodies' antipyrine method for determination of total body water to the AutoAnalyzer. Zak (*88C*) applied the 4,7-diphenyl-1,-10-phenanthroline sulfonate color reagent to an automated serum iron procedure. Pentz (*65C*) adapted a taurine method requiring a liquid-liquid extraction with an organic solvent to the AutoAnalyzer. Pellerin (*64C*) adapted a urea method based on the formation of a red color complex between urea, diacetyl monoxime, and thiosemicarbazide to the AutoAnalyzer. The carbonate uric acid method was adapted to the AutoAnalyzer by Crowley (*19C*). Lanzkowsky *et al.* (*46C*) adapted to the AutoAnalyzer a procedure for the determination of D-xylose in urine in which furfural formed from pentoses develops a pink color with *p*-bromoaniline. Robinson and Watts (*67C*) described an automated fluorometric analytical method for catecholamines. Walter and Gerarde (*83C*) developed a precision capillary in a plastic adapter for rapid semiautomatic measurement of microspecimens. Dwiggins (*22C*) used new automated x-ray methods for determining elements in organic materials.

CONTROL AND PRECISION OF CLINICAL CHEMISTRY METHODS

Nelson (*7D*) reviewed statistical developments and applications in chemistry for the period October 1962 to October 1964. Whitby (*13D*) reviewed various methods of controlling and improving the accuracy of clinical chemistry laboratories. Henry (*3D*) in his report on some applications of statistics to clinical chemistry discussed frequency distribution, standard deviation, overall error, and control of laboratory error in application of statistics to analytical chemistry data. Weinberg and Barnett (*12D*) found no evidence that analysts achieved a closer approach to average known values which indicated absence of analytic bias in quality control. Curnow and Sheard (*2D*) used probability graph paper to plot cumulative percentage frequency distribution. Calder (*1D*) illustrated the importance of the statistical approach in analytical chemistry by examples chosen from the conventional and photometric fields. Hoffmann and Wald (*5D*) discussed the number plus method of quality control of laboratory accuracy in a promising preliminary study. Young (*14D, 15D*) believed that the greatest immediate practical usefulness of quality control was to extend knowledge relating to the "concentration ranges" and "concentration profiles" of body constituents in clinically defined states of health and disease. Tonks (*10D*) in a survey of the accuracy and precision of 170 Canadian laboratories found that 40% of the clinical chemistry determinations reported fell outside the allowable limits of errors. Porter (*9D*) in an intermedical quality-analysis survey presented factors responsible for observed variations in results obtained. Peckham and Dimmette (*8D*) reported statistical data on quality control of automatic and manual chemistry methods for a 1700-bed hospital. Turpin (*11D*) presented a method for determining the number of medical technologists required to perform routine laboratory procedures. Hersey *et al.* (*4D*) described the preparation of a lyophilized alkaline phosphatase serum enzyme for quality control. Mull and Murphy (*6D*) found that tourniquets and light exercise of the forearm did not alter significantly routine blood chemistry determinations.

AMINO ACIDS

Rapp (*25E*) investigated the determination of amino acids by reaction with 1-fluoro-2,4-dinitrobenzene. Body fluid amino acids were estimated by complexing with copper in alkaline solution by Malangeau *et al.* (*17E*). Blackburn and Lee (*3E*) studied the cadmium acetate–ninhydrin amino acid color reaction as a micro method for estimation of amino acids. Frame (*6E*) presented a standard procedure for the determination of free amino acids in plasma and urine by the gasometric ninhydrin–carbon dioxide method with the Van Slyke–Neill Manometer. Jacobs (*10E*) determined amino acids in protein hydrolysates with Zeo-Karb 225 and Amberlite 120 ion exchange resins. Belen'kii and Orestova (*2E*) presented a gas chromatographic method for the micro determination of primary amino groups in amino acids and polypeptides. Methods for determination of individual amino acids were reported: by Warren *et al.* (*34E*) for alanine by using Leuconostoc citrovorum 8081 as the test organism; by Roston (*26E*) for cysteine by color reaction with noradrenochrome; by Gustus (*7E*) for combined cystine by a temperature dependent color reaction with liquid ammonia at 25° C. in the presence of oxygen; by Carson and Wong (*5E*) for detection of sulfur containing amino acids with a new color reaction; by Umberger and Fiorese (*32E*) for determination of glycine with *p*-nitro-benzoyl chloride and pyridine; by Meiss *et al.* (*19E*) and Sowerby and Ottaway (*30E*) by estimation of glutamic acid by enzymatic methods; by Ramadan and Greenberg (*24E*) who determined glutamine and asparagine by an enzymic micro method, and by Lack and Smith (*13E*) who developed a sensitive specific enzymic spectrophotometric assay of reduced glutathione using maleylpyruvic acid isomerase. Updike and Dounce (*33E*) described a reaction of histidine with oxygen and biuret reagent which yielded color. Baldridge and Greenberg (*1E*) determined histidine in blood by reacting with L-amino acid oxidase in the presence of borate ions to yield an enolborate complex of imidazolepyruvic acid. Summer and Hawes (*31E*) determined free proline in serum by a special ninhydrin method, and Myhill and Jackson (*21E*) separated proline and hydroxyproline by the use of the nitrous acid reaction and thin layer chromatography. Enzymatic colorimetric methods for determination of serum phenylalanine and tyrosine were reported by Knapp *et al.* (*12E*) and La Du *et al.* (*14E*). Opienska-Blauth *et al.* (*23E*) reported a new rapid colorimetric method for the determination of tryptophan. Saifer and Gerstenfeld (*29E*) modified and quantitated the glyoxylic method of Fischl for tryptophan. Jutisz and de la Llosa (*11E*) reviewed recent advances in the realm of amino acid chromatography. Said and Fleita (*28E*) reported the study of a new color reaction of naturally occurring alpha amino acids with 1,2,3-phenalenetrione. A technique for the chromatographic determination of free amino acids in small amounts of whole blood was described by Lebedeva (*15E*). Hamilton (*8E*) devised a fully automatic procedure to determine 10^{-8} mole of amino acids by ion exchange chromatography. Matheson (*18E*) reported an improved method of separation of ether soluble dinitrophenol (DNP)-amino acids by partition chromatography. Hara (*9E*) separated amino acids by using a 5-compartment ion exchange cell and Mougey and Mason (*20E*) used Sephadex in glass columns to separate iodoamino acids. Nybom (*22E*) analyzed amino acids by electrophoresis on cellulose coated glass. Amino acids were determined by high voltage electrophoresis in serum by Braun (*4E*) and Mabry and Karam (*16E*) and by Sackett (*27E*) in urine.

BLOOD PRESERVATION, CLOTTING FACTORS, GASOMETRIC ANALYSIS, pH, AND VOLUME

Astrup and Andersen (*3F*) reviewed micromethods for measuring acid–base values of blood including nomograms, and determination of total CO_2, pCO_2, pH, and calculation of acid–base values. Andersen (*1F, 2F*) published papers concerned with the interpretation and determination of blood acid–base status and oxygen saturation and acid–base alignment nomogram for calculation of base excess after direct measurement of pH, pCO_2, and hemoglobin concentration. Intracellular pH of erythrocytes was determined by Thomason (*31F*) by measuring the distribution of 5,5-dimethyl-2,4-oxazolidinedione between erythrocytes and plasma and Manfredi (*19F*) determined total body intracellular pH in normal subjects with this agent. Ware *et al.* (*35F*) suggested a dilution technique for capillary blood pH to prevent pH change. Rebar (*28F*) patented a bibulous carrier impregnated with a solution of indicator material. Johnston (*13F*) described standardized techniques for the determination of oxygen saturation of blood. Techniques for blood oxygen determination by gas chromatography were reported by Chambliss and Nouse (*6F*) and Russ *et al.* (*29F*). Severinghaus (*30F*) obtained fast response for respiratory gas sampling by heating a membrane covered polarographic oxygen electrode to 80° C. Laughlin *et al.* (*16F*) designed a micromethod for measurement of PO_2 in a drop of blood. Maås *et al.* (*18F*) described a rapid microspectrophotometric method for determining hemoglobin oxygen saturation. Tsao and Vadnay (*32F*) devised a system for continuous measurement of blood PO_2 and PCO_2, consisting of a countercurrent gas exchange unit, a microconductivity cell and an alternating-current conductivity bridge. Spectrophotometric methods for determination of oxygen saturation were reported by Gambino *et al.* (*11F*) who designed a reflection oximeter and by Falholt (*8F*) who measured blood oxygenated at 625 mμ and reduced at 805 mμ. Updegraff and Huckabay (*33F*) described an apparatus for rapid gas analysis for CO_2, O_2, hydrocarbon gases, and H_2 which was applicable to multicomponent systems. Berglund *et al.* (*4F*) described a method for the determination of CO_2 tension of whole blood by pH measurements and interpolation. Martinek (*20F*) eliminated the absorption of CO_2 with alkali in determining CO_2 with the Natelson microgastometer. Blood gas chromatographic methods for the determination of nitrogen were described by Farhi *et al.* (*9F*) and by Bowes (*5F*) for nitrous oxide, nitrogen, and oxygen.

An appraisal of the methodology of the prothrombin assay methods in common use was made by Ware (*36F*). Quick (*25F*) discussed the progress and problems of control of anticoagulant therapy, the analytical and clinical aspects of the one- and two-stage prothrombin methods (*27F*) and the experimental variables of the partial thromboplastic test (*26F*). The effect of warfarin, ethyl biscoumacetate, and phenindione on blood clotting factors were studied by Noren and Quick (*21F*). Owren (*22F*) discussed the criteria of response of different laboratory methods for anticoagulant therapy control to blood factors for selection of the proper method. Gollub and Ulin (*12F*) proposed a useful modification of the prothrombin time test. Lanchantin *et al.* (*15F*) studied some of the chemical and physical properties of purified human plasma prothrombin. Fenichel *et al.* (*10F*) used a stabilized partial thromboplastic reagent in the partial thromboplastic test. Kollath *et al.* (*14F*) improved the method of Monkhouse and Jaques for the determination of heparin in blood plasma. Heparin separated by paper electrophoresis was located by staining with Auramine O, Malachite green, or Brilliant green by Conti *et al.* (*7F*). Vermylen *et al.* (*34F*) assayed fibrinogen by fibrin polymerization time. Paloheimo (*23F*) studied the seasonal variations and plasma volumes of healthy men. Little and Williams (*17F*) determined Evans Blue in plasma by alkaline cadmium sulfate precipitation and extraction of dye with butyl alcohol. Pawan and Hoult (*24F*) determined total body water in man by ethanol dilution based on the fact that 1 to $1^{1}/_{2}$ hours after oral dose of ethanol, its concentration is practically the same in blood–water and urine–water (dose 0.35 gram/kg. body weight).

CARBOHYDRATES

Free (*12G*) reviewed enzymic determination of glucose, and Fales (*8G*) presented standardized procedures for the glucose–oxidase peroxidase methods. Studies of the glucose oxidase–peroxidase glucose methods were made by Hansen (*15G*) to investigate specificity, by Hjelm and de Verdier (*16G*) to search for substances causing unspecific oxidation of chromogen, by Rutman (*36G*) who studied the effect of manganese and by Meites and Bohman (*25G*) who evaluated the precision of an ultramicro method. Smith *et al.* (*40G*) described an accurate glucose method employing *Lactobacillus casei*. Achari *et al.* (*1G*) determined true blood glucose by condensation with benzidine. Relander and Raiha (*33G*) compared the enzymatic and *O*-toluidine blood glucose methods. Campbell and King (*5G*) modified the cuprous oxide–neocuprione method for the ultramicro determination of glucose. Jacobs (*17G*) improved the *O*-toluidine blood glucose method. Watson and Stevenson (*44G*) compared four micro blood glucose methods and found the *O*-aminotoluene condensation and the glucose oxidase–tolidine methods the most satisfactory. Klein and Weissman (*21G*) identified clinically important sugars as their phenylosazones by infrared absorption spectrometry. Meites and Bohman (*24G*) stabilized glucose in shed blood for 2 hours by dilution with water (1:80, v./v.). Ruiter *et al.* (*35G*) observed no loss of glucose in serum separated immediately from blood clot during 48 hours. Apparently considerable interest has arisen concerning patents on methods to determine glucose as shown by the number recently issued. Patents were obtained by or issued to: Free (*10G*) for determining glucose in blood and urine (*11G*) and to Miles Laboratories (*26G*, *27G*) by modification of the glucose–oxidase reagents for incorporation into paper strips, etc. Patents embodying the glucose–oxidase principle were also issued to Western Laboratories (*46G*), Teller (*41G*), and Smeby (*39G*). Other methods of determining glucose were patented by Momose (*28G*) who used 3,6-dinitrophthalic acid, by Gold (*14G*) and by Kantorovich (*19G*) who employed reduction of ferricyanide in the presence of zinc and cadmium salts. Scheffer and Kickuth (*38G*) separated 17 important sugars by paper chromatography. Gagliardo and Zambotti (*13G*) separated esterified sugars by gas chromatography and Wells, Chin and Weber (*45G*) determined serum and urinary sugars by gas liquid chromatography of their trimethylsilyl ethers. A new solvent was introduced by Pietrusiewicz and Pidek (*31G*) for better separation by paper chromatography of glucosamine, glucose, galactose, mannose, and rhamnose. Methods for the determination of blood galactose were reported; by Ford and Haworth (*9G*) who used a galactose oxidase–peroxidase system, by Bowden (*2G*) who employed a glucose oxidase–catalase reagent, and by Relander (*32G*) and Watson (*43G*) who used *O*-toluidine after removal of glucose by fermentation. Brewer and Tarlov (*3G*) described a clinical test for galactosemia which depends upon the inability of erythrocytes from the galactosemic patients to reduce methemoglobin when galactose is the sole source of energy. A patent was issued to Rorem and Lewis (*34G*) for a test paper to detect galactose in urine. Cessi and Serafini-Cessi (*6G*) determined D-galactosamine by condensation with acetylacetone in methanol in the presence of triethylamine and pyridine and reaction of the 2-methylpyrrole with *p*-dimethylaminobenzaldehyde. Percheron (*30G*) determined fructose colorimetrically by the theobarbituric acid reaction. Pakianathan (*29G*) estimated fructose colorimetrically by the pink color formed with thymol–HCl. McKay (*23G*) modified the orcinol method to determine plasma pentose and minimize glucose interference. Sato (*37G*) determined serum protein bound hexose with a phenol–sulfuric acid reagent. Klassen and Lanzkowsky (*20G*) determined D-xylose colorimetrically in blood. Mabry *et al.* (*22G*) described a method for the separate measurement of individual sugars by high-voltage paper electrophoresis. Enzymic methods for the determination of glycogen were reported by Johnson *et al.* (*18G*), Bueding and Hawkins (*4G*) and by Topi and Zane (*42G*). Di Ferrante (*7G*) separated three fractions of acid mucopolysaccharides in human urine by chromatography on ion exchanger cellulose.

CATIONS AND ANIONS

Scribner and Margoshes (*48H*) reviewed emission spectrometry covering emission flame photometry and absorption flame photometry for the period 1962 through December 1963. Flame photometry in biochemistry and biological analysis was reviewed by Krampitz and Doepfmer (*30H*). Valberg *et al.* (*57H*) determined calcium, magnesium, copper, and zinc in red blood cells by emission spectrometry. MacFate (*36H*) made an extensive review of methods for calcium determination in biologic materials, especially blood plasma. Zettner and Seligson (*61H*) used a special diluent to abolish the effect of depressors in atomic absorption determination of calcium in serum. Loken *et al.* (*33H*) modified the Beckman direct oxyacetylene sprayer burner by using acetylene as the spraying gas. Complexometric, chelating, colorimetric calcium methods were described by: Watson and Rogers (*60H*) who used 1,2-diaminocyclohexane-*N*-tetraacetate, Wallach and Steck (*59H*) who used a fluorescent indicator 2,4-bis[*N*,*N'*-d (carboxymethyl)aminomethyl] fluorescein, Vedso and Rud (*58H*) who used EDTA and cation exchange resin, Spach and Erny (*52H*) who used a substituted phenylazonaphthalenedisulfonate dye for urine and feces, Sherrick and De la Huerga (*49H*) who used *N*-hydroxynapthalimide, Mayr (*37H*) who determined calcium in erythrocytes with ammonium rhodanate and K–Na–Co hexanitrite, Kepner and Hercules (*29H*) who used calcein for fluorometric measurement, Herrero-Lancina and West (*26H*) who used calcichrome, Fingerhut and Miller (*20H*) who removed bilirubin with potassium ferricyanide and hydrogen peroxide and then used Calcein or Cal-red, Bowden and Patston (*5H*) who used Calcon, Dunsbach (*17H*) who employed ethylene glycol bis(beta-aminoethyl ether) tetraacetic acid, and Shimomura *et al.* (*50H*) who employed bioluminescent protein aequorin. Lindstrom and Milligan (*32H*) studied six derivatives of glyoxal bis(2-hydroxy-anil) as direct calcium reagents. Mueller (*41H*) identified the different nuclear fast red dyes. Lumb (*35H*) described a direct method for measuring the level of ionic calcium in serum. Decker *et al.* (*13H*) determined magnesium and calcium in spinal fluid by atomic absorption spectrometry. Munday and Mahy (*42H*) determined plasma ultrafiltrate calcium and magnesium by ultrafiltration and flame photometry. Ferguson *et al.* (*19H*) described a rapid sensitive spectrophotometric procedure for determining calcium and magnesium simultaneously with chlorophosphonazo III. Beale and Bostrom (*3H*) used sensitive titrimetric methods to determine calcium and magnesium. Stewart and Frazer (*54H*) reviewed the physiological relationships of magnesium in man. Stewart *et al.* (*53H*) and Horn and Latner (*27H*) determined magnesium in blood and urine by atomic absorption spectrometry. Sky-Peck (*51H*) determined magnesium in serum and urine by formation of a red thiazole-yellow magnesium hydroxide complex. Butler *et al.* (*7H*) compared the ammonium phosphate, Titan yellow, and EDTA methods for serum magnesium determination. Spectrophotometric magnesium methods for body fluids were reported by: Rice and La Para (*44H*) who employed the "Mann" dye; Gusev *et al.* (*24H*) who used picraminazo; Ellis and Bishop (*18H*) who substituted congo red for Titan yellow; Diehl *et al.* (*14H*) who employed *O,O′*-dihydroxyazobenzene for fluorometric measurement; and by Burcar *et al.* (*6H*) who used magon. Anast (*1H*) noted the unreliability of Titan yellow as a magnesium reagent in the presence of gluconate.

Cotlove (*10H*) reviewed the determination of chloride in biological materials. Crockson (*11H*) and Schoenfeld and Lewellen (*47H*) determined chloride ion spectrophotometrically with a mixed solution of ferric nitrate and mercuric thiocyanate. Rodkey and Sendroy (*45H*) extended the silver iodate method of Sendroy to the microspectrophotometric determination of chloride in plasma and cells. Baar (*2H*) estimated chlorides colorimetrically in serum with dichlorodihydroxybenzoquinone mercuric salt. Cotlove (*9H*) used an isotope Cl^{36} dilution method for determination of true chloride content of body fluids and tissues.

Hall (*25H*) determined submicrogram amounts of fluorine in biological specimens by a new direct color reaction with alizarin complexon. Teixeira *et al.* (*56H*) determined total cations in biological fluids by an ultramicro dialysis technique. A system for analysis of clinical acid–base states by micro methods was described by Freier *et al.* (*21H*). A simple method was presented by Rovner and Conn (*46H*) for the simultaneous estimation of plasma volume, extracellular fluid volume, total body sodium, and potassium in which T-1824, Br^{82}, Na^{22}, and K^{42} were utilized.

Moore and Wilson (*39H*) described a ternary glass system ($Na_2O–Al_2O_3–SiO_2$) for producing electrodes for determining sodium in biological fluids. Methods for the determination of sodium and potassium in biological fluid were presented by: Gregoriadis (*23H*) who used paper electrophoresis, Friedman *et al.* (*22H*) who used cation-response glass electrodes and Bloomer *et al.* (*4H*) who applied acetone as a solvent and CsCl and $(NH)_2\ HPO_4$ as radiation buffers for flame spectrophotometric measurement. Methods for potassium determination in serum were presented by Kaplar (*28H*) who titrated with sodium tetraphenyl boron and Variamine blue as indicator and by Lazarov (*31H*) who used a colorimetric method which depended upon the formation of insoluble $K_2Pb[Ca(NO_2)_6]$. Procedures for the determination of phosphorus in biological fluids were described by: Dryer and Routh (*15H*) who standardized a method employing semidine–HCl to reduce phosphomolybdic acid; Negrin (*43H*) who employed HI to reduce phosphomolybdic acid; Morrison (*40H*) who described a simple fast micro method; London and Marymont (*34H*) who used heat coagulation to separate inorganic phosphate from proteins; Davis *et al.* (*12H*) who utilized flame emission continuum centered at 540 mμ; and Christian and Knoblock (*8H*) who used direct argentometric titration. Miller *et al.* (*38H*) determined inorganic sulfate in urine by use of radioactive barium chloride. A submicro determination of sulfur in organic substances was described by Stoffyn and Keane (*55H*) in which Carius oxidation and barium chloranilate were used. Dugenetay *et al.* (*16H*) described a sensitive flame spectrophotometric method for the determination of rubidium in biological materials at 795 mμ.

DRUGS

p-Aminosalicylic acid was determined alone and in presence of isoniazid by Hamilton *et al.* (*6J*) by coupling with transcinnamaldehyde to form a Schiff base. Vercruysse (*16J*) measured doriden in autopsy material by thin layer chromatography on silica gel with petroleum ether–methyl alcohol. Street and McMartin (*15J*) studied the action of concentrated H_2SO_4 on 5,5-disubstituted barbituric acid derivatives to develop a satisfactory procedure. Sato (*13J*) used ascending paper chromatography to identify nonbarbiturate sedatives in urine. Koechlin and D'Arconte (*9J*) determined chlordiazepoxide (Librium) in plasma by a spectrofluorometric micro method in which a fluorescent 4,5-epoxide is formed. Kanter (*7J*) described a technique for removal of urea interference in determination of meprobmate with *p*-dimethylaminobenzaldehyde. Lindfors (*10J*) identified meprobamate by thin layer chromatography in the presence of sedatives other than barbiturates. Musil *et al.* (*12J*) determined 6-azauridine in serum by separation on Sephadex G25 column and measuring the differential absorbance at $A_{260}–A_{280}$. A screening test for isoniazid was described by Eidus and Hamilton (*3J*) in which a transcinnamaldehyde reacts to form a colored hydrazone derivative of isoniazid. Analytical procedures for the estimation of phenothiazines in urine were reported by: Gold *et al.* (*4J*) who studied six different tests; Mellinger and Keeler (*11J*) who measured the fluorescence characteristics of oxidized phenothiazine and by Eagleson (*2J*) who identified important phenothiazines by measurement of R_f values. Gothelf and Karczmar (*5J*) described a quantitative extraction procedure for the determination of chlorpromazine in tissues. Kern (*8J*) determined phenyl and tolylsulfonylurea derivatives in blood by nitration, reduction, diazotization, and coupling with *N*-(1-naphthyl)ethylenediamine to produce color for measurement. Sil'vestrov *et al.* (*14J*) reported a fluorometric technique for determination of tetracycline in biological fluids. Cramer and Isaksson (*1J*) determined quinidine in plasma photofluorometrically after extraction with benzene.

LIPIDS

Giarnieri (*20K*) reviewed oxidative, volumetric, gravimetric, nephelometric, gasometric, and chromatographic methods for the determination of total blood lipids. Sperry (*39K*) standardized solvent extraction and gravimetric techniques for the determination of total lipids in serum or plasma. Techniques for the determination of total lipids in biological specimens were described by: Searcy *et al.* (*37K*) who used a turbidimetric technique; Dales (*10K*) who quantitated tissue lipids with Red O colorant; and by Bowers *et al.* (*6K*) who used silicic acid column chromatography for total fecal fats. Prevot (*32K*) reviewed recent applications of gas chromatography to the analysis of lipids. Kuksis (*25K*) applied direct gas chromatographic fractionation for the measurement of neutral lipids of natural origin. Separation of minute amounts of lipids by thin layer chromatography and gas chromatography was made by Dobiasova (*13K*) in tissues, Araki (*1K*) in serum, and by Mangold *et al.* (*27K*) who labeled the lipids by reaction with diazomethane C^{14} or acetic anhydride–C^{14}. Schain (*34K*) obtained a patent for a blood serum fat reagent. Methods for the colorimetric micro determination of long-chain fatty acids were reported by Duncombe (*15K*) and Konitzer *et al.* (*22K*). Duncombe (*16K*) described a rapid colorimetric method for micro determination of nonesterified fatty acids in plasma. Reinhold *et al.* (*33K*) standardized the colorimetric hydroxylamine–HCl method for measurement of total esterified fatty acid and triglyceride in serum. Methods for the determination of triglycerides were reported by: Pinter *et al.* (*31K*) who used glass-fiber paper chromatography for isolation and colorimetric estimation by hydroxylaminolysis; Nicolaysen and Nygaard (*29K*) who simultaneously isolated phospholipids and triglycerides; Krell and Hashim (*23K*) who employed thin layer chromatography and infrared spectrophotometry; Freeman (*17K*) who used silicic adsorption and infrared measurement; and by Carlson (*7K*) who devised immediate dilution of plasma lipid extract and change of column to a batch procedure. Christophe and Matthijs (*9K*) developed a routine method for serum glyceride glycerol. Glycerol was determined in serum by: Galanos *et al.* (*19K*) who used a binary solvent system; Jellum and Bjornstad (*21K*) who employed gas liquid chromatography; Kreutz (*24K*) who used glycerol kinase; and by Wheeldon *et al.* (*40K*) who determined glycerol, inositol, serine, and ethanolamine in a phospholipid hydrolyzate simultaneously.

Barclay *et al.* (*4K*) determined the density range by ultracentrifugation of high-density serum lipoproteins. High and low density serum lipoproteins were measured fluorometrically, by the amount of protoporphyrin IX bound to the lipoprotein fraction separated immunochemically or electrophoretically by Searcy *et al.* (*36K*). Methods for the estimation of lipoproteins in biological specimens were described by: Schjeide *et al.* (*35K*) who studied the uptake of stains; McFarlane (*28K*) who prestained with Sudan blue prior to electrophoresis; Carmichael (*8K*) who employed tetrazolium salt reduction in tissues; Barre and Labat (*5K*) who separated alpha and beta lipoproteins by the phytic acid method; Dangerfield and Faulkner (*11K*) who used sulphated polysaccharides; Armstrong (*2K*) who presented electrophoretic and immunochemical procedures for beta lipoprotein; Fried and Hoeflmayr (*18K*) who proposed a simple indirect colorimetric method for beta lipoproteins; Di Leo (*12K*) who stained before and after electrophoresis with Sudan Black B; Peeters *et al.* (*30K*) who found Sudan Black B gave best staining among several dyes studied; and by Lees and Hatch (*26K*) who obtained sharper separations of lipoproteins by paper electrophoresis in an albumin-containing buffer. Ayrault-Jarrier *et al.* (*3K*) found detergents bound to purified alpha lipoprotein isolated from human serum by ultracentrifugation. Skipski *et al.* (*38K*) described a method for quantitative separation of phospholipids by thin layer chromatography and a similar method was described by Doizaki and Zieve (*14K*) for phosphatides.

ENZYMES

A standardized 5-minute turbidimetric serum amylase method using a stabilized commercial starch was presented by Ware *et al.* (*45L*). A saccrogenic serum amylase method employing ferricyanate reduction by the sugar formed was described by Fingerhut *et al.* (*10L*). Lambert and Zitomer (*26L*) simplified the preparation of cadmium iodide–linear starch reagent for colorimetric iodimetry. Ludvigsen (*27L*) developed a spectrophotometric DPNH method for the estimation of serum aldolase. Blood catalase methods were described by Kirk (*21L*) who developed a rapid gasometric method and by Feinstein *et al.* (*9L*) who used a rapid screening technique. Wetstone and Bowers (*46L*) standardized a serum cholinesterase method employing acetylcholine bromide incubation, formation of acethydroxamic acid and development of red-purple color with $FeCl_3$. Kirk (*22L*) determined the diaphorase activity of connective tissue by determination of decolorization time of 2,6-dichlorophenolindophenol in the presence of DPNH and NaCN. Kamaryt and Zazvorka (*18L*) obtained six different fractions of malic dehydrogenase activity by electrophoresis of serum. A simple and rapid determination of carbamoyl transferase in serum was made by Reichard (*37L*) by trapping the $C^{14}O_2$ evolved by incubation of paper moistened with KOH. Planta and Gruber (*34L*) used a new chromogenic substrate for simple estimation of cathepsin C. An improved method for determination of creatine kinase was described by Nielsen and Ludvigsen (*33L*). Metge *et al.* (*30L*) described a micro method for estimation of bilirubin glucuronyl transferase in hepatic tissue using sulfanilic acid-S^{35}. Martinek (*28L*) reported a micro method for estimation of serum adenosine deaminase. Guilbault *et al.* (*15L*) developed an electrochemical method for determination of glucose oxidase and glucose by using diphenylamine sulfonic acid as a potential poiser. Methods for the determination of lipase were described by: Vogel and Zieve (*44L*) who used a substrate of pure triglyceride in tris buffer and sodium deoxycholate as emulsifier and by Kramer and Guilbault (*12L*, *23L*) who hydrolyzed fluorescein esters catalyzed by lipase. Bowers (*6L*) chose the oxidation of $NADH_2$ to NAD as the standard spectrophotometric method for lactic dehydrogenase. Amador *et al.* (*2L*) made an analytical assessment of current assay methods for serum LDH activity. Lactic dehydrogenase studies and methods were reported by: Richterich *et al.* (*38L*) who separated the isoenzymes by column chromatography on Sephadex; Kamaryt and Zazvorka (*19L*) who separated the isoenzymes in serum by agar-gel electrophoresis on a microscope slide; Allison *et al.* (*1L*) who quantitated the enzyme in vertical starch gels with a microscope photometer; Guilbault and Kramer (*13L*) who measured the enzyme activity with resazurin in conjunction with the NAD^+-NADH system by conversion of resazurin to fluorescent resorufin; Raabo (*35L*) who determined LDH serum isoenzymes separated by electrophoresis with the tetrazolium salt INT; Bell (*4L*) who separated the enzyme in myocardial and hepatic tissue by means of heat fractionation; and by Kreutzer and Fennis (*24L*) who studied the stability of the enzyme at different temperatures and found the greatest stability at room temperature. Rosalki (*41L*) described a simple colorimetric method for the determination of serum *alpha*-hydroxybutyric dehydrogenase activity. Nachlas *et al.* (*31L*) simplified the colorimetric method for the measurement (TPNH) of isocitric dehydrogenase activity.

Alkaline phosphatase techniques were described by Bowers and McComb (*5L*) who used a semiautomated multiple cell changer with *p*-nitrophenyl phosphate substrate, and by Judd and Kwok (*17L*) who devised an ultramicro technique. A method for turbidimetric assay of phospholipase A based on the clearing effect of lysolecithin was developed by Doizaki and Zieve (*7L*). Babson (*3L*) obtained a patent for a serum acid phosphatase buffer. Ricketts (*39L*) improved a micro method for measurement of glucose-6-phosphatase. Fairbanks and Beutler

(*8L*) reported a simple method for erythrocyte glucose-6-phosphate dehydrogenase based on the reduction of tetrazolium dye by triphosphopyridine nucleotide. Kucera (*25L*) reviewed methods for the determination of proteolytic enzymes. Ritz and Rubin (*40L*) determined plasma proteolytic activity using chromium-51 tagged casein as substrate. Leucine aminopeptidase activity in biological fluids was determined by Roth (*42L*) by the fluorescence of *beta*-naphthylamine produced from 1-leucyl-*beta* naphthylamide, and by Hanson *et al.* (*16L*) who studied the specificity of this substrate. Fleisher *et al.* (*11L*) used a substrate of L-leucylglycine for determination of leucine aminopeptidase in serum. Glutamic oxaloacetic transaminase was studied by: Schwartz and Nisselbaum (*43L*) who described a method for staining for variants separated by electrophoresis with starch gel; McNair (*29L*) who evaluated a screening test based on the reaction between oxalacetic acid and 6-benzamido-4-methoxy-*m*-toluidine diazonium chloride to form a rose color; and by Ramaswamy and Radhakrishnan (*36L*) who used liquid scintillation counting for micro determination of transaminase. Katz (*20L*) determined urease activity by glass electrode measurement of ammonia. Ng *et al.* (*32L*) employed radioactive galactose 1-phosphate for assay of galactose-1-phosphate uridyl-transferase. Guilbault *et al.* (*14L*) developed an electrochemical method for determination of xanthine oxidase based on the oxidation of hypoxanthine by xanthine oxidase at pH 7.4.

FUNCTION TESTS

Kidney. Identification and analysis of urinary calculi was described by: Klein (*21M*) and Suecker (*33M*) who used infrared spectrometry, and by Beeler *et al.* (*5M*) who compared chemical and x-ray diffraction analysis. Urinary and serum creatinine was determined by: Murata *et al.* (*26M*) who used 3,5-dinitrobenzene-sulfonate and KOH; Beyermann (*6M*) who coprecipitated creatinine hydrogen borotetraphenol with $BuNH_3BPh_4$; Adams *et al.* (*1M*) who used ion exchange chromatography and ultraviolet spectrophotometry; and by Canturri and de Rivera (*11M*) who compared the alkaline picrate and 3,5-dinitrobenzoic acid methods.

Liver. Bodansky (*8M*) reviewed several blood quantitative biochemical procedures that have been used for the past few years as tests of liver function. A uniform bilirubin standard was recommended by the College of American Pathologists Standards Committee as reported by Larson (*23M*). Bilissis and Speer (*7M*) used an aqueous solution of *N*-(1-naphthyl) ethylenediamine dihydrochloride as a routine secondary "bilirubin" standard. Techniques for the determination of serum bilirubin were reported by: Jacobs *et al.* (*19M*) who pointed out the need of reference standards for bilirubin conjugates; Costa and Costa (*12M*) who used 20% HIO_3 as a new color reagent; Boutwell (*9M*) who studied the optimum conditions for use of acetamide as an accelerator in the bilirubin-diazotized sulfanilic acid reaction; Ferro and Ham (*13M*) who described a new colorimetric method based on the oxidation of bilirubin with $FeCl_3$ in an organic solvent mixture; Rand and di Pasqua (*27M*) who substituted 2,4-dichloroaniline for sulfanilic acid; and by Allen (*2M*) who simplified procedures for direct reading of bilirubin in the newborn. Brodersen and Vind (*10M*) determined unconjugated bilirubin by chloroform extraction in the cold at pH 8 after addition of ascorbic acid and salicylic acid. Gregory (*16M*) presented chromatographic evidence that the monoglucuronide of bilirubin is not present in serum. Stevenson *et al.* (*32M*) extracted serum total bilirubin with acidified ethylene glycol, then separated free bilirubin from this extract with chloroform. Weber and Schalm (*35M*) also described a method for determination of serum bilirubin and its conjugate. Garay and Argerich (*14M*) used a fluorometric method for the determination of biliverdin in body fluids based on the production of red fluorescent bilipurpurin zinc salt. Sjovall (*31M*) reviewed the methods for the separation and determination of bile acids. Bile acid conjugates were separated and detected by Gordon *et al.* (*15M*) by adsorption from 25% ethyl alcohol on a Dowex 1 anion exchange column. Kritchevsky *et al.* (*22M*) detected bile acids separated by thin layer chromatography with a spray reagent containing p-$CH_3OC_6H_4CHO$ in acetic and sulfuric acid solution.

Martinek *et al.* (*24M*) described an improved rapid turbidimetric method for determination of cephalin-cholesterol flocculation, and Rosenberg *et al.* (*29M*) shortened the method to 5 hours. Reinhold *et al.* (*28M*) replaced barbiturate buffer with tris-hydroxymethylaminomethane in the thymol turbidity test. Methods for the separation and identification of porphyrins were reported by: Baker *et al.* (*4M*) who resolved a number of porphyrins by column partition chromatography using dimethyl sulfoxide on silica support and cyclohexane as the mobile base; Hoschek (*18M*) who described a rapid method; Jensen (*20M*) who separated coproporphyrin I and III by thin layer chromatography; and by Aziz *et al.* (*3M*) who adapted the Eriksen paper chromatographic method to the quantitative analysis of porphyrin isomers I and III in urine. Taddeini *et al.* (*34M*) reported inhibition of the Ehrlich's reaction of porphobilinogen by indican and related compounds. Henry *et al.* (*17M*) obtained erratic recoveries of added urobilin in a "quantitative" method for the determination of urobilinogen in urine. Mizuta (*25M*) described a simple method for the estimation of urinary urobilinogen. Schmidt and Scholtis (*30M*) found favorable comparison of a modified Schlesingers reagent method for urobilin determination in urine with Ehrlich's extraction method.

HEMOGLOBIN

Smith (*15N*) outlined simplified steps to check the determination of hemoglobin to assure consistent reliable analysis. Weinberg (*18N*) prepared a stable colloidal suspension of hemoglobin as a reference standard for control of precision. Roughton (*14N*) described apparatus and technique for accurate gasometric microdetermination of oxygen-hemoglobin equilibrium in blood dilutions. A special freeze technique with liquid nitrogen was devised by Elliott and Doebbler (*6N*) to intensify 40-fold the absorbance of heme compounds. Adams and Peterson (*1N*) patented a test compound for detection of blood. Mickelsen *et al.* (*11N*) and Weatherburn and Logan (*17N*) observed the instability when frozen of the potassium ferricyanide-potassium cyanide reagent used for hemoglobin determination. Many different materials and techniques were reported for the separation and identification of human hemoglobins by: Nakamichi and Raymond (*13N*) who used acrylamide gel electrophoresis; Jones and Schroeder (*8N*) who applied Amberlite IRC-50 chromatography and tracer technique; Zade-Oppen (*19N*) who used cation exchange gels; Lionetti *et al.* (*10N*) and Berman and Kench (*5N*) who used dextran gel columns to measure hemoglobin binding capacity of human serum and separation of myoglobin and hemoglobin, respectively; Awad *et al.* (*2N*) who separated hemoglobin and myoglobin on Sephadex gel; Sunderman (*16N*) and Baur (*4N*) who separated hemoglobin by means of starch gel electrophoresis; Naiman and Gerald (*12N*) who resolved "fetal" hemoglobin into three components with modified agar gel electrophoresis; and cellulose acetate electrophoresis was employed by Graham and Grunbaum (*7N*), Bartlett (*3N*), and Kelsey and Kloss (*9N*).

METALS

Bertrand (*6P*) made a critical review of chemical methods for analysis of trace elements having biological significance. Fritz and Garralda (*15P*) titrated directly magnesium, calcium, strontium, and barium with a silver electrode and EDTA. Fukai and Meinke (*16P*) carried out activation analysis of ash of marine organisms for vanadium, arsenic, molybdenum tungsten, rhenium, and gold. Natelson (*28P*) reviewed methods for the determination of elements by x-ray emission spectrometry. Trace elements were determined by emission spectrometry by Voth (*39P*) in milk and by Tipton *et al.* (*37P*) in human tissues. Alkaline earths and cobalt, copper, nickel, and zinc were determined in micromolar concentration by fluorescence techniques by Wallach and Steck (*40P*). Sekido *et al.* (*35P*) studied the effectiveness of a new chelating agent, quinoline-8-selenol, as a reagent for group IV elements. Simon (*36P*) found tetraphenylboron ion an effective precipitant for alkali metals higher than sodium. Daly and Anstall (*11P*) determined chromium sesquioxide in feces by flame spectrophotometry by use of 4-methyl 2-pentanone extracts. Cobalt and nickel were determined spectrophotometrically by Ayres and Annand (*1P*) by complexing with 2,3-quinoxaline–dithiol, and by Mukhedkar and Deshpande (*26P*) by complexing with 8-quinolinol. Barling and Banks (*3P*) found 4-*tert*-butyl-1,2-cyclohexanedionedioxime to be an excellent reagent for the spectrophotometric determination of nickel. Rice (*32P*) selected the color complexing agent, oxalyldihydrazide for standardizing a copper method. Rice (*33P*) standardized ceruloplasmin activity assay in serum by using the enzymatic method of Houchin and colorimetric measurement with Bandrowski's base. Spectrophotometric methods for the determination of copper were reported by: Nebel and Boltz (*29P*) who measured the complex dipyrophosphatocuprate; Kilshaw (*24P*) and Giorgio *et al.* (*18P*) who complexed with zinc dibenzyldithiocarbamate; Billman and Chernin (*7P*) who complexed with *ortho*-(*p*-toluene sulfonamido) aniline (T-sulfonanidine); Beale and Croft (*4P*) who used oxalyldihydrazide–acetaldehyde complex; Mikac-Devic (*25P*) who complexed with 1,5-diphenylcarbohydrazide; van der Heyde and Henderickx (*38P*) who used dicyclohexanone oxalyldihydrazone as color reagent; and by Berman (*5P*) who used atomic absorption. Kaiser and Meinke (*21P*) utilized 5.1-minute copper-66 radioisotope for rapid activation analysis of trace copper in animal tissue. Werthemann (*41P*) detected gold in liver biopsy by emission spectrophotometry. Serum iron was determined spectrophotometrically by: Sanford (*34P*) with Teepol, ascorbic acid and orthophenanthroline; Nelson (*30P*) who used sulfonated diphenylphenanthroline; Forman (*14P*) who used a modification of the bathophenanthroline method; and by Fischer and Price (*13P*) who used a very sensitive chromogen, tripyridyl-*s*-triazine. Methods for the determination of both serum iron and iron binding capacity were described by Giovanniello and Peters (*19P*) and Card *et al.* (*10P*) who standardized with bathophenanthroline reagent and by Caraway (*9P*) who complexed with tripyridyltriazine at pH 4–5. Barber *et al.* (*2P*) used a gel filtration technique and radioactive iron for rapid determination of protein–iron binding. Nadeau and Fortin (*27P*) demonstrated that the addition of pure beta globulin solutions to serum did not change the iron-binding capacity. Ramsay and Fulton (*31P*) described a new method for the assay of small amounts of radioactive iron (Fe^{59}) and Katz *et al.* (*23P*) simplified a procedure for the simultaneous assay of Fe^{55} and Fe^{59} in a liquid scintillation system. Karvanek and Karvankova (*22P*) devised a photometric method for traces of manganese in biological materials by preventing the interference of color effects of other ions with Tiron reagent. Bingley (*8P*) found that thiourea prevents copper interference in the determination of molybdenum with dithiol. Elfers *et al.* (*12P*) determined strontium by flame photometry at 461 mμ. Direct determination of zinc in biological fluids by atomic absorption spectrophotometry was reported by Honegger (*20P*) and Fuwa *et al.* (*17P*).

NITROGEN COMPOUNDS

A direct titrimetric method of Kjeldahl digests for the microdetermination of nitrogen in biological materials was described by Ashraf *et al.* (*1Q*). Rodkey (*30Q*) suggested the use of tris-(hydroxymethyl) aminomethane as a convenient primary standard for Kjeldahl analysis. Several modifications of the phenolhypochlorite method for ammonia were reported by Konitzer and Voigt (*20Q*), Muftic (*26Q*), Mann (*21Q*) and by Dienst and Morris (*12Q*) who first separated the ammonia by ion exchange. Prochazkova (*29Q*) determined ammonia as rubazoic acid with bispyrazolone reagent. Howell and Boltz (*17Q*) determined ammonia by hypobromite oxidation to nitrogen and measurement of the differential absorbance at 330 mμ. Miller and Rice (*25Q*) adsorbed ammonia on ion exchange resin and eluted into a color complex. Kirsten *et al.* (*19Q*) made an enzymic microdetermination of ammonia in biological fluids using glutamic dehydrogenase. Orloff and Stevens (*28Q*) found that rapid freezing of blood stabilizes ammonia content for 72 hours, and Conn and Kuljian (*7Q*) confirmed this observation, but Wilder (*34Q*) observed a significant change in ammonia during frozen storage for 1 to 8 days. A rapid colorimetric method for the adenine compounds was described by Davis and Morris (*10Q*). An improved method for determination of blood glutathione based on the development of yellow color when 5,5′-dithiobis-(2-nitrobenzoic acid) reacts with sulfhydril compounds was reported by Beutler *et al.* (*4Q*). Davidson and Hird (*9Q*) described a new method for glutathione based on thiol-disulfide exchange reactions with cystamine and glutathione reductase. Grokholskaya (*14Q*) determined nitrates and nitrites in blood by the diphenylamine method. Hartley and Asai (*15Q*) spectrophotometrically determined nitrate with 2,6-xylenol reagent and nitrite (*16Q*) as 4-nitroso-2,6-xylenol. Pyridine nucleotides were determined in blood fluorometrically with dehydrogenase by Pande *et al.* (*27Q*) and Tabuchi *et al.* (*33Q*). The determination of urea by the phenol–hypochlorite reaction was reported by Gasanov (*13Q*) and by Chaney (*6Q*) who obtained a patent on a commercial reagent employing this reaction. A patent was obtained by Miles Laboratories, Inc. (*24Q*) on cellulose strips impregnated with urease, buffer, and pH indicator for urea determination. Spectrophotometric methods for the determination of urea were described by: Yatzidis *et al.* (*35Q*) who used Ehrlich's reagent; Roijers and Tas (*31Q*) who employed *p*-dimethylaminobenzaldehyde; Baylor (*3Q*) who used a stable diacetyl reagent; Ceriotti and Spandrio (*5Q*) who combined phenazone with diacetylmonoxine; Coulombe and Favreau (*8Q*) who used diacetylmonoxine and thiosemicarbazide together; McCleskey (*23Q*) who measured fluorescence produced by the urea–diacetyl–monoxime reaction; Kachani (*18Q*) who proposed a simple turbidimetric determination of urea with xanthydiol; and by de Jorge and de Ulhoa Cintra (*11Q*) who determined urea colorimetrically with $P–(CH_3)_2NC_6-H_4-CHO$. Simoes and Pereira (*32Q*) increased the color intensity of uric acid in the presence of tungstate and Folin reagents with hydrazine hydrate. Marymont (*22Q*) determined uric acid directly on heat coagulated blood and serum by ultraviolet absorption at 295 mμ. Bare and Wiseman (*2Q*) described a technique for distinguishing urate chromogen from nonurate chromogens colorimetrically before and after action of urease.

HORMONES

Weissbach (*45R*) standardized a method for measuring serotonin, 5-hydroxytryptamine in tissues by HCl extraction, activation at 295 mμ and fluorescent measurement at 550 mμ. Davis *et al.* (*11R*) presented a method for the microdetermination of serotonin in blood using ion exchange resins. Vanable (*42R*) assayed small amounts of 5-hydroxytryptamine fluorometrically by reaction with ninhydrin. Ashcroft *et al.* (*2R*) modified the method of Weissbach (*45R*) to increase sensitivity of 5-hydroxytryptamine blood measurement. Callingham and Cass (*6R*) reviewed methods for catechol amines in biological materials. Methods for the determination of catecholamines were reported by: Brunjes and Wybenga (*3R*) who used differential fluorometry; Marshall (*29R*) who used sephadex G-25 for separation of the catecholamines and by Brunjes *et al.* (*4R*) who isolated metanephrine and normetanephrine with cation exchange resin. Klotz *et al.* (*21R*) presented evidence that formaldehyde interferes with fluorometric measurement of urinary catecholamines. Methods for the determination of 5-hydroxy-indoleacetic acid in urine were described by: Weissbach (*44R*) who standardized the 1-nitroso-2-naphthol reaction; Pitkanen *et al.* (*32R*) who studied the specificity of this reaction; Davis (*10R*) who investigated the oxidation of homogentisic acid by nitrous acid and its interference in the determination of 5-hydroxy-indoleacetic acid; and by Sprince (*38R*) who studied the paper chromatography of several urinary indoles extracted under acid and alkaline conditions. Several methods were described for the estimation of 3-methoxy-4-hydroxymandelic acid (VMA) in urine by: Weil-Malherbe (*43R*) who estimated VMA by oxidation to vanillin by copper ion at pH 10; Hermann (*20R*) and Aguayo (*1R*) who used cellulose acetate electrophoresis; Georges (*17R*) who modified the method of Pisano by condensation of vanillin with 2,4-dinitrophenyl hydrazine to form a red hydrazone; Fellman *et al.* (*15R*) who developed a color with 4-chloro-*o*-phenylenediamine and H_3PO_4; Eichhorn and Rutenberg (*14R*) who employed low-voltage paper electrophoresis for separation of VMA from 0.05 ml. of urine; and by Rosano (*34R*) who utilized L-mandelic acid dehydrogenase preparation from Pseudomonas fluorescein A-312 to catalyze the transformation of VMA to vanillin. Campbell *et al.* (*7R*) noted false positive reactions due to methocarbamol in the screening test for vanilmandelic acid. A method for the determination of urinary 3,4-dihydroxymandelic acid (DOMA) was reported by DeQuattro *et al.* (*12R*) and Ruthven and Sandler (*36R*) described a technique for urinary homovanillic acid.

Yalow and Berson (*46R*) reviewed methods for the immunoassay of plasma insulin. Lee *et al.* (*23R*) determined free thyroxine content of serum by labeling the total serum thyroxine with thyroxine-I^{131} and separation of the free thyroxine on microcolumns of Sephadex G-25. Mitchell (*30R*) described elution techniques for determination of thyroxine (T$_4$) and triiodothyronine (T$_3$) in serum. Robbins (*33R*) reviewed methods for the measurement of thyroxine-binding proteins in serum by paper electrophoresis. Sunderman (*40R*) reviewed the techniques for measurement of serum protein-bound iodine. Foss (*16R*) standardized the alkaline ash method for the determination of protein-bound iodine in serum. Lieb (*26R*) reviewed the determination of iodine in biological material. Methods for the determination of protein-bound iodine in biological fluids were described by: Graham (*18R*) who carried out the entire determination in a single vial; Shtolts (*37R*) compared various methods of iodine determination in biological materials; Leffler and McDougald (*25R*) described improved techniques; and Routh (*35R*) shortened the time required for the performance of the alkaline ash method. Hamilton (*19R*) patented a PBI method which eliminated precipitation of proteins. Tamura and Kondo (*41R*) and Stolc and Knopp (*39R*) made microdeterminations of organic iodine by use of Schoenigers digestion method. La Roche *et al.* (*22R*) and Chakrabarti and White (*8R*) reported the use of an ion exchange resin to remove inorganic and contaminating iodide in serum prior to the determination of PBI. The use of ion exchange resins in the determination of PBI was reported by: Comar *et al.* (*9R*) who separated iodoprotein; Lee *et al.* (*24R*) who studied the variables affecting ion exchange resin uptake of I^{131}-labeled triiodothyronine of serum; Lowenstein *et al.* (*28R*) who used ion exchange paper to concentrate iodiated compounds in plasma; Lissitzky and Bismuth (*27R*) who separated serum iodine compounds by filtration on dextran gel; DiGiulio *et al.* (*13R*) who employed agar gel electrophoresis and autoradiography to measure thyroxine binding protein capacities; and Burke *et al.* (*5R*) who characterized thyroid function by determination of radiothyroxine partition among carrier plasma proteins. Pileggi *et al.* (*31R*) reported faslely elevated serum PBI from sulfobromphthalein.

ORGANIC ACIDS

Schmidt *et al.* (*24T*) described a new method for location of organic acids on paper chromatograms. Riddle (*20T*) described a gas liquid chromatography technique for determination of organic acids in small samples. Kesner and Muntwyler (*8T*) estimated the hydrazone derivatives of keto-acids by use of a partially hydrated silica gel column and gradient solvent elution system. Rink and Hermann (*21T*) detected the presence of acetoacetic acid and acetone with the aid of thin layer chromatography. Organic acids were determined by: Lester (*12T*) who determined acetic acid in blood by vacuum distillation and gas liquid chromatography; Meichen and Short (*15T*) who measured *beta*-aminoisobutyric acid in urine by column and paper chromatography; Wolosowicz *et al.* (*28T*) who measured E-aminocaproic acid in plasma by its antifibrinolytic properties; Jacobs and Lee (*7T*) who measured citric acid by conversion to pentabromoacetone with Br^{82}; Chambon (*5T*) who determined citric acid in biological fluids by formation of a yellow color with pyridine and acetic anhydride; Lavender *et al.* (*10T*) who measured EDTA in plasma and urine by reaction with 1-(2-pyridylazo-)-2-naphthol and nickel to form a red dithiooxalate complex; Qureshi *et al.* (*18T*) who described a new color reaction of formic acid in which a violet color is formed with anisol and lead nitrate; Shinriki and Nambara (*25T*) who determined glucuronic acid in the presence of oxidized starch by an ion-exchange resin, elution and color reaction with orcinol; Nir (*17T*) who determined glucuronic acid by modification of the naphthoresorcinol procedure; and by Umberger and Fiorese (*26T*) who determined hippuric acid colorimetrically by reaction with benzenesulfonyl chloride in pyridine to form a red-orange color. Williams and Leonard (*27T*) performed microdeterminations of dihydroxy aromatic acids by gas chromatography. Ramsey (*19T*) separated *beta*-hydroxy butyric acid by silicic acid column chromatography. Matulis and Guyon (*13T*) developed a method for determination of *alpha*-hydroxy acids based on the bleaching effect of the blue hue of a reduced molybdate solution. Rosan and Nieland (*22T*) suggested precautions for more accurate oxalacetic acid determination in the formation of its hydrazone. Berry and Campbell (*2T*) separated free *alpha*-keto acids electrophoretically and used a 2,4-dinitrophenyl-hydrazine spray for their location. Hoffman *et al.* (*6T*) measured lactic acid in blood by oxidation with HIO_4 and determination of acetic acid produced by gas chromatography. Rosenbloom and Seegmiller (*23T*) determined orotic acid in urine and serum by measuring the decrease in optical density at 295 mμ when the acid is converted to uridylic acid by orotidylic pyrophosphorylase and decarboxylase. Mayer *et al.* (*14T*) determined oxalic acid in urine with oxalic decarboxylase. Barness *et al.* (*1T*), by suitable preparation and separation, fractionated quantitatively free phenols and aromatic acid phenols. Legrand *et al.* (*11T*) described the chromatographic identification of the lactonic derivative of 2,5-dihydroxyphenylpyruvic acid in pathological conditions. Landon *et al.* (*9T*) measured blood pyruvate by a specific method employing the oxidation of reduced nicotinamide–adenine dinucleotide in the presence of lactic dehydrogenase. Woodring *et al.* (*29T*) described a simple specific microprocedure for the estimation of urinary 4-pyridoxic acid in urine. Zak and Weiner (*30T*) used a simple electrophoretic technique for the rapid separation of ribonucleic acid. Millican (*16T*) developed a sensitive colorimetric assay for shikmic acid based on periodide oxidation in concentrated phosphoric acid. Cabezas and Porto (*3T*) described a simple spectrophotometric method for sialic acid based on its reaction with resorcinol and extraction with butyl acetate-*n*-butanol, and by Caraway (*4T*) who standardized the uric acid method employing sodium carbonate and phosphotungstic acid.

ORGANIC COMPOUNDS

Madonia (*9U*) described a sensitive, specific technique for determination of acetone and acetoacetic acid in serum. Cooper (*1U*) determined acetylcholine fluorometrically by reducing to ethyl alcohol then oxidizing to CH_3CHO with alcoholic dehydrogenase with the concomitant reduction of nicotin–amide–adenine dinucleotide. Hashmi *et al.* (*6U*) discovered that aldehydes give a characteristic yellow color with chlorite with formation of chlorine dioxide. Guilbault (*4U*) determined *p*-aminophenol by oxidation with cerium perchlorate in 3*F* perchloric acid to a purple compound. Harada (*5U*) described a new color reaction of sugars (aldoses) which react with ethyl malonate in weak alkali to form a stable purple chromogen. Mortensen (*10U*) revised the nitroprusside method for the determination of reduced glutathione. Simple chemical methods were devised by Peden (*11U*) for the individual determination of acetone and alpha hydroxybutyrate. Tanayama and Ui (*13U*) determined colorimetrically small amounts of ketone bodies in blood by oxidation to $(CH_3)_2$-CO which reacted with salicylaldehyde in strong base to form 1,5-bis-(2 - hydroxyphenyl) - 3 - pentadienone. Selleck *et al.* (*12U*) used a specific enzymic assay (glutamic dehydrogenase coupled with NH_4^+ and $NADH_2$) for determination of alpha ketoglutarate in biological fluids. Luke *et al.* (*8U*) used a gas liquid chromatographic procedure for identification of the methyl esters of the stable Krebs Cycle acids. Indole and indole-like compounds were assayed by: Kupfer and Atkinson (*7U*) who used anthranilic acid and *p*-dimethylaminobenzaldehyde in acid to form colored products; Fischl and Rabiah (*3U*) who described a new method employing Fischl's tryptophan reaction to determine free and total indole-3-acetic acid; and by Durkee and Sirois (*2U*) who detected 27 indoles by paper chromatography, Ehrlich aldehyde, and *p*-dimethylaminocinnamaldehyde reagents.

PROTEINS

Sunderman and Sunderman (*43V*) reviewed "Serum Proteins and the Dysproteinemias" in a book with this title. Spiro (*39V*) reviewed structure, metabolism and biology of glycoproteins. A comparison study of several protein methods in clinical use was made by Sakamoto and Takahira (*37V*). A book on "Techniques in Protein Chemistry" was written by Bailey (*22V*) for chemists who have had limited practical experience in the chemistry of proteins. van de Loo (*44V*) investigated and compared different protein methods. A new method for gravimetric analysis for total protein of human serum to determine the total protein–protein N ratio was devised by Chiaraviglio (*6V*) by which the ratio 6.54 was confirmed. Minari and Zilversmit (*28V*) used KCN to stabilize Nessler reagent for direct application to Kjeldahl digest. Stanley (*40V*) determined the extent of the interference of ammonium sulfate in estimation of proteins by the biuret reaction. Inchiosa (*20V*) suggested a technique for measurement of total protein content of whole tissue homogenates with the biuret reaction. London and Marymont (*24V*) investigated the effect of 14 common organic solvents in lowering the heat coagulation temperature of proteins and Dawson and Zajtchuk (*11V*) studied the use of halogenated acetates and formaldehyde in inhibiting heat denaturation of plasma proteins. Methods for concentrating proteins in biological solutions were reported by: McFarlane (*27V*) who concentrated urine five-fold in 90–120 minutes by dialysis against sucrose using cellulose acetate membrane; Curtain (*9V*) who used rods of dried polyacrylamide gel; and by Peetoom and Gerald (*31V*) who described a simple inexpensive method for concentration by means of ultrafiltration. Serum albumin was determined by: Rodkey (*34V*) who bound albumin specifically with bromcresol green; Slutskii (*38V*) who removed globulins from albumin by precipitation with 3% CCl_3COOH in 96% alcohol; and by Kaplan and Johnstone (*21V*) who used specific fluorescent dyes, 1-anilino-naphthalene-6-sulfonic acid and vasoflavine. Plasma fibrinogen was determined by Goodwin (*16V*) who made a comparative study of three methods and by Foreman *et al.* (*14V*) who bound fibrin to Ponceau S. Cerebrospinal fluid proteins were determined spectrophotometrically at 220 mμ by Watson (*46V*) and Patrick and Thiers (*30V*). Lundh (*26V*) determined B_{1c}-globulin by the effect of hydrazine treatment on the electrophoretic mobility of this globulin. Botvinik and Troshko (*5V*) determined the esters of acylated peptides by the hydroxamic reaction. Techniques for distinguishing macroglobulins were presented by: Steel (*41V*) who treated serum with 2-mercapto-ethanol which serves as a valuable supplementary viscosity test; Saifer (*36V*) who added Rivanol solution to serum and compared the electrophoretic pattern of the supernatant fraction to the original serum; Ratcliff *et al.* (*32V*) who used a specific antiserum to the 19S component of gamma globulin for estimating pathological macroglobins; and by Roskes and Thompson (*35V*) who used a Sephadex G-200 molecular sieve column to differentiate macroglobulinemic, hyperglobulinemic, and normal sera. Reynafarje (*33V*) simplified a method for the determination of myoglobin.

Sunderman (*42V*) reviewed the recent advances in clinical interpretation of electrophoretic fractionation of serum proteins. Electrophoretic methods for the detection of proteins were described by: Lloyd and Meares (*23V*) who recovered proteins from starch by electrophoresis; Hase (*18V*) who used a simple starch–agar gel to obtain higher resolution; Acharya *et al.* (*1V*) who used a simple and rapid procedure for the separation of serum protein by agar-gel electrophoresis on microscope slides; Lorber (*25V*) who increased globulin fractionation with an improved vertical gel electrophoretic technique; Bartlett (*3V*) who reported equal dye-binding characteristics of amido black with albumin and gamma globulin separated by cellulose acetate electrophoresis; and by Collens *et al.* (*7V*) who employed polyethylene glycol to concentrate urine for paper electrophoresis. Methods for immunoelectrophoresis of proteins in biological fluids were reported by: Betsuyaku *et al.* (*4V*) who made direct simultaneous quantitation of subfractions of serum globulins; Glass *et al.* (*15V*) who employed an immunochemical technique based on hemagglutination-inhibition reaction to measure urinary albumin; and by Afonso (*2V*) who described a quantitative immunoelectrophoresis in which antiserum diffusion gradient was eliminated. Hochwald and Thorbecke (*19V*) studied the effect of storage and enzymes on the electrophoretic mobility of gamma-trace and beta-trace proteins in cerebrospinal fluid. Curtain (*10V*) chromatographed proteins and peptides on diethylaminoethyl methacrylate gels.

Grasbeck and Karlsson (*17V*) described a detector column of high specific activity Cu^{64} complexed with alkaline Sephadex for continuous microdetermination of protein. Morris (*29V*) presented a new protein chromatographic method employing a two-phase liquid-liquid system. Wachter (*45V*) demonstrated protein fractions after paper electrophoresis by their reduction of $KMnO_4$ to MnO_2. de St. Groth *et al.* (*12V*) suggested two new staining procedures for protein estimation of electrophoretic strips which employ Procion Brilliant Blue RS and Coomassee Brilliant Blue R 250. Fischl and Gabor (*13V*) used a new stain for electrophoresis composed of light green, Ponceau red, and amido black in sulfosalic and trichloroacetic acids. Investigation by Cupic and Topalovic-Avramov (*8V*) of the affinity of various dyes for serum proteins indicated Ponceau S was most evenly distributed in the protein fractions.

STEROLS

Photometric methods for serum cholesterol determination were reviewed by Vanzetti (*53W*). Webster (*55W*) determined esterified cholesterol without preliminary hydrolysis with $FeCl_3$–*p*-toluenesulfonic acid reagent, but Antonini *et al.* (*1W*) found the direct Zak method unreliable and Moore and Boyle (*33W*) also noted errors resulting from the use of $FeCl_3$ color reagent in the determination of serum cholesterol. Several different reagents for serum cholesterol determination were described by: Zurkowski (*59W*) who used a sulfosalicylic acid, acetic anhydride, and sulfuric acid reagent; Jamieson (*23W*) who used toluene–*p*-sulfonic acid; Ness *et al.* (*38W*) who stabilized the Liebermann-Burchard reagent with the addition of anhydrous sodium sulfate; Mose *et al.* (*35W*) who obtained stable color with a reagent of perchloric acid, phosphoric acid, and $FeCl_3$; Claude (*8W*) who used dimethylbenzenesulfonic acid and H_2SO_4 for measurement at 650 mμ and at 560 for correction of icterus and hemolysis; Richterich and Dauber (*42W*) who employed a reagent of 2,5-dimethylphenolsulfonic acid, acetic anhydride, acetic acid, and H_2SO_4; and by Wrigley (*58W*) who devised a reagent of methyl alcohol, SbI_3, and H_2SO_4. Kritchevsky *et al.* (*27W*) determined serum cholesterol by isotope dilution analysis with cholesterol–C^{14}. Grande *et al.* (*15W*) studied the effect of various anticoagulants on serum cholesterol determination. Cholesterol was determined by the digitonin moiety of cholesterol digitonide with phloroglucinol in acid solution by Sahagian and Levine (*44W*). Hernandez and Axelrod (*17W*) described a method for chromatographic purification of steroids from total lipid extracts. Morris (*34W*) fractionated cholesterol esters by silica gel thin layer chromatography and visualization with 2,7-dichlorofluorescein. Shin (*47W*) used silicic acid column chromatography for microdetermination of cholesterol and cholesterol ester. Webster (*54W*) fractionated cholesterol by application of a petroleum ether extract of serum to a column of alumina. Horning *et al.* (*19W*) reviewed the methodology of separation and determination of steroids by gas chromatography. Landowne and Lipsky (*30W*) described a technique of detecting ultramicro quantities of sterols by gas chromatography by electron capture spectrometry of haloacetates. Chamberlain *et al.* (*7W*) gas chromatographed steroids on columns coated with neopentylglycol adipate, the fluoralkyl silicone polymer QFI and methyl silicone polymer SE-30. Cawley *et al.* (*6W*) analyzed serum cholesterol by gas liquid chromatography with argon detector. Kritchevsky *et al.* (*26W*) made a comparison of alpha and beta lipoprotein cholesterol levels of lipoproteins of serum by dextran sulfate precipitation. Huang *et al.* (*20W*) reported a new method of precipitating free cholesterol with Tomatine reagent.

Silber (*48W*) standardized a technique for free and conjugated 17-hydroxy corticosteroids in urine using methylene solvent extraction, beta glucuronidase hydrolysis, and color development with phenylhydrazine hydrochloride. Steenburg and Thomasson (*51W*) used 2,2,4-trimethylpentane, chloroform extraction and fluorescent reagent [75% ethanolic H_2SO_4 (v./v.)] for serum corticosteroid determination. Methods for the determination of 17-ketogenic steroids (corticoids) were described by: Murphy *et al.* (*36W*) who employed corticosteroid competitive protein binding and gel filtration; Liu (*32W*) who modified the Porter-Silber technique by use of a single ethyl ether extraction and stabilization of the phenylhydrazene-H_2SO_4 reagent with NaCl; Kornel (*25W*) who applied the Porter-Silber method to eluate from paper chromatograms; Wilson and Lipsett (*56W*), James and Caie (*22W*), and Rutherford and Nelson (*43W*) who studied the use of periodate oxidation; Copinschi *et al.* (*9W*) who used the bismuthate oxidation in the determination of the 17-ketogenic steroids; Brown (*5W*) who evaluated several procedures for their simplicity, accuracy, and reproducibility for urinary 17-ketogenic steroid determination; Copinschi *et al.* (*10W*) who described a procedure for the simultaneous determination of the excretion rate of 11-deoxy and 11-oxysteroids; and by Thomas and Bulbrook (*52W*) who rapidly estimated total 11-deoxy-17-oxosteroids in urine by heptane elution from filter paper saturated with propylene glycol.

A technique for urinary estrogens was standardized by Hobkirk and Metcalfe-Gibson (*18W*) using a fluorometric reagent, *p*-nitrophenol in tetrabromoethane. Chromatographic methods for the determination of estrogens were described by: Kroman *et al.* (*28W*) who employed a combination of chemical extraction and gas chromatography for human plasma; Wotiz and Chattoraj (*57W*) who determined seven estrogens in urine using thin layer and gas liquid chromatography; Jacobsohn (*21W*) who separated estrogens by thin layer chromatography and one-step photographic procedure; and by Epstein and Zak (*14W*) who used glass paper chromatography and *p*-toluene sulfonic acid color reagent. Barlow (*3W*) combined a fluorescent and radioactive method for urinary estrone, estradiol-17 beta, and estriol.

Peterson (*40W*) standardized a method for determination of urinary neutral 17-ketosteroids by extraction with petroleum ether benzene and colorimetric measurement with *m*-dinitrobenzene. Schulz *et al.* (*45W*) developed a new spectrophotometric technique for the determination of 3-ketosteroids by development of the chromophore with 4-aminoantipyrine HCl. Methods for the determination of 17-ketosteroids were described by: Epstein and Zak (*13W*) who eluted the Zimmerman reactive chromogens with dimethyl sulfoxide and measured photometrically and by Eberlein (*12W*) who used a transesterification method for measurement of plasma 17-ketosteroid sulfates. Borushek and Gold (*4W*) investigated 10 groups of commonly used medications which interfere in the colorimetric determination of 17-ketosteroids and 17-ketogenic steroid determinations. Gas chromatographic methods for the determination of 17-ketosteroids were described by Nair *et al.* (*37W*) who used a mixture of a nonpolar silicone rubber and a polar polyester phase and by Sparagana *et al.* (*49W*) who determined individual $C_{19}O_2$ and $C_{19}O_3$ individual urinary 17-ketosteroids by gas chromatography. Other chromatographic methods for 17-ketosteroids were reported by: Lim *et al.* (*31W*) who first separated by glass paper chromatography the total 11-oxy and 11-desoxy groups and then the individual 11-desoxy 17-ketosteroid groups with three additional chromatographic systems; Starka *et al.* (*50W*) who separated the steroids by thin layer adsorption chromatography on alumina and evaluated the chromatogram by Zimmerman's reaction; Oertel (*39W*) who obtained striking differences in the chromatographic distribution of 17-ketosteroid conjugates in chromatography of pooled serum; and by Schumacher (*46W*) who obtained complete fractionation of urinary 17-ketosteroids by thin layer chromatography on horizontal plates of activated silica gel. Haust and Beveridge (*16W*) made a differential photometric estimation of coprostanol in feces by change in extraction coefficients of cholesterol and coprostanol with time in the Liebermann-Burchard reaction, and a similar technique was used by Pottier and Busigny (*41W*) for differentiation of cholesterol and desmosterol in serum. Lachese *et al.* (*29W*) detected pregnanolone after paper chromatography in a ligroin–propanediol system of benzene eluates with pyridylazonaphthol reagent. Methods for the determination of pregnanediol in urine were described by: Jansen (*24W*) who applied gas liquid chromatography to crude urinary extracts; Bang (*2W*) who applied sulfuric acid reaction directly on material from the thin layer chromatograph; and by Crepy *et al.* (*11W*) who reported a comparative study of four methods of estimation of urinary pregnanediol.

TOXICOLOGY

Clavell (*10X*) reviewed the most common chemical methods for the determination of blood alcohol. Spectrophotometric methods for the determination of ethanol were reported by de Oliveira Menditsch (*16X*) who used the iodide of methylene blue in aqueous solution, and by Scoggins (*57X*) who determined trace alcohols by ultraviolet spectrophotometry of alkyl nitrobenzoates formed by esterification of alcohols with 3,5-dinitrobenzoyl chloride. Hessell and Modglin (*24X*) determined ethanol in blood by gas liquid partition chromatography and McCord and Gadsden (*41X*) by gas chromatography. A low cost gas chromatograph for the simultaneous determination of methanol, ethanol, and isopropanol in biologic samples was described by Lyons and Bard (*36X*). Leithoff and Chan (*34X*) estimated blood alcohol by an ultramicro enzymic method employing diphosphopyridine nucleotide and alcohol dehydrogenase and Malmstadt and Hadjiioannou (*37X*) developed an automatic spectrophotometric reaction rate method employing the same enzymic method. A rapid quantitative method for methanol determination in biologic fluids was developed by Hindberg and Wieth (*25X*) by oxidizing methanol to formaldehyde in the presence of added alcohol. Mantel (*38X*) reported a technique for microdetermination of isopropyl alcohol in aqueous solutions. Winefordner and Latz (*76X*) determined aspirin in serum by chloroform extraction, evaporation, and measurement of phosphorescence in a mixed solvent of ethyl ether, isopentane, and ethyl alcohol. Crippen and Freimuth (*13X*) described a gas chromatographic method for determination of aspirin in drugs, tissues, and solutions. Chromatographic methods for the determination of narcotics and alkaloids were described by: Mule (*47X*) who applied ultraviolet spectrophotometry to thin layer and gas liquid chromatography; Parker *et al.* (*50X*) who identified 41 alkaloids by rapid gas chromatography on SE-30 column at five temperatures; Brochmann-Hanssen and Svendsen (*7X*) who determined phenolic alkaloids by gas chromatography as the trimethylsilyl ethers on silicone rubber SE-30; Frahm *et al.* (*19X*) who applied thin layer chromatography in the identification of 18 narcotics, 12 barbiturates, and six nonbarbiturates; and by Dadlez and Wojciak (*15X*) who determined alkaloids in animal material with cationitized (phenolformaldehyde) paper. Methods for the determination of individual narcotics were reported by: Wojciak (*78X*) who extracted atropine from animal material with ion exchange paper; Kuhn and Friebel (*32X*) who traced codein and its metabolites in urine by complexing with bromocresol green and determining photometrically at 615 mμ; Mills (*44X*) who described a new color test for the infrared identification of micro amounts of heroin–HCl; and by Wojciak (*79X*) who used ion exchange paper for the determination of morphine in animal material. Methods for the identification and separation of barbiturates in blood by thin layer chromatography techniques were described by Petxold and Camp (*51X*), Cochin and Daly (*12X*), Lehmann and Karamustafaoglu (*33X*), Sunshine *et al.* (*67X*) and by Kelleher (*30X*) who separated long, intermediate and short acting barbiturates with a 10-minute development. Methods of application of gas chromatography to the separation and detection of micro quantitative of barbiturates were reported by Kazyak and Knoblock (*29X*) and Jain *et al.* (*28X*). A microspectrophotometric method for determination of diphenylhydantoin and phenobarbital was reported by Svensmark and Kristensen (*68X*). Street and McMartin (*65X*) outlined a procedure for estimation and identification of blood barbiturates in emergency cases in which the extract is divided into three parts for ultraviolet, elevated temperature, chromatography, and H_2SO_4 treatment examination. Swagzdis and Flanagan (*69X*) used the Aminco-Bowman spectrophotofluorometer for determination of low concentrations of amobarbital in plasma. Guzak and Caraway (*22X*) determined serum barbiturate and salicylate by ultraviolet absorption spectrophotometry. Mantel and Molco (*39X*) used the red color produced by the reaction of pyridine with chloroform in presence of NaOH for the determination of micro amounts of chloroform. Amenta (*3X*) standardized the determination of carbon monoxide in blood by treatment with ammonia and measuring absorbance at 575, 560, and 498 mμ. Coburn *et al.* (*11X*) described the sources of error in the blood carbon monoxide method in which the CO is disassociated from carboxyhemoglobin with ferricyanide and the CO extracted with oxygen for infrared absorption. Stewart and Evans (*64X*) determined carbon monoxide in the Orsat gas analysis apparatus with copper sulfate–beta-naphthol. Terao (*70X*) determined carboxyhemoglobin in heat coagulated blood by microdiffusion analysis. Zitomer and Lamber (*81X*) determined bromide ion spectrophotometrically in water as a function of its inhibition of the chlorination of ammonia to form trichloramine. The bromides normally present in serum after suitable preliminary preparation were assayed in the x-ray spectrophotometer by Natelson *et al.* (*48X*). Miller *et al.* (*43X*) used the spontaneous response of a rapidly rotating gold electrode to cyanide ion in lithium hydroxide for the determination of micro amounts of cyanide. Powell and Ellam (*52X*) determined dichromate in body fluids by use of the ethyl alcohol–$K_2Cr_2O_7$ reaction in acid with KI–starch indicator. Moore (*46X*) used a novel radiotracer method for rapid fluoride ion determination by forming a complex with tantalum-182 and measurement of radio activity after diisobutylketone extraction. Dubois *et al.* (*17X*) measured fluoride in urine by microdiffusion. Buettner *et al.* (*9X*) determined micro amounts of fluorine in bone by diffusion separation and colorimetric measurement with $ZrOCl_2$ and Eriochrom Cyanine R. Micro estimation of inorganic fluoride was made by Aldous (*1X*) by precipitating as a calcium fluoride complex containing thorium ion on a filter paper disk and straining the thorium–fluoride complex after suitable elution with solochrome brilliant blue.

Merkus (*42X*) used cellulose gel thin layer chromatography and suitable color reagents to detect most metallic ions. Winefordner and Staab (*77X*) applied atomic fluorescence flame spectrometry for the determination of zinc, cadmium, and mercury in less than 1 p.p.m. in aqueous solutions. Methods for the determination of arsenic were reported by: Hanke (*23X*) who extracted arsenic from urine residue with toluene and after suitable preparation measured colorimetrically with ammonium molybdate; Tertian *et al.* (*71X*) who determined traces of arsenic in organic substrates by x-ray fluorescence; Savel (*56X*) and Sjostrand (*60X*) who determined arsenic by activation analysis. Rozenberg (*54X*) determined beryllium in biological media after proper preparation, colorimetrically with sodium EDTA and Beryllon-P. Mohilner (*45X*) determined beryllium by a spot test based on a color reaction with Eriochrome Cyanine R. After proper digestion and preparation Sanz-Pedrero and Hermoso (*55X*) determined cadmium colorimetrically in biological media with a dithizone chloroform reagent. Methods for the determination of gold were described by: White and Zuber (*74X*) who used trimethylphenyl–ammonium iodide as a quantitative precipitant; Holbrook and Rein (*26X*) who extracted a bromoaurate–trioctylphosphine oxide complex into chloroform and measuring its absorbance; and by Solvsten (*62X*) and Rakovic (*53X*) who used neutron activation analysis to determine gold in serum and urine. Methods for the determination of lead were reported by: Sereda and Vorontsova (*58X*) who used ion exchange and a new reagent, sulfarsazen for colorimetric determination of urinary lead; Ilcewicz *et al.* (*27X*) who determined lead in bone ash by the absorbance of the chloro complex of lead in 9*M* HCl at 271 mμ; and by Natelson and De (*49X*) who used x-ray emission spectrometry to measure lead and bismuth in whole blood. Vignoli *et al.* (*73X*) reviewed modern techniques for the micro determination of mercury in body fluids. Methods for the determination of mercury in biological material were described by: Yablochkin (*80X*) and Fukuyama *et al.* (*20X*) who modified methods using dithizone for colorimetric determination; Matusiak *et al.* (*40X*) who deposited mercury on copper dust as in column chromatography; Smith (*61X*) who employed neutron activation analysis; and by Brooks *et al.* (*8X*) who determined mercury in urine by x-ray spectrometry. A method for determination of less than 0.4 μg. of nickel by ultraviolet spectrophotometry was devised by Sunderman (*66X*) in a study of nickel carcinogenesis. Ayres and McCrory (*6X*) determined platinum spectrophotometrically with 2, 3-quinoxalinedithiol which reacts with platinum–tin chloride in *N,N*-dimethylformamide to develop a blue color. Methods for the determination of selenium in biological materials were reported by: Allaway and Cary (*2X*) and Lott *et al.* (*35X*) who used 2,3-diaminonaphthalene reagent to determine microgram amounts; Cummins *et al.* (*14X*) who described a new digestion mixture for rapid determination; and by Kirkbright and Yoe (*31X*) who devised a new spectrophotometric method based on the formation of an azo dyestuff from the coupling of 1-naphthylamine with the diazonium oxidation product of the oxidation of phenylhydrazine *p*-sulfonic acid by selenous acid. Trudell and Boltz (*72X*) determined silicon by a method based on the UV absorptivity of molybdate originating from molybdosilic acid. Methods for the determination of thallium in biological fluids were described by: Wilson and Hausman (*75X*) who used flame spectrophotometry after proper oxidative preparation of specimen; Gabriel and Dubin (*21X*) who used a simple technique of shaking specimen with bromine water, then sulfosalicylic acid and finally with rhodamine B; Duvivier *et al.* (*18X*) who also coupled thallium with Rhodamine B after proper mineralization; and by Ariel and Bach (*5X*) who formed a colored complex with Brilliant Green after digestion and extraction of specimen. Sill and Willis (*59X*) described a micro method for the determination of thorium in biological samples by precipitation with barium sulfate and fluorometric measurement in alkaline solution. Fluorometric methods for the determination of uranium were reported by Andersen and Hercules (*4X*) who utilized the reaction between uranylbenzoic acid complex and Rhodamine B, and by Sommer (*63X*) who used a fluorometer working on the reflection principle.

VITAMINS

Kuznetsova and Vendt (*9Y*) described a new method for determination of vitamin A based on the fact that vitamin A in the form of the alcohol dissolved in a water free solvent is converted into anhydrovitamine A which is photometrically measured with *p*-toluenesulfonic acid. The turbidity produced by the Carr-Price reagent was avoided by Neeld and Pearson (*12Y*) and Dugan *et al.* (*1Y*) who used trifluoroacetic acid instead of $SbCl_3$. Lichtenthalter (*10Y*) developed a rapid method for the separation of vitamins and naturally occurring quinones. Rindi and Perri (*14Y*) described a simple fluorometric method for the determination of thiamine and thiamine sulfides in blood. Nelp *et al.* (*13Y*) measured the radioactivity of a single plasma sample 8 hours after an oral dose of Co^{57} vitamin B_{12} to test for pernicious anemia or malabsorption of vitamin B_{12}. Hall and Allen (*3Y*) described an improved method for extraction of vitamin B_{12} from serum by papain digestion and heat. Uchino *et al.* (*16Y*) used paper electrophoretic analysis of $Co^{60}B_{12}$ binding in human gastric juice. Kemper (*7Y*) made a critical evaluation of work since 1949 on bound ascorbic acid in animal tissues. Methods for the estimation of ascorbic acid were reported by: Nair *et al.* (*11Y*) who shortened the 2,4-dinitrophenylhydrazine coupling reaction time to 45 minutes by raising temperature to 57° C., Kum-Tatt and Leong (*8Y*) who titrated ascorbic acid in urine by a method based on the quantitative reduction of mercuric chloride to mercurous chloride; Huber and Stapelfeldt (*4Y*) who developed a constant current potentiometric titration method for ascorbic acid–glutathionine mixtures using 2,6-dichlorophenol–indophenol, *N*-bromosuccinimide and other oxidizing reagents; and by Hughes (*5Y*) who removed interferring substances with a cation exchange resin before color measurement with 2,6-dichloroindophenol. Katsui and Nishimoto (*6Y*) found both 2,4,6-tripyridyl-*S*-triazine and bathophenanthrolene satisfactory for colorimetric determination of vitamin E by the Emmlrie-Engel method. Erickson and Dunkley (*2Y*) determined tocopherol with 4,7-diphenyl-1,10-phenanthroline after separation from interferring compounds by silicic acid chromatography. Shul'gin *et al.* (*15Y*) described the construction of a slide rule with graduations for rapid estimation of chlorophyll a and b from the absorbance of ethyl alcohol extracts at 662 and 642 mμ.

LITERATURE CITED

Reviews and New Books

(1A) Bodansky, O., *Clin. Chem.* **9,** 1 (1963).
(2A) Boltz, D. F., Mellon, M. G., Anal. Chem. **36,** 257R (1964).
(3A) Campbell, W. J., Brown, J. D., *Ibid.*, p. 312R (1964).
(4A) C & EN Special Rept. *Chem. & Eng. News* **41,** 102 (1963).
(5A) Cocks, G. G., Anal. Chem. **36,** 163R (1964).
(6A) Escriu, J. M., Heer, E. E., *Semana Med.* (*Buenos Aires*) **123,** 965 (1963).
(7A) Evans, J. C., Anal. Chem. **36,** 240R (1964).
(8A) Hamilton, P. B., *Ibid.*, 3R (1964).
(9A) Heftmann, E., *Ibid.*, 14R (1964).
(10A) Henry, R. J., "Clinical Chemistry, Principles and Techniques," Harper and Row, New York, 1964.
(11A) Hirt, R. C., Anal. Chem. **36,** 308R (1964).
(12A) Hobbs, A. P., *Ibid.*, 130R (1964).
(13A) Horning, E. C., Van den Heuvel, W. J. A., *Ann. Rev. Biochem.* **32,** 709 (1963).
(14A) Juvet, R. S., Jr., Anal. Chem. **36,** 36R (1964).
(15A) Kingsley, G. R., *Ibid.*, **35,** 11R (1963).
(16A) Korenman, I. M., *Tr. po Khim. i Khim. Tekhnol.* **2,** 437 (1962).
(17A) Kunin, R., McGarvey, F. X., Anal. Chem. **36,** 142R (1964).
(18A) Lynch, M. J., Raphael, S. S., Mellow, L. D., Spare, P. D., Hills, P., Inwood, M. J. H., "Medical Laboratory Technology," Reprinted September, 1963 by W. B. Saunders Co., Philadelphia and London.
(19A) Ma, T. S., Gutterson, M., Anal. Chem. **36,** 150R (1964).
(20A) Mangold, H. K., Schmid, H. H. O., Stahl, E., *Methods Biochem. Anal.* (David Glick, ed., Interscience) **12,** 393 (1964).
(21A) Mason, W. B., *Chem. Eng. News* **42,** 107 (1964).
(22A) Morrison, G. H., Anal. Chem. **36,** 93R (1964).

(23A) Schoen, H., *Z. Anal. Chem.* **201,** 81 (1964).
(24A) Strickland, R. D., ANAL. CHEM. **36,** 80R (1964).
(25A) Tomas, S. M. E., Heer, E. E., *Semana Med.* (*Buenos Aires*) **122,** 33 (1963).
(26A) Thompson, R. H. S., King, E. J., "Biochemical Disorders in Human Disease," 2nd ed. (1066 pp., 153 illus.) Academic Press, New York, 1964.
(27A) Udenfriend, S., "Fluorescence Assay in Biology and Medicine," Academic Press, New York, 1962.
(28A) Van Kampen, E. J., *Z. Anal. Chem.* **205,** 381 (1964).
(29A) Walton, H. F., ANAL. CHEM. **36,** 51R (1964).
(30A) West, P. W., *Ibid.*, 144R (1964).
(31A) Whitby, L. G., *Lancet.* **1963,** 1239.
(32A) White, C. E., ANAL. CHEM. **36,** 116R (1964).
(33A) Williams, R. T., Bridges, J. W., *J. Clin. Path.* **17,** 371 (1964).

Apparatus and Equipment

(1B) Afonso, E., *Clin. Chim. Acta* **9,** 173 (1964).
(2B) Aikawa, J. K., Reardon, J. Z., Plym, A., *Am. J. Clin. Path.* **39,** 326 (1963).
(3B) Boling, E. A., *J. Lab. of Clin. Med.* **63,** 501 (1964).
(4B) Booman, G. L., Holbrook, W. B., ANAL. CHEM. **35,** 1793 (1963).
(5B) Chen, H. P., Myers, R., *Am. J. Clin. Path.* **38,** 522 (1962).
(6B) Danzer, L. A., Cohn, J. E., *J. Lab. & Clin. Med.* **63,** 355 (1964).
(7B) Draper, L. R., Mencken, C. R., *Ibid.*, 325 (1964).
(8B) Ewing, G. W., Brayden, T. H., Jr., ANAL. CHEM. **35,** 1826 (1963).
(9B) Faulkner, W. R., O'Mara, T. F., Jr., *Am. J. Clin. Path.* **39,** 209 (1963).
(10B) Hansen, W. N., Horton, J. A., ANAL. CHEM. **36,** 783 (1964).
(11B) Hochstrasser, H., Skeggs, L. T., Jr., Lentz, K. E., Kahn, J. R., *Anal. Biochem.* **6,** 13 (1963).
(12B) Holmes, F. E., *Chem. Anal.* **53,** 20 (1964).
(13B) Houtsmuller, A. J., *Clin. Chim. Acta* **9,** 94 (1964).
(14B) Lapaeva, L. A., *Lab. Delo* **1,** 58 (1964).
(15B) Lauer, G., Schlein, H., Osteryoung, R. A., ANAL. CHEM. **35,** 1789 (1963).
(16B) Lewis, U. J., Clark, M. O., *Anal. Biochem.* **6,** 303 (1963).
(17B) Ludvigsen, B., *Biochem. Biophys. Acta* **74,** 532 (1963).
(18B) Lyman, D. J., Loo, B. H., Crawford, R. W., *Biochem.* **3,** 985 (1964).
(19B) Mansell, R. E., Hunemorder, E. J., ANAL. CHEM. **35,** 1981 (1963).
(20B) Morrison, C. F., *Ibid.*, 1820 (1963).
(21B) Mueller, R. H., *Ibid.*, **36,** 140 (1964).
(22B) Natelson, S., Stellate, R. L., *Ibid.*, **35,** 847 (1963).
(23B) Onishi, T., Hagihara, B., Okunuki, K., *J. Biochem.* (*Tokyo*) **54,** 287 (1963).
(24B) Pasieka, A. E., *Can. J. Biochem.* **42,** 219 (1964).
(25B) Rockenmacher, M., Farr, A. F., *Clin. Chem.* **9,** 554 (1963).
(26B) Schwarz, W. M., Shain, I., ANAL. CHEM. **35,** 1770 (1963).
(27B) Shipotofsky, S. H., *Anal. Biochem.* **7,** 233 (1964).
(28B) Spurr, H. W., Jr., Hildebrandt, A. C., Riker, A. J., *Phytopath.* **53,** 782 (1963).
(29B) Stevenson, G. W., Carlsen, E. N., Arthur, E. P. (to Beckman Instruments, Inc.) U. S. Patent **3,147,081 (Cl. 23-230)** (Sept. 1, 1964) Appl. Feb. 27, 1961.
(30B) Taylor, B. W. (to Fisher Scientific Co.). U. S. Patent **3,111,390 (Cl. 23-253)** (Nov. 19, 1963) Appl. June 30, 1959.
(31B) Underwood, A. L., Robertson, T. M., ANAL. CHEM. **35,** 1761 (1963).
(32B) West, C. D., Hume, D. N., *Ibid.*, **36,** 412 (1964).
(33B) Williams, R. C., Ruffin, R. S., Berry, E. R., *Anal. Biochem.* **8,** 293 (1964).
(34B) Winsten, S., Friedman, H., Schwartz, E. E., *Ibid.*, **6,** 404 (1963).
(35B) Zak, B., Holland, J., Williams, L. A., *Clin. Chem.* **8,** 530 (1962).

Automation

(1C) ANAL. CHEM. **36,** No. 6, 31A (1964).
(2C) *Chem. Eng. News* **41,** 112 (1963).
(3C) Antonis, A., *Technicon Int'l Sym.* **1964,** 16.
(4C) "Autochemist," *Lab. World* **15,** 1204 (1964).
(5C) Baginski, E., Epstein, E., Zak, B., *Clin. Chim. Acta* **10,** 76 (1964).
(6C) Bell, J. L., Collier, M., Hartmann, A. M., *J. Clin. Path.* **17,** 301 (1964).
(7C) Benotti, J., Benotti, N., *Clin. Chem.* **9,** 408 (1963).
(8C) Block, R. J., Mandl, R. H., *Ann. N. Y. Acad. Sci.* **102,** 87 (1962).
(9C) Bonnafe, M., *Chromatog. Symp.* **2,** 285 (1962).
(10C) Boy, J., *J. Clin. Pathol.* **16,** 178 (1963).
(11C) Brooks, L., *Technicon Int'l Symp.* **1964,** 17.
(12C) Burns, T. W., Bregant, R. E., Vanpeenan, H. J., Hood, T. E., *J. Lab. Clin. Med.* **62,** 863 (1963).
(13C) Carstairs, E. A., *J. Sci. Technol.* **9,** 80 (1963).
(14C) Catanzaro, E. W., *Technicon Int'l Sym.* **1964,** 6.
(15C) Catravas, G. N., ANAL. CHEM. **36,** 1146 (1964).
(16C) Chen, H. P., Sharton, H., *Am. Jour. Clin. Pathol.* **40,** 651 (1963).
(17C) Cooper, G. R., Roland, D. M., Eavenson, E., *Technicon Int'l Sym.* **1964,** 16.
(18C) Crofford, O. B., Lacy, W. W., *J. Lab. Clin. Med.* **61,** 708 (1963).
(19C) Crowley, L. V., *Clin. Chem.* **10,** 838 (1964).
(20C) Curtain, C. C., *Biol. Eng.* **1,** 227 (1963).
(21C) Discombe, G., *J. Clin. Pathol.* **16,** 170 (1963).
(22C) Dwiggins, C. W., Jr., ANAL. CHEM. **36,** 1577 (1964).
(23C) Epstein, E., Baginski, E. S., Zak, B., *Clin. Chem.* **9,** 441 (1963).
(24C) Etienne, G., Papin, J. P., Renault, H., *Ann. Biol. Clin.* **21,** 85 (1963).
(25C) Everard, D., Seymour, G., *J. Clin. Pathol.* **16,** 170 (1963).
(26C) Fingerhut, B., Ferzola, R., Marsh, W. H., *Clin. Chim. Acta* **8,** 953 (1963).
(27C) Frings, C. S., Pardue, H. L., ANAL. CHEM. **36,** 2477 (1964).
(28C) Gambino, S. R., *Technicon Int'l Sym.* **1964,** 14.
(29C) Getchell, G., Kingsley, G. R., Schaffert, R. R., *Clin. Chem.* **10,** 540 (1964).
(30C) Glenn, J. H., *Clin. Chim. Acta* **9,** 501 (1964).
(31C) Golub, M., *Clin. Chem.* **10,** 399 (1964).
(32C) Griffiths, W. J., *Clin. Chim. Acta* **9,** 210 (1964).
(33C) Grossman, A., Grossman, G. F., Pollack, R. L., *Anal. Biochem.* **8,** 124 (1964).
(34C) Hill, J. B., *Ann. N. Y. Acad. Sci.* **102,** 108 (1962).
(35C) Hochella, N. J., Weinhouse, S., *Technicon Int'l Sym.* **1964,** 14.
(36C) Hochstrasser, H., *Ibid.*, 11 (1964).
(37C) Jones, J. D., McGuckin, W. F., *Clin. Chem.* **10,** 767 (1964).
(38C) Kabara, J. J., Zyskowski, D., Pokornicki, H., *Ibid.*, 950 (1964).
(39C) Keay, H., Trew, J. A., *Ibid.*, 75 (1964).
(40C) Kenny, A. P., Jamieson, A., *Clin. Chim. Acta* **10,** 536 (1964).
(41C) Kessler, G., Wolfman, M., *Clin. Chem.* **10,** 686 (1964).
(42C) King, J. S., Jr., *Clin. Chim. Acta* **9,** 441 (1964).
(43C) Kirkpatrick, C. H., Anderson, R. A., *J. Chromatog.* **14,** 295 (1964).
(44C) Kolbezen, M. J., Eckert, J. W., ANAL. CHEM. **36,** 593 (1964).
(45C) Krampitz, G., Wieneke, J., *Z. Tierphysiol., Tierenaehr. Futtermittelk.* **18,** 147 (1963).
(46C) Lanzkowsky, P., Klassen, W. H., Wilson, J. F., *J. Lab. Clin. Med.* **62,** 341 (1963).
(47C) Leithoff, H., *Med. Welt* **1963,** 959.
(48C) Leonards, J. R., Davoren, P. R., *Ann. N. Y. Acad. Sci.* **102,** 83 (1962).
(49C) Lofland, H. B., Jr., *Technicon Int'l Sym.* **1964,** 15.
(50C) Lowell, D. J., Aroner, J., Pierce, R., Danielson, G., Marshall, W. *Clin. Chem.* **10,** 463 (1964).
(51C) Malmstadt, H. V., Hadjiioannou, T. P., ANAL. CHEM. **35,** 14 (1963).
(52C) Malmstadt, H. V., Hadjiioannou, T. P., *Clin. Chem.* **9,** 423 (1963).
(53C) Malmstadt, H. V., Hadjiioannou, T. P., ANAL. CHEM. **35,** 2157 (1963).
(54C) Malmstadt, H. V., Pardue, H. L., *Clin. Chem.* **8,** 606 (1962).
(55C) McKay, D. K., Seligson, D., Jewett, W. R., *Ibid.*, **10,** 907 (1964).
(56C) McNair, R. D., *Ibid.*, 648 (1964).
(57C) Merrills, R. J., *Anal. Biochem.* **6,** 272 (1963).
(58C) Meschia, G., *Technicon Int'l Sym.* **1964,** 14.
(59C) Morganstern, S., Kessler, G., Auerbach, J., Flor, R. V., Klein, B., *Clin. Chem.* **10,** 635 (1964).
(60C) Natho, V., Frankel, S., *Lab. World* **15,** 692 (1964).
(61C) Nevius, D. B., Lanchantin, G. F., *Clin. Chem.* **10,** 651 (1964).
(62C) Pardue, H. L., ANAL. CHEM. **35,** 1240 (1963).
(63C) Pardue, H. L., Simon, R. K., Malmstadt, H. V., *Ibid.*, **36,** 735 (1964).
(64C) Pellerin, J., *Clin. Chem.* **10,** 374 (1964).
(65C) Pentz, E. I., *Anal. Biochem.* **8,** 328 (1964).
(66C) Riley, M., Gochman, N., *Clin. Chem.* **10,** 649 (1964).
(67C) Robinson, R. L., Watts, D. T., *Technicon Int'l Sym.* **1964** 17.
(68C) Russell, S., Bryant, M., Morrison, D. B., *Clin. Chem.* **10,** 641 (1964).
(69C) Skeggs, L. T., Jr., *Ibid.*, **9,** 442 (1963).
(70C) *Ibid.*, p. 443.
(71C) Skeggs, L. T., Jr., Hochstrasser, H., *Ibid.*, **10,** 918 (1964).
(72C) Schaffert, R. R., Kingsley, G. R., Getchell, G., *Ibid.*, 519 (1964).
(73C) Schwartz, M. K., Bodansky, O., *Biochem. Anal.* **11,** 211 (1963).
(74C) Sobolewski, G., *Clin. Chem.* **10,** 563 (1964).
(75C) Stevens, C. O., Levandoski, N. G., *Ibid.*, **9,** 400 (1963).
(76C) Strandjord, P. E., Clayson, K. J., *Ibid.*, **10,** 635 (1964).
(77C) Summers, R. M., Mefferd, R. B., Jr., Littleton, H. R., *Technicon Int'l Sym.* **1964,** 14.
(78C) Thiers, R. E., *Clin. Chem.* **10,** 651 (1964).

(79C) *Ibid.*, p. 651.
(80C) Thiers, R. E., Oglesby, K. M., *Ibid.*, 246 (1964).
(81C) Tietz, N. W., Green, A., *Clin. Chim. Acta* **9**, 392 (1964).
(82C) Walborg, E. F., Jr., Cobb, B. F., III, Adams-Mayne, M., Ward, D. N., *Anal. Biochem.* **6**, 367 (1963).
(83C) Walter, A. R., Gerarde, H. W., *Clin. Chem.* **10**, 509 (1964).
(84C) Whitley, R. W., Alburn, H. E., *Technicon Int'l Sym.* **1964**, 16.
(85C) Widdowson, G. M., Northam, B. E., *Clin. Chim. Acta* **8**, 636 (1963).
(86C) Wieme, R. J., van Raepenbusch, F. R., *Ibid.*, **7**, 883 (1962).
(87C) Winsten, S., *Ann. N. Y. Acad. Sci.* **102**, 127 (1962).
(88C) Zak, B., *Technicon Int'l Sym.* **1964**, 14.
(89C) Zak, B., Epstein, E., *Chem. Anal.* **52**, 45 (1963).

Control and Precision of Clinical Chemistry Methods

(1D) Calder, A. B., ANAL. CHEM. **36**, 25 A (1964).
(2D) Curnow, D. H., Sheard, K., *Clin. Chem.* **9**, 462 (1963).
(3D) Henry, R. J., *Std. Methods Clin. Chem.* **4**, 205 (1963).
(4D) Hersey, M. Q., Hartwell, K., Doe, R. P., *Clin. Chem.* **9**, 557 (1963).
(5D) Hoffmann, R. G., Wald, M. E., *Am. J. Clin. Pathol.* **40**, 263 (1963).
(6D) Mull, J. D., Murphy, W. R., *Am. J. Clin. Pathol.* **39**, 134 (1963).
(7D) Nelson, B. N., ANAL. CHEM. **36**, 344R (1964).
(8D) Peckham, S., Dimmette, R. M., *Clin. Chem.* **10**, 656 (1964).
(9D) Porter, C. J., *Ibid.*, **9**, 460 (1963).
(10D) Tonks, D. B., *Ibid.*, 217 (1963).
(11D) Turpin, B. C., *Am. J. Med. Technol.* **29**, 181 (1963).
(12D) Weinberg, M. S., Barnett, R. N., *Am. J. Clin. Pathol.* **38**, 468 (1962).
(13D) Whitby, L. G., *Lancet* 1239 (1963).
(14D) Young, D. M., *Clin. Chem.* **9**, 461 (1963).
(15D) Young, D. M., *Am. J. Med. Technol.* **30**, 99 (1964).

Amino Acids

(1E) Baldridge, R. C., Greenberg, N., *J. Lab. Clin. Med.* **61**, 700 (1963).
(2E) Belen'kii, B. G., Orestova, V. A., *Izv. Akad. Nauk SSSR, Ser. Khim.* I 182 (1964).
(3E) Blackburn, S., Lee, G. R., *Biochem. J.* **87**, 1P (1963).
(4E) Braun, L., *Biochem. Z.* **339**, 8 (1963).
(5E) Carson, J. F., Wong, F. F., *J. Chromatog.* **12**, 408 (1963).
(6E) Frame, E. G., *Standard Methods Chem.* **4**, 1 (1963).
(7E) Gustus, E. L., *J. Biol. Chem.* **239**, 115 (1964).
(8E) Hamilton, P. B., ANAL. CHEM. **35**, 2055 (1963).
(9E) Hara, Y., *Bull. Chem. Soc. Japan* **36**, 1373 (1963).
(10E) Jacobs, S., *Biochem. J.* **87**, 30 P (1963).
(11E) Jutisz, M., de la Llosa, P., *Bull. Soc. Chim. (France)* **12**, 2913 (1963).
(12E) Knapp, A., Hothmann, M., Richter, B., Foerster, W., *Deut. Gesundheitsw.* **18**, 2285 (1963).
(13E) Lack, L., Smith, M., *Ann. Biochem.* **8**, 217 (1964).
(14E) La Du, B. N., Howell, R., Michael, P. J., Sober, E. K., *Pediatrics* **31**, 39 (1963).
(15E) Lebedeva, Z. N., *Lab. Delo* **8**, 451 (1964).
(16E) Mabry, C. C., Karam, E. A., *Am. J. Clin. Pathol.* **42**, 421 (1964).
(17E) Malangeau, P., Bourdon, R., Nicaise, A. M., Masson, B., *Ann. Biol. Clin. (Paris)* **21**, 3 (1963).
(18E) Matheson, N. A., *Biochem. J.* **88**, 146 (1963).
(19E) Meiss, M., Peyser, D. P., Miller, A., *J. of Lab. & Clin. Med.* **64**, 512 (1964).
(20E) Mougey, E. H., Mason, J. W., *Anal. Biochem.* **6**, 223 (1963).
(21E) Myhill, D., Jackson, D. S., *Ibid.*, 193 (1963).
(22E) Nybom, N., *Physiol. Plantarum* **17**, 434, (1964).
(23E) Opienska-Blauth, J., Charezinski, M., Berbec, H., *Anal. Biochem.* **6**, 69 (1963).
(24E) Ramadan, M. E. A., Greenberg, D. M., *Ibid.*, 144 (1963).
(25E) Rapp, R. D., *Clin. Chem.* **9**, 27 (1963).
(26E) Roston, S., *Anal. Biochem.* **6**, 486 (1963).
(27E) Sackett, D. L., *J. of Lab. & Clin. Med.* **63**, 306 (1964).
(28E) Said, A., Fleita, D. H., *Chemist-Analyst* **52**, 79 (1963).
(29E) Saifer, A., Gerstenfeld, S., *Clin. Chem.* **10**, 970 (1964).
(30E) Sowerby, J. M., Ottaway, J. H., *Biochem. J.* **79**, 21 (1961).
(31E) Summer, G. K., Hawes, J. A., *Proc. Soc. Exptl. Biol. Med.* **112**, 402 (1963).
(32E) Umberger, C. J., Fiorese, F. F., *Clin. Chem.* **9**, 79 (1963).
(33E) Updike, S. S., Dounce, A. L., *Anal. Biochem.* **6**, 1 (1963).
(34E) Warren, H. W., Horn, M. J., Blum, A. E., *Ibid.*, **5**, 70 (1963).

Blood Preservation, Clotting Factors, Gasometric Analysis, pH, and Volume

(1F) Andersen, O. S., *Scandinav. J. Clin. Lab. Invest.* **15**, 211 (1963).
(2F) *Ibid.*, p. 116.
(3F) Astrup, P., Andersen, O. S., *Advances in Clin. Chem.* **6**, 1 (1963).
(4F) Berglund, E., Malmberg, R., Stenhagen, S., *Scandinav. J. Clin. Lab. Invest.* **16**, 185 (1964).
(5F) Bowes, J. B., *Anaesthesia* **19**, 40 (1964).
(6F) Chambliss, K. W., Nouse, D. C., *Clin. Chem.* **8**, 654 (1962).
(7F) Conti, A. M., Grisi, A. M., Cresseri, A., *Boll. Soc. Ital. Biol. Sper.* **38**, 1196 (1962).
(8F) Falholt, W., *Scandinav. J. Clin. Lab. Invest.* **15**, 67 (1963).
(9F) Farhi, L. E., Edwards, A. W. T., Homma, T., Passke, M., *J. Appl. Physiol.* **18**, 97 (1963).
(10F) Fenichel, R. L., Baker, W. J., Rose, L., *Clin. Chem.* **10**, 69 (1964).
(11F) Gambino, S. R., Goldberg, H. E., Polanyi, M. L., *Am. J. Clin. Pathol.* **42**, 364 (1964).
(12F) Gollub, S., Ulin, A. W., *Clin. Chem.* **10**, 730 (1964).
(13F) Johnston, G. W., *Std. Methods Clin. Chem.* **4**, 183 (1963).
(14F) Kollath, H. H., Fischer, H., Breddin, K., *Klin Wochschr.* **41**, 1165 (1963).
(15F) Lanchantin, G. F., Friedmann, J. A., DeGroot, J., Mehl, J. W., *J. Biol. Chem.* **238**, 238 (1963).
(16F) Laughlin, D. E., McDonald, J. S., Bedell, G. N., *J. Lab. Clin. Med.* **64**, 330 (1964).
(17F) Little, J. M., Williams, C., *Proc. Soc. Exptl. Biol. Med.* **115**, 564 (1964).
(18F) Maas, A. H. J., Zuijdgeest, L. P. W. A., Kreukniet, J., *Clin. Chim. Acta* **9**, 236 (1964).
(19F) Manfredi, F., *J. Lab. Clin. Med.* **61**, 1005 (1963).
(20F) Martinek, R. G., *Clin. Chem.* **10**, 153 (1964).
(21F) Noren, I., Quick, A. J., *J. Lab. Clin. Med.* **61**, 302 (1963).
(22F) Owren, P. A., *Arch. Internal. Med.* **3**, 248 (1963).
(23F) Paloheimo, J. A., *Scandinav. J. Clin. Lab. Invest.* **15**, 563 (1963).
(24F) Pawan, G. L. S., Hoult, W. H., *Biochem. J.* **87, 1963** 6.
(25F) Quick, A. J., *Arch. Internal. Med.* **3**, 234 (1963).
(26F) Quick, A. J., Geppert, M., *Am. J. Clin. Pathol.* **40**, 465 (1963).
(27F) Quick, A. J., *Am. J. Med. Sci.* **246**, 517 (1963).
(28F) Rebar, J., Jr., Oakes, P. W., Freeby, W. A. (to Miles Labs., Inc.). U. S. Patent **3,122,420** (**Cl. 23-253**) (Feb. 25, 1964) Appl. Feb. 28, 1962.
(29F) Russ, C., Lee, J., Carr, Z., Taylor, B., *J. Gas Chromatog.* **2**, 18 (1964).
(30F) Severinghaus, J. W., *Clin. Chem.* **9**, 727 (1963).
(31F) Thomason, R., *Scand. J. Clin. Lab. Invest.* **15**, 45 (1963).
(32F) Tsao, M. U., Vadnay, A., *J. Lab. Clin. Med.* **63**, 1041 (1964).
(33F) Updegraff, D. M., Huckabay, W. B., *Anal. Biochem.* **5**, 28 (1963).
(34F) Vermylen, C., De Vreker, R. A., Verstraete, M., *Clin. Chim. Acta* **8**, 418 (1963).
(35F) Ware, A. G., Nowack, J., Westover, L., *Clin. Chem.* **9**, 340 (1963).
(36F) Ware, A. G., Sterling, R. E., Stragnell, R., *Angiology* **15**, 11 (1964).

Carbohydrates

(1G) Achari, G., Mishra, K. C., Rudra, M. N., *Indian Practitioner* **13**, 469 (1960).
(2G) Bowden, C. H., *J. Clin. Pathol.* **16**, 470 (1963).
(3G) Brewer, G. J., Tarlov, A. R., *Am. J. Clin. Pathol.* **39**, 579 (1963).
(4G) Bueding, E., Hawkins, J. T., *Anal. Biochem.* **9**, 115 (1964).
(5G) Campbell, D. M., King, E. J., *J. Clin. Pathol.* **16**, 173 (1963).
(6G) Cessi, C., Serafini-Cessi, F., *Biochem. J.* **88**, 132 (1963).
(7G) Di Ferrante, N., *J. of Lab. and Clin. Med.* **61**, 633 (1963).
(8G) Fales, F. W., *Standard Methods Clin. Chem.* **4**, 101 (1963).
(9G) Ford, J. D., Haworth, J. C., *Clin. Chem.* **10**, 1002 (1964).
(10G) Free, A. H. (to Miles Laboratories, Inc.). U. S. Patent **3,061,523** (**Cl. 195-103.5**) (Oct. 30, 1962) Appl. Nov. 7, 1956. Continuation-in-part of U. S. Patent **2,848,308** (*CA*, **53**, 11503*i*).
(11G) *Ibid.*, U. S. Patent **3,099,605** (**Cl. 195-103.5**) (July 30, 1963) Appl. Feb. 1, 1960.
(12G) Free, A. H., *Advances in Clin. Chem.* **6**, 67 (1963).
(13G) Gagliardo, E., Zambotti, V., *Boll. Soc. Ital. Biol. Sper.* **38**, 1791 (1962).
(14G) Gold, H., U. S. Patent **2,963,350** (Dec. 6, 1960).
(15G) Hansen, O., *Scand. J. Clin. & Lab. Invest.* **14**, 651 (1962).
(16G) Hjelm, M., de Verdier, C. H., *Ibid.*, **15**, 415 (1963).
(17G) Jacobs, Ph., *Chem. Weekblad* **59**, 465 (1963).
(18G) Johnson, J. A., Nash, J. D., Fusaro, R. M., *Anal. Biochem.* **5**, 379 (1963).
(19G) Kantorovich, A. S., U.S.S.R. Patent **158,399** (Oct. 19, 1963) Appl. Nov. 16, 1962.
(20G) Klassen, W. H., Lanzkowsky, P., *Clin. Chim. Acta* **9**, 183 (1964).
(21G) Klein, B., Weissman, M., *Clin. Chem.* **10**, 741 (1964).

(22G) Mabry, C. C., Gryboski, J. D., Karam, E. A., *J. Lab. Clin. Med.* **62**, 817 (1963).
(23G) McKay, E., *Clin. Chim. Acta,* **10**, 320 (1964).
(24G) Meites, S., Bohman, N., *Clin. Chem.* **9**, 594 (1963).
(25G) Meites, S., Bohman, N., *Am. J. Med. Technol.* **29**, 327 (1963).
(26G) Miles Laboratories, Inc., British Patent **911,181** (Nov. 21, 1962) U. S. Appl., March 25, 1960.
(27G) Miles Laboratories, Inc. (by Edward K. Harvill), Belg. Patent **627,415** (May 16, 1963) U. S. Appl. Jan. 24, 1962.
(28G) Momose, T. (Chugai Pharmaceutical Co., Ltd.) Japan Patent **15,900** (Oct. 6, 1962) Appl. July 27, 1959.
(29G) Pakianathan, S. W., *Enzymologia* **26**, 155 (1963).
(30G) Percheron, F., *Compt. Rend.* **255**, 2521 (1962).
(31G) Pietrusiewicz, M., Pidek, E., *Chem. Anal. (Warsaw)* **7**, 1007 (1962).
(32G) Relander, A., *Scandinav. J. Clin. & Lab. Invest.* **15**, 218 (1963).
(33G) Relander, A., Raiha, C. E., *Ibid.*, 221 (1963).
(34G) Rorem, E. S., Lewis, J. C. (to U. S. Dept. Agriculture). U. S. Patent **3,066,081 (Cl. 195-103.5)** (Nov. 27, 1962) Appl. May 5, 1961.
(35G) Ruiter, J., Weinberg, F., Morrison, A., *Clin. Chem.* **9**, 356 (1963).
(36G) Rutman, J. Z., *Anal. Biochem.* **8**, 179 (1964).
(37G) Sato, T., *Seikagaku* **35**, 331 (1963).
(38G) Scheffer, F., Kickuth, R., *Z. Anal. Chem.* **191**, 116 (1962).
(39G) Smeby, R. R. (to Miles Laboratories, Inc.) U. S. Patent **3,123,443 (Cl. 23-253)** (Mar. 3, 1964) Appl. Apr. 18, 1960.
(40G) Smith, M. D., St. Rose, J. E. M., Radomski, M. W., *Anal. Biochem.* **9**, 116 (1964).
(41G) Teller, J. D. (to Worthington Biochemical Corp.). U. S. Patent **3,099,606 (Cl. 195-103.5)** (July 30, 1963) Appl., Nov. 7, 1961.
(42G) Topi, G. C., Zane, V. A., *Arch. Studio Fisiolpatol. Clin. Ricambio* **25**, 93 (1961).
(43G) Watson, D., *Anal. Biochem.* **5**, 260 (1963).
(44G) Watson, D., Stevenson, M. E. K., *Australian J. Exptl. Biol. Med. Sci.* **41**, 211 (1963).
(45G) Wells, W. W., Chin, T., Weber, B., *Clin. Chim. Acta* **10**, 352 (1964).
(46G) Western Laboratories, Inc., British Patent **920,186** (Mar. 6, 1963) U. S. Appl. Nov. 2, 1959.

Cations and Anions

(1H) Anast, C. S., *Clin. Chem.* **9**, 544 (1963).
(2H) Baar, S., *Clin. Chim. Acta* **7**, 642 (1962).
(3H) Beale, R. N., Bostrom, J. O., *J. Clin. Pathol.* **16**, 252 (1963).
(4H) Bloomer, H. A., Trammell, D. D., Rector, F. C., Jr., *J. Lab. Clin. Med.* **61**, 692 (1963).
(5H) Bowden, C. H., Patston, V. J., *J. Clin. Pathol.* **16**, 18 (1963).
(6H) Burcar, P. J., Boyle, A. J., Mosher, R. E., *Clin. Chem.* **10**, 1028 (1964).
(7H) Butler, E. J., Forbes, D. H. S., Munro, C. S., Russell, J. C., *Anal. Chim. Acta* **30** 524 (1964).
(8H) Christian, G. D., Knoblock, E. C., ANAL. CHEM. **35**, 1869 (1963).
(9H) Cotlove, E., *Ibid.*, 95 (1963).
(10H) Cotlove, E., *Methods Biochem. Anal.* **12**, 277 (1964).
(11H) Crockson, R. A., *J. Clin. Pathol.* **16**, 473 (1963).
(12H) Davis, A., Dinan, F. J., Lobbett, E. J., Chazin, J. D., Tufts, L. E., ANAL. CHEM. **36**, 1066 (1964).
(13H) Decker, C. F., Aras, A., Decker, L. E., *Anal. Biochem.* **8**, 344 (1964).
(14H) Diehl, H., Olsen, R., Spielholtz, G. I., Jensen, R., ANAL. CHEM. **35**, 1144 (1963).
(15H) Dryer, R. L., Routh, J. L., *Std. Meth. Clin. Chem.* **4**, 191 (1963).
(16H) Dugenetay, C., Lubochinsky, B., Stolkowski, J., *J. Physiol. (Paris)* **55**, 523 (1963).
(17H) Dunsbach, F., *Aerztl. Lab.* **10**, 153 (1964).
(18H) Ellis, N. J., Bishop, D. M., *Can. J. Biochem.* **42**, 1225 (1964).
(19H) Ferguson, J. W., Richard, J. J., O'Laughlin, J. W., Banks, C. V., ANAL. CHEM. **36**, 796 (1964).
(20H) Fingerhut, B., Miller, H., *Clin. Chem.* **9**, 360 (1963).
(21H) Freier, E. F., Clayson, K. J., Benson, E. S., *Clin. Chim. Acta* **9**, 348 (1964).
(22H) Friedman, S. M., Wong, S. L., Walton, J. H., *J. Appl. Physiol.* **18**, 950 (1963).
(23H) Gregoriadis, G., *Chim. Chronika (Athens, Greece)* **28**, 15 (1963).
(24H) Gusev, S. I., Sokolova, E. V., Bitovt, Z. A., *Sb. Nauchn, Tr. Permsk. Gos. Med. Inst.* **38**, 293 (1963).
(25H) Hall, R. J., *Analyst* **88**, 76 (1963).
(26H) Herrero-Lancina, M., West, T. S., ANAL. CHEM. **35**, 2131 (1963).
(27H) Horn, D. B., Latner, A. L., *Clin. Chem.* **9**, 458 (1963).
(28H) Kaplar, L., *Acta Pharm. Hung.* **33**, 152 (1963).
(29H) Kepner, B. L., Hercules, D. M., ANAL. CHEM. **35**, 1238 (1963).
(30H) Krampitz, G., Doepfmer, R., *Rev. Univ. Ind. Santander* **5**, 361 (1963).
(31H) Lazarov, N., *Lab Delo* **1**, 9 (1964).
(32H) Lindstrom, F., Milligan, C. W., ANAL. CHEM. **36**, 1334 (1964).
(33H) Loken, H. F., Teal, J. S., Eisenberg, E., *Ibid.*, **35**, 875 (1963).
(34H) London, M., Marymont, J. H., Jr., *Clin. Chem.* **10**, 417 (1964).
(35H) Lumb, G. A., *Clin. Chim. Acta* **8**, 33 (1963).
(36H) MacFate, R. P., *Proc. Appl. Seminar Assoc. Clin. Scientists, 3rd,* Washington, D. C., 1961, 180–98 (pub. 1963).
(37H) Mayr, K., *Wien. Med. Wochschr.* **113**, 519 (1963).
(38H) Miller, E., Hlad, C. J., Jr., Levine, S., Holmes, J. H., Elrick, H., *J. Lab. & Clin. Med.* **62**, 710 (1963).
(39H) Moore, E. W., Wilson, D. W., *J. Clin. Invest.* **42**, 293 (1963).
(40H) Morrison, W. R., *Anal. Biochem.* **7**, 218 (1964).
(41H) Mueller, J., *Cesk. Morfol.* **11**, 376 (1963).
(42H) Munday, K. A., Mahy, B. W. J., *Clin. Chim. Acta* **10**, 144 (1964).
(43H) Negrin, A., *Ibid.*, **10**, 262 (1964).
(44H) Rice, E. W., La Para, C. Z., *Clin. Chem.* **10**, 465 (1964).
(45H) Rodkey, F. L., Sendroy, J., Jr., *Ibid.*, **9**, 668 (1963).
(46H) Rovner, D. R., Conn, J. W., *J. Lab. & Clin. Med.* **62**, 492 (1963).
(47H) Schoenfeld, R. G., Lewellen, C. J., *Clin. Chem.* **10**, 533 (1964).
(48H) Scribner, B. F., Margoshes, M., ANAL. CHEM. **36**, 329 (1964).
(49H) Sherrick, J. C., De la Huerga, J., *Proc. Appl. Seminar Assoc. Clin. Scientists, 3rd,* Washington, D. C., 1961, 208–12 (pub. 1963).
(50H) Shimomura, O., Johnson, F. H., Saiga, Y., *Science* **140**, 1339 (1963).
(51H) Sky-Peck, H. H., *Clin. Chem.* **10**, 391 (1964).
(52H) Spach, C., Erny, A., *Bull. Soc. Chim. Biol.* **45**, 1191 (1963).
(53H) Stewart, W. K., Hutchinson, F., Fleming, L. W., *J. Lab. & Clin. Med.* **61** 858 (1963).
(54H) Stewart, C. P., Frazer, S. C., *Advances in Clin. Chem.* **6**, 29 (1963).
(55H) Stoffyn, P., Keane, W., ANAL. CHEM. **36**, 397 (1964).
(56H) Teixeira, F., Martins, A., Preza, F. M., *Clin. Chem.* **9**, 452 (1963).
(57H) Valberg, L. S., Holt, J. M., Szivek, J., ANAL. CHEM. **36**, 790 (1964).
(58H) Vedso, S., Rud, C., *Scand. J. Clin. Lab. Invest.* **15**, 395 (1963).
(59H) Wallach, D. F. H., Steck, T. L., *Anal. Biochem.* **6**, 176 (1963).
(60H) Watson, D., Rogers, J. A., *Clin. Chim. Acta* **8**, 168 (1963).
(61H) Zettner, A., Seligson, D., *Clin. Chem.* **10**, 869 (1964).

Drugs

(1J) Cramer, G., Isaksson, B., *Scandinav. J. Clin. & Lab. Invest.* **15**, 533 (1963).
(2J) Eagleson, D. A., *Am. J. Clin. Pathol.* **39**, 648 (1963).
(3J) Eidus, L., Hamilton, E. J., *Clin. Chem.* **10**, 581, (1964).
(4J) Gold, S., Griffiths, P. D., Huntsman, R. G., *J. Mental Sci.* **108**, 88 (1962).
(5J) Gothelf, B., Karczmar, A. G., *Intern. J. Neuropathacol.* **2**, 95 (1963).
(6J) Hamilton, E. J., Eidus, L., Lee, Y. S., *Clin. Chim. Acta* **8**, 763 (1963).
(7J) Kanter, S. L., *Ibid.*, **8**, 2 (1963).
(8J) Kern, W., ANAL. CHEM. **35**, 50 (1963).
(9J) Koechlin, B. A., D'Arconte, L., *Anal. Biochem.* **5**, 195 (1963).
(10J) Lindfors, R., *Ann. Med. Exptl. Biol. Fenniae (Helsinki)* **41**, 355 (1963).
(11J) Mellinger, T. J., Keeler, C. E., ANAL. CHEM. **35**, 554 (1963).
(12J) Musil, J., Kafka, V., Knobloch, E., Pavlovska, J., *Clin. Chim. Acta* **7**, 875 (1962).
(13J) Sato, K., *Kagaku Keisatsu Kenkyusho Hokoku* **15**, 237 (1962).
(14J) Sil'vestrov, V. P., Malov, Y. S., Feofilova, V. G., *Lab. delo* **9**, 16 (1963).
(15J) Street, H. V., McMartin, C., *Clin. Chim. Acta* **9**, 301 (1964).
(16J) Vercruysse, A., *Chromatog. Symp., 2nd,* Brussels 207 (1962).

Lipids

(1K) Araki, E., *Nisshin Igaku* **50**, 85 (1963).
(2K) Armstrong, S. J., *Am. J. Med. Technol.* **30**, 47 (1964).
(3K) Ayrault - Jarrier, M., Walt, R., Polonovski, J., *Bull. Soc. Chim. Biol.* **45**, 361 (1963).
(4K) Barclay, M., Barclay, R. K., Terebus-Kekish, O., Shah, E. B., Skipski, V. P., *Clin. Chim. Acta* **8**, 721 (1963).
(5K) Barre, R. Labat, J., *Ann. Biol. Clin. (Paris)* **20**, 679 (1962).
(6K) Bowers, M. A., Lund, P. K., Mathies, J. C., *Clin. Chim. Acta* **9**, 344 (1964).
(7K) Carlson, L. A., *J. Atherosclerosis Res.* **3**, 334 (1963).
(8K) Carmichael, G. G., *J. Histochem. Cytochem.* **11**, 738 (1963).
(9K) Christophe, A., Matthijs, F., *Bull. Soc. Chim. Belges* **73**, 592 (1964).
(10K) Dales, R. P., *Nature* **197**, 1120 (1963).
(11K) Dangerfield, W. G., Faulkner, G., *Clin. Chim. Acta* **10**, 123 (1964).
(12K) Di Leo, F. P., *SIPS (Soc. Ital. Progr. Sci.), Sci. Tech.* **4**, 110 (1960).

(13K) Dobiasova, M., *J. Lipid Res.* **4,** 481 (1963).
(14K) Doizaki, W. M., Zieve, L., *Proc. Soc. Exptl. Biol. Med.* **113,** 91 (1963).
(15K) Duncombe, W. G., *Biochem. J.* **88,** 7 (1963).
(16K) Duncombe, W. G., *Clin. Chim. Acta* **9,** 122 (1964).
(17K) Freeman, N. K., *J. Lipid Res.* **5,** 236 (1964).
(18K) Fried, R., Hoeflmayr, J., *Klin. Wochschr.* **41,** 246 (1963).
(19K) Galanos, D. S., Alivazis, G. A. M., Kapoulas, V. M., *J. Lipid Res.* **5,** 242 (1964).
(20K) Giarnieri, D., *Lab. Diagnosi Med.* **8,** 1 (1963).
(21K) Jellum, E., Bjornstad, P., *J. Lipid Res.* **5,** 314 (1964).
(22K) Konitzer, K., Voight, S., Solle, M. Moeschler, D., *Acta Biol. Med. Ger.* **12,** 502 (1964).
(23K) Krell, K., Hashim, S. A., *J. Lipid Res.* **4,** 407 (1963).
(24K) Kreutz, F. H., *Clin. Chem.* **9,** 492 (1963).
(25K) Kuksis, A., *Can. J. Biochem.* **42,** 419 (1964).
(26K) Lees, R. S., Hatch, F. T., *J. Lab. Clin. Med.* **61,** 518 (1963).
(27K) Mangold, H. K., Kammereck, R., Malins, D. C., *Microchem. J., Symp. Ser.* **2,** 697 (1961) (pub. 1962).
(28K) McFarlane, R. G., *Irish J. Med. Sci. Ser.* **6,** 423 (1962).
(29K) Nicolaysen, R., Nygaard, A. P., *Scandinav. J. Clin. Lab. Invest.* **15,** 79 (1963).
(30K) Peeters, H., de Keersgieter, W., Lamote, E., *Chromatog. Symp., 2nd,* Brussels 113 (1962).
(31K) Pinter, K. G., Hamilton, J. G., Miller, O. N., *Anal. Biochem.* **8,** 158 (1964).
(32K) Prevot, A., *Bull. Soc. Chim. France* **1963,** 314.
(33K) Reinhold, J. G., Yonan, V. L., Gershman, E. R., *Std. Methods Clin. Chem.* **4,** 85 (1963).
(34K) Schain, P., U. S. Patent **3,062,623** (*Cl.* **23-230**) (Nov. 6, 1962) Appl. Oct. 13, 1958. Continuation-in-part of U. S. Patent **2,863,734 (CA 53,5539h).**
(35K) Schjeide, O. A., Rivin, A. U., Yoshino, J., *Am. J. Clin. Pathol.* **39,** 329 (1963).
(36K) Searcy, R. L., Korotzer, J. L., Bergquist, L. M., *Clin. Chim. Acta* **8,** 148 (1963).
(37K) *Ibid.,* p. 376.
(38K) Skipski, V. P., Peterson, R. F., Barclay, M., *Biochem. J.* **90,** 374 (1964).
(39K) Sperry, W. M., *Std. Methods Clin. Chem.* **4,** 173 (1963).
(40K) Wheeldon, L. W., Brinley, M., Turner, D. A., *Anal. Biochem.* **4,** 433 (1962).

Enzymes

(1L) Allison, M. J., Gerszten, E., Sanchez, B., *Am. J. Clin. Pathol.* **40,** 451 (1963).
(2L) Amador, E., Dorfman, L. E., Wacker, W. E. C., *Clin. Chem.* **9,** 391 (1963).
(3L) Babson, A. L. (to Warner-Lambert Pharmaceutical Co.). U. S. Patent **3,096,251 (Cl. 167-84.5)** (Mar. 10, 1961).
(4L) Bell, R. L., *Am. J. Clin. Pathol.* **40,** 216 (1963).
(5L) Bowers, G. N., Jr., McComb, R. B., *Clin. Chem.* **10,** 636 (1964).
(6L) Bowers, G. N., Jr., *Std. Methods Clin. Chem.* **4,** 163 (1963).
(7L) Doizaki, W. M., Zieve, L., *J. Lab. Clin. Med.* **63,** 524 (1964).
(8L) Fairbanks, V. P., Beutler, E., *Blood* **20,** 591 (1962).
(9L) Feinstein, R. N., Howard, J. B., Ballonoff, L. B., Seaholm, J. E., *Anal. Biochem.* **8,** 277 (1964).
(10L) Fingerhut, B., Ferzpla, R., Poock, A., Marsh, W. H., *Clin. Chem.* **10,** 633 (1964).
(11L) Fleisher, G. A., Pankow, M., Warmka, C., *Clin. Chim. Acta* **9,** 254 (1964).
(12L) Guilbault, G. G., Kramer, D. N., *Anal. Chem.* **36,** 409 (1964).
(13L) *Ibid.,* p. 2497.
(14L) Guilbault, G. G., Kramer, D. N., Cannon, P. L., Jr., *Ibid.,* **36,** 606 (1964).
(15L) Guilbault, G. G., Tyson, B. C., Jr., Kramer, D. N., Cannon, P. L., Jr., *Ibid.,* **35,** 582 (1963).
(16L) Hanson, H., Bohley, P., Mannsfeldt, H. G., *Clin. Chim. Acta* **8,** 555 (1963).
(17L) Judd, R. E. H., Kwok, G., *Am. J. Med. Technol.* **30,** 87 (1964).
(18L) Kamaryt, J., Zazvorka, Z., *Clin. Chim. Acta* **9,** 559 (1964).
(19L) Kamaryt, J., Zazvorka, Z., *Sci. Tools LKB Instru. J.* **10,** 21 (1963).
(20L) Katz, S. A., ANAL. CHEM. **36,** 2500 (1964).
(21L) Kirk, J. E., *Clin. Chem.* **9,** 763 (1963).
(22L) *Ibid.,* p. 776.
(23L) Kramer, D. N., Guilbault, G. G., ANAL. CHEM. **35,** 588 (1963).
(24L) Kreutzer, H. H., Fennis, W. H. S., *Clin. Chim. Acta* **9,** 64 (1964).
(25L) Kucera, M., *Prumsyl Potravin* **15,** 297 (1964).
(26L) Lambert, J. L., Zitomer, F., ANAL. CHEM. **35,** 405 (1963).
(27L) Ludvigsen, B., *J. Lab. Clin. Med.* **61,** 329 (1963).
(28L) Martinek, R. G., *Clin. Chem.* **9,** 620 (1963).
(29L) McNair, R. D., *Ibid.,* **10,** 631 (1964).
(30L) Metge, W. R., Owen, C. A., Jr., Foulk, W. T., Hoffman, H. N., II., *J. Lab. Clin. Med.* **64,** 335 (1964).
(31L) Nachlas, M. M., Davidson, M. B., Goldberg, J. D., Seligman, A. M., *Ibid.,* **62,** 148 (1963).
(32L) Ng, W. G., Bergren, W. R., Donnell, G. N., *Clin. Chim. Acta* **10,** 337 (1964).
(33L) Nielsen, L., Ludvigsen, B., *J. Lab. Clin. Med.* **62,** 159 (1963).
(34L) Planta, R. J., Gruber, M., *Anal. Biochem.* **5,** 360 (1963).
(35L) Raabo, E., *Scandinav. J. Clin. Lab. Invest.* **15,** 405 (1963).
(36L) Ramaswamy, K., Radhakrishnan, A. N., *Clin. Chim. Acta* **10,** 271 (1964).
(37L) Reichard, H., *J. Lab. Clin. Med.* **63,** 1061 (1964).
(38L) Richterich, R., Schafroth, P., Aebi, H., *Clin. Chim. Acta* **8,** 178 (1963).
(39L) Ricketts, T. R., *Ibid.,* **8,** 160 (1963).
(40L) Ritz, N. D., Rubin, H., *J. Lab. Clin. Med.* **63,** 344 (1964).
(41L) Rosalki, S. B., *J. Clin. Pathol.* **15,** 566 (1962).
(42L) Roth, M., *Clin. Chim. Acta* **9,** 448 (1964).
(43L) Schwartz, M. K., Nisselbaum, J. S., Bodansky, O., *Am. J. Clin. Pathol.* **40,** 103 (1963).
(44L) Vogel, W. C., Zieve, L., *Clin. Chem.* **9,** 168 (1963).
(45L) Ware, A. G., Walberg, C. B., Sterling, R. E., *Std. Methods Clin. Chem.* **4,** 15 (1963).
(46L) Wetstone, H. J., Bowers, G. N., Jr., *Ibid.,* **4,** 47 (1963).

Function Tests

(1M) Adams, W. S., Davis, F. W., Hansen, L. E., ANAL. CHEM. **36,** 2209 (1964).
(2M) Allen, T. E., Jr., *Am. J. Med. Technol.* **29,** 395 (1963).
(3M) Aziz, M. A., Schwartz, S., Watson, C. J., *J. Lab. Clin. Med.* **63,** 585 (1964).
(4M) Baker, E. W., Lachman, M., Corwin, A. H., *Anal. Biochem.* **8,** 503 (1964).
(5M) Beeler, M. F., Veith, D. A., Morriss, R. H., Biskind, G. R., *Am. J. Clin. Pathol.* **41,** 553 (1964).
(6M) Beyermann, K., *Z. Anal. Chem.* **205,** 416 (1964).
(7M) Bilissis, P. K., Speer, R. J., *Clin. Chem.* **9,** 552 (1963).
(8M) Bodansky, O., *Bull. N. Y. Acad. Med.* **38,** 711 (1962).
(9M) Boutwell, J. H., Jr., *Clin. Chem.* **10,** 197 (1964).
(10M) Brodersen, R., Vind, I., *Scandinav. J. Clin. Lab. Invest.* **15,** 225 (1963).
(11M) Canturri, F. J. G., de Rivera, M. P. S., *Med. Seguridad Trabajo* **10,** 43 (1962).
(12M) Costa, A. L., Costa, F. D. V., *Lab. Diagnosi Med.* **8,** 79 (1963).
(13M) Ferro, P. V., Ham, A. B., *Techn. Bull. Reg. Med. Technol.* **33,** 111 (1963).
(14M) Garay, E. A. R., Argerich, T. C., *J. Lab. Clin. Med.* **62,** 141 (1963).
(15M) Gordon, B. A., Kuksis, A., Beveridge, J. M. R., *Can. J. Biochem. Physiol.* **41,** 77 (1963).
(16M) Gregory, C. H., *J. Lab. Clin. Med.* **61,** 917 (1963).
(17M) Henry, R. J., Fernandez, A. A., Berkman, S., *Clin. Chem.* **10,** 440 (1964).
(18M) Hoschek, R., *Aerztl. Lab.* **8,** 228 (1962).
(19M) Jacobs, S. L., Henry, R. J., Segalove, M., *Clin. Chem.* **10,** 433 (1964).
(20M) Jensen, J., *J. Chromatog.* **10,** 236 (1963).
(21M) Klein, B., *Std. Methods Clin. Chem.* **4,** 23 (1963).
(22M) Kritchevsky, D., Martak, D. S., Rothblat, G. H., *Anal. Biochem.* **5,** 388 (1963).
(23M) Larson, E. F., *Am. J. Clin. Pathol.* **39,** 90 (1963).
(24M) Martinek, R. G., Vimba, E., Mann, J. D., *Clin. Chem.* **10,** 781 (1964).
(25M) Mizuta, W., *Sogo Rinsho* **12,** 828 (1963).
(26M) Murata, T., Eto, S., Yamatsu, K., Sugiura, K., *Chem. Pharm. Bull (Tokyo)* **12,** 770 (1964).
(27M) Rand, R. N., di Pasqua, A., *Clin. Chem.* **8,** 570 (1962).
(28M) Reinhold, J. G., Young, J. H., Daitz, E., *Ibid.,* **8,** 475 (1962).
(29M) Rosenberg, A. A., Eimann, L. G., O'Leary, J., *Ibid.,* **9,** 450 (1963).
(30M) Schmidt, N. A., Scholtis, R. J. H., *Clin. Chim. Acta* **10,** 574 (1964).
(31M) Sjovall, J., *Methods Biochem. Anal.* **12,** 97 (1964).
(32M) Stevenson, G. W., Jacobs, S. L., Henry, R. J., *Clin. Chem.* **10,** 95 (1964).
(33M) Suecker, I., *Aerztl. Lab.* **9,** 260, 306 (1963).
(34M) Taddeini, L., Kay, I. T., Watson, C. J., *Clin. Chim. Acta* **7,** 890 (1962).
(35M) Weber, A. Ph., Schalm, L., Ruben, A. Th., *Ibid.,* **7,** 805 (1962).

Hemoglobin

(1N) Adams, E. C., Jr., Peterson, J. A., (to Miles Laboratories, Inc.) U. S. Patent **3,092,464 (Cl. 23-253)** (June 4, 1963) Appl. Nov. 2, 1959.
(2N) Awad, E., Cameron, B., Kotite, L., *Nature* **198,** 1201 (1963).
(3N) Bartlett, R. C., *Clin. Chem.* **9,** 325 (1963).
(4N) Baur, E. W., *J. Lab. Clin. Med.* **61,** 166 (1963).

(5N) Berman, M. C., Kench, J. E., *J. Clin. Pathol.* **16**, 385 (1963).
(6N) Elliott, W. B., Doebbler, G. F., *Nature* **198**, 690 (1963).
(7N) Graham, J. L., Grunbaum, B. W., *Am. J. Clin. Path.* **39**, 567 (1963).
(8N) Jones, R. T., Schroeder, W. A., *J. Chromatog.* **10**, 421 (1963).
(9N) Kelsey, J. R., Kloss, R. A., *Clin. Chem.* **10**, 424 (1964).
(10N) Lionetti, F. J., Valeri, C. R., Bond, J. C., Fortier, N. L., *J. Lab. Clin. Med.* **64**, 519 (1964).
(11N) Mickelsen, O., Woolard, H., Ness, A. T., *Clin. Chem.* **10**, 611 (1964).
(12N) Naiman, J. L., Gerald, P. S., *J. Lab. Clin. Med.* **61**, 508 (1963).
(13N) Nakamichi, M., Raymond, S., *Clin. Chem.* **9**, 135 (1963).
(14N) Roughton, F. J. W., *Ibid.*, **9**, 682 (1963).
(15N) Smith, T. B., *Am. J. Med. Technol.* **29**, 226 (1963).
(16N) Sunderman, F. W., Jr., *Am. J. Clin. Pathol.* **40**, 227 (1963).
(17N) Weatherburn, M. W., Logan, J. E., *Clin. Chim. Acta* **9**, 581 (1964).
(18N) Weinberg, M. S., *Am. J. Clin. Pathol.* **39**, 659 (1963).
(19N) Zade-Oppen, A. M. M., *Scandinav. J. Clin. Lab. Invest.* **15**, 491 (1963).

Metals

(1P) Ayres, G. H., Annand, R. R., ANAL. CHEM. **35**, 33 (1963).
(2P) Barber, A. A., Dempster, C., Anderson, N. G., *Clin. Chim. Acta* **8**, 143 (1963).
(3P) Barling, M. M., Banks, C. V., ANAL. CHEM. **36**, 2359 (1964).
(4P) Beale, R. N., Croft, D., *J. Clin. Path.* **17**, 260 (1964).
(5P) Berman, E., *Clin. Chem.* **9**, 459 (1963).
(6P) Bertrand, D., *Ann. Nutr. Aliment.* **18**, A1 (1964).
(7P) Billman, J. H., Chernin, T., ANAL. CHEM. **36**, 552 (1964).
(8P) Bingley, J. B., *J. Agr. Food Chem.* **11**, 130 (1963).
(9P) Caraway, W. T., *Clin. Chem.* **9**, 188 (1963).
(10P) Card, R. T., Brown, G. M., Valberg, L. S., *Can. Med. Assoc. J.* **90**, 618 (1964).
(11P) Daly, J. R., Anstall, H. B., *Clin. Chim. Acta* **9**, 576 (1964).
(12P) Elfers, L. A., Hallbach, P. F., Velten, R. J., ANAL. CHEM. **36**, 540 (1964).
(13P) Fischer, D. S., Price, D. C., *Clin. Chem.* **10**, 21 (1964).
(14P) Forman, D. T., *Am. J. Clin. Pathol.* **42**, 103 (1964).
(15P) Fritz, J. S., Garralda, B. B., ANAL. CHEM. **36**, 737 (1964).
(16P) Fukai, R., Meinke, W. W., *Limnol. Oceanog.* **7**, 186 (1962).
(17P) Fuwa, K., Pulido, P., McKay, R., Vallee, B. L., ANAL. CHEM. **36**, 2407 (1964).
(18P) Giorgio, A. J., Cartwright, G. E., Wintrobe, M. M., *Am. J. Clin. Pathol.* **41**, 22 (1964).
(19P) Giovanniello, T. J., Peters, T., Jr., *Std. Methods Clin. Chem.* **4**, 139 (1963).
(20P) Honegger, N., *Aerztl. Lab.* **9**, 41 (1963).
(21P) Kaiser, D. G., Meinke, W. W., *Anal. Biochem.* **6**, 77 (1963).
(22P) Karvanek, M., Karvankova, J., *Prumysl Potravin* **14**, 271 (1963).
(23P) Katz, J. H., Zoukis, M., Hart, W. L., Dern, R. J., *J. Lab. Clin. Med.* **63**, 885 (1964).
(24P) Kilshaw, D., *J. Med. Lab. Technol. (London)* **20**, 295 (1963).
(25P) Mikac-Devic, D., *Clin. Chim. Acta* **7**, 788 (1962).
(26P) Mukhedkar, A. J., Deshpande, N. V., ANAL. CHEM. **35**, 47 (1963).
(27P) Nadeau, G., Fortin, B., *Clin. Chem.* **10**, 862 (1964).
(28P) Natelson, S., Whitford, W. R., *Methods Biochem. Anal.* **12**, 1 (1964).
(29P) Nebel, M. L., Boltz, D. F., ANAL. CHEM. **36**, 144 (1964).
(30P) Nelson, C. V., *Am. J. Med. Tech.* **30**, 71 (1964).
(31P) Ramsay, W. N. M., Fulton, J. V., *Anal. Biochem.* **8**, 319 (1964).
(32P) Rice, E. W., *Std. Methods Clin. Chem.* **4**, 57 (1963).
(33P) *Ibid.*, p. 39.
(34P) Sanford, R., *J. Clin. Pathol.* **16**, 174 (1963).
(35P) Sekido, E., Fernando, Q., Freiser, H., ANAL. CHEM. **35**, 1550 (1963).
(36P) Simon, V. A., *Chemist-Analyst* **53**, 16 (1964).
(37P) Tipton, I. H., Cook, M. J., Steiner, R. L., Boye, C. A., Perry, H. M., Jr., Schroeder, H. A., *Health Phys.* **9**, 89 (1963).
(38P) van der Heyde, H., Henderickx, H., *Mededel. Landbouwhogeschool Opzoekingsstas Staat Gent* **26**, 687 (1961).
(39P) Voth, J. L., ANAL. CHEM. **35**, 1957 (1963).
(40P) Wallach, D. F. H., Steck, T. L., *Ibid.*, **35**, 1035 (1963).
(41P) Werthemann, A., *Pathol. Microbiol.* **26**, 319 (1963).

Nitrogen Compounds

(1Q) Ashraf, M., Illahi, M., Bhatty, M. K., Shash, R. A., *Pakistan J. Sci. Ind. Res.* **6**, 17 (1963).
(2Q) Bare, L. N., Wiseman, R. F., *Am. J. Clin. Pathol.* **40**, 556 (1963).
(3Q) Baylor, C., *Am. J. Med. Technol.* **29**, 9 (1963).
(4Q) Beutler, E., Duron, O., Kelly, B. M., *J. Lab. Clin. Med.* **61**, 882 (1963).
(5Q) Ceriotti, G., Spandrio, L., *Clin. Chim. Acta* **8**, 295 (1963).
(6Q) Chaney, A. L., U. S. Patent **3,119,751** (**Cl. 195-103.5**) (Jan. 28, 1964) Appl. May 8, 1962.
(7Q) Conn, H. O., Kuljian, A. A., *J. Lab. Clin. Med.* **63**, 1033 (1964).
(8Q) Coulombe, J. J., Favreau, L., *Clin. Chem.* **9**, 102 (1963).
(9Q) Davidson, B. E., Hird, F. J. R., *Biochem. J.* **93**, 232 (1964).
(10Q) Davis, J. R., Morris, R. N., *Anal. Biochem.* **5**, 64 (1963).
(11Q) de Jorge, F. B., de Ulhoa Cintra, A. B., *Rev. Paulista Med.* **62**, 289 (1963).
(12Q) Dienst, S. G., Morris, B., *J. Lab. Clin. Med.* **64**, 495 (1964).
(13Q) Gasanov, S. G., *Lab. Delo* **8**, 3 (1962).
(14Q) Grokholskaya, N. V., *Ibid.*, **9**, 37 (1963).
(15Q) Hartley, A. M., Asai, R. I., ANAL. CHEM. **35**, 1207 (1963).
(16Q) *Ibid.*, p. 1214.
(17Q) Howell, J. H., Boltz, D. F., *Ibid.*, **36**, 1799 (1964).
(18Q) Kachani, Z. F. Ch., *Aerztl. Lab.* **9**, 81 (1963).
(19Q) Kirsten, E., Gerez, C., Kirsten, R., *Biochem. Z.* **337**, 312 (1963).
(20Q) Konitzer, K., Voigt, S., *Z. Ges. Inn. Med. Ihre Grenzgebiete* **17**, 951 (1962).
(21Q) Mann, L. T., Jr., ANAL. CHEM. **35**, 2179 (1963).
(22Q) Marymont, J. H., Jr., London, M., *Clin. Chem.* **10**, 937 (1964).
(23Q) McCleskey, J. E., ANAL. CHEM. **36**, 1646 (1964).
(24Q) Miles Laboratories, Inc., British Patent **922,655** (Apr. 3, 1963) Appl. May 5, 1960.
(25Q) Miller, G. E., Rice, J. D., Jr., *Am. J. Clin. Pathol.* **39**, 97 (1963).
(26Q) Muftic, M. K., *Nature* **201**, 622 (1964).
(27Q) Pande, S. V., Bhan, A. K., Venkitasubramanian, T. A., *Anal. Biochem.* **8**, 446 (1964).
(28Q) Orloff, M. J., Stevens, C. O., *Clin. Chem.* **10**, 991 (1964).
(29Q) Prochazkova, L., ANAL. CHEM. **36**, 865 (1964).
(30Q) Rodkey, F. L., *Clin. Chem.* **10**, 606 (1964).
(31Q) Roijers, A. F. M., Tas, M. M., *Clin. Chim. Acta* **9**, 197 (1964).
(32Q) Simoes, M. S., Pereira, M. J. R., *Arquiv. Port. Bioquim.* **6**, 192 (1962–63).
(33Q) Tabuchi, S., Nishida, K., Nose, Y., Ueda, K., *Bitamin* **27**, 303 (1963).
(34Q) Wilder, C. E., *Clin. Chem.* **8**, 593 (1962).
(35Q) Yatzidis, H., Garidi, M., Vassilikos, C., Mayopoulou, D., Akilas, A., *J. Clin. Path.* **17**, 163 (1964).

Hormones

(1R) Aguayo, S. E., *Clin. Chem.* **10**, 637 (1964).
(2R) Ashcroft, G. W., Crawford, T. B. B., Binns, J. K., MacDougall, E. J., *Clin. Chim. Acta* **9**, 364 (1964).
(3R) Brunjes, S., Wybenga, D., *Clin. Chem.* **9**, 626, (1963).
(4R) Brunjes, S., Wybenga, D., Johns, V. J., Jr., *Ibid.*, **10**, 1 (1964).
(5R) Burke, G., Metzger, B. E., Goldstein, M. S., *J. Lab. Clin. Med.* **63**, 708 (1964).
(6R) Callingham, B. A., Cass, R., *West-European Symp. Clin. Chem.* **2**, 19 (1963).
(7R) Campbell, D. J., Sherbaniuk, R., Rigby, J., *Clin. Chem.* **10**, 447 (1964).
(8R) Chakrabarti, S. G., White, D., *Can. J. Med. Technol.* **26**, 111 (1964).
(9R) Comar, D., Blanquet, P., Riviere, R., Kellershohn, C., *Ann. Fac. Sci. Univ. Clermont Phys.* **5**, 23 (1963).
(10R) Davis, R. B., *Clin. Chem.* **8**, 660 (1962).
(11R) Davis, V. E., Huff, J. A., Brown, H., Alfrey, C. P., *Clin. Chim. Acta* **9**, 419 (1964).
(12R) DeQuattro, V., Wybenga, D., von Studnitz, W., Brunjes, S., *J. Lab. Clin. Med.* **63**, 864 (1964).
(13R) DiGiulio, W., Michalak, Z., Weinhold, P. A., Hamilton, J. R., Thoma, G. E., *Ibid.*, **64**, 349 (1964).
(14R) Eichhorn, F., Rutenberg, A., *Clin. Chem.* **9**, 615 (1963).
(15R) Fellman, J. H., Severson, L. J., Robinson, E. H., Fujita, T. S., *Am. J. Clin. Pathol.* **38**, 651 (1962).
(16R) Foss, O. P., *Std. Methods Clin. Chem.* **4**, 125 (1963).
(17R) Georges, R. J., *Clin. Chim. Acta* **10**, 583 (1964).
(18R) Graham, V., *Am. J. Med. Technol.* **30**, 251 (1964).
(19R) Hamilton, C. H., U. S. Patent **3,055,742** (**Cl. 23-230**) (Sept. 25, 1962), Appl. Jan. 28, 1959.
(20R) Hermann, G. A., *Am. J. Clin. Pathol.* **41**, 373 (1964).
(21R) Klotz, M. O., Richter, H., Meuffels, M., *Clin. Chem.* **10**, 372 (1964).
(22R) La Roche, G., Oakes, C., Blanquet, P., *U. S. At. Energy Comm. UCRL-***10211**, 160–7 (1962).
(23R) Lee, N. D., Henry, R. J., Golub, O. J., *J. Clin. Endocrin. Metab.* **24**, 486 (1964).
(24R) Lee, N. D., Pileggi, V. J., Segalove, M., *Clin. Chem.* **10**, 136 (1964).
(25R) Leffler, H. H., McDougald, C. H., *Am. J. Clin. Pathol.* **41**, 95 (1964).
(26R) Lieb, H., *Farmaco (Pavia), Ed. Sci.* **19**, 286 (1964).

(27R) Lissitzky, S., Bismuth, J., *Pathol. Biol., Semaine Hop.* **11**, 653 (1963).

(28R) Lowenstein, J. M., Greenspan, F. S., Spilker, P., *J. Lab. Clin. Med.* **62**, 831 (1963).

(29R) Marshall, C. S., *Biochim. Biophys. Acta* **74**, 158 (1963).

(30R) Mitchell, W. D., *Clin. Chem. Acta* **10**, 96 (1964).

(31R) Pileggi, V. J., Segal, H. A., Lanchatin, G. F., *Ibid.*, **8**, 547 (1963).

(32R) Pitkanen, E., Airaksinen, M. M., Mustala, O. O., Paloheimo, J., *Scand. J. Clin. & Lab. Inv.* **14**, 571 (1962).

(33R) Robbins, J., "Evaluation Thyroid Parathyroid Functions," *Proc. Appl. Seminar Assoc. Clin. Scientists, 3rd,* Washington, D. C., 1961, 95 (pub. 1963).

(34R) Rosano, C. L., *Clin. Chem.* **10**, 673 (1964).

(35R) Routh, J. I., *Proc. Iowa Acad. Sci.* **70**, 178 (1963).

(36R) Ruthven, C. R., Jr., Sandler, M., *Anal. Biochem.* **8**, 282 (1964).

(37R) Shtolts, V., *Probl. Endokrinol. i Gormonoterap.* **9**, 56 (1963).

(38R) Sprince, H., Parker, C., Dawson, J. T., Jr., Jameson, D., Dohan, F. C., *J. Chromatog.* **8**, 457 (1962).

(39R) Stolc, V., Knopp, J., *Mikrochim Ichanoanal. Acta* **5**, 941 (1963).

(40R) Sunderman, F. W., Jr., "Evaluation Thyroid Parathyroid Functions," *Proc. Appl. Seminar Assoc. Clin. Scientists, 3rd.* Washington, D. C., 1961, 53 (pub. 1963).

(41R) Tamura, H., Kondo, Y., *Seikagaku* **35**, 278 (1963).

(42R) Vanable, J. W., Jr., *Anal. Biochem.* **6**, 393 (1963).

(43R) Weil-Malherbe, H., *Ibid.*, **7**, 484 (1964).

(44R) Weissbach, H., *Std. Methods Clin. Chem.* **4**, 121 (1963).

(45R) *Ibid.*, p. 197.

(46R) Yalow, R. S., Berson, S. A., *Methods Biochem. Anal.* **12**, 69 (1964).

Organic Acids

(1T) Barness, L. A., Mellman, W. J., Tedesco, T., Young, D. G., Nocho, R., *Clin. Chem.* **9**, 600 (1963).

(2T) Berry, S. A., Campbell, J. N., *Anal. Biochem.* **8**, 495 (1964).

(3T) Cabezas, J. A., Porto, J. V., *Clin. Chem.* **10**, 986 (1964).

(4T) Caraway, W. T., *Std. Methods Clin. Chem.* **4**, 239 (1963).

(5T) Chambon, P., *Ann. Pharm. Franc.* **21**, 613 (1963).

(6T) Hoffman, N. E., Barboriak, J. J., Hardman, H. F., *Anal. Biochem.* **9**, 175 (1964).

(7T) Jacobs, S. L., Lee, N. D., *J. Nucl. Med.* **5**, 297 (1964).

(8T) Kesner, L., Muntwyler, E., *J. Lab. Clin. Med.* **61**, 604 (1963).

(9T) Landon, J., Fawcett, J. K., Wynn, V., *J. Clin. Path.* **15**, 579 (1962).

(10T) Lavender, A. R., Pullman, T. N., Goldman, D., *J. Lab. Clin. Med.* **63**, 299 (1964).

(11T) Legrand, J. C., Gonnard, P., Grupper, C., *Clin. Chim. Acta* **8**, 26 (1963).

(12T) Lester, D., ANAL. CHEM. **36**, 1810 (1964).

(13T) Matulis, R. M., Guyon, J. C., *Ibid.*, **36** 118 (1964).

(14T) Mayer, G. G., Markow, D., Karp, F., *Clin. Chem.* **9**, 334 (1963).

(15T) Meichen, F. W., Short, A. W., *Intern. J. Radiation Biol.* **6**, 495 (1963).

(16T) Millican, R. C., *Anal. Biochem.* **6**, 181 (1963).

(17T) Nir, I., *Ibid.*, **8**, 20 (1964).

(18T) Qureshi, M., Husain, W., Rawat, J. P., ANAL. CHEM. **35**, 1592 (1963).

(19T) Ramsey, H. A., *J. Dairy Sci.* **46**, 480 (1963).

(20T) Riddle, V. M., ANAL. CHEM. **35**, 853 (1963).

(21T) Rink, M., Hermann, S., *J. Chromatog.* **12**, 249 (1963).

(22T) Rosan, R. C., Nieland, M. L., *Anal. Biochem.* **6**, 125 (1963).

(23T) Rosenbloom, F. M., Seegmiller, J. E., *J. Lab. Clin. Med.* **63**, 492 (1964).

(24T) Schmidt, G. C., Fischer, C., McOwen, J. M., *J. Pharm. Sci.* **52**, 468 (1963).

(25T) Shinriki, N., Nambara, T., *Yakugaku Zasshi* **83**, 322 (1963).

(26T) Umberger, C. J., Fiorese, F. F., *Clin. Chem.* **9**, 91 (1963).

(27T) Williams, C. M., Leonard, R. H., *Anal. Biochem.* **5**, 362 (1963).

(28T) Wolosowicz, N., Niewiarowski, S., Czerepko, K., *Thromb. Diath. Haemorrhag.* **10**, 309 (1964).

(29T) Woodring, M. J., Fisher, D. H., Storvick, C. A., *Clin. Chem.* **10**, 479 (1964).

(30T) Zak, B., Weiner, L. M., *Anal. Biochem.* **8**, 349 (1964).

Organic Compounds

(1U) Cooper, J. R., *Biochem. Pharmacol.* **13**, 795 (1964).

(2U) Durkee, A. B., Sirois, J. C., *J. Chromatog.* **13**, 173 (1964).

(3U) Fischl, J., Rabiah, S., *Clin. Chem.* **10**, 281 (1964).

(4U) Guilbault, G. G., ANAL. CHEM. **35**, 828 (1963).

(5U) Harada, T., *Biochim. Biophys. Acta* **63**, 334 (1962).

(6U) Hashmi, M. H., Ayaz, A. A., Shmad, H., ANAL. CHEM. **36**, 2029 (1964).

(7U) Kupfer, D., Atkinson, D. E., *Anal. Biochem.* **8**, 82 (1964).

(8U) Luke, H. H., Freeman, T. E., Kier, L. B., ANAL. CHEM. **35**, 1916 (1963).

(9U) Madonia, J. P., *Am. J. Clin. Pathol.* **39**, 206 (1963).

(10U) Mortensen, E., *Scand. J. Clin. & Lab. Investig.* **16**, 87 (1964).

(11U) Peden, V. H., *J. Lab. Clin. Med.* **63**, 332 (1964).

(12U) Selleck, B., Cohen, J. J., Randall, H. M., Jr., *Anal. Biochem.* **7**, 178 (1964).

(13U) Tanayama, S., Ui, M., *Chem. Pharm. Bull. (Tokyo)* **11**, 835 (1963).

Proteins

(1V) Acharya, U. S. V., Swaminathan, M., Srenivasan, A., Subrahmanyan, V., *Indian J. Med. Res.* **52**, 224 (1964).

(2V) Afonso, E., *Clin. Chim. Acta* **10**, 114 (1964).

(3V) Bartlett, R. C., *Clin. Chem.* **9**, 317 (1963).

(4V) Betsuyaku, T., Yachi, A., Fukuda, M., Anzai, T., Wada, T., Carpenter, C. M., *Proc. Soc. Exptl. Biol. Med.* **115**, 267 (1964).

(5V) Botvinik, M. M., Troshko, E. V., *Zh. Obshch. Khim.* **33**, 3813 (1963).

(6V) Chiaraviglio, E. D. C., Wolf, A. V., Prentiss, P. G., *Am. J. Clin. Pathol.* **39**, 42 (1963).

(7V) Collens, R., Meyers, H., Lange, K., *Clin. Chem.* **9**, 330 (1963).

(8V) Cupic, M., Topalovic-Avramov, R., *Arhiv. Farm.* **13**, 29 (1963).

(9V) Curtain, C., *Nature* **203**, 1380 (1964).

(10V) Curtain, C. C., *Anal. Biochem.* **6**, 512 (1963).

(11V) Dawson, D., Zajtchuk, R., Allen, J. G., *Clin. Chem.* **10**, 793 (1964).

(12V) de St. Groth, S., Webster, R. G., Datyner, A., *Biochim. Biophys. Acta* **71**, 377 (1963).

(13V) Fischl, J., Gabor, J., *Clin. Chim. Acta* **8**, 330 (1963).

(14V) Foreman, J. A., Douglas, G. L., Bergquist, L. M., Searcy, R. L., *Clin. Chem.* **9**, 456 (1963).

(15V) Glass, R. H., Risinger, C., Wide, L., Gemzell, C. A., *Scandinav. J. Clin. Lab. Invest.* **15**, 266 (1963).

(16V) Goodwin, J. F., *Clin. Chem.* **9**, 455 (1963).

(17V) Grasbeck, R., Karlsson, R., *Acta Chem. Scand.* **17**, 1 (1963).

(18V) Hase, T., *Clin. Chem.* **10**, 62 (1964).

(19V) Hochwald, G. M., Thorbecke, G. J., *Clin. Chim. Acta* **8**, 678 (1963).

(20V) Inchiosa, M. A., Jr., *J. Lab. Clin. Med.* **63**, 319 (1964).

(21V) Kaplan, A., Johnstone, M. A., *Clin. Chem.* **9**, 505 (1963).

(22V) Bailey, J. L., "Techniques in Protein Chemistry," Elsevier Pub. Co., Amsterdam-London-New York (1962).

(23V) Lloyd, H. M., Meares, J. D., *Clin. Chim. Acta* **9**, 192 (1964).

(24V) London, M., Marymont, J. H., Jr., Field, J., *Clin. Chem.* **10**, 789 (1964).

(25V) Lorber, A., *J. Lab. Clin. Med.* **64**, 133 (1964).

(26V) Lundh, B., *J. Clin. Lab. Invest.* **16**, 108 (1964).

(27V) McFarlane, H., *Clin. Chim. Acta* **9**, 376 (1964).

(28V) Minari, O., Zilversmit, D. B., *Anal. Biochem.* **6**, 320 (1963).

(29V) Morris, C. J. O. R., *Protides Biol. Fluids, Proc. Colloq.* **10**, 325 (1962) (pub. 1963).

(30V) Patrick, R. L., Thiers, R. E., *Clin. Chem.* **9**, 283 (1963).

(31V) Peetoom, F., Gerald, P. S., *Clin. Chim. Acta* **10**, 375 (1964).

(32V) Ratcliff, P., Soothill, J. F., Stanworth, D. R., *Ibid.*, **8**, 91 (1963).

(33V) Reynafarje, B., *J. Lab. Clin. Med.* **61**, 138 (1963).

(34V) Rodkey, F. L., *Clin. Chem.* **10**, 643 (1964).

(35V) Roskes, S. D., Thompson, T. E., *Clin. Chim. Acta* **8**, 489 (1963).

(36V) Saifer, A., *J. Lab. Clin. Med.* **63**, 1054 (1964).

(37V) Sakamoto, M., Takahira, H., *Kotsu Igaku* **17**, 136 (1963).

(38V) Slutskii, L. I., *Klinich. Biokhim., Rizhsk. Med. Inst.* **1962** 141.

(39V) Spiro, R. G., *N. E. J. Med.* **269**, (11, 12) 566–616 (1963).

(40V) Stanley, P. G., *Nature* **197**, 1108 (1963).

(41V) Steel, A. E., *Clin. Chim. Acta* **8**, 86 (1963).

(42V) Sunderman, F. W., Jr., *Am. J. Clin. Pathol.* **42**, 1 (1964).

(43V) Sunderman, F. W., Sunderman, F. W., Jr., "Serum Proteins and the Dysproteinemias," Lippincott, Philadelphia, Pa., 1964.

(44V) van de Loo, J., *Protides Biol. Fluids, Proc. Colloq.* **10**, 335 (1962).

(45V) Wachter, H., *Z. Physiol. Chem.* **333**, 256 (1963).

(46V) Watson, D., *Clin. Chem.* **10**, 412 (1964).

Sterols

(1W) Antonini, F. M., Tinti, P., Scolari, L., *Giorn. Arteriosclerosi* **1**, 119 (1963).

(2W) Bang, H. O., *J. Chromatog.* **14**, 520 (1964).

(3W) Barlow J. J., *Anal. Biochem.* **6**, 435 (1963).

(4W) Borushek, S., Gold, J. J., *Clin. Chem.* **10**, 41 (1964).

(5W) Brown, W. R., *Ibid.*, **9**, 468 (1963).

(6W) Cawley, L. P., Musser, B. O., Campbell, S., Faucette, W., *Am. J. Clin. Pathol.* **39**, 450 (1963).

(7W) Chamberlain, J., Knights, B. A., Thomas, G. H., *J. Endocrin.* **26**, 367 (1963).

(8W) Claude, J. R., *Pathol. Biol. Semaine Hop.* **10**, 1599 (1962).

(9W) Copinschi, G., Cornil, A., Franckson, J. R. M., *Clin. Chim. Acta* **7**, 817 (1962).

(10W) Copinschi, G., Cornil, A., Franckson, J. R. M., *Ibid.*, **8**, 749 (1963).

(11W) Crepy, O., Lachese, B., Fermanian, J., Jayle, M. F., *Ibid.*, **8**, 227 (1963).

(12W) Eberlein, W. R., *J. Clin. Endocrinol. Metab.* **23**, 990 (1963).

(13W) Epstein, E., Zak, B., *Ibid.*, **23**, 355 (1963).

(14W) Epstein, E., Zak, B., *Clin. Chem.* **9**, 70 (1963).

(15W) Grande, F., Amatuzio, D. S., Wada, S., *Ibid.*, **10**, 619 (1964).

(16W) Haust, H. L., Beveridge, J. M. R., *Can. J. Biochem.* **42**, 251 (1964).

(17W) Hernandez, R., Jr., Axelrod, L. R., ANAL. CHEM. **35**, 80 (1963).

(18W) Hobkirk, R., Metcalfe-Gibson, A., *Std. Methods Clin. Chem.* **4**, 65 (1963).

(19W) Horning, E. C., Van den Heuvel, W. J. A., Creech, B. G., *Methods Biochem. Anal.* **11**, 69 (1963).

(20W) Huang, T. C., Wefler, V., Raftery, A., ANAL. CHEM. **35**, 1757 (1963).

(21W) Jacobsohn, G. M., *Ibid.*, **36**, 2030 (1964).

(22W) James, V. H. T., Caie, E., *J. Clin. Enocr. Metab.* **24**, 180 (1964).

(23W) Jamieson, A., *Clin. Chim. Acta* **10**, 530 (1964).

(24W) Jansen, A. P., *Ibid.*, **8**, 785 (1963).

(25W) Kornel, L., *Anal. Biochem.* **7**, 345 (1964).

(26W) Kritchevsky, D., Tepper, S. A., Alaupovic, P., Furman, R. H., *Proc. Soc. Exptl. Biol. Med.* **112**, 259 (1963).

(27W) Kritchevsky, D., Tepper, S. A., Shapiro, I. L., *J. Lab. Clin. Med.* **63** 511 (1964).

(28W) Kroman, H. S., Bender, S. R., Capizzi, R. L., *Clin. Chim. Acta* **9**, 73 (1964).

(29W) Lachese, B., Crepy, O., Jayle, M. F., *Ibid.*, **8**, 538 (1963).

(30W) Landowne, R. A., Lipsky, S. R., ANAL. CHEM. **35**, 532 (1963).

(31W) Lim, N. Y., Fesler, K. W., Dingman, J. F., *J. Clin. Endocrinol. Metab.* **24**, 68 (1964).

(32W) Liu, J. H. C., *Clin. Chem.* **10**, 103 (1964).

(33W) Moore, R. V., Boyle, E., Jr., *Ibid.*, **9**, 156 (1963).

(34W) Morris, L. J., *J. Lipid Res.* **4**, 357 (1963).

(35W) Mose, T., Ueda, Y., Yamamoto, K., Masumura, T., Ohta, K., ANAL. CHEM. **35**, 1751 (1963).

(36W) Murphy, B. E. P., Pattee, C. J., Inglis, E., Sun, W. C., *J. Clin. Endocr. Metab.* **24**, 919 (1964).

(37W) Nair, P. P., Sarlos, I. J., Solomon, D., Turner, D. A., *Anal. Biochem.* **7**, 96 (1964).

(38W) Ness, A. T., Pastewka, J. V., Peacock, A. C., *Clin. Chim. Acta* **10**, 229 (1964).

(39W) Oertel, G. W., *Ibid.*, **8**, 154 (1963).

(40W) Peterson, R. E., *Std. Methods Clin. Chem.* **4**, 151 (1963).

(41W) Pottier, J., Busigny, M., *Clin. Chim. Acta* **10**, 406 (1964).

(42W) Richterich, R., Dauber, K., *Klin. Wochschr.* **40**, 1252 (1962).

(43W) Rutherford, E. R., Nelson, D. H., *J. Clin. Endocrinol. Metab.* **23**, 533 (1963).

(44W) Sahagian, B., Levine, V. E., *Clin. Chem.* **10**, 116 (1964).

(45W) Schulz, E. P., Diaz, M. A., Lopez, G., Guerrero, L. M., Barrera, H., Pereda, A. L., Aguilera, A., ANAL. CHEM. **36**, 1624 (1964).

(46W) Schumacher, R. D., *Experientia* **19**, 84 (1963).

(47W) Shin, Y. S., *Anal. Biochem.* **5**, 369 (1963).

(48W) Silber, R. H., *Std. Methods Clin. Chem.* **4**, 113 (1963).

(49W) Sparagana, M., Keutmann, E. H., Mason, W. B., *Am. J. Med. Technol.* **29**, 1231 (1963).

(50W) Starka, L., Sulcova, J., Riedlova, J., Adamec, O., *Clin. Chim. Acta* **9**, 168 (1964).

(51W) Steenburg, R. W., Thomasson, B. H., *J. Clin. Endocrinol. Metab.* **24**, 875 (1964).

(52W) Thomas, B. S., Bulbrook, R. D., *J. Chromatog.* **14**, 28 (1964).

(53W) Vanzetti, G., *Clin. Chim. Acta* **10**, 389 (1964).

(54W) Webster, D., *Ibid.*, **8**, 19 (1963).

(55W) *Ibid.*, p. 731.

(56W) Wilson, H., Lipsett, M. B., *Anal. Biochem.* **5**, 217 (1963).

(57W) Wotiz, H. H., Chattoraj, S. C., ANAL. CHEM. **36**, 1466 (1964).

(58W) Wrigley, D. W., *J. Med. Lab. Technol.* **21**, 129 (1964).

(59W) Zurkowski, P., *Clin. Chem.* **10**, 451 (1964).

Toxicology

(1X) Aldous, J. G., Hall, R. J., Sapp, P., ANAL. CHEM. **36**, 335 (1964).

(2X) Allaway, W. H., Cary, E. E., *Ibid.*, **36**, 1359 (1964).

(3X) Amenta, J. S., *Std. Methods Clin. Chem.* **4**, 31 (1963).

(4X) Andersen, N. R., Hercules, D. M., ANAL. CHEM. **36**, 2138 (1964).

(5X) Ariel, M., Bach, D., *Analyst* **88**, 30 (1963).

(6X) Ayres, G. H., McCrory, R. W., ANAL. CHEM. **36**, 133 (1964).

(7X) Brochmann-Hanssen, E., Svendsen, A. B., *J. Pharm. Sci.* **51**, 1095 (1962).

(8X) Brooks, E. J., Gates, O. R., Nottingham, M., Jr., *Am. J. Clin. Pathol.* **41**, 154 (1964).

(9X) Buettner, W., Schuelke, S., Soyka, S., *Deut. Zahnaerztl. Z.* **18**, 24 (1963).

(10X) Clavell, L. A., *Rev. Fac. Farm. Univ. Central Venezuela* **5**, 40 (1964).

(11X) Coburn, R. F., Danielson, G. K., Blakemore, W. S., Forster, R. E. II, *J. Appl. Physiol.* **19**, 510 (1964).

(12X) Cochin, J., Daly, J. W., *J. Pharmacol. Exptl. Therap.* **139**, 154 (1963).

(13X) Crippen, R. C., Freimuth, H. C., ANAL. CHEM. **36**, 273 (1964).

(14X) Cummins, L. M., Martin, J. L., Maag, G. W., Maag, D. D., *Ibid.*, **36**, 382 (1964).

(15X) Dadlez, J., Wojciak, Z., *Bull. Soc. Amis Sci. Lettres Poznan Ser. C No.* **12**, 87 (1962).

(16X) de Oliveira-Menditsch, J., *Chem. Anal.* **53**, 102 (1964).

(17X) Dubois, L., Teichman, T., Monkman, J. L., *Clin. Chem.* **9**, 454 (1963).

(18X) Duvivier, J., Versie, R., Bonnard, J., Noirfalise, A., *Clin. Chim. Acta* **9**, 454 (1964).

(19X) Frahm, M., Gottesleben, A., Soehring, Kl., *Pharm. Acta Helv.* **38**, 785 (1963).

(20X) Fukuyama, T., Yamada, M., *Koshu Eiseiin Kenkyu Hokoku* **11**, 56 (1962).

(21X) Gabriel, K. L., Dubin, S., *J. Am. Vet. Med. Assoc.* **143**, 722 (1963).

(22X) Guzak, R., Caraway, W. T., *Am. J. Med. Technol.* **29**, 231 (1963).

(23X) Hanke, M. E., *Clin. Chem.* **10**, 647 (1964).

(24X) Hessell, H. W., Modglin, F. R., *J. Forensic Sci.* **9**, 255 (1964).

(25X) Hindberg, J., Wieth, J. O., *J. Lab. Clin. Med.* **61**, 355 (1963).

(26X) Holbrook, W. B., Rein, J. E., ANAL. CHEM. **36**, 2451 (1964).

(27X) Ilcewicz, F. H., Holtzman, R. B., Lucas, H. F., Jr., *Ibid.*, **36**, 1132 (1964).

(28X) Jain, N. C., Fontan, C. R., Kirk, P. L., *Microchem. J.* **8**, 28 (1964).

(29X) Kazyak, L., Knoblock, E. C., ANAL. CHEM. **35**, 1448 (1963).

(30X) Kelleher, J., *Clin. Chim. Acta* **9**, 92 (1964).

(31X) Kirkbright, G. F., Yoe, J. H., ANAL. CHEM. **35**, 808 (1963).

(32X) Kuhn, H. F., Friebel, H., *Arch. Pharm.* **296**, 232 (1963).

(33X) Lehmann, J., Karamustafaoglu, V., *Scandinav. J. Clin. Lab. Invest.* **14**, 554 (1962).

(34X) Leithoff, H., Chan, S. Y., *Med. Welt* **38**, 2011 (1964).

(35X) Lott, P. F., Cukor, P., Moriber, G., Solga, J., ANAL. CHEM. **35**, 1159 (1963).

(36X) Lyons, H., Bard, J., *Clin. Chem.* **10**, 429 (1964).

(37X) Malmstadt, H. V., Hadjiioannou, T. P., ANAL. CHEM. **34**, 455 (1962).

(38X) Mantel, M., *Ibid.*, **36**, 936 (1964).

(39X) Mantel, M., Molco, M., Stiller, M., *Ibid.*, **35**, 1737 (1963).

(40X) Matusiak, W., Cefola, M., Cortivo, L. D., *Anal. Biochem.* **8**, 463 (1964).

(41X) McCord, W. M., Gadsden, R. H., *J. Gas Chromatog.* **2**, 38 (1964).

(42X) Merkus, F. W. H. M., *Pharm. Weekblad* **98**, 947 (1963).

(43X) Miller, G. W., Long, L. E., George, G. M., Sikes, W. L., ANAL. CHEM. **36**, 980 (1964).

(44X) Mills, A. L., *Ibid.*, **35**, 416 (1963).

(45X) Mohilner, P. R., *Ibid.*, **35**, 1103 (1963).

(46X) Moore, F. L., *Ibid.*, **35**, 1032 (1963).

(47X) Mule, S. J., *Ibid.*, **36**, 1907 (1964).

(48X) Natelson, S., Sheid, B., Leighton, D. R., *Clin. Chem.* **8**, 630 (1962).

(49X) Natelson, S., De, P. K., *Microchem. J.* **7**, 448 (1963).

(50X) Parker, K. D., Fontan, C. R., Kirk, P. L., ANAL. CHEM. **35**, 356 (1963).

(51X) Petxold, J. A., Camp, W. J. R., *Science* **52**, 1106 (1963).

(52X) Powell, F. J. N., Ellam, B., *Clin. Chim. Acta* **10**, 472 (1964).

(53X) Rakovic, M., *Atompraxis* **9**, 177 (1963).

(54X) Rozenberg, P. A., *Lab. Delo* **9**, 11 (1963).

(55X) Sanz-Pedrero, P., Hermoso, M. D., *Med. Seguridad Trabajo* **11**, 28 (1963).

(56X) Savel, P., *Ann. Pharm. Franc.* **21**, 303 (1963).

(57X) Scoggins, M. W., ANAL. CHEM. **36**, 1152 (1964).

(58X) Sereda, G. A., Vorontsova, A. S., *Gigiena Truda i Prof. Zabolevaniya* **8**, 55 (1964).

(59X) Sill, C. W., Willis, C. P., ANAL. CHEM. **36**, 622 (1964).

(60X) Sjostrand, B., *Ibid.*, **36**, 814 (1964).

(61X) Smith, H., *Ibid.*, **35**, 635 (1963).

(62X) Solvsten, S., *Scandinav. J. Clin. Lab. Invest.* **16**, 39 (1964).

(63X) Sommer, J., *Chem. Tech.* **15**, 38 (1963).

(64X) Stewart, R., Evans, D. G., ANAL. CHEM. **35**, 1315 (1963).

(65X) Street, H. V., McMartin, C., *Nature* **199**, 456 (1963).

(66X) Sunderman, F. W., Jr., *Am. J. Clin. Pathol.* **39**, 549 (1963).

(67X) Sunshine, I., Rose, E., LeBeau, J., *Clin. Chem.* **9**, 312 (1963).

(68X) Svensmark, O., Kristensen, P., *J. Lab. Clin. Med.* **61**, 501 (1963).

(69X) Swagzdis, J. E., Flanagan, T. L., *Anal. Biochem.* **7**, 147 (1964).
(70X) Terao, K., *Kagaku Keisatsu Kenkyusho Hokoku* **15**, 311 (1962).
(71X) Tertian, R., Fagot, C., Thiele, B., *Compt. Rend.* **258**, 4769 (1964).
(72X) Trudell, L., Boltz, D. F., ANAL. CHEM. **35**, 2122 (1963).
(73X) Vignoli, L., Badre, R., Morel, M. C., Ardorino, J., *Chim. Anal.* **45**, 53 (1963).
(74X) White, W. W., Zuber, J. R., ANAL. CHEM. **36**, 2363 (1964).
(75X) Wilson, W. J., Jr., Hausman, R., *J. Lab. Clin. Med.* **64**, 154 (1964).
(76X) Winefordner, J. D., Latz, H. W., ANAL. CHEM. **35**, 1517 (1963).
(77X) Winefordner, J. D., Staab, R. A., *Ibid.*, **36**, 165 (1964).
(78X) Wojciak, Z., *Bull. Soc. Amis Sci. Lettres Poznan Ser. C* **12**, 75 (1962).
(79X) *Ibid.*, p. 87.
(80X) Yablochkin, V. D., *Farmatsevt. Zh. (Kiev)* **18**, 68 (1963).
(81X) Zitomer, F., Lamber, J. L., ANAL. CHEM. **35**, 1731 (1963).

Vitamins

(1Y) Dugan, R. E., Frigerio, N. A., Siebert, J. M., ANAL. CHEM. **36**, 114 (1964).
(2Y) Erickson, D. R., Dunkley, W. L., *Ibid.*, **36**, 1055 (1964).
(3Y) Hall, C. A., Allen, E. S., *J. Lab. Clin. Med.* **63**, 517 (1964).
(4Y) Huber, C. O., Stapelfeldt, H. E., ANAL. CHEM. **36**, 315 (1964).
(5Y) Hughes, R. E., *Analyst* **89**, 618 (1964).
(6Y) Katsui, G., Nishimoto, Y., *Bitamin* **27**, 279 (1963).
(7Y) Kemper, A., *Nahrung* **7**, 449 (1963).
(8Y) Kum-Tatt, L., Leong, P. C., *Clin. Chem.* **10**, 575 (1964).
(9Y) Kuznetsova, L. M., Vendt, V. P., *Ukr. Biokhim. Zh.* **34**, 608 (1962).
(10Y) Lichtenthalter, H. K., *J. Chromatog.* **13**, 166 (1964).
(11Y) Nair, K. K. G., Tewari, C. P., Krishnan, P. S., *Indian J. Chem.* **1**, 131 (1963).
(12Y) Neeld, J. B., Jr., Pearson, W. N., *J. Nutr.* **79**, 454 (1963).
(13Y) Nelp, W. B., McAfee, J. G., Wagner, H. N., Jr., *J. Lab. Clin. Med.* **61**, 158 (1963).
(14Y) Rindi, G., Perri, V., *Intern. Z. Vitaminforsch.* **32**, 398 (1962).
(15Y) Shul'gin, I. A., Podol'nyi, V. Z., Sokolova, S. V., *Fiziol. Rast.* **10**, 383 (1963).
(16Y) Uchino, H., Schwartz, G. H., Glass, G. B. J., *Clin. Chim. Acta* **9**, 461 (1964).

Coatings

M. H. Swann, M. L. Adams, and G. G. Esposito

Coating and Chemical Laboratory, Aberdeen Proving Ground, Md.

THE IMPORTANT CONTRIBUTIONS to the analysis of coating materials as selected by the reviewers since the previous summary (*137*) are contained in this biennial review. The period covered extends from November 1962 through December 1964, although a few significant foreign publications, located by abstracts, may predate this coverage. In this attempt to be selective, it is hoped that valuable publications have not been omitted. Other similar reviews were made within this period (*102–104, 106*) in addition to some studies of special subjects. The annual reviews of analytical methods for the examination of oils and fatty acids (*81, 82*) are very comprehensive. Firestone (*35*) reviewed available methods for analyzing polymerized fats and oils and James (*57*) prepared a comprehensive review of analytical procedures for separating naturally occurring fatty acids including *cis*- and *trans*-isomers.

The fifth edition of "Standard Methods for the Analysis of Oils, Fats and Soaps" (*133*) has been published and includes a number of physical and chemical test methods not found in the 10-year old fourth edition. Over one-half of the 122 pages of "Progress in the Chemistry of Fats and Other Lipids" (*52*) are devoted to the applications of gas chromatography in this field.

Boekenoogen (*15*) has edited the first volume of a work surveying the entire field of analysis of oils and fats. A review of the uses of gas liquid chromatography in the field of drying oils was prepared (*40*) while several reviews (*53, 105, 118*) are concerned with application of gas chromatography to analysis and research in the field of coatings in general.

Two new books on the subject of thin layer chromatography (*14, 113*) include methods for separations of such coating materials as oils, fatty acids, plasticizers, and carboxylic acids. Rybicka (*119*) prepared a survey of thin layer chromatography with special reference to the problems of the paint analyst. A review that also includes some original methods for the analysis of alkyd resins (*115*) was prepared in which the analytical scheme was divided into physical tests and chemical tests.

Two reviews (*72, 127*) of the analysis of synthetic resins in general were published; the one by Kupfer contains 227 references. Lucchesi (*78*) prepared a paper on the role of analytical tools in coating research, commenting mostly on the applications of nuclear magnetic resonance, gel permeation chromatography, and attenuated total reflectance for surface analysis. Valentine (*146*) discussed the use of the electron microsope and the Coulter counter, as well as reflectance spectrophotometry for the study of paint films.

The Office of Technical Services of the U. S. Department of Commerce prepared a selective bibliography (*145*) of its available reports on paints and varnishes, collected from 1945 through 1963; unfortunately, however, many available reports on the subject were omitted from the listing.

GENERAL ANALYTICAL SCHEMES

By far the greatest number of contributions for general analytical applications involve gas chromatography and particularly the separation of pyrolysis products. Schweppe (*126*) discussed the use of established chemical methods for the qualitative analysis of synthetic binders in general and presented some new methods for differentiating polyvinyl esters, polyacrylates, polymethacrylates, cellulose esters and ethers, and chlorinated vinyl resins. Jordan (*59*) contributed a useful technique for relatively rapid quantitative saponification of highly hindered esters using alcoholic solvent and perchlorate anion catalysis.

A number of papers have illustrated the application of paper chromatographic analysis to synthetic resins. Weigel treated the subject broadly and included details for its application to differentiating modified alkyds (*151*); separating copolymers of vinyl acetate, vinyl chloride, maleic adducts, and poly(butyl methacrylate) from alkyds (*150*); and tabulating (*149*) a wide range of developing solvents for a large number of resins. Ingram (*56*) explored the use of attenuated total reflectance in infrared studies of polymeric materials and McGowan (*80*) compared quantitative results obtained in three laboratories by attenuated total reflectance and by transmission infrared spectrometry on alkyd vehicle films. The reproducibility and lack of film thickness sensitivity of ATR was illustrated.

An extensive report (*55*) describes the analytical applications of the infrared spectra of polymers, resins, waxes, plasticizers, monomers, and solvents, incorporating the absorption bands of important chemical families into correlation charts. The preparation of disposable silver chloride disks for examining water soluble and suspended polymers by infrared spectrometry in both wet and dry states has been described by Metzler (*87*).

A recent publication (*47*) explains a technique for applying attenuated total reflection to the examination of both the pigment and vehicle portion of a paint after separation of the two parts by the use of millipore filters. The samples studied were not sufficiently diversified to indicate whether the technique will have general application. Brako and Wexler (*16*) demonstrated the detection of carboxylate, ester, and olefinic groups in copolymers by changes in the infrared spectra before and after treatment with various reagents.

There is much similarity in the papers on identification of polymers from their pyrolysis products by gas chromatography. Cox and Ellis (*23*) examined 39 polymers using a packed tubular furnace. Stanley and Peterson (*134*) separated the pyrolyzates on a capillary column. In another investigation (*33*), the effect of temperature on thermal degradation was the prime concern. Two articles on the subject were concerned only with the identification of plastics (*21, 95*). Two studies (*48, 110*) compared the mass spectra of the pyrolysis products of the rubber polymers for identification purposes.

A report is available (*32*) on the application of pyrolytic gas chromatography to the analysis of thermosetting acrylic coating resins. A technique for determining polymer ratios of poly(ethylene ethyl acrylate) and poly(ethylene vinyl acetate) from pyrolyzates was described (*10*). An extensive investigation of polymer characterization by the application of pyrolytic gas chromatography was presented by Groten (*41*), in which the identification of 150 polymers was described and quantitative data for some of the cellulosics and styrene in vulcanizates were given.

Approximately 25 resins were included in the study made by Sadowski and Kuhn (*120*); the subject was also treated by Braun (*17*) and Hippe (*51*). A brief but recent publication (*29*) demonstrated the applicability of the internal standardization technique to the semiquantitative determination of some polymethacrylates and polystyrene in various coating systems and tabulated its contrast with the "total area" method. Eighteen polymers were included in a study (*136*) of thermogravimetric analysis, and a discussion of the points of decomposition was included.

SPECIFIC CLASSES OF HIGH POLYMERS

The basic theory of nuclear magnetic resonance was reviewed by Opp (*101*), who described its application to alkyd resin analysis. His work indicated that resins prepared from different phthalic acid isomers produced characteristic magnetic resonance spectra, and that the technique was applicable to the analysis of simple polymer mixtures. NMR was also used to study the factors affecting the degree of maleate–fumarate isomerization in unsaturated polyester formation (*25*).

Two significant contributions for the analysis of polyester resins utilized gas chromatography. Percival (*107*) isolated the coating resins from monomers by precipitation and used gas chromatography to analyze the liberated glycols and dimethyl esters formed by methanolysis. Luce *et al.* (*79*) characterized the polyesters in cured laminates by pyrolysis and gas chromatography. The simplified pyrolysis furnace described could be attached to a conventional chromatographic unit.

April (*6*) claimed complete analysis of alkyd and polyester resins by a combination of spectrometry and gas chromatography. Various phenol–formaldehyde resins were examined by infrared spectrometry (*19*) and the methods of identification developed utilized the 6- to 7-micron and the 8.1- to 8.2-micron regions of the spectrum. Wurst (*155*) outlined the separation and the determination of linear and cyclic poly(dimethyl siloxanes) by gas chromatography. An internal standard was used for quantitative work.

Paper chromatographic methods (*111*) were employed for the differentiation of urea- and melamine–formaldehyde adhesives. The samples were hydrolyzed with hydrochloric acid in the presence of hydrogen peroxide and the chromatograms developed with butanol–water–acetic acid. A report (*4*) is available that describes an adaptation of a previous method (*89*) for improved accuracy in measuring nitrogen resins in modified alkyd resins by infrared spectrometry extended to include benzoguanamine–formaldehyde.

Three papers on rosin analysis were presented. Nestler and Zinkel (*96*) separated seven methyl esters of rosin acids by gas liquid chromatography and listed relative retention values for both polyester and nitrile silicone rubber columns. Sandermann *et al.* (*121*) used differential thermal analysis in their investigation of rosin acids and their maleic anhydride adducts. A quantitative analytical procedure for rosin acids (*9*) uses column chromatography (silicic acid) followed by spectrophotometry and gravimetric determination of the cyclohexylamine salts of the eluted acids.

The quantitative determination of the composition of vinyl acetate copolymers by high resolution nuclear magnetic resonance was described by Dietrich and Keller (*27*). In another study (*20*) NMR was employed for quantitative determination of the vinyl acetate content of ethylene–vinyl acetate copolymers. Ferrocene was used as an internal standard and the analytical results for blends and commercial copolymers were presented.

The polarographic determination of vinyl acetate was reported (*54*) in which the resin was first hydrolyzed with lithium hydroxide. Schroder (*124, 125*) explained his procedure for the analysis of linear polyurethanes in which the material is hydrolyzed after 2 hours of boiling with 50% sulfuric acid. He then uses paper chromatography, gas chromatography, and oxidation techniques to identify the products of hydrolysis.

The determination of alkylcellulose ethers by gas chromatography was published by Cobler *et al.* (*22*). They reacted the samples with hydriodic acid to form alkyl iodides which were separated by gas chromatography. Newmann and Nadeau (*98*) analyzed polyether and polyolefin polymers by controlled pyrolysis in evacuated glass vials, determining the volatile products with a gas chromatograph having a flame ionization detector. The composition of butadiene copolymers of styrene and vinyl cyanide was determined (*65*) by infrared spectrometric examination of the swollen rubbers.

Wexler (*152*) analyzed insoluble butadiene–styrene copolymers by infrared examination of pressed or cast films. A comparison (*63*) was made of pitch resins from different sources through a combination of pyrolysis and gas liquid chromatography. The pyrolysis products were collected as they emerged and identified by infrared spectrometry.

SPECIFIC CONSTITUENTS

Procedures for both the polarographic and gas chromatographic determination of styrene monomer in polystyrene resins have been outlined (*112*) and results tabulated for samples containing up to 0.5% styrene. Two methods specific for styrene monomer and ethyl benzene in polystyrene were described (*109*). In one method the polystyrene is precipitated with methanol from *o*-dichlorobenzene and in the other the monomer and solvents are removed by distillation; both methods are concluded with gas chromatography.

The selective determination of residual monomers in copolymers of vinyl acetate and 2-ethylhexyl acrylate (*90*) was based on the difference in the rate of reaction of both monomers with bromine, and the application to other copolymers was proposed. The spectrophotometric determination of vinyl cyanide in the presence of styrene was proposed (*13*) using differential absorption at 204 and 247 mμ.

Two gas chromatographic methods appeared for determining residual monomers in polymer emulsions. In one (*143*), water and toluene were added and 3 ml. of distillate collected. The solvent layer was chromatographed, the toluene acting also as internal standard. In the other procedure (*153*), a solvent is added which reacts with the water present and the sample is injected onto glass wool in the injection port. Qualitative and quantitative data are presented with indications that the method may be applicable to a variety of latex systems.

A number of papers published in this period concern the analysis of polyester resins. Novak (*99*) described a polarographic technique for simultaneous determination of citraconic, mesaconic, and phthalic acids. Percival and Stevens (*108*) made qualitative and semiquantitative identification of commonly used acids and glycols in polyesters from NMR spectra.

For paper chromatographic analysis of linear polyesters (*34*), two new reagents were used for detection of the acids and higher glycols. Some of the more rarely used polyhydric alcohols in polyesters were identified (*93*) with gas chromatography. Wittendorfer (*154*) published a detailed procedure for quantitative measure of trimethylolpropane in polyesters, with a modification for polyurethane foams. The samples are treated with amine, then acetic anhydride; the triacetate is extracted and separated by gas liquid chromatography.

Smith and Carlsson (*130*) separated polyhydric compounds as trimethylsilyl ethers by gas chromatography. The 1-meter column they used was packed with 20% SE-30 or Apiezon M on 60- to 100-mesh Celite 545. The gas chromatographic determination of phthalic anhydride in alkyd resins by methoxide transesterification was reported (*30*). The analysis of alkyd resins for benzoic acid and its *p*-tertiary butyl homologue by gas chromatographic separation of methyl esters was discussed (*44*) but no quantitative data were presented.

A report on the direct measure, by gas chromatography, of styrene content during the cooking of styrenated alkyds (*129*) is available. Robb and Westbrook (*116*) used formation of methyl esters within the injection port of a gas chromatograph by conversion from the tetramethylammonium salts of carboxylic acids for qualitative purposes.

Unger (*144*) suggested colorimetric procedures for the micro determination of acids, glycols, glycerine, and formaldehyde in resins. Reed *et al.* (*114*) have presented a procedure for determining hydroxyl equivalent that is applicable to polyfunctional polyols used in producing urethane resins. Phenyl isocyanate catalyzed with stannous octoate is used as the reagent.

Adams (*5*) proposed infrared absorbance at 3.08 microns, with corrections for water, for measuring hydroxyl content of epoxy resins. A method for measuring epoxy groups was published (*148*) that is claimed to be free from interference from other resins. The resin is treated with hydrochloric acid in dioxane and the excess back-titrated with mercuric nitrate in the presence of diphenylcarbazone.

Jay (*58*) contributed a rapid method for the determination of oxirane ring in epoxy resins. A gas chromatographic method for determining free phenol and free formaldehyde in phenolic resins (*135*) was described. Formaldehyde and acetaldehyde in some of their polymers were measured (*39*) by thermal decomposition and absorption of aldehydes in water, followed by oxidation to respective acids.

A direct titration in anhydrous acetic acid with perchloric acid (*66*) was claimed to be suitable for measuring the melamine content of melamine–formaldehyde resins. Lee (*73*) outlined spectrophotometric methods for determining free and total formaldehyde in amino resins. A study was made which claims (*92*) that the composition of melamine–formaldehyde resins can be established from colorimetric and iodometric measure of butoxy and hydroxy–methyl groups, respectively.

Neubauer and associates (*97*) used gas chromatography to measure the free tolylene diisocyanate in adducts with trimethylol propane. They devised a plan for compensating for TDI that formed from thermal decomposition of polymers and claimed the method has advantages of greater speed and accuracy over chemical methods. Lowe (*77*) combined column chromatography and colorimetric analysis to determine free tolylene diisocyanate in polyisocyanates.

Levitsky and Norwitz (*74*) conducted a study to establish the best procedure for the quantitative estimation of nitrogen in nitrocellulose and concluded that the 6-micron band of the infrared spectrum should be used with the samples dissolved in tetrahydrofuran. Both electrometric titration and ion exchange chromatography were described (*43*) as suitable for measuring the fluoride ion in polymers. Miles (*88*) has supplied specific tests for antimony, phosphorus, and vinyl chloride that he uses to identify some flame-resistant finishes.

OILS AND FATTY ACIDS

The interlaboratory testing of a modified Rosenmund - Kuhnhenn method for application to the determination of total unsaturates of drying oils, bodied oils, and fatty acids was described by Spagnola (*131*). A comparison of well known iodine value methods was made (*60*) with a new procedure using *n*-bromosuccinimide as reagent. The oil or fatty acid in 10 ml. of chloroform was treated with 20 ml. of a 1.8% solution of reagent in anhydrous acetic acid for 24 hours. The application of paper chromatography to the unsaponifiable portion was used (*64*) to identify tall oil fatty acids in lacquers.

Scholfield and associates (*123*) fractionated mixtures of fatty acid methyl esters by countercurrent distribution between hexane and methanolic silver nitrate to separate the geometric isomers. Reversed phase circular paper chromatography has been applied (*147*) to the analysis of the fatty acids commonly found in drying oils.

Five significant papers concerned the separation of triglycerides. Trowbridge *et al.* (*142*) recommended the use of rubber powder chromatographic columns. Swartout and Gross (*138*) found that triglycerides may be separated by ascending chromatography on uncoated glass fiber filter paper or silica gel coated paper using pyridine–water solvent system. Two thin-layer chromatographic techniques for determining glycerides were developed by Gunstone and associates (*42*). Horizontal thin layer chromatography was also used (*26*) for the complete triglyceride analysis of palm oil and soyabean oil using silica gel and silver nitrate. Barrett *et al.* (*11*) used a similar method for triglyceride mixtures.

Hammonds and Shone (*46*) obtained complete separation of methyl linoleate, laurate, linolenate, and myristate with thin layer chromatography. Fatty acid methyl esters have been separated by thin layer chromatography (*117*) and by column chromatography (*156*).

Many analytical contributions for oil and fatty acids make use of gas liquid chromatography. Mason and associates (*83, 84*) contributed procedures for the simultaneous quantitative determination of the glycerol and fatty acid contents of fats and oils. In their method, derivatives were formed by transesterification with dimethoxypropane. Two authors (*31, 158*) have reported separately on the application of the internal standardization method for the quantitative determination of drying oil fatty acids and polymer content.

Ackman (*1, 2*) has correlated structure of fatty acids for identification purposes with GLC retention times. He used polyester substrates and tabulated (*3*) specific response factors for methyl esters of fatty acids for gas chromatographic analysis with flame ionization detectors. Miwa (*91*) made a similar study. Two gas chromatographic studies (*36, 100*) were conducted with drying oils of high unsaturation that had been treated by heat-bodying or dehydration. A study was made (*75, 76*) of *cis-trans* isomerization of oleic, linoleic, and linolenic acids and their gas chromatographic separation.

Ast (*8*) has investigated the inadvertent isomerization of polyunsaturated acids during ester preparation for subsequent chromatographic separation. Metcalfe (*86*) reported on separation of unesterified fatty acids on phosphoric acid–treated polyester columns and proposed other uses for such columns. Stannic phosphate was also proposed (*67*) as a support for the gas chromatographic analysis of fatty acids.

Kuemmel (*71*) conducted gas chromatographic analysis of the products of oxidation of fatty acids, esters, hydrogenated fats, and mixtures of isomeric olefins. Oxidation was also used to determine the distribution of saturated acids within the glycerides of certain oils, including soyabean and cottonseed (*157*).

ASSOCIATED MATERIALS

A greater number of papers than usual have appeared on separation or identification of plasticizers in plastics and coating materials, probably because of the adaptability of some of the newer analytical techniques. Infrared spectra for 21 common plasticizers were included in a publication (*85*) on their extraction from plastics. Criddle (*24*) combined chromatographic and infrared analysis in his work. Thin layer chromatography was used by Braun (*18*) who tabulated R_f values for 40 plasticizers.

A simple, rapid, and accurate method was presented (*28*) for identifying and determining seven common plasticizers in nitrocellulose, vinyl, and acrylic-type lacquers by programmed-temperature gas chromatography. Adipate and phthalate plasticizers were separated and identified (*159*) by the use of two columns of glass microbeads coated with polar and nonpolar liquid phases.

Haken and McKay (*45*) gave relative retention times for chromatographic separation of over 90 solvents using polar and nonpolar columns. Gatrell (*37*) prepared a mixed substrate column for gas chromatographic analysis of lacquer thinners. Mortimer and Gent (*94*) separated an 18-component solvent mixture which included xylene isomers and ethyl benzene with a 12-foot column prepared from silicone oil, Bentone 34, and Celite. Spencer (*132*) used a column prepared from diisodecyl phthalate modified with Bentone 34 to separate toluene, ethyl benzene, and xylene isomers in about 12 minutes.

A variety of techniques have been used to analyze isolated carboxylic acids, raw materials for the preparation of alkyds, polyesters, and plasticizers. Two USSR patents (*69*) describe the potentiometric titration of mixtures of the three phthalic acid isomers and of a mixture of the iso- and tere-isomeric acids. A polarographic determination (*12*) of terephthalic acid and its potassium salts in the presence of other phthalic acids, toluic acid, and benzoic acid was described. Chromatography on a silica gel column was used to separate mixtures of iso- and terephthalic acids (*61*). Trachman and Zucker (*141*) determined isothermally maleic anhydride, benzoic acid, naphthalene, and 1,4-naphthoquinone in commercial phthalic anhydride by gas chromatography using a silicone column and a hydrogen flame detector. Twelve benzene carboxylic acids were separated (*122*) as their methyl esters by gas chromatography.

Three publications (*38, 62, 140*) concerned the analysis of bisphenol A and its impurities, and all used gas chromatography. Thin layer chromatography was employed to measure the amounts of stabilizers in poly(vinyl chloride) (*68*).

Thaw and Hirn (*139*) reported an investigation of ashing under controlled temperatures for rapid estimation of the pigment content of emulsion paints. They obtained good results with samples of certain known pigment composition, but the method is not applicable with accuracy to unknown materials in general. Ashton (*7*) made a study of analytical procedures for measuring pigment volume concentration to obtain improved accuracy.

A review was made of the methods for analyzing synthetic organic pigments (*128*), followed by tabulation of their reactions and a description of methods for separating mixtures. An interesting description was published (*49*) of a method by which it should be possible to identify a mixture of four to eight pigments as their compounds form within a few hours. This analytical scheme was republished in its English translation (*50*).

The volumetric determination of zinc in mixed paint pigments by chelate formation was published (*70*) as an improved modification of earlier methods.

LITERATURE CITED

(1) Ackman, R. G., *J. Am. Oil Chemists' Soc.* **40,** 558 (1963).
(2) *Ibid.*, p. 564.
(3) Ackman, R. G., Sipos, J. C., *Ibid.*, **41,** 377 (1964).
(4) Adams, M. L., U. S. Dept. Commerce, Off. Tech. Serv., *Rept.* No. **AD-430-724** (1964).
(5) Adams, M. R., ANAL. CHEM. **36,** 1688 (1964).
(6) April, A., *Ingr. Chimiste* **45,** 74 (1963).
(7) Ashton, H. E., *Offic. Dig. Federation Soc. Paint Technol.* **34,** 1343 (1962).
(8) Ast, H. J., ANAL. CHEM., **35,** 1539 (1963).
(9) Bardyshev, I. I., Cherches, K. A., Meerson, L. A., *Zhur. Anal. Khim.* **18,** 895 (1963).
(10) Barrall, E. M., II, Porter, R. S., Johnson, J. F., ANAL. CHEM. **35,** 73 (1963).
(11) Barrett, C. B., Dallas, M. S. J., Padley, F. M., *J. Am. Oil Chemists' Soc.* **40,** 580 (1963).
(12) Bellen, Z., Kochel, I., *Chem. Anal. (Warsaw)* **8,** 411 (1963).
(13) Birr, K. H., Zieger, G., *Z. Anal. Chem.* **196,** 351 (1963).
(14) Bobbitt, J. M., "Thin Layer Chromatography," Reinhold, New York, 1963.
(15) Boekenoogen, H. A., "Analysis and Characterization of Oils, Fats and Fat Products," Vol. **I,** Interscience, New York, London 1964.
(16) Brako, F. D., Wexler, A. S., ANAL. CHEM. **35,** 1944 (1963).
(17) Braun, D., *Farbe Lack* **69,** 820 (1963).
(18) Braun, D., *German Plastics* **52,** 1 (1962).
(19) Cazes, J., *Double Liaison* **88,** 17 (1962).
(20) Chen, H. Y., Lewis, M. E., ANAL. CHEM. **36,** 1394 (1964).
(21) Cobler, J. G., Samsel, E. P., *Trans. Soc. Plast. Engrs.* **2,** 145 (1962).
(22) Cobler, J. G., Samsel, E. P., Beaver, G. H., *Talanta* **9,** 473 (1962).
(23) Cox, B. C., Ellis, B., ANAL. CHEM. **36,** 90 (1964).
(24) Criddle, W. J., *Brit. Plast.* **36,** 242 (1963).
(25) Curtis, L. G., Edwards, D. L., Simons, R. M., Trent, P. J., Von Bramer, P. T., *Ind. Eng. Chem., Prod. Res. Dev.* **3,** 218 (1964).
(26) De Vries, B., Jurriens, G., *J. Chromatog.* **14,** 525 (1964).
(27) Dietrich, M. W., Keller, R. E., ANAL. CHEM. **36,** 2174 (1964).
(28) Esposito, G. G., *Ibid.*, **35,** 1439 (1963).
(29) Ibid., **36,** 2183 (1964).
(30) Esposito, G. G., U. S. Dept. Commerce, Off. Tech. Serv. *Rept.* No. **AD-293-160** (1963).
(31) *Ibid.*, No. **AD-402-178** (1963).
(32) *Ibid.*, No. **AD-432-590** (1964).
(33) Ettre, K., Varadi, P. F., ANAL. CHEM. **35,** 69 (1963).
(34) Fijolka, P., Radowitz, W., Runge, F., *Plaste Kautschuk* **10,** 521 (1963).
(35) Firestone, D., *J. Am. Oil Chemists' Soc.* **40,** 247 (1963).
(36) Gast, L. E., Schneider, W. J., Forest, C. A., Cowan, J. C., *Ibid.*, p. 287.
(37) Gatrell, R. L., ANAL. CHEM. **35,** 923 (1963).
(38) Gill, H. H., *Ibid.*, **36,** 1201 (1964).
(39) Giuffre, L., Capizzi, F. M., *Chim. Ind. (Milan)* **45,** 40 (1963).
(40) Greaves, J. H., *J. Oil Colour Chemists' Assoc.* **47,** 499 (1964).
(41) Groten, B., ANAL. CHEM. **36,** 1206 (1964).
(42) Gunstone, F. D., Padley, F. B., Qureshi, M. I., *Chem. Ind. (London)* **1964,** p. 483.
(43) Gurvich, D. B., Balandina, V. A., *Plastmassy v Mashinostr. Mosk. Dom Nauchn. Tekhn. Propagandy* **1962** (6), p. 54.
(44) Haken, J. K., *J. Gas Chromatog.* **2,** 263 (1964).
(45) Haken, J. K., McKay, T. R., *J. Oil Colour Chemists' Assoc.* **47,** 517 (1964).
(46) Hammonds, T. W., Shone, G., *J. Chromatog.* **15,** 200 (1964).
(47) Hannah, R. W., Dwyer, J. L., ANAL. CHEM. **36,** 2341 (1964).

(48) Happ, G. P., Maier, D. P., *Ibid.*, p. 1678.
(49) Hezel, E., *Farbe Lack* **69,** 361 (1963).
(50) Hezel, E., *Paint Technol.* **28,** (6), 16 (1964).
(51) Hippe, Z., *Polimery* **8,** (11), 414 (1963).
(52) Holman, R. T., "Progress in the Chemistry of Fats and Other Lipids," Vol. **7,** Part 2, Macmillan, New York, 1964.
(53) Hoover, W. S., Jr., *Paint Varnish Production* **54,** (7), 69; (8), 41; (9), 61 (1964).
(54) Horacek, J., *Plaste Kautschuk* **9,** 116 (1962).
(55) Hummel, D. O., U. S. Dept. Commerce, Off. Tech. Serv. *Rept.* No. **AD-600-552** (1964).
(56) Ingram, B. M., *Research Development* **34,** 18 (1964).
(57) James, A. T., *Analyst* **88,** 572 (1963).
(58) Jay, R. R., ANAL. CHEM. **36,** 667 (1964).
(59) Jordan, D. E., *Ibid.*, p. 2134.
(60) Jovchev, A., Bonchev, N., Ionkov, D., *Nahrung* **7,** 375 (1963).
(61) Kamneva, A. I., Panfilova, E. S., *Zavod. Lab.* **29,** 666 (1963).
(62) Kanne, F., Stange, K., *Z. Anal. Chem.* **189,** 261 (1962).
(63) Karr, C., Comberiati, J. R., Warner, W. C., ANAL. CHEM. **35,** 1441 (1963).
(64) Kaufmann, H. P., Guldenpfennig, R., *Fette, Seifen, Anstrichmittel* **64,** 334 (1962).
(65) Kimmer, W., *Plaste Kautschuk* **9,** 135 (1962).
(66) Knappe, E., Peteri, D., *Z. Anal. Chem.* **194,** 417 (1963).
(67) Konishi, K., Kano, Y., *Bunseki Kagaku* **13,** 299 (1964).
(68) Korn, O., Woggon, H., *Nahrung* **8,** 351 (1964).
(69) Kreshkov, A. P., Bykova, L. N., Smolova, N. T., USSR patent **161,725; 161,726** (1964).
(70) Kruijsse, L., *Verfkroniek* **36,** 446 (1963).
(71) Kuemmel, D. F., ANAL. CHEM. **36,** 426 (1963).
(72) Kupfer, W., *Z. Anal. Chem.* **192,** 219 (1962).
(73) Lee, Min-Dar, *Chemistry (Taipei)* **1963** (1), p. 27.
(74) Levitsky, H., Norwitz, G., *Offic. Dig. Federation Soc. Paint Technol.* **35,** 390 (1963).
(75) Litchfield, C., Lord, J. E., Isbell, A. F., Reiser, R., *J. Am. Oil Chemists' Soc.* **40,** 553 (1963).
(76) Litchfield, C., Reiser, R., Isbell, A. F., *Ibid.*, p. 302.
(77) Lowe, A., *J. Oil Colour Chemists' Assoc.* **46,** 842 (1963).
(78) Lucchesi, C. A., *Offic. Dig. Federation Soc. Paint Technol.* **35,** 975 (1963).
(79) Luce, C. C., Humphrey, E. F., Guild, L. V., Norrish, H. H., Coull, J., Castor, W. W., ANAL. CHEM. **36,** 482 (1964).
(80) McGowan, R. J., *Ibid.*, **35,** 1664 (1963).
(81) Mahadevan, V., *J. Am. Oil Chemists' Soc.* **40,** 372 (1963).
(82) *Ibid.*, **41,** 559 (1964).
(83) Mason, M. E., Eager, M. E., Waller, G. R., ANAL. CHEM. **36,** 587 (1964).
(84) Mason, M. E., Waller, G. R., *Ibid.*, p. 583.
(85) Meise, W., Ostromow, H., *Kunststoffe* **54,** 213 (1964).
(86) Metcalfe, L. D., *J. Gas Chromatog.* **1,** 7 (1963).
(87) Metzler, R. K., ANAL. CHEM. **36,** 2378 (1964).
(88) Miles, T. D., *Am. Dyestuff Reptr.* **51,** 442 (1962).
(89) Miller, C. D., Shreve, O. D., ANAL. CHEM. **28,** 200 (1956).
(90) Mintzer, I., Montzoi, I., *Z. Anal. Chem.* **196,** 27 (1963).
(91) Miwa, T. K., *J. Am. Oil Chemists' Soc.* **40,** 309 (1963).
(92) Miyauchi, N., *Chem. High Polymers (Tokyo)* **20,** 42 (1963).
(93) Mlejnek, O., *Chem. Prumysl* **13,** 105 (1963).
(94) Mortimer, J. V., Gent, P. L., *Nature* **197,** 789 (1963).
(95) Nelson, D. F., Yee, J. L., Kirk, P. L., *Microchem. J.* **6,** 225 (1962).
(96) Nestler, F. H. M., Zinkel, D. F., ANAL. CHEM. **35,** 1747 (1963).
(97) Neubauer, N. R., Skreckoski, G. R., White, R. G., Kane, A. J., *Ibid.*, p. 1647.
(98) Newmann, E. W., Nadeau, H. G., *Ibid.*, p. 1454.
(99) Novak, V., *Chem. Prumysl* **14,** 200 (1964).
(100) O'Neill, L. A., Rybicka, S. M., *Chem. Ind.* **10,** 390 (1963).
(101) Opp, C. J., *Offic. Dig. Federation Soc. Paint Technol.* **36,** 42 (1964).
(102) *Paint Manuf.* **33** (2), 68 (1963).
(103) *Ibid.*, **34** (5), 69; (10), 56 (1964).
(104) *Paint Varnish Prod.* **53** (4), 122 (1963).
(105) *Ibid.*, (7), p. 29.
(106) *Ibid.*, **54** (3), 138 (1964).
(107) Percival, D. F., ANAL. CHEM. **35,** 236 (1963).
(108) Percival, D. F., Stevens, M. P., *Ibid.*, **36,** 1574 (1964).
(109) Pfab, W., Noffz, D., *Z. Anal. Chem.* **195,** 37 (1963).
(110) Phillips, J. K., *Appl. Spectry.* **17,** 9 (1963).
(111) Plath, L., *Holz Roh-Werkst.* **19,** 489 (1961).
(112) Ragelis, E. P., Gajan, R. J., *J. Assoc. Offic. Agri. Chemists* **45,** 918 (1962).
(113) Randerath, K., "Thin Layer Chromatography," Academic Press, New York, 1963.
(114) Reed, D. H., Critchfield, F. E., Elder, D. K., ANAL. CHEM. **35,** 571 (1963).
(115) Rente, M. A., Preza, M. A., *Peintures Pigments, Vernis* **39,** 359 (1963).
(116) Robb, E. W., Westbrook, J. J., ANAL. CHEM. **35,** 1644 (1963).
(117) Ruggieri, S., *Nature* **193,** 1282 (1962).
(118) Rybicka, S. M., *J. Oil Colour Chemists' Assoc.* **47,** 475 (1964).
(119) Rybicka, S. M., *Rev. Current Lit. Paint Allied Ind.* **36,** 437 (1963).
(120) Sadowski, F., Kuhn, E., *Farbe Lack* **69,** 267 (1963).
(121) Sandermann, W., Weissmann, G., Augustin, H., *Ibid.*, p. 428.
(122) Schnitzer, M., Desjardins, J. G., *J. Gas Chromatog.* **2,** 270 (1964).
(123) Scholfield, C. R., Jones, E. P., Butterfield, R. O., Dutton, H. J., ANAL. CHEM. **35,** 1588 (1963).
(124) Schroder, E., *Plaste Kautschuk* **9,** 121 (1962).
(125) *Ibid.*, **10** 25 (1963).
(126) Schweppe, H., *F.A.T.I.P.E.C.* **VI,** Congress 219 (1962).
(127) Schweppe, H., *Paint Technol.* **27** (7), 14 (1963).
(128) *Ibid.*, (8), 12 (1963).
(129) Seifert, W. K., Percival, D. F., *Ind. Eng. Chem., Prod. Res. Develop.* **3,** 222 (1964).
(130) Smith, B., Carlsson, O., *Acta Chem. Scand.* **17,** 455 (1963).
(131) Spagnola, F., *Mater. Res. Std.* **2,** 899 (1962).
(132) Spencer, S., ANAL. CHEM. **35,** 592 (1963).
(133) "Standard Methods for the Analysis of Oils, Fats and Soaps," 5th ed., Butterworth, London (1964).
(134) Stanley, C. W., Peterson, W. R., *Trans. Soc. Plast. Engrs.* **2,** 298 (1962).
(135) Stevens, M. P., Percival, D. F., ANAL. CHEM. **36,** 1023 (1964).
(136) Sung, Chwa-Kyung, *J. Korean Chem. Soc.* **7,** 96 (1963).
(137) Swann, M. H., Adams, M. L., Esposito, G. G., *Anal. Chem.* **35,** 35*R* (1963).
(138) Swartout, J. R., Gross, R. J., *J. Am. Oil Chemists' Soc.*, **41,** 378 (1964).
(139) Thaw, W. M., Hirn, C. F., *Offic. Dig. Federation Soc. Paint Technol.* **35,** 1036 (1963).
(140) Tominaga, S., *Japan Analyst* **12,** 137 (1963).
(141) Trachman, H., Zucker, F., ANAL. CHEM. **36,** 269 (1964).
(142) Trowbridge, J. R., Herrick, A. B., Bauman, R. A., *J. Am. Oil Chemists' Soc.* **41,** 306 (1964).
(143) Tweet, O., Miller, W. K., ANAL. CHEM. **35,** 852 (1963).
(144) Unger, P., *Paint Manuf.* **34** (1), 49 (1964).
(145) U. S. Dept. Comm., Off. Tech. Serv., "Selective Bibliography of Reports on Paints and Varnishes," *Rept.* No. **SB-518** (1963).
(146) Valentine, L., *Peintures Pigments, Vernis* **39,** 211 (1963).
(147) Viswanathan, C. V., Bai, B. M., *J. Chromatog.* **7,** 507 (1962).
(148) Vorobjov, V., *Chem. Prumysl* **13,** 381 (1963).
(149) Weigel, K., *Dtsch. Farben-Z.* **16,** 297, 339 (1962).
(150) Weigel, K., Gum., Asb., *Kunststoffe* **15,** 20 (1962).
(151) Weigel, K., *Plaste Kautschuk* **9,** 123 (1962).
(152) Wexler, A. S., ANAL. CHEM. **36,** 1829 (1964).
(153) Wilkinson, L. B., Norman, C. W., Buettner, J. P., *Ibid.*, p. 1759.
(154) Wittendorfer, R. E., *Ibid.*, p. 930.
(155) Wurst, M., *Collection Czech. Chem. Commun.* **29,** 1458 (1964).
(156) Wurster, C. F., Jr., Copenhaver, J. H. Jr., *J. Am. Oil Chemists' Soc.* **40,** 513 (1963).
(157) Youngs, C. G., Subbaram, M. R., *Ibid.*, **41,** 218 (1964).
(158) Zielinski, W. L., Jr., *Ibid.*, p. 249.
(159) Zulaica, J., Guichon, G., ANAL. CHEM. **35,** 1724 (1963).

Essential Oils and Related Products

Ernest Guenther, James A. Rogers, Jr., Gilbert Gilbertson, and Roman T. Koenig
Fritzsche Brothers, Inc., New York, N. Y.

THIS TENTH review of analytical procedures for essential oils and related products covers the literature from September 1962 to August 1964, inclusive. It follows the general pattern previously established (*217*).

During this period a continuing increase in the use of spectrophotometric methods of analysis has been noted, and chromatographic methods, particularly thin layer and paper chromatography, as well as gas chromatographic procedures with modified columns and new detectors, are continually being adapted to the solution of difficult analytical problems.

High resolution mass spectroscopy has become one of the important tools of the analyst, greatly simplifying the identification and structure elucidation of organic compounds.

It becomes increasingly apparent that, because of the high degree of specialization required for the effective use of modern chemical, physical, and instrumental methods of analysis, most of the outstanding work is being accomplished by those laboratories in which teams of experts successfully correlate the data obtained with the diverse techniques.

Many books of interest to the analyst have appeared, but only those specifically dealing with essential oils and related products are listed:

Ourisson, G., Crabbe, P., "Les Triterpenes Tetracycliques," Hermann et Cie., Paris, 1961, 194 pp.

Gildemeister, E., Hoffmann, Fr., "Die Aetherischen Oele," Vol. 3b, 4th ed., 1962, 436 pp., and Vol. 3c, 4th ed., 1963, 510 pp., Akademie-Verlag, Berlin.

A new periodical, *Food and Cosmetics Toxicology* published quarterly by Pergamon Press Ltd., Oxford, appeared, the first issue being dated September 1963.

OFFICIAL COMPENDIA

The Scientific Committee of the Essential Oil Association of the U.S.A. has published specifications for nine additional essential oils, oleoresins or natural products, and twelve aromatic chemicals, increasing the total number of specifications from 224 to 245 as of January 1965. The natural products monographs added since the last review are:

No.	
229	Oil cedarwood atlas
238	Oleoresin cloves
239	Oleoresin paprika
240	Oleoresin black pepper
241	Oleoresin mace
242	Oleoresin celery
243	Oleoresin ginger
244	Oleoresin capsicum
245	Oleoresin red pepper

Specifications for the following aromatics were issued:

No.	
225	Linalyl acetate—synthetic
226	Linalool—synthetic
227	Citronellal
228	Methyl benzyl acetate
230	Ethyl heptoate
231	*p*-Cresyl phenyl acetate
232	*p*-Cresyl isobutyrate
233	Terpinyl propionate
234	Santalol
235	Decahydro beta naphthol
236	Ethyl salicylate
237	Benzyl isoeugenol

Specifications for three essential oils and three aromatic chemicals were rewritten:

No.	
14	Oil citronella Java type
23	Oil patchouly
32	Oil spruce
21	Indole
27	Amyl salicylate
28	Benzyl acetate

Specifications were revised for six essential oils and six aromatic chemicals under R-10 to R-13.

No.	
2	Oil bois de rose Brazilian
7	Oil lemongrass
49	Oil geranium reunion
50	Oil Siberian fir needle
137	Oil galbanum
215	Oil bitter almond *ffpa*
26	Aldehyde C-10
35	Dimethyl anthranilate
109	Aldehyde C-16 (so-called)
127	Cedryl acetate
203	Methyl cinnamic alcohol
211	Anisyl acetate

New test procedures were added to Notes for Specifications and Standards:

1-ID-3 Gas Chromatographic Analysis.

A determination based on ID-3 was added to

No.

16 Geranoil.

1-ID-4 Ultraviolet Absorbance Test for Expressed Citrus Oils

Specifications based on ID-4 were added for

No.	
30	Oil grapefruit expressed
88	Oil limes expressed
94	Oil tangerine expressed
95	Oil mandarin expressed
155	Oil bitter orange

Tests were revised under 1-7 for:

A. Determination of Esters
B. Determination of Total Alcohols
E. Determination of Acid Value

Infrared Spectra for identification purposes have been included in 63 additional monographs, bringing the total to 83.

ANALYTICAL PROCEDURES FROM THE LITERATURE

Essential Oils. GENERAL PROCEDURES. Gas, thin layer, and paper chromatography were compared as to efficiency, sensitivity, specificity, accuracy, and cost by Seher (*628*). Aratani and Komae (*12*) reviewed the application of gas chromatography to terpenic compounds and perfume materials.

Porcaro (*549*) reported the resolution of common oxygenated essential oil constituents, using an empty copper capillary column coated with polyisobutylene. Self, Land, and Casey (*630*) gave details for the construction and operation of capillary columns made with nylon tubing. Bevitt and Cheshire (*43*) discussed conditions which gave optimum performance in the gas chromatographic analysis of essential oils. A new method for the continuous direct analysis of natural fragrances was devised by Sprecker and Strackenbrock (*662*). Franc and Michajlova (*177*) described a new gas chromatographic method for the separation of organic compounds; Bassette, Ozeris, and Whitnah (*27*) adapted gas chromatography to the analysis of complex mixtures of volatile compounds in headspace gas.

Asphalt was employed as the stationary phase in the high temperature gas chromatography of oxygenated terpenes by Aratani, Komae, and Matsuura (*13*).

Gas chromatography with two different columns, alone or in series, was used to separate the components of the light fractions of essential oils by Ricciardi, Burgos, and Cassano (*571*), and Ricciardi and Cassano (*572*). Didecyl phthalate, polyethylene glycol 1500, dicyclohexyl phthalate, and ethylene glycol polysuccinate were evaluated as stationary phases. The two column technique of Oster was employed by Drawert and Rapp (*144*) for the separation of alcohols, esters, ketones, and terpenes.

A new technique in reaction gas chromatography was demonstrated by Hoff and Feit (*266, 267*), who caused the chemical reaction to occur in the injection syringe. Procedures were described for carbonyl compounds, alcohols, ethers, olefins, aromatic hydrocarbons, and unsaturated compounds. Okamura, Noriishi, and Tanaka (*505*) applied the Walsh method to the detection of functional groups in the effluent from gas chromatography. Alcohols, ketones, aldehydes, and ethers were detected by nichromic acid, *o*-dianisidine, 2,4-dinitrophenylhydrazine, and ferric hydroxamate methods. Beroza (*39, 40*), and Beroza and Acree (*41*) discussed new micro and semimicro techniques for the determination of structure by gas chromatography. A hot catalyst-containing tube that attaches to the injection port was employed.

A continuous separation method was achieved by Pichler *et al.* (*536*), who applied a countercurrent elution procedure to gas chromatography. The separation of compounds which are difficult to fractionate, such as *cis*- and *trans*-2-butene, was successfully undertaken.

An apparatus for the collection of gas chromatographic fractions for infrared analysis was described by Nigam *et al.* (*472*); Rath (*568*) discussed an instrument for preparative gas chromatography capable of separating 15 to 20 ml. during one operation.

Gas chromatography together with infrared spectroscopy was employed by Nigam and Levi (*468*) to separate and identify many closely related sesquiterpene compounds such as α- and β-santalol from sandalwood oil, and Nigam, Sahasrabudhe, and Levi (*471*) coupled gas and thin layer chromatography to determine simultaneously piperitone and piperitone oxide in various oils. Wellendorf (*746*) combined gas and thin layer chromatography in the study of the oils described in the Pharmacopeia Danica. Datta *et al.* (*117*) determined the geographical origin of cassia, black pepper, nutmeg, and ginger by micro-infrared spectroscopy of the effluent from gas chromatography of the respective essential oils.

The problems encountered in the application of gas chromatography to perfumery raw materials and the errors which may result were reviewed by Naves (*451*). Johnston (*301*) stressed that results of instrumental analysis must be applied with care. Mitzner (*424*) described the decomposition of oxygenated terpenes in injection heaters, as exemplified by the breakdown of α-terpineol, and demonstrated an injection technique which prevents it. Stedman and Miller (*666*) cautioned that the concentration of extracts may lead to losses of natural volatile aromatic compounds.

Chromatography on an Al_2O_3 column was employed by Rothe (*589*) to separate oxygenated compounds from hydrocarbons. The oxygenated fraction was recovered by aqueous extraction. Sherma, Locke, and Bassett (*640*) used the salting-out chromatographic technique to separate water-insoluble alcohols and ketones. Nigam and Kumari (*476*) discussed chromatography as applied to the analysis of *citrus decumana*, carrot, clove, thyme, rosemary, and lemon oils, and tabulated the compounds determined. They isolated gratissimol, a new sesquiterpene alcohol from oil of *ocimum gratissimum*.

Thin layer chromatography was applied to essential oils by Jaspersen-Schib and Flueck (*298, 299*) for identification, characterization of constituents, and purity determination. The technique was applied to rosemary and rose oils. Pertsev and Pivnenko (*530*) similarly analyzed the oils of coriander, lavender and nutmeg; Paris and Godon (*522*), who examined 21 oils, demonstrated how the method may be used to differentiate between closely related products such as anise oil and star anise oil. Thin layer chromatography on alumina was used by Kucera (*355*) to separate alcohols, glycols, and diketones; and by Kheifits, Moldovanskaya, and Shulov (*324*) to separate alkylphenols and alkylcyclohexanones. Knappe and Peteri (*338*) used silica gel to chromatograph organic peroxides. Halpaap (*226*) presented a detailed procedure for preparative film chromatography, which is a scaled up technique permitting the separation of up to 100-gram mixtures.

Hoerhammer, Richter, and Wagner (*264*), and Richter and Muscholl (*575*) described in detail new paper chromatographic procedures and gave the chromatograms resulting from their application to several important essential oils and mixtures of aromatic chemicals. Paraformaldehyde-impregnated paper was employed. Edwards (*148*) developed a markedly different two-dimensional paper chromatographic separation of hydroxyl compounds wherein esterification was brought about on the paper by formic acid.

The routine analysis of essential oils by infrared spectroscopy was illustrated in relation to geranium and citronella oils by Carroll and Price (*87*). Mann (*400*) discussed its application, together with gas chromatography, to the evaluation of ambergris, civet, jasmine, orris, and rose oils. The infrared absorption spectra of a number of isolates from essential oils, including apiole and myristicin, were discussed by Biernacka *et al.* (*50*). Hayden *et al.* (*237*) gave infrared, ultraviolet, and visible absorption spectra for many U.S.P. and N.F. reference standards.

Ultraviolet absorption spectra were given by Minutilli (*420*) for oils of star anise, cinnamon, clove, lavender, crispmint, and peppermint.

The Raman spectra of fifteen essential oils were discussed by Mohan (*425, 426*), who found the data to be adequate for the identification of major constituents in the oils. Michel (*416*) studied the carbonyl Raman bands of a large number of esters, ketones, and aldehydes. Briner *et al.* (*69*) investigated the Raman and infrared spectra of derivatives of eugenol, anethole, and estragole.

The x-ray diffraction patterns of various essential oils were obtained by Mohan (*427*), and the interplanar distances were utilized to identify the main constituents in the oils. An attempt was made to classify essential oils into those that contain compounds having aliphatic, cyclic, and sesquiterpenic structures.

Calvarano (*82*) reviewed the theory of rotatory dispersion and its experimental application to essential oils.

Luedde (*385*) proposed the drum number according to Gordienko as a new constant for the characterization of essential oils; Franchi and Franchi (*178*) proposed the propionyl number.

The methods of colorimetry and their application to the analysis of essential oils were discussed by Nigam and Kumari (*478*).

A semimicro and micro steam distillation method for the estimation of oil content of small samples of botanicals was devised by Franklin and Keyzer (*179*). Kartha (*311*) and Wasicky (*745*) both reported modifications of oil traps used in such determinations. A new iodometric method for the estimation of oil content was exemplified by Kartha and Misra (*313*). The method, being operable on a micro and semimicro scale, is particularly suitable for oils which are difficult to distill, such as vetiver oil, or which are present in low percentage, such as rose and jasmine oils. Kiseleva and Voitkevich (*326*) reported an extraction technique for estimating oil content. The Essential Oils Subcommittee, as reported in

The Analyst (*157*), compared drying agents for essential oils and found $MgSO_4$ to be superior.

Individual Essential Oils. From *Abies sibirica* oil Porsch and Farnow (*552*) isolated tricyclene and established its structure. Sesquiterpenes from the same plant were identified by Chirkova and Pentegova (*93*).

The oil of *Achryocline satureioides* was investigated by Ricciardi, Cassano, and Burgos (*573*) and some of its constituents were identified.

The composition of agarwood oil was examined by Jain, Maheshwari, and Bhattacharyya (*294*), and Maheshwari *et al.* (*391*, *392*) isolated and characterized several sesquiterpenic furans from the oil.

Benzyl acetate and benzoate were found in the oil from *Albizzia lebbeck* flowers by Jain and Mishra (*293*).

The properties of the oil of *Alpinia galanga* were ascertained by Trabaud (*716*) who identified several of its main constituents. Nigam and Radhakrishnan (*483*, *484*) reported quite different properties for an oil from the same botanical grown in Bengalore, from which they isolated a new sesquiterpene hydrocarbon of unusually low specific gravity (25/25 = 0.7743).

Oil of *Amomum subulatum* was examined chromatographically by Nigam and Purohit (*479*), and several constituents were identified. The removal of cineol and coloring matter was necessary before chromatography could be successfully accomplished.

Amorpha fruiticosa oil consists mostly of hydrocarbons, according to the spectrophotometric analysis conducted by Topalov, Ivanov, and Georgiev (*715*).

Angelica oil was investigated by Parczewski (*517*) who found seven constituents. Mitsuhashi and Itoh (*422*) isolated edultin from *Angelica edulis* oil; Tanaka (*704*) reported five compounds, including tiglic and angelic acid, in an oil obtained from *Angelica pubescens*.

The "thymene" fraction from ajowan oil was examined by Nigam, Skakum, and Levi (*473*), using column and liquid partition chromatography, and by Bhargava and Haksar (*45*) employing fractional distillation.

Araucaria oil was shown by Bates and Hendrickson (*30*) to contain several major constituents, among which was γ-eudesmol.

Oils from the Artemisia species have been extensively investigated. Nakajima (*440*) isolated a number of components from oil of *A. annula;* Suchy (*673*) determined the structure of balchanin from oil of *A. balchanorum;* Guven (*223*) reported the properties and quantitative composition of the oil of *A. campestria;* Vashist, Nigam, and Handa (*729*) investigated an oil distilled from *A. grata* and quantitatively determined many of its components; Goryaev and Bazalitskaya (*197*) reported the composition of oil of *A. kaschgarica;* Fujita, Ueda, and Maruyama (*183*) analyzed the oil of *A. kurramensis;* Gimaddinov, Lishtvanova, and Goryaev (*189*) found the percentages of many constituents in oil of *A. porrecta;* Goryaev and Ignatova (*199*) similarly investigated oil of *A. tennisecta;* and Rao and Sood (*565*) determined the properties and some components of *A. vulgaris*. Specific new constituents identified in Artemisia oils were absinthin and artabsin in oil of *A. sieversiana*, by Novotny and Herout (*494*); estafiatin in oil of *A. mexicana*, by Sanchez-Viesca and Roma (*615*); austricin, erivanine, and other sesquiterpene lactones from *A. austriaca*, *A. fragrans*, and *A. leucodes*, by Rybalko and his coworkers (*604*, *603*, *602*, respectively). The lactone, deacetoxymatricarin was isolated from *A. leucodes* by Holub and Herout (*268*), the structure of hydroxycostunolide from *A. balchanorum* was elucidated by Krasch *et al.* (*348*), and Pigulevskii and Kovaleva (*540*) found a new sesquiterpene in the oil of *A. taurica*.

The composition of the oil from *Aster tataricus* was reported by Nishimura and Hirose (*486*).

Balsam Peru was shown, by thin layer and gas chromatography, to contain benzyl benzoate and cinnamate, nerolidol, and vanillin, according to Frauendorf and Auterhoff (*180*). Several other constituents were absent.

Five alcohols from bay oil were separated and identified by preparation of the borates, distillation, and chromatography, as reported by Lamparsky (*373*).

Theile, Dean, and Suffis (*711*) correlated ultraviolet and infrared data with gas chromatography to provide an accurate and rapid method for the detection of adulteration in bergamot oil. Sundt, Willhalm, and Stoll (*680*) analyzed the acid–lactonic portion of bergamot oil, confirming past results, and discovering numerous new components including *cis*- and *trans*-jasmone and dihydrojasmone. Calvarano (*85*) quantitatively determined the monoterpene hydrocarbon composition of the oil.

Bois de rose oil was intensively investigated by Nigam and Levi (*469*), who identified citral and linalool oxides among many new constituents found in the oil. Gottlieb *et al.* (*207*) conducted a detailed study of the oils from different parts of the tree and of various species. Chiurdoglu *et al.* (*94*) isolated and characterized several oxygenated sesquiterpene compounds from the oil.

The calamus oil constituents, acorone, isoacorone, and cryptoacorone, were characterized with the aid of the Hudson-Klyne rule by Vrkoc *et al.* (*737*). The same authors (*739*) also determined the structure of the newly isolated calarene, and Vrkoc, Herout, and Sorm (*736*) clarified the structure of acorone and its stereoisomers. Indian calamus oils from Jammu and Cashmir were compared by Vashist and Handa (*726*) who gave the constants and the compositions of both oils.

Rudman (*601*) showed that *Callitris columellaris* contained guaiol, α-, β-, and γ-eudesmol, and cryptomeridiol.

A camphor oil distilled from the leaves and twigs of *Cinnamomum camphora* was found by Maurel (*411*) to consist of 90% alchohols, mostly linalool. Twenty-four components were detected. Hirota and Hiroi (*260*), investigating the leaf oil from the same plant grown in Formosa, reported finding nerolidol, fumurene, α-pinene, and cineol, but little linalool and no terpineol or safrol. Fujita (*182*) distinguished between *Cinnamomum micranthum* and *C. kawahirai* by the differences observed in the composition of their essential oils.

An oil distilled from *Canarium euphyllum* was described by Das and Saha (*116*) who recommended its use for perfume.

Brazilian carqueja oil was investigated by Naves (*448*). Though many constituents were identified, no hydrocarbons having the *o*-menthane structure were present.

The composition of oil of carrot seed was quantitatively ascertained by Zalkow, Park, and Ellis (*772*), using gas chromatography and nuclear magnetic resonance. Pigulevskii and Motskus (*541*, *542*) found complex esters of geraniol as the main part of the oil of wild carrot growing in Osetinsk. They also determined many other minor constituents and isolated a sesquiterpene alcohol of the selinane type and determined its structure. Pigulevskii, Motskus, and Rodina (*543*, *544*) also reported on the properties and composition of an oil from seeds grown in the Tashkent region and elucidated the structure of carotol. Parczewski and Rajkowski (*519*) identified some constituents, separated by fractionation, and determined the properties of an oil from the ripe fruits of carrot. Soucek (*658*) studied the biosynthesis and structure of carotol and daucol by radioactivity distribution. Starkovsky (*665*) found 2,4,5-trimethoxybenzaldehyde to be the bitter principle in the seeds. Talwar, Nigam, and Handa (*698*) determined the properties and many constituents of Indian carrot seed oil, and Nigam and Radhakrishnan (*481*) identified daucol and carotol in the same oil.

A method for the determination of *o*-methoxy cinnamaldehyde, in cassia oil based on infrared absorption, was described by Chowdhury and Williams

(*104*). They also distinguished between oils from the bark of *Cinnamomum cassia* and those from the leaves and twigs by a comparison of their infrared spectra.

The yield, properties, and composition of Russian catnip oil were investigated by Kulesza, Roth, and Jaworski (*358*). Gopinath, Prakash, and Kidwai (*196*) examined the oil from *Nepeta hindustani* and isolated oleanolic acid. Bates and Sigel (*31*) separated the steroisomers of nepetalactone from catnip oil by gas chromatography.

Caucalis antriscus oil was chromatographically and chemically examined by Talwar and Handa (*695*) who reported many constituents including 4% *p*-methoxy benzyl acetate.

Cedrol and α- and β-cedrene in red cedarwood oil were estimated chromatographically by Nigam and Handa (*474*).

Among 38 volatile compounds identified in celery, Gold and Wilson (*192, 193*) found that several phthalides, *cis*-3-hexenyl pyruvate, and diacetyl are primarily responsible for its flavor. Barton and De Vries (*25*) found *n*-butyl phthalide and sedanonic acid in the high fractions from celery oil, but no sedanolide.

Oil of *Ceratocystis coerulescens*, according to Sprechler (*661*), contained 6-methyl-5-hepten-2-one, among many other constituents.

The yield of oil from German chamomile grown in Egypt was determined by Fahmy and Hamidi (*158*). The oil and azulene content in French and Hungarian chamomile infusion bags was colorimetrically estimated by Lewe (*377*).

The properties and many constituents of champaca oil were reported by Chopra and Handa (*95*).

Chrysanthemum indicum oil distilled from the whole plant was found by Pascual, Sanchez, and Sanchez (*525*), to represent a new source of chrysanthenone. Demura (*123*) isolated β-3-carene from the oil distilled from the flowers.

Kerala and Mysore cinnamon leaf oils were examined by Bhramaramba and Mahboob (*48*) who identified some of the constituents. From the nonphenolic portion of the oil, Bhramaramba and Sidhu (*49*) isolated, using gas and thin layer chromatography, caryophyllene, isocaryophyllene, and humulene, and, by column chromatography, linalool, *o*-methyleugenol, caryophyllene, humulene, isocaryophyllene, and benzyl benzoate.

Vashist, Nigam, and Handa (*728*) reported that the distillation waters from citronella grass grown in Jammu and Kashmir yielded an oil containing, among many other compounds, 12.1% perillaldehyde, 10.7% *l*-carvotanacetone, 8.4% phellandral, and 2.3% furfural. Schmidt, Bernhardt, and Muehlstaedt (*621*) found Vietnamese citronella oil to contain more citral than the commercial Java variety. Johnston (*302*) found that the product of the acetylation of citronellal in the total alcohol determination is principally citronellal enol acetate.

The applications to citrus oil analysis of the latest techniques involving thin layer, gas chromatography, and spectrographic methods were discussed and exemplified by Stanley (*664*). Determination of the terpene composition was illustrated by Ikeda *et al.* (*283*). DiGiacomo, Rispoli, and Tracuzzi (*136*) used gas chromatography and infrared spectroscopy to elucidate the terpene composition of Italian citrus oils. Rogers (*583*) evaluated instrumental methods as related to citrus oil analysis and reported *p*-cymene to be the odor principle of spoiled lemon oil. Safina (*610*) discussed methods of analysis of citrus oils. Wolford and Attaway (*757*) investigated the volatile flavor compounds of citrus juice by gas chromatography. Two instruments with several different columns were employed, and infrared and chemical identifications were utilized. Temperature programmed runs showed 35 to 40 resolved constituents, while isothermal analyses indicated only 18 to 23. Rispoli, DiGiacomo, and Tracuzzi (*579*) studied and interpreted the compositions of the 10% distillate. Monselise (*429*) developed a rapid colorimetric estimation of peroxides in citrus oils, and Hunter (*275*) identified monoethoxydiethylene glycol as an adulterant.

The properties and composition of Argentinian oil of *Cordia chacoensis* were determined by Ricciardi, Cassano, and Burgos (*574*).

Coriander oil separated from aqueous solutions was examined and its main constituents were determined by Makarova and Borisyuk (*393*). Citral in oxidized coriander oil was determined spectrophotometrically by Polyakov and Falina (*548*), who reported that the method is more precise than determination by polarography or oximation.

Costus root oil was examined by Kulkarni *et al.* (*360*), who found a number of lactones in one of the fractions. Sharma, Nigam, and Handa (*637*) determined the properties and composition of the same oil. Hikino *et al.* (*259*) elucidated the structure of mokko and dehydrocostus lactones.

An essential oil from *Cocculus hirsutus* was partially examined by Merchant, Naik, and Hirwe (*415*).

The properties and composition of oils of *Crypotcarya aschersoniana* and *C. moschata* were reported by Naves *et al.* (*453*).

Seventeen compounds comprising 71% of the oil of black currant leaves were identified by Andersson, Bosvik, and Sydow (*5*).

Cymbopogon densiflorus oil was found by Burger (*76*) to contain a high percentage of perilla alcohol and carvone.

Patchoulenone was isolated from *Cyperus rotundus* oil, and its structure was determined by Motl *et al.* (*437*). Senic (*631*) found the oil to contain two new sesquiterpenes, cyperene-1 and cyperene-2. Kapadia *et al.* (*307*) established the structure of muskatone and copaene isolated from the same oil.

A cypress wood oil was shown by Naves (*452*) to contain about 66% carvacrol methyl ether, identified by infrared spectrography.

Dill oil recovered from cohobation waters contained 76% carvone, as well as other oxygenated constituents, according to Talwar, Nigam, and Handa (*699*).

Loder (*383*) isolated polygodial and guaiol from *Drimys lanceolata* oil.

Ephedra sinica gave a fragrant oil from which Liu (*382*) isolated *l*-α-terpineol.

Eucalyptus citriodora oil from Jammu was found by Sharma and Handa (*635*) to contain 50% citronellal, among other substances.

The composition of *Eucalyptus dives* oil was quantitatively determined by Farnow and Porsch (*160*) who employed a combination of techniques. Blumann *et al.* (*53*) determined the structure of *p*-menthenediols isolated from the oil; and Blumann, Gallagher, and Sutherland (*54*) identified terpinene-4-ol in it.

Hellyer and his coworkers (*240–242*) determined many constituents of oils of *Eucalyptus crenulata*, *E. ablonga*, and *E. mitchelliana*, and established the structure of tasmanone which they isolated from the oils.

A greenish blue fragrant oil was obtained from *Ferula jaeschkeana* roots by Vashist *et al.* (*727*) who found it contained a compound similar to *S*-guaiazulene.

Geranium oil was analyzed, using gas chromatography, by Howard (*273*), who found that polyethylene glycol 400 was the most effective of five stationary phases employed. The functional groups were identified by comparing chromatograms of the oil before and after treatment with specific reagents. Peyron (*534*) determined the properties and composition of Moroccan geranium oil obtained by extraction of the distillation water. He also (*535*) compared Moroccan oil with Algerian oil, and gave methods for determining various compounds including sulfur compounds. Sharma and Handa (*634*) investigated the characteristics and composition of the oil from geraniums raised in Jammu and Kashmir. van Os (*512*) applied the formulation technique to the determination of citronellol and geraniol and studied methods to compensate for its inaccuracies. Ognyanov, Mkhailov, and Burogudzkiev (*501*) determined germacrone in geranium oil by ultraviolet spectroscopy. Ota, Nishimura, and Hirose (*514*) reported seven new constituents in the monoterpene fraction; Benesova *et al.* (*36*) established the structure of 3,7-guaiadiene; Naves *et al.* (*460*) isolated and characterized 2-acetonyl-4-methyl tetrahydropyran; Romanuk *et al.* (*585*) isolated and elucidated the structure of pelargone; and Wolff, Ma, and Lukas (*755*) isolated pelargones A and B and determined their structures, reporting their ultraviolet and infrared spectra, and rotatory dispersions.

Some new constituents of wild ginger oil were identified and estimated by Nigam and Levi (*467*), using column and gas chromatography. Ramaswami and Bhattacharyya (*561*) separated and characterized humulene monoxide and dioxide from the sesquiterpene fraction of the same oil. Varma, Jain, and Bhattacharyya (*725*) established the stereochemistry of zingiberol and juniper camphor from the higher boiling fractions of ginger oil.

Hyderabad gingergrass oil was shown to contain (+)-perillyl alcohol as well as two *p*-menthadieneols by Swaleh, Bhushan, and Sidhu (*685*), who used paper chromatography and other techniques. Swaleh, Srivastava, and Bhushan (*686*) reported color reaction which distinguished between gingergrass and palmarosa oils.

Goryaev and Yurima (*206*) determined several compounds in the terpenes from the oil of *Glycyrrhiza triphylla*.

Oils related to goldenrod were investigated by Krepinsky and Herout (*349*), who elucidated much of the composition of the oil from the roots of *Solidago canadensis*, and by Knuetter and Pohloudek-Fabini (*339*) who, by paper chromatography and ultraviolet spectrophotometry of the eluate, identified matricaria ester from the roots of *S. virgaurea*.

Oils of red and white grapefruits were compared as to properties, ultraviolet absorption, and composition by Kesterson and Hendrickson (*322*). The same authors (*321*) correlated organoleptic evaluations of the oil of marsh grapefruit at various stages of maturity with detailed chemical and instrumental analyses. Hunter and Brogden (*276*) developed a rapid method for isolating and identifying the sesquiterpenes from grapefruit oil. It involves molecular stripping of low-boiling components and removal of oxygenated compounds with basic Al_2O_3. Rispoli, DiGiacomo, and Tracuzzi (*578*) determined the composition of Sicilian grapefruit oil by gas and thin layer chromatography and infrared spectroscopy.

Components of guaiacwood and patchouli oils were isolated and characterized by Bates and Slagel (*32*), who ascertained the structures of β-bulnesene, α-guaiene, β-patchoulene, and guaioxide. β-Gurjunene from oil of gurjun balsam was shown to be identical with calarene by Vrkoc *et al.* (*738*), and by Streith, Pesnelle, and Ourisson (*671*), who also (*672*) clarified the stereochemical structure of α-gurjunene.

The physicochemical properties of oil from *Helichrysum graveolens* were reported by Baytop (*33*). Romo, Joseph-Nathan, and Diaz (*586*, *587*) isolated aromatin and aromaticin from oil of *Helenium aromaticum* and established their structures, utilizing nuclear magnetic resonance spectra. Romo, Romo de Vivar, and Herz (*588*) isolated mexicanin E from *Helenium mexicanum*.

The physicochemical properties and main constituents of the oil of *Heracleum candicans* were given by Charma, Nigam, and Handa (*90*).

The structures of compounds in oil of hiba wood have been clarified by Norin (*487*, *489*), who determined the configurations of thujopsene and hinokiic acid, and by Kitahara and Yoshikoshi (*329*, *330*), who established the structures of hibaene and dolabradiene.

The monoterpene constituents of hinoki oil were quantitatively determined by Hayashi, Yano, and Matsuura (*236*).

Extensive investigations have been conducted on the composition of hop oil. Jahnsen (*291*, *292*) found about 200 compounds. He used chromatography, solvent extraction, and the formation of chemical derivatives prior to resolution by gas chromatography. Most of the identifications, based on retention time, were tentative. Buttery *et al.* (*79–81*) combined gas chromatography with mass spectrography and identified many constituents, including methyl dec-4-enoate and methyl deca-4,8-dienoate.

Roberts (*581*) identified many components of the nonsaponifiable fraction. Shigematsu and Kitazawa (*641*) investigated Japanese hop oil. Likens and Nicherson (*378*) studied the influence of compression on the oil and found that the myrcene content varied directly with compression. Burton and Stevens (*77*) elucidated the structure of hulupinic acid; Rillaers and Verzele (*576*), that of prehumulone; and Stevens and Wright (*668*), those of the lupulones and the hulupones.

Sutherland and Waters (*682*) studied the structure of humulene; McPhail, Reed, and Sim (*390*) established its stereochemistry; and Hartsuk and Paul (*233*) used x-ray analysis to clarify its molecular structure. Stevens (*667*) investigated the oil from an abnormal hop variety, and found it to consist principally of myrcene and β-selinene; and Spetzig (*660*) described an apparatus for partition chromatography and its application to the separation of the bitter substances of hops.

Many constituents of oils from 35 Hypericum species were identified by Mathis and Ourisson (*407*), using gas and thin layer chromatography. They described a technique for obtaining chromatograms of the volatile oil directly from the plant material.

Hyssop oil from Indian plants was analyzed, using classic chemical methods, by Sharma, Nigam, and Handa (*638*), who identified a number of its terpene constituents.

The oil of *Inula grandis*, like the oil of *I. helenium*, has alantolactone as its main constituent, as reported by Yudovich (*771*).

Jasmine oil was found by Demole (*122*) to contain methyl jasmonate, but not the ketolactone reported by Naves. However, Naves and Grampoloff (*456*, *457*) reaffirmed their previous findings with new data, and the three authors (*458*) agreed that the ketolactone actually occurs in the oil of jasmine and that methyl jasmonate could be identified in absolute of jasmine.

Winter *et al.* (*752*) reported the isolation and structure determination of (−) *cis*-5-(2-pentenyl) pentanolide-(5,1) from the same oil.

The major chemical constituents of the oils of *Juniperus depressa* and *J. ablonga* were identified by Goryaev *et al.* (*200*), who (*205*) also identified β-cedrene in oil of *J. semiglobosa Rgl.* Talwar, Nigam, and Handa (*701*) reported the major components of *J. macropoda* oil. Tucakov (*720*) measured the optical rotation of 100 samples of Yugoslavian juniper oils and found that it varied between −16° 18′ and +11° 56′.

Kewda oil, one of the popular perfume oils in India, was investigated by Sadgopal (*609*). He found the methyl ether of phenyl ethyl alcohol as its main constituent.

Several hydroxy compounds and esters were identified in the neutral fraction of concrete of labdanum by Tabacik-Wlotzka, Mousseron, and Chafai (*688*) and by Tabacik-Wlotzka alone (*687*).

The composition of *Laserpitium hispidum* oil was partially ascertained by Deryng and Parczewski (*125*) using fractionation and column chromatography.

Lavender and lavandin oils were studied by gas chromatography with different stationary phases by Kolsek and Maticic (*345*). Castor wax gave the most efficient separation. Naves and Tullen (*461*) identified (+)-nopinene and sabinene in lavender oil. Thin layer chromatography was employed by Hoerhammer and Wagner (*265*) to determine the quality and purity of commercial lavender oils.

Montes (*432*) used gas chromatography for the same purpose.

A new sesquiterpene, for which they proposed the empirical formula $C_{15}H_{25}$, was isolated from lawang oil by Suga and Watanabe (*676*).

Lemon oil production around the world was described by Guenther (*215*, *216*). DiGiacomo and Rispoli (*132*) compared sponge and machine pressed lemon oils by gas chromatography. DiGiacomo, Rispoli, and Crupi (*134*) determined the quantitative terpene composition of Sicilian lemon oil. Slater (*649*) showed that infrared spectra together with other data can be used to evaluate Sicilian lemon oils and to detect certain adulterants. Thin layer chromatography was employed to analyze lemon and caraway oils by El-Deeb, Karawya, and Wahba (*152*). Ikeda *et al.* (*281*) used a combination of techniques to isolate and identify whole series of new aldehydes in lemon oil, and Ikeda and Spitler (*282*) determined new esters and alcohols. Montes (*431*) detected the addition of lemongrass citral by gas chromatographic determination of the geranial-to-neral ratio. DiGiacomo and Rispoli (*133*) found heptenone in distilled lemon oil; and Rispoli, DiGiacomo, and Crupi (*577*) related *p*-cymene concentration to adulteration of the oil.

The physicochemical properties and main constituents of lemongrass oil from Ceylon were reported by De Silva (*128*).

An aromatic oil from the fruit of *Libanotis intermedia* was examined by Solodovnichenko (*655*) who reported its properties and some of its constituents. Pigulevskii and Borovkov (*538*, *539*) isolated and identified β-elemene and several other sesquiterpenes from oil of *Libanotis transcaucasica*.

Distilled lime oil was subjected to exhaustive investigation by Kovats (*347*). Using various extractions followed by distillation and chromatography, he identified 44 compounds, including 2,6,6-trimethyl-2-vinyltetrahydropyran which was found for the first time in essential oils. Srivas, Pruthi, and Siddappa (*663*) studied the effect of maturity and storage of the fruit on the volatile oil content.

The composition of the oil of *Lindera strychnifolia* leaves was investigated by Motl and Lukes (*436*).

Lippia geminata leaves yielded an oil containing 50% lippione, as found by Kunth, Shukla, and Rao (*364*). Fester *et al.* (*167*, *171*) distilled and compared the oils from *Lippia alba* growing in various parts of Argentina. Though some were similar, the composition of others varied surprisingly.

Gas chromatography of the oil of *Litsea odorifera*, conducted by Mathews, Pickering, and Umoh (*406*), showed it to contain a high proportion of methoxy monanyl acetylene.

Mandarin oil was fractionated and examined by gas chromatography by Kugler and Kovats (*356*). The 48 identified compounds constituted 99.2% of the volatile part of the oil. Mukherjee and Bose (*438*) compared mandarin oils from Sikhim, Nagpur, Sylhet, Coorg, Italy, and Brazil, as well as tangerine oils from Brazil and Florida. Parekh *et al.* (*521*) suggested the active oxygen manometric method as a measure of stability and quality of mandarin oil. Parekh *et al.* (*520*) and Pruthi *et al.* (*558*) made extensive studies of storage stability as evidenced by physicochemical properties including the A.O.M. value and the peroxide number.

The monoterpene constituents of the oil from the leaves of *Mangifera indica* were indentified by Nigam, Nigam, and Dhingra (*470*).

Some main constituents of oil of Marjoram from plants grown in Jammu were determined by Vashist *et al.* (*731*).

Oils of the Mentha species, namely peppermint, Japanese mint, spearmint, pennyroyal, and peperina were analyzed by Manfredini and Montes (*398*), using gas chromatography as a quality control method, with particular emphasis on relative retention volumes to methone. Quantitative analysis of peppermint and *Mentha arvensis* oils by ultraviolet spectrophotometry was performed by Manfredini, Mizrahi, and Montes (*397*). Menthol, menthone, pulegone, and carvone were determined. Nine new compounds were found in Japanese mint oil by Kobayashi, Yoshigi, and Kasuhara (*343*), using gas chromatography and infrared spectrometry; four more compounds were identified by Katsuhara, Hashimoto, and Kobayashi (*316*). Katsuhara and Kobayashi (*317*) found myrcene in Japanese mint, Mitcham type peppermint, and spearmint oils. Shimizu and Ikeda (*643*) found (+)-isomenthone in various Mentha species.

Eight varieties of mint oils were subjected to detailed chemical examination, and their important constituents were determined by Chopra, Vashist, and Handa (*98*). Gulati, Singh, and Dimri (*218*) investigated the oil and menthol content of *Mentha arvensis* grown in various parts of India. Tanker (*705*) determined the physicochemical properties of peppermint oil from Akhisar. Talwar *et al.* (*703*) gave the properties and composition as ascertained after chromatography of oil from *Mentha arvensis* grown in Kashmir. Malla *et al.* (*396*) similarly investigated oils from *Mentha arvensis* cultivated in Jammu and Kashmir. Malik and Khan (*394*) determined the oil and menthol content of *Mentha piperita*, *M. arvensis*, and *M. viridis*. Onogaki and Uruishido (*511*) investigated the metal content of mint oil which had become colored upon storage. Talwar, Nigam, and Handa (*700*) studied the composition of an oil from the distillation water of *Mentha arvensis*. Menthone and menthol in *Mentha arvensis* were determined with an accuracy of 0.4% by Malla, Nigam, and Handa (*395*). The technique was based on the conversion of menthone to its dinitrophenyl hydrazone which was separated by chromatography and assayed by absorbance. The linalool content of six species of Mentha was calculated by the difference between total alcohol and primary and secondary alcohols by Kubrak (*354*). Kulesza, Cybulska, and Gora (*357*) investigated the same types of oils and reported their properties and chemical composition. The oil content of milfoil in various stages of growth was reported by Oswiecimska (*513*).

An essential oil of *Morina longifolia* was obtained in a 0.34% yield, and its composition was investigated by Talwar and Handa (*693*). Levand (*376*) determined the principal chemical constituents of *Morinda citrifolia oil.*

Mustard oils from Cruciferae seeds were tabulated with respect to composition by Kharchenko (*323*), who investigated oils of Indian mustard seeds, black mustard seed, and rape seed. Delaveau (*121*) investigated the mustard oil of *Dentaria pinnata Lmk.*, and found that it contained (*s*)-2-methylbutyl isothiocyanate.

An analysis of Indian myrtle oil was reported by Singh and Gupta (*647*) who identified some of its constituents.

The composition of an oil from *Narcissus tazetta var. chinensis* was elucidated by Sakai, Nishimura, and Hirose (*612*), who employed infrared spectra and gas and elution chromatography.

Nardostachys jatamansi oil, Chinese spikenard oil, was investigated by Pesnelle and Ourisson (*531*) who, by repeated chromatography, isolated aristolene, and by Naves (*449*) who established the presence of maaliol and valeranone.

Nepeta ciliaris oil was distilled and characterized by Talwar and Handa (*692*), and *Nepeta leucophylla* oil by Gupta *et al.* (*221*).

Neroli oil from Calabria was subjected to gas, paper, and column chromatography, as well as to spectrometric procedures, by Calvarano (*84*), who concluded that it was similar to the best French oils.

The essential oil from *Nigella sativa* seeds was shown by El-Dakhakhny (*151*) to contain thymoquinone, as indicated by mixed melting point, R_f value, and ultraviolet and infrared spectra.

The organoleptic quality of nutmeg oil was related to its chemical composition, as indicated by gas chromatography, by Lee *et al.* (*375*), who identified 15 of the 33 constituents found. Organoleptically unacceptable oils contained significantly less limonene and linalool. Shulgin (*645*) identified several compounds in the myristicin fraction. Jaureguiberry and Wolff (*300*) isolated (+)-sabinene and gave its ultraviolet, infrared, and nuclear magnetic resonance spectra. Bejnarowicz and Kirch (*34*) analyzed East Indian and West Indian nutmeg oils by gas chromatography, and reported significant differences in composition.

Ocimum gratissimum oil from Taiwan was found by Lin *et al.* (*379*) to be the eugenol type. Chowdhri and Haksar (*102, 103*) found a high percentage of camphor in oil *Ocimum kilimandscharicum.*

Ocotea cymbarum oils from different regions of Brazil and from different parts of the tree were studied as to odor and physicochemical properites by Mollan (*428*). Gottlieb, Fineberg, and Magalhaes (*208*) continued their work on the odor and composition of oils from *Ocotea pretiosa.*

Some constituents and properties of Olibanum oil from the Sheopur region were reported by Bhargava, Chowdhri, and Haksar (*44*).

The sesquiterpene fraction of *Olearia paniculata* oil was shown by Corbett, Jamieson, and Murray (*109*) to contain aromadendrene, arcurcumene, and curcumene.

Cold-pressed and distilled orange oils were fractionated and their hydrocarbons were identified by infrared, nuclear magnetic resonance, and mass spectra, by Teranishi *et al.* (*708*). The terpene fraction was also investigated by Mehlitz and Minas (*414*). Kesterson and Hendrickson (*320*) related the composition of Valencia orange oil to fruit maturity and flavor. They determined many oxygenated constituents, and found citronellal in Florida oil but not in California oil. Fernandez, Fagerson, and Nawar (*165*) applied gas chromatography to study the changes in orange oil upon aging and reported that the myrcene, isopulegol, and carvone content was affected. Boehm and Voelcker (*57*) found sterol esters, tangeretin, and another flavonoid, but no coumarins, in the nonvolatile fraction of cold-pressed orange oil. Hunter and Parks (*279*) isolated β-elemene, and Hunter and Brogden (*277*) identified 2,4-*p*-menthadiene, a new monoterpene, from cold-pressed Valencia orange oil.

The oil recovered from orange juice was analyzed by Wolford, Alberding, and Attaway (*756*), who resolved and identified 35 to 40 constituents using programmed temperature gas chromatography and three different columns. The method was general for the analysis of citrus fruit oils. Wolford *et al.* (*758*) compared the oils recovered from the juices of Hamlin, Pineapple, and Valencia oranges. About 50 constituents were identified, but no significant qualitative differences were found, and quantitative differences appeared to be responsible for the variation in flavor among the three varieties. Attaway, Wolford, and Edwards (*18*) used liquid-liquid extraction to obtain the oil from orange essence and identified the carbonyl compounds in the oil by gas chromatography, and by their dinitrophenylhydrazones. Attaway, Wolford, and Alberding (*16*) also identified many volatile alcohols and acids by programmed temperature gas chromatography and other techniques.

The terpenes from bitter orange oils were extensively studied by Mehlitz and Minas (*413*) who used column and gas chromatography, along with chemical treatment. The terpenes from Greek oils contained less β-pinene and more *p*-cymene than those from Spanish oils. DiGiacomo, Rispoli, and Tracuzzi (*135*) employed ultraviolet and infrared spectrometry and gas chromatography to distinguish between pure Sicilian bitter orange oil and adulterated oils.

The oil from Polish parsley seed was described by Rajkowski (*559*). He found it to contain 38% tetramethoxyallylbenzene besides other constituents.

The effect of light and heat on the volatile constituents of pepper were investigated by Wrolstad and Jennings (*759*). Many constituents of *Piper longum* oil were identified by Handa, Sharma, and Nigam (*230*), who, using repeated chromatography, isolated two new monocyclic sesquiterpenes. The structures of these compounds were later proposed by Handa, Nigam, and Sharma (*228*). An oil from pepper husks was examined by Sharma, Nigam, and Handa (*636*). Cortez (*112*) determined piperine in 10 kinds of black pepper spice; Genest, Smith, and Chapman (*187*) critically evaluated an ultraviolet method and a colorimetric method for the determination of piperine as well as its analog piperettine. Duro (*147*) described a new volumetric determination of piperine in pepper berries.

Peppermint oil was shown by McCarthy *et al.* (*388*) to contain isobutyraldehyde, isovaleraldehyde, and *trans*-2-hexenal in the low boiling fraction. Porsch and Farnow (*551*) discovered new differences between the composition of peppermint oil and that of *Mentha arvensis* oil. Karawya and Wahba (*308, 309*) separated the hydrocarbons, menthone, menthol, and menthyl acetate by double column and thin layer chromatography. Ognyanov and Vlahov (*502*) identified several nonterpene compounds in the terpene fraction from Bulgarian peppermint oil. Vashist *et al.* (*732*) chromatographed oils from plants grown in Jammu and Kashmir and reported the percentages of the main constituents. Deryng, Parczewski, and Walewska (*126*) determined the constants of Polish peppermint oil and many of its components, separated by fractionation. Sviderskaya (*684*) studied the ratio of crystalline to liquid menthol in relation to plant development. The menthols were isolated with borates. Sadgopal (*607*) studied the changes in the oil during maturation of the plant. Schroeder (*623*) related time of harvest to yield and quality of German peppermint oil. Solodovnichenko and Borisyuk (*656*) found azulenes and menthol in the residue from rectification of the oil.

An oil from the leaves of *Piper betle* was characterized by Nigam and Purohit (*480*) who quantitatively determined its composition.

Naves (*446*) demonstrated that the so-called pulespenone reported in pennyroyal oil by Pascual and Sanchez (*526*) is actually piperitenone.

Two new types of perilla oils from *Perilla naginataketoniferum* and *P. dillapioliferum* were investigated by Yeh (*764, 765*), who reported their constants and composition. Ueda (*722*) found matsutake alcohol, and Ueda and Fujita (*723*) observed egomaketone in the oil of *Perilla frutescens.*

A structure was proposed for abrotanol by Goryaev, Serkebaeva, and Bazalitskaya (*201*) who isolated it from the sesquiterpene fraction of oil of *Perovskia abrotanoides.* Goryaev *et al.* (*202, 203*) also determined the constants and much of the constitution of oils of *Perovskia scrophulariaefolia, P. abrotanoides,* and *P. angustifolia.*

A number of hydrocarbons from *Petasites albus* oils were separated by fractionation and identified by Hochmannova, Novotny, and Herout (*262*). Novotny, Herout, and Sorm (*496*) reported furanoeremophylan, petasalbin, and albopetasol in the same oil. Novotny *et al.* (*497*) also determined much of the composition of *Petasites officinalis* oil. Hochmannova, Novotny, and Herout (*263*) identified components in the sesquiterpene fraction. Novotny and Herout (*495*) studied the sesquiterpene composition of *Petasites spurius* oil and characterized albopetasin and petasalbin.

Petitgrain oils, as well as peel oils from four citrus species cultivated in Jammu and Kashmir, were described by Chopra *et al.* (*100*) who reported the constants and main constituents.

Pine needle oil from the West Himalayan *Picea morinda* was described by Rao and Sood (*566*), who determined a number of its main constituents. Chaudhary, Nazir, and Handa (*91*) ascertained the constants and composition of the oils of *Pinus excelsa, P. longifolia,* and *Cedrus deodara,* all of which are native to India. Ishikawa and Tsuchiya (*287*) determined 14 compounds in each of 11 oils from various Japanese conifers. Smith (*652*) found three terpenes, not previously reported, in the volatile oil from lodgepole pine oleoresin: limonene, sabinene, and α-phellandrene. Blight and McDonald (*52*) isolated *l*-sabinene from the oil of *Pinus muricata* and identified it by infrared and nuclear magnetic resonance spectra. The composition of a volatile oil from the heartwood of Norway pine was investigated by von Rudloff (*595*). Wang and Weinstein (*743*) studied the structure and relationship of some minor constituents of *Pinus parviflora* oil. Smith (*653*) found wide variation in the terpene composition of the oil from 64 different *Pinus ponderosa* trees. Enzell and Theander (*155*) isolated and assigned a structure to pinofolic acid from *Pinus silvestris* needles.

The composition of oil of *Pseudowintera colorata* was investigated by Corbett and Young (*110*), who elucidated much of its composition, and by Corbett (*108*) who identified four additional compounds separated by chromatography from a distilled fraction.

The $C_{10}H_{18}O$ compound previously isolated from Bulgarian rose oil was shown by Seidel and Stoll (*629*) to be (−) (4R)-*cis*-*2*-(2-methyl-1-propenyl)-4-methyl tetrahydropyran. El-Sakaa (*153*) isolated *n*-eicosane from the stearoptene of rose Damascus. Dranovskaya and Kondratskaya (*143*) showed that the concentration of ethyl ether in rose oil is directly related to the refractive index. Sakai, Nishimura, and Hirose (*613*) investigated commercial Japanese oil of *Rosa rugosa* and reported 15 constituents. Previous studies of the same oil were reviewed by Teshima (*709*).

Rosemary oil was investigated by Graf and Hoppe (*209*), who identified nine components including camphor and borneol. Schwenker and Kloehn (*626*) used fractionation, gas chromatography, and infrared spectrometry to identify 14 compounds in the same oil.

The oil of *Rhododendron adamsi* was investigated by Pigulevskii and Belova (*537*), who reported the infrared and ultraviolet spectra of the isolated compounds.

Sicilian rue oil was compared to other rue oils by La Bruto (*366*). Gonzalez and Estevez Reyes (*195*) quantitatively determined the main constituents of the oil from the fruit of *Ruta pinnata*.

The quality and chemical composition of Dalmatian sage oil was related to time of harvest by Janackovic (*296*), who found the best oil was obtained in October. The physicochemical properties of Greek sage oil were given by La Bruto (*367*), who reported that extraction with diacetin to remove terpenes was unsatisfactory. Calvarano and DeLeo (*83*) compared the oils of Italian sage obtained after the first and second year of seeding. Ultraviolet and infrared spectra and gas chromatograms were given.

The structures of β-santalol from sandalwood oil, as well as that of the lactone of tricycloekasantalic acid, were reviewed by Bhati (*46*).

The main constituents of Chinese sassafras oil were determined by Moryashchev and Voronin (*434*), who employed gas chromatography.

Commercial oil of savin was shown by von Rudloff (*594*), with the aid of gas chromatography, to be very similar to a Canadian juniper oil. He determined the composition of the oils quantitatively, and pointed out that a fraction having the typical pungent odor of juniper leaves consisted primarily of methyl *d*-citronellate.

The composition of *Selinum vaginatum* oil was determined by Handa, Nigam, and Sharma (*229*) using liquid and gas chromatography, infrared spectrometry, and chemical derivatives. The infrared spectra showed similarities to that of *Seseli sibiricum* oil, which Handa, Smith, and Levi (*231*) examined by gas chromatography, identifying many constituents, including fenchone, fenchyl alcohol, and fenchyl acetate, which were found for the first time in a plant of the Umbelliferae family.

Oil of *Schinus molle* was investigated by Bernhard and Wrolstad (*38*). Application of flame ionization detection with capillary column gas chromatography resulted in the identification of six terpene hydrocarbons not previously reported in this oil.

The oils of *Sium latifolium* obtained at various stages of maturity were examined and compared by Andrzej (*6*). *Sium latigugum* leaf oil and fruit oil were analyzed for the first time by Talwar and Handa (*694*), and several constituents were identified.

Formosan spearmint oil was fractionated and its principal components were chemically identified by Yeh (*767*). He (*766*) also studied the composition of oils from "Ryan-Fong" and a Japanese improved variety of plants, and isolated rotundifolone. Smith, Skakum, and Levi (*650*) employed gas chromatography and ultraviolet spectrometry to correlate the composition of spearmint oils with botanical and geographic origin. Shimizu and Ikeda (*642*) reported on the composition of the oil from various spearmint hybrids. Chopra, Nigam, and Handa (*96*) identified 11 components of spearmint oil from Kashmir.

Spruce oils from black, white, Colorado, Sitka, and Engelman spruce varieties were described by von Rudloff (*596*, *598*), who used gas chromatography employing several different columns to identify many constituents.

A number of Tagetes oils were investigated by chromatographic and spectrographic methods. Guzman and Manjarrez (*224*) isolated four main components from an ether extract of *T. florida* oil. Handa, Chopra, and Nigam (*227*) quantitatively determined twelve compounds in oil of *T. minuta*. Nigam, Kapoor, and Handa (*475*) described several Indian Tagetes oils. Chopra, Nigam, and Handa (*97*) determined nine constituents of *T. signata* oil. Boehm, Thaller, and Whiting (*56*) showed that tagetone from *T. glandulifera* is composed of *cis*- and *trans*-isomers.

A commercial and a local tansy oil were analyzed by means of gas chromatography by von Rudloff (*593*). Over twenty compounds were identified, and most of them quantitatively estimated. Yakunina (*761*) reported the oil content of various parts of the plant at different stages of development. Talwar *et al.* (*696*), described an oil from plants cultivated in Srinagar.

Oil of green tea leaves was analyzed by gas chromatography by Yamanishi *et al.* (*763*), who found, in addition to previously reported constituents, isoamyl acetate, salicylaldehyde, benzyl acetate, indole, skatole, and four unidentified compounds. Brandenberger and Mueller (*64*) identified a series of acids in the oil of black tea, and Gogiya (*191*) determined the oil content at various processing stages.

Oil of *Thuja plicata*, western red cedar leaf oil, was examined by von Rudloff (*597*) who used gas chromatography and infrared spectroscopy to determine its composition. Vashist *et al.* (*730*) described the oil of *Thuja orientalis* and determined the major constituents. In the same oil, Chetty and Dev (*92*) found new sesquiterpenoid ketones having cuparene-based structures. Nakazaki (*441*) determined the structure of (+)-occidol from thuja oil, and von Rudloff and Erdtman (*599*) isolated occidentalol and established its stereochemistry. von Rudloff and Nair (*600*) identified several minor constituents of the volatile compounds from the heartwood of *Thuja occidentalis*, and Sandermann and Schweers (*618*) studied the biogenesis of thujone in the tree by C^{14} labeling and gas chromatography.

Several varieties of thyme oil were discussed and their properties reported by Granger, Passet, and Verdier (*211*).

The recent investigations of the terpene constituents of tobacco were reviewed by Bhati (*47*). Roberts and Rowland (*580*) reported two macrocyclic diterpene diols. Tsonev and Chenikov (*718*, *719*) evaluated various methods for oil determination, and found that the oil content of tobacco decreased during fermentation.

Torreya nucifera oil from the leaves was investigated by Sakai *et al.* (*611*), who quantitatively reported the composition of the oil, and established the structure of a new compound, torreyol. The essential oil from the wood was also extensively investigated by Sakai, Nishimura, and Hirose (*614*).

In his investigation of turpentine oil from *Pinus khasya*, Fisher (*172*) found that only the laevorotatory isomer of β-pinene was present. Hirsjarvi and Pirila (*261*) reported differences in the α-pinene and 3-carene content of turpentine oils from Sweden and Finland. Mirov, Zaharin, and Bicho (*421*) identified several constituents of the turpentines from *Pinus nelsonii* and *P. occidentalis*. Witek and Bukala (*753, 754*) fractionated Polish turpentine from *Pinus silvestris* and reported its percentage composition. They identified *p*-cymene and γ-terpinene by infrared spectra. From Swedish turpentine, Erdtman and Westfelt (*156*) isolated α-longipinene, a sesquiterpene with a new skeleton structure. Kobayashi and Akiyoshi (*340, 341*) elucidated the structure of thunbergene from the turpentine oil of *Pinus thunbergii*. Nathorst-Westfelt (*445*) clarified the structures of (+)-ε-muurolene and (−)-ε-cadinene from Swedish sulfate turpentine, and Nayak and Dev (*462*) established the structure of longicyclene, the first tetracyclic sesquiterpene isolated from the turpentine oil of *Pinus longifolia*.

Recent research on the essential oils of Polish Umbelliferae was reviewed by Parczewski (*518*).

In studies on the composition of valerian oil, Krepinsky *et al.* (*350–352*) found jatamansone to be identical with valeranone from *Valeriana officinalis* oil, and elucidated the structure and configuration of valeranone using chemical evidence, the Hudson-Klyne lactone rule, and x-ray analysis. Sood (*657*) isolated maaliol from Indian valerian oil. Schultz and Eckstein (*624*) reported a new diester in the same oil. Narayanan *et al.* (*444*), made an extensive investigation of Indian valerian oil and identified many of its constituents. Kulkarni *et al.* (*362*) established the structure of the newly isolated β-bergamotene. Kulkarni, Paknikar, and Bhattacharyya (*361*) clarified the structure and stereochemistry of hydroxyvaleranone and acetylhydroxyvaleranone, new sesquiterpenes from Indian valerian oil. Seed and Karnek (*627*) reported the physicochemical properties of oil from cultivated roots.

Hikino *et al.* (*255*) distilled the oils from three varieties of Japanese valerian roots, as well as from kesso root (*253*), and determined their constants and many of their constituents. Ito *et al.* (*289*) assigned absolute configurations to α-kessyl alcohol and kessyl glycol. Hikino *et al.* (*254, 256–258*) established the structures of kanokonol, valeranone, kessane, and kessanol, all newly isolated from Japanese valerian oils.

Preliminary examinations of the oils from *Vateria indica* and *Saccopetalum tomentosum* were conducted by Nigam and Radhakrishnan (*482*).

The geographic origin of vetiver oils was detected by Rao *et al.* (*564*), who applied differential infrared spectroscopy to oils from the Reunion Islands, Haiti, Java, and India. Sadgopal (*608*) determined the yield of oil from vetiver roots of various origins and listed their physicochemical properties. Anh and Fetizon (*8*) reviewed the present state of knowledge of the sesquiterpenoids in vetiver oil. Kalsi, Chakravarti, and Bhattacharyya (*305, 306*) established the structures of isobisabolene and khusol; Kartha *et al.* (*314*) determined the structure and stereochemistry of (−)-γ_2-cadinene; Rao *et al.* (*563*) established the structure of khusinol; and Shaligram, Rao, and Bhattacharyya (*632*) proved the absolute configuration of levojunenol and junenol, isolated, like the other compounds mentioned, from vetiver oil.

Nilov (*485*) obtained an oil from grape vines in 0.002% yield. This oil when added to wine enhanced its bouquet and stability.

The structure of aritasone, isolated from wormseed oil, was clarified by Nakajima (*439*).

Fractionation and chromatography of wormwood oil were employed by Goryaev, Bazalitskaya, and Lishtvanova (*198*) to establish the percentage content of myrcene, *d*-α-pinene, thujyl alcohol, nerol, and isothyjyl acetate.

Various qualities of commercial ylang ylang oils were extensively analyzed by Naves and Ardizio (*454*), who compared them with previously described absolute oils, and found the geranyl acetate content to be low. Katague and Kirch (*315*) also identified a number of constituents from ylang ylang oil by means of gas chromatography.

New humulene based sesquiterpenoids were isolated from oil of *Zingiber zerumbet* by Damodaran and Dev (*115*), who established their structures with the aid of nuclear magnetic resonance and infrared spectra, as well as chemical studies.

Essential oils from plants grown in West Pakistan were discussed as to yield and quality of oil by Ahmad, Bhatty, and Karimullah (*1*). Orange, lemon, *Cymbopogon jwarancusa*, Artemisia, juniper, ajowan, black and white caraway, fennel, dill, corander, *Lantana indica*, and peppermint oils were described. The oils obtained from four Umbelliferae plants, also grown in Pakistan, were analyzed by Wahid and Ikram (*742*). Lukic, Savin, and Gorunovic (*387*) distilled the oils from several wild herbs native to Yugoslavia. La Bruto and Calvarano (*368*) reported the properties, including ultraviolet spectra, of oils from lemongrass, myrtle, basil, and thyme grown in Sicily. Fester *et al.* (*169*) reviewed results of the examination of 77 Argentine essential oils, and also (*170*) described two oils from *Lippia alba* from the Santa Fe area. Fonseca (*176*) gave the properties and some constituents of several other oils obtained from aromatic plants in the state of Corrientes. Montes (*433*) reported a study of the oils of cinnamon, cassia, ylang ylang, and lemongrass. Jansen and Van der Holk (*297*) identified small quantities of rue oil and peppermint oil by characteristic peaks.

Acids. Aromatic acids were detected by a new paper chromatographic technique developed by Grant (*212*), in which a 0.3% solution of H_2O_2 is used for development. Rogozinski (*584*) found that the H_2SO_4–methanol method compared favorably with the diazomethane procedure as a rapid esterification technique for determining carboxylic acids. Shelley, Salwin, and Horwitz (*639*) determined volatile carboxylic acids in foods by steam distillation, neutralization, and drying, followed by gas chromatography.

Methods for making the oxime, semicarbazone, and thiosemicarbazone of pinolic acid were given by Avotins, Osipov, and Vitols (*19*).

Indole-3-acetic acid was found in the bark of balsam fir by Clark and Bonga (*106*).

Aldehydes and Ketones. GENERAL METHODS. A gas chromatographic column containing poly(ethylene isophthalate) and polyethylene glycol 4000, operating at 205° C., was employed by Prabucki and Lenz (*556*) to separate aromatic aldehydes. Ralls (*560*) described a useful technique for increasing the recovery of carbonyl compounds regenerated from their 2,4-dinitrophenylhydrazones in the flash exchange gas chromatographic technique. Ramshaw (*562*) studied the retention times of vinyl ketones and alcohols on stationary phases of various polarities. The data obtained can be used as an aid in identification.

Liquid-liquid partition chromatography was employed by Corbin (*111*) to separate 2,4-dinitrophenylhydrazones of dicarbonyl and other compounds.

Paper chromatography of 2,4-dinitrophenylhydrazones was accomplished by Onoda (*510*) using a specially pretreated paper and dioxane–ligroine as the developer. Diemair and Schams (*131*) characterized trace quantities of carbonyl compounds which had first been separated by low temperature, high vacuum fractionation, followed by paper chromatography and identification of their 2,4-dinitrophenylhydrazones by ultraviolet or infrared absorption. Fuerst and Fuestel (*181*) identified and quantitatively determined carbonyl compounds in essential oils by applying paper chromatography and ultraviolet spectroscopy to their 2,4-dinitrophenylhydrazones, and Suzuki, Takeuchi, and Maruta (*683*) also employed ultraviolet absorption to determine the 2,4-dinitrophenylhydrazones of aliphatic aldehydes which had been separated by paper chromatography. Brandt, Kouines, and Cheronis (*66*) detected carbonyl compounds down to 0.1 μg. by treating them with 2-diphenylacetyl-1,3-indandione-1-hydrazone and applying ascending paper chromatography. Bush and Hockaday (*78*) distinguished *cis* and *trans* isomers of carbonyl compounds by chromatography of their 2,4-dinitrophenylhydrazones using various solvent systems. Brandt, Kouines, and Cheronis (*65*) used fluorescent detection and determination of carbonyl compounds which were separated chromatographically.

Thin layer chromatography of 2,4-dinitrophenylhydrazones was employed by Schwartz and Parks (*625*) to separate aliphatic carbonyl compounds into classes, by Bordet and Michel (*60*) as a rapid method to identify methyl ketones of low molecular weight, and by Anet (*7*) to separate hydroxy carbonyl compounds. For complex mixtures Anet employed a two-dimensional method.

Thin layer chromatography was used by Badings and Wassink (*20*) to effectively separate aliphatic aldehydes and ketones into classes, and by Klouwen, Heide, and Kok (*337*) to separate 24 substituted benzaldehydes.

Ultraviolet spectrophotometric changes resulting from acetal formation were utilized to characterize and determine aldehydes by Crowell, Powell, and Varsel (*113*). Aldehydes and ketones in the same mixture could readily be differentiated. Crowell and Varsel (*114*) further studied the effect of various substituents on the ultraviolet fluorescence of aromatic aldehydes and their acetals. Montes (*430*) assembled the ultraviolet absorption spectra of the 2,4-dinitrophenylhydrazones of 35 carbonyl compounds from natural and synthetic perfume materials.

Polarography for the determination of aldehydes was discussed by Nigam and Kumari (*477*), and illustrated by application to citral and cineol in essential oils. Bodyu and Lyalikov (*55*) determined aromatic and furan aldehydes by the impulse polarographic method. The method is applicable to vanillin in food products.

Polarography was employed by Devyatnin and Solunina (*129*) to determine methyl vinyl ketone in the presence of formaldehyde and acetone. Marciszewski *et al.* (*401*) utilized the technique to determine pseudoionone in the presence of hexahydropseudoionone. The method was compared with spectrophotometric determinations.

Spectropolarimetric analysis, a new quantitative method, was described by Potapov, Terent'ev, and Avakyan (*555*). Optically inactive substances were determined with the aid of optically active reagents. The determination of benzaldehyde served to illustrate the technique. Potapov, Moiseeva, and Terent'ev (*554*) applied the method to the analysis of furfural and salicylaldehyde. The same authors (*553*) investigated the use of other optically active reagents for the determination of benzaldehyde, acetophenone, butyric, and propionic aldehydes.

Optical rotatory dispersion studies were used to establish the stereochemistry of the isomeric isopulegones by Ohloff, Osiecki, and Djerassi (*503*).

An improved hydroxylamine hydrochloride determination of aldehydes was described by Hashmi, Elahi, and Huda (*234*). The method relies on pH determination after the reaction, instead of titration of the liberated hydrochloric acid. Ketones interfere. Omboly (*509*) studied the assay by oximation of less active carbonyl compounds and ascertained the conditions under which the reaction became quantitative. Camphor was successfully determined. Belcher and Fleet (*35*) devised a submicromethod for determining carbonyl compounds based on oximation and titration of the excess reagent with $HClO_4$.

A rapid colorimetric determination of carbonyl compounds based on the finding that 4-nitro- or 2,4-dinitrophenylhydrazones become red to blue in $HCONMe_2$ on addition of Me_4NOH, was described by Yoshida and Nakamura (*769*). Zozulya and Novikova (*777*) devised a colorimetric estimation of small amounts of methyl vinyl ketone.

Optimum procedures for the dichromatometric determination of some aldehydes were ascertained by Karpov (*310*).

The use of 2,4-dinitrophenylhydrazine in the analysis and separation of flavor ingredients was reviewed by Patton (*527*).

Spot tests for aromatic and α,β-unsaturated aldehydes using thiobarbituric acid were described by Feigl and Libergott (*163*).

The reaction with aniline was used to determine aldehydes in the presence of ketones by Petrova, Skvortsova, and Novikova (*532*). They (*648*) subsequently elaborated on the procedure and proved that acetals do not interfere with this determination.

A silver oxide–*tert*-butylamine complex was employed by Mayes, Kuchar, and Siggia (*412*) to determine aldehydes in the presence of acids, acetals, and ketones.

INDIVIDUAL COMPOUNDS. Benzaldehyde in the presence of benzoic acid, benzoquinone, and maleic acid was determined by a combination of extraction and titration techniques, by Kumar, Bhat, and Kuloor (*363*).

A microanalytical technique to determine carvone was given in detail by Pohloudek-Fabini and Goeckeritz (*546*).

Citral was estimated in food and drug products by the barbituric acid condensation method by Laughton, Skakum, and Levi (*374*). Shukla, Nigam, and Handa (*644*) used a spectrophotometric determination of the eluted solution of its 2,4-dinitrophenylhydrazone. Citral was also determined colorimetrically, after separation by thin layer chromatography from lemongrass and other oils, by a method described by Martinez, Casillas, and Lecumberry (*403*).

Formaldehyde in essential oils was estimated by Talwar, Nigam, and Handa (*697*) by determination of the optical density of a derivative at 355 mμ.

5-Hydroxymethyl furfural was determined in grape juice by Boetticher (*58*).

The infrared spectra of nine ionones and methyl ionones were correlated with their structures for identification purposes by Theimer *et al.* (*712*). Kergomard, Pigeret, and Renard (*319*) studied the equilibrium in the isomerization of ionones, using gas chromatography, spectroscopy, distillation, and the preparation of derivatives.

Lippione, lippiophenol, and dihydrolippione, and their reaction products were discussed in relation to earlier work by Fester *et al.* (*168*). An inflection in the infrared spectrum at 1709 cm.$^{-1}$ was attributed to dihydrolippione.

The Girard reaction for the isolation of *l*-menthone results in isomerization as shown by Nigam and Levi (*466*). They recommended that the reaction therefore is not reliable for examining complex mixtures containing labile carbonyls, and that data based on the Girard reaction with such mixtures should be reappraised in accordance with the new findings.

Myrtenal and verbenone were determined by Retamar and Mazzola (*569*) based on the ultraviolet absorption spectra of their semicarbazones.

Minute quantities of α- or β-thujone were detected by Nano and Sancin (*443*) using a method which may be applied to vermouths.

Vanillin was determined by ultraviolet absorption by Feeny (*161*). The method is satisfactory in the absence of ethyl vanillin and coumarin. Vanillin and ethyl vanillin in food flavors were separated by Obata *et al.* (*499*) using gas chromatography with a hydrogen flame ionization detector. A quantitative gas chromatographic determination of vanillin and ethyl vanillin was developed by Martin, Feeny, and Scaringeli (*402*).

Alcohols and Phenols. GENERAL METHODS. The difficulties encountered in the gas chromatography of terpene alcohols and esters were discussed by Petrowitz (*533*), with particular emphasis on isomerization. Day and Miller (*120*) observed decomposition of α-terpineol and linalool if the injection temperature was too high. Robinson (*582*) described a gas chromatographic method for determining monohydric alcohols, using programmed temperature of 50° to 300° C. Baron and Maume (*24*) conducted a study to find the best conditions for the separation of menthol, menthone, borneol, menthoglycol, their stereoisomers, and camphor. von Rudloff (*592*) studied the hydrogenation products of α-terpineol, as well as the dehydration products of *p*-menthan-8-ol, by gas chromatography.

Reaction gas chromatography with boric acid to remove primary and secondary terpene alcohols was described by Hefendehl (*238*).

A paper chromatographic method for the analysis of terpene alcohols, based on their *o*-nitrophenol and *p*-phenylazophenylurethan derivatives, was developed by Attaway *et al.* (*17*). Quantities too small or too impure for infrared spectrographic determination may be analyzed by this method.

Infrared spectroscopy distinguishes among primary, secondary and tertiary alcohols, as explained by Habermehl (*225*). Douthit, Garska, and Yarborough (*142*) developed a method for identifying alcohols by the unique infrared spectra of their 3,5-dinitrobenzoates.

The ultraviolet absorption spectra of many terpene alcohols were reported by Naves and Frei (*455*).

The mass spectrometry of monoterpene alcohols was discussed, with data given, by Von Sydow (*735*).

Nuclear magnetic resonance was employed by Gaudemer, Polonsky, and Wenkert (*184*) to determine the conformation of primary terpene alcohols and their acetates.

Twenty alcohols were subjected to 25 color-inducing agents and the results were recorded by Christensen (*105*). The data are useful for identification. Gutnikov and Schenk (*222*) developed a spectrophotometric determination of hydroxyl compounds based on the formation of their ferric hydroxamates.

The hot pyridinic phthalization determination of alcohols was applied to a number of alcohols with good results by Kishore, Lal, and Kushwaha (*328*); by Lal, Kushwaha, and Kishore (*370*) to various mixtures; and by Kishore (*327*) to other alcohols such as santalol and geraniol. Results are comparable with those of the acetylation technique.

Stearic anhydride was employed for the determination of a wide range of hydroxyl compounds by Sully (*679*). The method is rapid and accurate.

Acetylation, bromination, and oxidation by cerium, as methods for determining alcohols, were discussed by Dryhurst (*145*).

Direct titration with lithium aluminum amide was described by Jordan (*303*) as a technique for selective hydroxyl group determination.

Based on the additive property of specific volumes, the percentage of component alcohols of a binary system was estimated by Lal, Patwardhan, and Lal (*371*). The method was extended to ternary and quaternary systems by Lal and Lal (*372*).

Phenol, guaiacol, and catechol were quantitatively determined, and phenols and their esters were separated by Lipina (*380*), using thin layer chromatography on specially prepared silica gel plates. Klouwen and Heide (*335*) separated phenols and their ethers, by thin layer chromatography, from fractions of essential oils obtained by means of gas chromatography and distillation. They suggested in detail the most suitable solvent systems and methods for identification.

Sublimation was employed by Yoshimura (*770*) to separate mixtures of polyhydric phenol isomers.

A novel apparatus which effected chromatographic separation of phenols by steam distillation was described by Dumazert and Ghiglione (*146*). A constant flow of steam was employed in place of the usual carrier gases.

Infrared analysis was used by Rashkes and Melkanovitskaya (*567*) to determine eugenol, chavibetol, and *o*-eugenol in a mixture. Lippmaa (*381*) described infrared determination of phenolic hydroxy groups.

The preparation of various phenylazo esters and their use in the characterization and determination of phenols occurring in essential oils was described by Pohloudek-Fabini and Munchow (*547*).

INDIVIDUAL COMPOUNDS. The structure and stereochemistry of α-caryophyllene alcohol was established both by Nickon *et al.* (*465*) and Gemmell *et al.* (*186*).

The conformational stereochemistry of carvomenthols according to their infrared and nuclear magnetic resonance spectra was elucidated by Naves (*450*); Royals and Leffingwell (*590*) assigned absolute configurations to isocarvomenthol and neoisocarvomenthol.

The cognac alcohols were separated by means of their 3,5-dinitrobenzoates by Egorov and Rodopulo (*149*).

The stereoisomeric farnesols and their derivatives were separated by Tyihak, Vagujfalvi, and Hagony (*721*), using gas and thin layer chromatography. Bates, Gale, and Gruner (*29*) established their stereochemical configurations by means of nuclear magnetic resonance spectra and chemical reactions.

Geraniol in rose oil was determined by Talwar, Nigam, and Handa (*702*), based on the absorbance at 550 mμ of the geraniol–phloroglucinol complex. Okazawa (*506*) postulated that geraniol is a mixture of two structural isomers.

Isopulegol was identified in acetic anhydride cyclized β-citronellal by Houlihan (*272*), using gas chromatography.

Linalool in linalyl acetate was determined by thin layer chromatography by Brud and Daniewski (*71*).

The physical processes for separating and analyzing isomeric menthols were reviewed by Mignat, Farnow, and Porsch (*417*). Mignat and Porsch (*418*) discussed the stereochemistry and stereochemical behavior of menthols. Bose, Harrison, and Farber (*62*) correlated the stereochemical structures of menthylamines and menthols. Klouwen and Heide (*336*) separated stereoisomeric menthols by gas chromatography. Terada, Tsuda, and Shono (*707*) similarly separated methols synthesized from thymol, and Houlihan (*271*) described the separation of menthone–menthol isomers by gas chromatography. Yoshida, Komatsu, and Indo (*768*) ascertained the purity of menthols by converting them to delta-3-menthone enol acetate and comparing the optical rotatory values. Yamaguchi, Yamaguchi, and Sakai (*762*) assayed free menthol in Japanese mint oil as the *p*-toluidinium salt of menthyl sulfate. Pertsev and Pivnenko (*529*) determined menthol, its esters, and cineole by column chromatography. Naves and Ochsner (*459*) studied the structures of the menthoglycols and their acetals.

The absolute configuration of nerolidol was established by Vlad and Soucek (*734*).

The structure and configuration of patchouli alcohol was indicated by x-ray studies conducted by Dober *et al.* (*139*).

Chemical studies of sclareol by Soucek and Vlad (*659*) elucidated its absolute configuration.

A spot test for *p*-cresol in which the other isomers do not interfere was devised by Feigl and Anger (*162*).

Phenol in salicylaldehyde was determined by precipitating the aldehyde, then colorimetrically assaying the phenol, by Gershuns and Rastrepina (*188*).

Gas chromatography using lanolin was employed by Porcaro and Johnston (*550*) to separate the four isomers of thymol.

Esters and Lactones. GENERAL METHODS. Gas chromatography was used by Shaposhnikov *et al.* (*633*) to determine free butanol in butyl acetate. Mattick *et al.* (*410*) used an electron affinity detector to determine methyl anthranilate in less than 0.1 p.p.m. concentrations.

Thin layer chromatography was employed by Korte and Vogel (*346*) to separate lactones, lactams, and thiolactones. R_f values were tabulated for numerous compounds of these classes.

An infrared spectrophotometric method was employed by Ruch'eva and Fillippov (*591*) to analyze mixtures of vinyl and vinyl-alkyl esters of dicarboxylic acids. Bory and Fetizon (*61*) established an empirical correlation between the infrared spectra and the stereochemistry of di- and triterpene esters. Mitsuhashi *et al.* (*423*) investigated the distribution of alkyl phthalides in the distillates from several members of the family Umbelliferae and reported their infrared and ultraviolet spectra.

A photometric determination of esters in essential oils based on the ferric hydroxamate reaction was developed by Graf and Hoppe (*210*).

The correlation of the Hudson-Klyne lactone rule with the optical rotation of sesquiterpenoid lactones was presented by Cocker and Nisbet (*107*).

Ethanolysis was employed by Heitler (*239*) to determine the number of ester groups in a molecule, as well as the saponification equivalent and degree of polymerization. The method is rapid but only moderately accurate.

Fifteen naturally occurring coumarins and those from *Angelica archangelica* and *Heracleum sibiricum* were separated by a gas chromatographic technique devised by Brown and Shyluk (*70*). Saxby (*620*) described a new paper chromatographic method for the characterization of flavonoids and coumarins by the shifts in their absorption spectra with the application of developing agents. Perel'son (*528*) reported a study of the infrared spectra of a series of coumarins and furocoumarins. Vul'fson, Zaretsky, and Zaikin (*740*) conducted a mass spectrometric investigation of natural coumarins. Prokopenko and Tarasenko (*557*) devised a colorimetric determination of coumarins which may be applied to plant materials. Denisova and Dranitsyna (*124*) used extraction and luminescence methods to show that coumarin compounds were present in all the oil channels of the fruit of *Archangelica decurrens*. The co-occurrence of coumarin, *o*-coumaric acid, and melilotic acid in the acid hydrolyzed extracts of *Gliricidia sepium* and *Dipteryz odorata* was proved, using paper chromatography, by Griffiths (*214*). The route of biosynthesis of coumarin from C^{14} labeled *trans*-cinnamic acid was traced by Stoker and Bellis (*670*).

Ethers, Oxides, and Peroxides. GENERAL METHODS. The mass spectra of aromatic ethers, in which the oxygen is part of a heterocyclic ring, were studied by Willhalm, Thomas, and Gautschi (*751*).

The Zeisel methoxyl determination was modified to permit determination of micro quantities by Schole (*622*), and also by Wu (*760*) who described a modified apparatus for this purpose. Klimova and Zabrodina (*332*) also devised a modified Zeisel-Vieboeck method for microdetermination of alkoxyl groups. In their modification KI and H_3PO_4 were used instead of HI. Anderson and Zaidi (*4*) studied the behavior of propoxyl and butoxyl groups in the Zeisel determination by means of infrared spectroscopy. Anderson *et al.* (*3*) also applied a modified procedure to the determination of tertiary butoxyl groups. The method, however, does not distinguish between tertiary butoxyl and tertiary butylated phenols. Ehrlich-Rogozinski and Patchornik (*150*) presented a simple and rapid method for determining higher alkoxyl groups by direct chemical reaction and nonaqueous titration. The distillation procedure inherent in all modifications of the Zeisel determination is eliminated.

The alkoxyl group in an ester was determined colorimetrically by Obtemperanskaya and Volodz'ko (*500*).

Peroxides in essential oils were estimated by Waginaire and Guillot (*741*) using an iodometric technique. The result may be employed to indicate the age of an oil. Bakker and Zwaving (*21*) compared the iodometric peroxide determinations of Sully and van Os-Sholten. Both methods gave corresponding and reproducible results. Using chemical reaction, photometric measurement, and titrimetry, Nettesheim (*463*) analyzed aqueous solutions containing H_2O_2, aldehydes, and organic peroxides for these components. Novikova, Plyushchevskii, and Makarevich (*493*) determined the reaction of antioxidants with α-pinene hydroperoxide by iodometric and infrared methods.

INDIVIDUAL COMPOUNDS. Quantitative determinations of *cis*- and *trans*-anethole were conducted by Ferroni, Ficalbi, and Secci (*166*).

Ascaridole was determined spectrophotometrically, on the basis of the colored complex with bis(4-dimethylaminophenyl) methane, by Chopra, Vashist, and Handa (*99*).

Cineole can be determined, according to Retamar and Ricciardi (*570*) by the Van der Driessen Mareuw reaction only when no interfering substances are present; therefore the method can not be generally applied to essential oils. Kohlmuenzer (*344*) analyzed 52 different essential oils for 1,8-cineole by microchemical reaction with tetraiodopyrrole, a modified plate chromatography method, and the orthocresol method.

Dimethyl ethers of pyrogallol and guaiacol were separated from liquid smoke, by paper chromatography, and identified by Kurko and Kel'man (*365*).

Terpenes and Hydrocarbons. GENERAL METHODS. Gas chromatographic separation of 14 terpene hydrocarbons was studied by Bernhard (*37*), utilizing capillary columns employing various liquid phases and flame ionization detection. He concluded that the stationary liquid phase is the most significant operational parameter. Haslam and Jeffs (*235*) found tritolyl phosphate and didecyl phthalate to be the most effective stationary phases in the gas chromatography of 28 terpenes. Jain, Varma, and Bhattacharyya (*295*) reported the retention times of monoterpenes obtained from 9 essential oils by fractionation with an ice cold condenser. Silicone and carbowax columns were used. Klouwen and Heide (*333, 334*) in an extensive study identified 22 monoterpene hydrocarbons in thirty essential oils. Five different column substrates were employed. Graphs derived from the results clearly separated the monoterpenes into three classes, and the position of a terpene on the graphs can generally be related to its structure, as well as to its boiling point. Matsuura *et al.* (*408*) determined the relative retention values of nine terpene hydrocarbons, and in a subsequent investigation (*409*), listed the values of 22 more. Seven liquid phases were compared and tritolyl phosphate gave the best resolution, but it isomerized or partially destroyed β-pinene. Sidorov, Baboshin, and Rudakov (*646*), using a column packed with brick and coated with castor oil, found the optimum conditions for separating common monoterpenes. Valkanas and Iconomou (*724*) conducted a detailed systematic study of conditions for the analysis of terpene hydrocarbons by gas chromatography. Wang *et al.* (*744*) used a 6-meter long, 6-mm. diameter column packed with petrolatum on firebrick at 100–10° C. for the gas chromatography of terpenes. The separation of 22 sesquiterpene hydrocarbons by gas chromatography using four stationary phases of different polarity was studied by Lukes and Komers (*386*). Polyethylene glycol adipate proved to be best for practical use.

Reaction gas chromatography was employed by Okamoto and Onaka (*504*) to determine skeletal structures. Hydrogenation, dehydrogenation, and alkylation techniques were investigated.

A temperature-programmed preparative gas chromatograph utilizing 1.5-inch diameter, 10-foot long tubes connected in series was described by Hunter and Veldhuis (*280*), and its application to the separation of terpene hydrocarbons was illustrated.

Thin layer chromatography followed by gas chromatography was employed by Ikeda *et al.* (*284*) to determine the content of 15 monoterpene hydrocarbons and other unknown terpenes in 31 different essential oils. Gupta and Dev (*219*) used silver nitrate–silica gel mixtures to separate many terpene olefins by the thin layer technique.

Partition chromatography for the separation of sesquiterpenes was described in some detail by Wickberg (*750*).

Infrared spectrophotometry was applied to the quantitative estimation of cymene isomers by Doerffel and Geyer (*140*).

Mass spectra of monoterpene hydrocarbons were recorded and discussed by Thomas and Willhalm (*713*). Thirty-two cyclic terpenes could be distinguished from each other by their fragmentation patterns, but two allo-ocimenes gave identical spectra. Ryhage and Von Sydow (*606*) also reported mass spectra of monoterpene hydrocarbons.

A specific spot test color reaction for aliphatic unsaturated organic compounds with benzaldehyde and sulfuric acid was described by Okuda (*507*).

A manometric method for the microdetermination of double bonds by catalytic hydrogenation was developed by Horacek and Pechanec (*270*).

INDIVIDUAL COMPOUNDS. Azulene was determined spectrophotometrically with new electrophilic reagents by Sawicki, Stanley, and Elbert (*619*), who gave the structures for the chromogens, and showed that the methods can detect less than 1 p.p.m. Tetenyl *et al.* (*710*) studied the azulene compounds of the Achillea species.

Camphene was iodometrically estimated in essential oils by Kartha and Duggal (*312*).

Limonene and its oxidation products were shown by Kenney and Fisher (*318*) to undergo chemical changes during gas chromatography on an alkaline carbowax-20 M column. Newhall and Kesterson (*464*) noted an increase in refractive index and acid value and a decrease in optical rotation of deteriorating *d*-limonene.

The biogenesis of longifolene was studied by Sandermann and Bruns (*616*), with the aid of C^{14} labeled compounds and gas chromatography.

α-Phellandrene was estimated by Sugathan and Verghese (*678*), who used a reaction with maleic anhydride followed by distillation of the remaining oil.

The biogenesis of α-pinene was studied by Sandermann and Schweers (*617*) using radioactive tracer chemistry in a manner similar to that which they applied to longifolene (*618*). The state of present knowledge of α-pinene was reviewed by Indo (*285*).

The Raman spectrum of pure sabinene was reported by Goryaev and Tolstikov (*204*).

Structure Determinations. GENERAL METHODS. The steadily increasing application of the latest instrumental methods of analysis has resulted in a great amount of work to establish structural configurations. Some of this work has already been mentioned in the foregoing sections. The papers which will be reviewed here appeared to be of interest primarily because they clarify the structural configurations of compounds. This review has been confined to the more volatile compounds, which are of greater interest to our industry. Many structural determinations of diterpenes and triterpenes have therefore been omitted.

Hydroboration as a method for the assignment of configurations was demonstrated by Zweifel, Ayyangar, and Brown (*778*).

The octant rule was discussed by Djerassi and Klyne (*138*) as applied to structural and stereochemical problems and related to optical rotatory dispersion.

INDIVIDUAL COMPOUNDS. Achillin and one other sesquiterpene lactone of the guaianolide type were isolated from *Achillea lanulosa* and their structures were established by White and Winter (*749*).

Amaraline, a new sesquiterpene lactone having potent analgesic activity, was characterized by Lucas *et al.* (*384*).

The constitution of anhydrotaxininol was further elucidated by Taga (*689*).

The structure of arborescin was established by Bates *et al.* (*28*) by correlation with artabsin and by nuclear magnetic resonance spectra.

Aristolactone, isolated from Texas and Virginia snakeroot, was characterized by Martin-Smith *et al.* (*404*).

Bigelovin, a new sesquiterpene lactone isolated from *Helenium bigelovii*, was assigned a structure by Parker and Geissman (*524*).

Bilobanone is a bicyclic ketone from *Ginkgo biloba*, whose structure was established by Kimura (*325*).

The structure of cadinane sesquiterpenes was related to that of the monoterpenes of the *p*-menthane series by Soffer *et al.* (*654*) using chemical procedures.

The stereochemistry of calarene and aristolone was established with the aid of nuclear magnetic resonance, mass spectrography, and other techniques, by Buechi, Greuter, and Tokoroyama (*72*).

The configurations of capsanthin and capsorubin were elucidated by Faigle *et al.* (*159*).

The stereoisomerism of 3,4-caranediols was clarified by Chabudzinski and Kuczynski (*89*).

The structure of 3-carene epoxide, previously proposed by Penfold, Ramase, and Simonsen, was confirmed by Desikan (*127*).

A synthesis was employed by Pinder and Williams (*545*) to confirm the structure and absolute configuration of carissone.

The conformations of carvomenthols were deduced by Chabudzinski and Kuczynski (*88*), based on chemical reactions.

The structure of cembrene, a 14-membered ring diterpene hydrocarbon, was elucidated by Dauben, Thiessen, and Resnick (*118*).

The absolute configurations of chamic, chaminic, and isochamic acids were established by Norin (*490*), who also (*491*) determined the structure of chanootin, a bicyclic sesquiterpene tropolone.

The structure of colensenone, a norditerpene oxide isolated from *Dacrydium colensoi*, was established by Grant and Carman (*213*).

The stereochemistry of cyclocolorenone was elucidated by Buechi and Loewenthal (*73*).

Cyperene from *Cyperus rotundus* was structurally defined by Trivedi *et al.* (*717*).

Damsin, a sesquiterpene lactone from *Ambrosia maritima*, was shown to consist of a single isomer, by Suchy, Herout, and Sorm (*675*).

Nakazaki (*442*) clarified the configuration of desmotroposantonin.

The structure of α-elemene isolated from Java citronella oil was studied by Paknikar and Bhattacharyya (*515*).

A possible structure for eremolactone, a new type of diterpene from *Eremophilia freelingii*, was proposed by Birch, Grimshaw, and Turnbull (*51*). Novotny *et al.* (*498*) clarified the constitution and absolute configurations of eremophilenolide, a sesquiterpenoid from *Petasites hybridus*.

Geijerene, a sesquiterpene isolated from the essential oil of *Geijera parviflora*, was structurally defined by Sutherland (*681*).

Grosshemine, a new sesquiterpene lactone, was characterized on the basis of its infrared spectra by Rybalko, Ban'kovskii, and Kibalchich (*605*).

A new guaianolide was isolated from the root of *Laser trilobum* and partially characterized by Holub *et al.* (*269*).

The absolute configuration of a portion of the guaiol molecule was established by Minato (*419*).

The structure and stereochemistry of α-gurjunene was determined by Palmade *et al.* (*516*).

The structure of helenalin and mexicanin A were established in accordance with new spectroscopic, chemical, and physical data by Herz *et al.* (*251*).

Helenine was isolated from oil of the root of *Inula helenium* and characterized by Olechnowicz-Stepien (*508*).

Hinesol, isolated from oil of *Atractylodes lancea*, was clarified as to constitution and configuration on the basis of degradation reactions, by Chow, Motl, and Sorm (*101*).

The absolute configuration of intermedeol, a new sesquiterpene alcohol isolated from oil of *Bothriochloa intermedia,* was established by Zalkow, Zalkow, and Brannon (*774*). From the same oil, Zalkow, Shaligram, and Zalkow (*773*) isolated *neo*-intermediol and established its stereochemistry.

The conformation of iridomyrmecin and isoiridomyrmecin was studied with the aid of low temperature x-ray crystallography by McConnell, Mathieson, and Shoenborn (*389*).

Carbon-14 tracer studies were utilized by Goetschel (*190*) to study the isocamphane series.

The stereochemistry of isophotosantonic lactone was defined by Asher and Sim (*15*), based on a detailed crystal-structure analysis.

The structure of isopimaric acid was elucidated by Ireland and Newbould (*286*).

The structure and stereochemistry of ivalin, obtained from *Iva microcephala,* was established by Herz and Hoegenauer (*247*) after extensive chemical investigation.

The stereochemistry of *l*-lanceol was established by Manjarrez, Rios, and Guzman (*399*), who used several instrumental techniques as well as chemical studies.

The conformation of ring C of levopimaric acid was shown to be primarily the folded structure, by Weiss, Ziffer, and Charney (*747*).

The structure of limonenylcarbinol acetate was deduced by Suga and Watanabe (*677*), based on infrared spectra, rotatory power, and chemical properties.

Linalool oxides were shown to comprise two derivatives of tetrahydrofuran and two of tetrahydropyran by Felix *et al.* (*164*). Further elucidation of structure of linalool oxides was offered by Klein, Farnow, and Rojahn (*331*).

Linderane, from *Lindera strichnifolia,* is the first example of a sesquiterpene lactone of its type having a furan ring in the molecule, according to the structure determined by Takeda, Minato, and Horibe (*691*).

The structure of linderene was established by Takeda and Ikuta (*690*).

The structures of the crystalline HCl salts of the manool series: manool, sclareol, manoyl oxide, epimanoyl oxide, and biformene, were established by Carman (*86*) with the aid of nuclear magnetic resonance spectra. From the heartwood of *Xylia dolabriformis,* Laidlaw and Morgan (*369*) isolated and characterized manoyl oxide, 3-oxomanoyl oxide, and sandaracopimaradien-3-one.

The absolute configuration of 3- and 4-methylcycloheptanone was determined by Djerassi *et al.* (*137*).

A new sesquiterpene lactone, mexicanin, related to tenulin was characterized by Dominguez and Romo (*141*).

The structure of mutilin, a new type tricyclic diterpene, was established by Arigoni (*14*).

The most probable absolute stereochemistry of occidentalol was suggested by Ziffer *et al.* (*775*).

A tentative structure was proposed for otobain, isolated from *Myristica otoba,* by Stevenson (*669*).

The structures of several volatile ketones from paprika were indicated by Barber *et al.* (*22*).

Herz *et al.* (*252*) established the structural formula of parthenin and compared it to ambrosin; partheniol, by interpretation of its nuclear magnetic resonance spectra, was shown by Hendrickson and Rees (*245*) to consist of two isomers.

The configuration of patchouli alcohol was verified by a synthesis reported by Buechi and MacLeod (*74*).

Stereochemical structures for the perhydroazulenic sesquiterpenes: lactucin, artabsin, arborescin, tenulin, baldulin, and helenanin, were derived by Hendrickson (*243*).

The conformational mobility of (−)-α-phellandrene was deduced by Ziffer, Charney, and Weiss (*776*) from its optical rotatory dispersion.

The structure of phyllocladene was established by Briggs *et al.* (*68*).

The constitution of the sesquiterpene lactone from *Helenium pinnatifidum,* pinnatifidin, was delineated by Herz *et al.* (*249*).

The configurations of pinonic and pinononic acids were clarified by Harispe, Mea, and Horeau (*232*).

The stereochemistry of piperitenone and its 2,4-dinitrophenylhydrazone was elucidated by Naves (*447*), as deduced from nuclear magnetic resonance and infrared spectra.

Polygodial, a new sesquiterpene dialdehyde, was isolated from *Polygonum hydropiper* and structurally characterized by Barnes and Loder (*23*).

The structure of pulchellin was established by Herz, Ueda, and Imayama (*250*); two other new sesquiterpene lactone isolates from *Gaillardia pulchella,* pulchellin B and pulchellin C, were also structurally characterized by Herz and Imayama (*248*).

The absolute configuration of sabinol and its esters was established by Norin (*488*).

Nuclear magnetic resonance spectrography along with other techniques were utilized by Brieger (*67*) to elucidate the stereochemistry of α- and β-santalol.

Double-bond derivatives of santonin were reexamined by several techniques by Hendrickson and Bogard (*244*), and stereochemical structures were assigned. X-ray data and degradative evidence were employed by Huffman (*274*) to establish the configurations of santonin and the desmotroposantonins.

Scabiolide was shown by Suchy, Herout, and Sorm (*674*) to be another sesquiterpenic lactone with a ten-membered ring in the molecule.

Sesquiterpene lactones from some southwestern *Helenium* species were isolated by Herz (*246*), who proposed structures for six of them: tenulin, isotenulin, helenalin, neohelanin, linofolin A, and linofolin B.

The structure of spathulenol, isolated from oil of *Eucalyptus spathulata* var. *grandiflora,* was established by Bowyer and Jefferies (*63*).

Sugiol, a diterpene phenolic ketone from *Juniperus macropoda* fruit, was characterized by Gupta, Vishwa, and Handa (*220*).

Sylvestrene was studied using nuclear magnetic resonance spectra along with other methods, and its structure was established by Mathew and Verghese (*405*).

The structure and stereochemistry of thujic acid was elucidated by Davis and Tulinsky (*119*) by means including x-ray analysis and nuclear magnetic resonance spectra. The steric relationship of thujone and isothujone to other members of the thujane series was demonstrated by Norin (*492*). Thujopsene was shown to be a tricyclic hydrocarbon by Kobayashi, Nagahama, and Akiyoshi (*342*).

Totarol, a diterpenoid from *Podocarpus mannii,* was characterized by Taylor (*706*).

The absolute configuration of umbellulone and the thujane terpenes as reported by Walborsky was verified by Smith and Gordon (*651*).

The constitution and stereochemistry of valdiviolide, fuegin, winterin, and futronolide from Drimys species was established by Appel, Bond, and Overton (*10*).

The stereochemical formula of verbenalin from *Verbena officinalis* was derived by Buechi and Manning (*75*).

The structure of an ester from *Verbesina virginica* was proved by Park (*523*).

The configuration of widdrol was elucidated by Ito, Endo, and Nozoe (*288*). Enzell (*154*) also submitted proof of the structure and absolute configuration of widdrol as well as that of widdrol-α-epoxide.

The structure of xanthinin was substantiated by Geissman (*185*) according to nuclear magnetic resonance spectra.

Three possible structures were proposed for ylangene by Motl *et al.* (*435*) as indicated by infrared and nuclear magnetic resonance spectra and chemical data. Hunter and Brogden (*278*) proved that ylangene is a stereoisomer of copaene and suggested a somewhat different structure.

The structure of zierone, isolated from *Zieria macrophylla*, was established by Barton and Gupta (*26*). Infrared, ultraviolet, and nuclear magnetic resonance spectra were utilized.

Miscellaneous. Osmo piles and surface-active receivers were used by Berton (*42*) for detecting and determining traces of volatile compounds of essential oils in the air.

Gas chromatography of pyrolyzed straight chain organic compounds having a single functional group was used by Dhont (*130*) to identify constituents of food odors.

Chemical reactions with malodorous compounds, resulting in their elimination were discussed by Kulka (*359*).

Six methods for measuring the small vapor pressures of aromatic materials were described by Appell (*11*).

Organic sulfides were separated with paper chromatography after reaction with methyl iodide by Kronrad and Panek (*353*). The infrared spectra of asymmetric disulfides from garlic were obtained by Jacobsen *et al.* (*290*), who used gas chromatography to separate the vapors from chopped garlic. Methyl allyl, methyl propyl, and allyl propyl disulfides were identified. Akerfeldt and Loevgren (*2*) described a method for the quantitative spectrophotometric determination of disulfides, sulfinic acid, thio ethers, and thiols, utilizing colored complexes with palladium. Golikov (*194*) determined organic disulfides by treatment with base, and subjecting the reaction products to polarography based on the anode wave.

A titrimetric determination of the isothiocyanates based on their reaction with piperidine in anhydrous dioxane was reported by Venkataraghavan and Rao (*733*).

Halogens in very low concentrations may be determined by a tube combustion procedure utilizing the oxygen flask method as recommended by Tomlinson (*714*).

The chemical and analytical characteristics of maltol were reviewed by Bohnsack (*59*).

Four vanilla extracts, one pure and three adulterated, were analyzed, by eleven collaborators, for alcohol, lead number, resins, and vanillin as reported by Wendt (*748*). A well prepared adulterated extract could not easily be detected. A rapid thin layer chromatographic method for the detection of ethyl vanillin, veratraldehyde, piperonal, and vanitrope was developed by Kahan and Fitelson (*304*), after a collaborative study, and was officially adopted by the Association of Official Agricultural Chemists. Fitelson (*174*) also described in detail an official gradient elution method for the separation of organic acids from vanilla extracts. In a subsequent paper he (*175*) further elaborated on the same subject. Fitelson (*173*) also described the adopted version of the two-dimensional paper chromatography of vanilla extracts. Anwar (*9*) separated the monohydroxy phenols from vanilla by paper chromatography and identified them by color under ultraviolet light with Millons reagent.

LITERATURE CITED

(1) Ahmad, M. N., Bhatty, M. K., Karimullah, *Pakistan J. Sci.* **14** (1), 12 (1962).
(2) Akerfeldt, S., Loevgren, G., *Anal. Biochem.* **8,** 223 (1964).
(3) Anderson, D. M. W., Duncan, J. L., Herbich, M. A., Zaidi, S. S. H., *Analyst* **88,** 353 (1963).
(4) Anderson, D. M. W., Zaidi, S. S. H., *Talanta* **10,** 1235 (1963).
(5) Andersson, J., Bosvik, R., Sydow, E. V., *J. Sci. Food Agr.* **14,** 834 (1963).
(6) Andrzej, P., *Farm. Polska* **18,** (6) 136 (1962).
(7) Anet, E. F. L. J., *J. Chromatog.* **9,** 291 (1962).
(8) Anh, N. T., Fetizon, M., *France Parfums* **6,** 203 (1963).
(9) Anwar, M. H., ANAL. CHEM. **35,** 1974 (1963).
(10) Appel, H. H., Bond, R. P. M., Overton, K. H., *Tetrahedron* **19,** 635 (1963).
(11) Appell, L., *Am. Perfumer Cosmet.* **79** (5), 29 (1964).
(12) Aratani, T., Komae, H., *Koryo* (64), 45 (1961).
(13) Aratani, T., Komae, H., Matsuura, T., *Kogyo Kagaku Zasshi* **65,** 1565 (1962).
(14) Arigoni, D., *Gazz. Chim. Ital.* **92,** 884 (1962).
(15) Asher, J. D. M., Sim, G. A., *Proc. Chem. Soc.* **1962,** p. 111.
(16) Attaway, J. A., Wolford, R. W., Alberding, G. E., *J. Agr. Food Chem.* **12,** 118 (1964).
(17) Attaway, J. A., Wolford, R. W., Alberding, G. E., Edwards, G. J., ANAL. CHEM. **35,** 234 (1963).
(18) Attaway, J. A., Wolford, R. W., Edwards, G. J., *J. Agr. Food Chem.* **10,** 102 (1962).
(19) Avotins, F., Osipov, L., Vitols, O., *Uch. Zap. Rizhsk. Poletekhn, Inst. Khim. Fak.* **2** (7), 53 (1959).
(20) Badings, H. T., Wassink, J. G., *Neth. Milk Dairy J.* **17,** 132 (1963).
(21) Bakker, H. K., Zwaving, J. H., *Pharm. Weekblad* **99,** 361 (1964).
(22) Barber, M. S., Jackman, L. M., Warren, C. K., Weedon, B. C. L., *Proc. Chem. Soc.* **1960,** p. 19.
(23) Barnes, C. S., Loder, J. W., *Australian J. Chem.* **15,** 322 (1962).
(24) Baron, C., Maume, B., *Bull. Soc. Chim. France* **1962,** p. 1113.
(25) Barton, D. H. R., De Vries, J. X., *J. Chem. Soc.* **1963,** p. 1916.
(26) Barton, D. H. R., Gupta, G. S., *Ibid.,* **1962,** p. 1961.
(27) Bassette, R., Ozeris, S., Whitnah, C. H., ANAL. CHEM. **34,** 1540 (1962).
(28) Bates, R. B., Cekan, Z., Prochazka, V., Herout, V., *Tetrahedron Letters* **1963,** p. 1127.
(29) Bates, R. B., Gale, D. M., Gruner, B. J., *J. Org. Chem.* **28,** 1086 (1963).
(30) Bates, R. B., Hendrickson, E. K., *Chem. Ind.* (*London*) **1962,** p. 1759.
(31) Bates, R. B., Sigel, C. W., *Experientia* **19,** 564 (1963).
(32) Bates, R. B., Slagel, R. C., *Chem. Ind.* (*London*) **1962,** p. 1715.
(33) Baytop, A., *Folia Pharm.* (*Istanbul*) **5,** 594 (1963).
(34) Bejnarowicz, E. A., Kirch, E. R., *J. Pharm. Sci.* **52,** 988 (1963).
(35) Belcher, R., Fleet, B., *J. Chem. Soc.* **1963,** p. 5720.
(36) Benesova, V., Chou, P. N., Herout, V., Naves, Y.-R., Lamparsky, D., *Collection Czech. Chem. Commun.* **29,** 1042 (1964).
(37) Bernhard, R. A., ANAL. CHEM. **34,** 1576 (1962).
(38) Bernhard, R. A., Wrolstad, R., *J. Food Sci.* **28,** 59 (1963).
(39) Beroza, M., ANAL. CHEM. **34,** 1801 (1962).
(40) Beroza, M., *Nature* **196,** 768 (1962).
(41) Beroza, M., Acree, F., Jr., *J. Assoc. Offic. Agr. Chemists* **47,** 1 (1964).
(42) Berton, A., *France Parfums* **6,** 391 (1963).
(43) Bevitt, R. N., Cheshire, J. D., *J. Soc. Cosmetic Chemists* **14,** 173 (1963).
(44) Bhargava, P. P., Chowdhri, B. L., Haksar, C. N., *Perfumery Essent. Oil Record* **54,** 740 (1963).
(45) Bhargava, P. P., Haksar, C. N., *Ibid.,* **53,** 379 (1962).
(46) Bhati, A., *Ibid.,* p. 15.
(47) *Ibid.,* **55,** 124 (1964).
(48) Bhramaramba, A., Mahboob, S. *Ibid.,* **54,** 30 (1963).
(49) Bhramaramba, A., Sidhu, G. S., *Ibid.,* p. 732.
(50) Biernacka, T., Kontnik, B., Parczewski, A., Rajkowski, Z., *Dissertationes Pharm.* **15,** 419 (1963).
(51) Birch, A. J., Grimshaw, J., Turnbull, J. P., *J. Chem. Soc.* **1963,** p. 2412.
(52) Blight, M. M., McDonald, I. R. C., *New Zealand J. Sci.* **6,** 229 (1963).
(53) Blumann, A., Della, E. W., Henrich, C. A., Hodgkin, J., Jefferies, P. R., *Australian J. Chem.* **15,** 290 (1962).
(54) Blumann, A., Gallagher, M. J., Sutherland, M. D., *Perfumery Essent. Oil Record* **54,** 743 (1963).
(55) Bodyu, V. I., Lyalikov, Y. S., *Zh. Analit. Khim.* **18,** 1007 (1963).
(56) Boehm, E. E., Thaller, V., Whiting, M. C., *J. Chem. Soc.* **1963,** p. 2535.
(57) Boehm, H., Voelcker, P. E., *Arch. Pharm.* **292,** 529 (1959).
(58) Boetticher, W., *Deut. Lebensm. Rundschau* **59,** 201 (1963).
(59) Bohnsack, H., *Riechstoffe Aromen* **14,** 33 (1964).
(60) Bordet, C., Michel, G., *Compt. Rend.* **256,** 3482 (1963).
(61) Bory, S., Fetizon, M., *Bull. Soc. Chim. France* **1964,** p. 570.
(62) Bose, A. K., Harrison, S., Farber, L., *J. Agr. Chem.* **28,** 1223 (1963).
(63) Bowyer, R. C., Jefferies, P. R., *Chem. Ind.* (*London*) **1963,** p. 1245.
(64) Brandenberger, H., Mueller, S., *J. Chromatog.* **7,** 137 (1962).
(65) Brandt, R., Kouines, J. C., Cheronis, N. D., *Ibid.,* **12,** 380 (1963).
(66) Brandt, R., Kouines, J. C., Cheronis, N. D., *Microchem. J.* **6,** 519 (1962).
(67) Brieger, G., *Tetrahedron Letters* **1963,** p. 2123.
(68) Briggs, L. H., Cain, B. F., Cambie, R. C., Davis, B. R., *J. Chem. Soc.* **1962,** p. 1840.
(69) Briner, E., Christol, C., Christol, H., Fliszar, S., Rosetti, G., *Helv. Chim. Acta* **46,** 2249 (1963).
(70) Brown, S. A., Shyluk, J. P., ANAL. CHEM. **34,** 1058 (1962).
(71) Brud, W., Daniewski, W., *Chem. Anal.* (*Warsaw*) **8,** 753 (1963).
(72) Buechi, G., Greuter, F., Tokoroyama, T., *Tetrahedron Letters* **1962,** p. 827.
(73) Buechi, G., Loewenthal, H. J. E., *Proc. Chem. Soc.* **1962,** p. 280.
(74) Buechi, G., MacLeod, Wm., Jr., *J. Am. Chem. Soc.* **84,** 3205 (1962).

(75) Buechi, G., Manning, R. E., *Tetrahedron* **18**, 1049 (1962).

(76) Burger, M., *Riechstoffe Aromen* **10**, 4 (1960).

(77) Burton, J. S., Stevens, R., *J. Chem. Soc.* **1964**, p. 952.

(78) Bush, I. E., Hockaday, T. D. R., *J. Chromatog.* **8**, 433 (1962).

(79) Buttery, R. G., Black, D. R., Kealy, M. P., McFadden, W. H., *Nature* **202**, 701 (1964).

(80) Buttery, R. G., Lundin, R. E., McFadden, W. H., Jahnsen, V. J., Kealy, M. P., *Chem. Ind.* (*London*) **1963**, 1981.

(81) Buttery, R. G., McFadden, W. H., Teranishi, R., Kealy, M. P., Mon, T. R., *Nature* **200**, 435 (1963).

(82) Calvarano, I., *Essenze Deriv. Agrumari* **33**, 102 (1963).

(83) Calvarano, I., DeLeo, M., *Ibid.*, p. 147.

(84) Calvarano, M., *Ibid.*, p. 5.

(85) Calvarano, M., *Ibid.*, p. 67.

(86) Carman, R. M., *Tetrahedron* **18**, 285 (1962).

(87) Carroll, M. F., Price, W. J., *Perfumery Essent. Oil Record* **55**, 114 (1964).

(88) Chabudzinski, Z., Kuczynski, H., *Bull. Acad. Polon. Sci., Ser. Sci. Chim.* **12**, 91 (1964).

(89) Chabudzinski, Z., Kuczynski, H., *Roczniki Chemii* **36**, 1173 (1962).

(90) Charma, M. L., Nigam, M. S., Handa, K. L., *Riechstoffe Aromen* **13**, 325 (1963).

(91) Chaudhary, S. S., Nazir, B. N., Handa, K. L., *Indian Oil Soap J.* **25**, 306 (1960).

(92) Chetty, G. L., Dev, S., *Tetrahedron Letters* **1964**, p. 73.

(93) Chirkova, M. A., Pentegova, V. A., *Izv. Sibirsk. Otd. Akad. Nauk. SSSR* **1962** (4), p. 125.

(94) Chiurdoglu, G., Smolders, R. R., Geeraerts, J., Kleber, R., Lecoq, R., Van Wuytswinkel, R., *Industrie Chim. Belge* **28**, 636 (1963).

(95) Chopra, M. M., Handa, K. L., *Perfumery Essent. Oil Record* **54**, 817 (1963).

(96) Chopra, M. M., Nigam, M. C., Handa, K. L., *Parfuem. Kosmetik* **44**, 123 (1963).

(97) Chopra, M. M., Nigam, M. C., Handa, K. L., *Perfumery Essent. Oil Record* **54**, 238 (1963).

(98) Chopra, M. M., Vashist, V. N., Handa, K. L., *Ibid.*, **55**, 323 (1964).

(99) Chopra, M. M., Vashist, V. N., Handa, K. L., *Riechstoffe Aromen* **14**, 36 (1964).

(100) Chopra, M. M., Vashist, V. N., Nigam, M. C., Handa, K. L., *Ibid.*, **13**, 253 (1963).

(101) Chow, W. Z., Motl, O., Sorm, F., *Collection Czech. Chem. Commun.* **27**, 1914 (1962).

(102) Chowdhri, B. L., Haksar, C. N., *Indian Oil Soap J.* **27**, 187 (1962).

(103) Chowdhri, B. L., Haksar, C. N., *Perfumery Essent. Oil Record* **55**, 121 (1964).

(104) Chowdhury, M. A., Williams, W. D., *J. Pharm. Pharmacol.* **16**, 347 (1964).

(105) Christensen, G. M., ANAL. CHEM. **34**, 1030 (1962).

(106) Clark, J., Bonga, J. M., *Can. J. Botany* **41**, 165 (1963).

(107) Cocker, W., Nisbet, M. A., *Chem. Ind.* (*London*) **1962**, p. 2121.

(108) Corbett, R. E., *J. Sci. Food Agr.* **13**, 158 (1962).

(109) Corbett, R. E., Jamieson, G. A., Murray, J., *Ibid.*, **14**, 349 (1963).

(110) Corbett, R. E., Young, H., *Australian J. Chem.* **16**, 250 (1963).

(111) Corbin, E. A., ANAL. CHEM. **10**, 1244 (1962).

(112) Cortez, F. F., *Rev. Brazil. Quim.* (*Sao Paulo*) **52**, 304 (1961).

(113) Crowell, E. P., Powell, W. A., Varsel, C. J., ANAL. CHEM. **35**, 184 (1963).

(114) Crowell, E. P., Varsel, C. J., *Ibid.*, p. 189.

(115) Damodaran, N. P., Dev. S., *Tetrahedron Letters* **1963**, 1941.

(116) Das, S. K., Saha, A. N., *Bull. Reg. Res. Lab., Jammu, India* **1**, 190 (1963).

(117) Datta, P. R., Sussi, H., Higman, H. C., Filipic, V. J., *Food Technol.* **16**, 116 (1962).

(118) Dauben, W. G., Thiessen, W. E., Resnick, P. R., *J. Am. Chem. Soc.* **84**, 2015 (1962).

(119) Davis, R. E., Tulinsky, A., *Tetrahedron Letters* **1962**, p. 839.

(120) Day, E. A., Miller, P. H., ANAL. CHEM. **34**, 869 (1962).

(121) Delaveau, P. G., *Acta Chem. Scand.* **17**, 2562 (1963).

(122) Demole, E., *Helv. Chim. Acta* **45**, 1951 (1962).

(123) Demura, Y., *Mem. Fac. Sci. Kyushu Univ. Ser.* **C4** (2), 145 (1961).

(124) Denisova, G. A., Dranitsyna, Y. A., *Dokl. Akad. Nauk SSSR* **146**, 954 (1962).

(125) Deryng, J., Parczewski, A., *Dissertationes Pharm.* **14**, 513 (1963).

(126) Deryng, J., Parczewski, A., Walewska, E., *Pharmazie* **17**, 400 (1962).

(127) Desikan, P., *Indian J. Chem.* **1**, 186 (1963).

(128) De Silva, M. G., *Mfg. Chemist* **30**, 415 (1959).

(129) Devyatnin, V. A., Solunina, I. A., *Tr. Vses. Nauchn.-Issled. Vitamin. Inst.* **7**, 104 (1961).

(130) Dhont, J. H., *Nature* **200**, 882 (1963).

(131) Diemair, W., Schams, F., *Z. Anal. Chem.* **189**, 149 (1962).

(132) DiGiacomo, A., Rispoli, G., *Essenze Deriv. Agrumari* **32**, 260 (1962).

(133) DiGiacomo, A., Rispoli, G., *Riv. Ital. Essenze, Profumi, Piante Offic.-Oli Vegetali-Saponi* **44**, 610 (1962).

(134) DiGiacomo, A., Rispoli, G., Crupi, F., *Essenze Deriv. Agrumari* **32**, 126 1962.

(135) DiGiacomo, A., Rispoli, G., Tracuzzi, M. L., *Ibid.*, **34**, 3 (1964).

(136) DiGiacomo, A., Rispoli, G., Tracuzzi, M. L., *Riv. Ital. Essenze, Profumi, Piante Offic.-Aromi-Saponi-Cosmet.* **45**, 269 (1963).

(137) Djerassi, C., Burrows, B. F., Overberger, C. G., Takekoshi, T., Gutsche, C. D., Chang, C. T., *J. Am. Chem. Soc.* **85**, 949 (1963).

(138) Djerassi, C., Klyne, W., *J. Chem. Soc.* **1962**, p. 4929.

(139) Dobler, M., Dunitz, J. D., Gubler, B., Weber, H. P., Buechi, G., Padilla, O. J., *Proc. Chem. Soc.* **1963**, p. 383.

(140) Doerffel, K., Geyer, R., *Wiss. Z. Tech. Hochsch. Chem. Leurna-Merseburg* **5** (1), 7 (1963).

(141) Dominguez, E., Romo, J., *Tetrahedron* **19**, 1415 (1963).

(142) Douthit, R. C., Garska, K. J., Yarborough, V. A., *Appl. Spectry.* **17** (4), 85 (1963).

(143) Dranovskaya, L. M., Kondratskaya, G. P., *Maslob.-Zhir. Prom.* **28** (12), 34 (1962).

(144) Drawert, F., Rapp, A., *Chemiker-Ztg.* **88**, 267 (1964).

(145) Dryhurst, G., *Ind. Chemist* **40**, 41, 97 (1964).

(146) Dumazert, C., Ghiglione, C., *Bull. Soc. Pharm. Marseille* **11**, 26 (1962).

(147) Duro, F., *Boll. Sedute Accad. Gioenia Sci. Nat. Catania* **7**, 120 (1961).

(148) Edwards, R. W. H., *Some Gen. Probl. Paper Chromatog., Symp., Liblice, Czech.* **1961**, p. 213 (pub. 1962); *C.A.* **59**, 12148.

(149) Egorov, I. A., Rodopulo, A. K., *Dokl. Akad. Nauk. SSSR* **146**, 210 (1962).

(150) Ehrlich-Rogozinski, S., Patchornik, A., ANAL. CHEM. **36**, 840 (1964).

(151) El-Dakhakhny, M., *Planta Med.* **11**, 465 (1963).

(152) El-Deeb, S. R., Karawya, M. S., Wahba, S. K., *J. Pharm. Sci. U. Arab Rep.* **3** (1), 81 (1962).

(153) El-Sakaa, H., *Egypt. Pharm. Bull.* **42**, 561 (1960).

(154) Enzell, C., *Acta. Chem. Scand.* **16**, 1553 (1962).

(155) Enzell, K., Theander, O., *Ibid.*, p. 607.

(156) Erdtman, H., Westfelt, L., *Ibid.*, **17**, 2351 (1963).

(157) Essential Oils Subcommittee, *Analyst* **89**, 233 (1964).

(158) Fahmy, I. R., Hamidi, A. E., *Egypt. Pharm. Bull.* **42**, (4), 195 (1960).

(159) Faigle, J. W., Mueller, H., Philipsborn, W. V., Karrer, P., *Helv. Chim. Acta* **47**, 741 (1964).

(160) Farnow, H., Porsch, F., *Dragoco Rept.* **11**, 99 (1964).

(161) Feeny, F. J., *J. Assoc. Offic. Agr. Chemists* **47**, 555 (1964).

(162) Feigl, F., Anger, V., ANAL. CHEM. **33**, 89 (1961).

(163) Feigl, F., Libergott, E., *Ibid.*, **36**, 132 (1964).

(164) Felix, D., Malera, A., Seibl, J., Kovats, E. Sz., *Helv. Chim. Acta* **46**, 1513 (1963).

(165) Fernandez, B., Fagerson, I. S., Nawar, W. W., *J. Gas Chromatog.* **1** (9), 21 (1963).

(166) Ferroni, E., Ficalbi, A., Secci, M., *Gazz. Chim. Ital.* **92**, 1198 (1962).

(167) Fester, G. A., Fonseca, L. R., Ricciardi, A. I. A., Cassano, A., Burgos, J., *Rev. Fac. Ing. Quim., Univ. Nacl. Litoral, Santa Fe, Arg.* **30**, 11 (1961).

(168) Fester, G. A., Martinuzzi, E. A., Retamar, J. A., Ricciardi, A. I. A., *Ibid.*, **29**, 17 (1960).

(169) *Ibid.*, p. 21.

(170) Fester, G. A., Retamar, J. A., Ricciardi, A. I. A., Cassano, A., *Ibid.*, p. 9.

(171) *Ibid.*, **30**, 5 (1961).

(172) Fisher, G. S., *Chem. Ind.* (*London*) **1963**, p. 1761.

(173) Fitelson, J., *J. Assoc. Offic. Agr. Chemists* **46**, 624 (1963).

(174) *Ibid.*, p. 626.

(175) *Ibid.*, **47**, 558 (1964).

(176) Fonseca, L. R., *Rev. Fac. Ing. Quim., Univ. Nacl. Litoral, Santa Fe, Arg.* **29**, 69 (1960).

(177) Franc, J., Michajlova, S., *J. Chromatog.* **12**, 22 (1963).

(178) Franchi, G., Franchi, R. R., *Congr. Sci. Farm., Conf. Comun., 21°, Pisa* **1961**, p. 532; *C.A.* **59**, 3713*g*.

(179) Franklin, W. J., Keyzer, H., ANAL. CHEM. **34**, 1650 (1962).

(180) Frauendorf, H., Auterhoff, H., *Deut. Apotheker-Ztg.* **103**, 1299 (1963).

(181) Fuerst, H., Fuestel, G., *Z. Chem.* **2**, 106 (1962).

(182) Fujita, Y., *Acta Phytotaxon. Geobotan.* **18**, 178 (1960).

(183) Fujita, Y., Ueda, T., Maruyama, S., *Nippon Kagaku Zasshi* **84**, 429 (1963).

(184) Gaudemer, A., Polonsky, J., Wenkert, E., *Bull. Soc. Chim. France* **1964**, p. 407.

(185) Geissman, T. A., *J. Org. Chem.* **27**, 2692 (1962).

(186) Gemmell, K. W., Parker, W., Roberts, J. S., Sim, G. A., *J. Am. Chem. Soc.* **86**, 1438 (1964).

(187) Genest, C., Smith, D. M., Chapman, D. G., *J. Agr. Food Chem.* **11**, 508 (1963).
(188) Gershuns, A. L., Rastrepina, I. A., USSR Patent **149,423** (Aug. 28, 1962); from *Byul. Izobret.* **1962** (16), p. 24.
(189) Gimaddinov, Zh. K., Lishtvanova, L. N., Goryaev, M. I., *Izv. Akad. Nauk Kaz. SSR, Ser. Khim.* **1962** (1), p. 112.
(190) Goetschel, C. T., *Dissertation Abstr.* **23**, 3120 (1963).
(191) Gogiya, V. T., *Biokhimiya* **28**, 308 (1963).
(192) Gold, H. J., Wilson, C. W., III, *J. Food Sci.* **28**, 484 (1963).
(193) Gold, H. J., Wilson, C. W., III, *J. Org. Chem.* **28**, 985 (1963).
(194) Golikov, A. A., USSR Patent **149,615** (Aug. 28, 1962); from *Byul. Izobret.* **1962**, (16), p. 55.
(195) Gonzalez, A. G., Estevez Reyes, R., *Anales Real Soc. Espan. Fis. Quim. (Madrid) Ser.* **B59**, 649 (1963).
(196) Gopinath, K. W., Prakash, L., Kidwai, A. R., *J. Sci. Ind. Res. (India)* **21B**, 404 (1962).
(197) Goryaev, M. I., Bazalitskaya, V. S., *Zh. Priklad. Khim.* **35**, 1360 (1962).
(198) Goryaev, M. I., Bazalitskaya, V. S., Lishtvanova, L. N., *Ibid.*, p. 2799.
(199) Goryaev, M. I., Ignatova, L. A., *Izv. Akad. Nauk Kaz. SSR, Ser. Khim.* **1962** (1), p. 109.
(200) Goryaev, M. I., Lishtvanova, L. N., Bazalitskaya, V. S., Teppeev, S. B., *Ibid.* (2), p. 103.
(201) Goryaev, M. I., Serkebaeva, T. E., Bazalitskaya, V. S., *Izv. Akad, Nauk Kaz. SSR, Ser. Tekhn. i Khim. Nauk*, **1963** (2), p. 3.
(202) Goryaev, M. I., Serkebaeva, T. E., Sharipova, F. S., Volkova, V. S., *Zh. Prikl. Khim.* **35**, 1144 (1962).
(203) Goryaev, M. I., Sharipova, F. S., Serkebaeva, T. E., El'chibekova, L. A., *Izv. Akad. Nauk Kaz. SSR, Ser. Khim.* **1962** (2), p. 106.
(204) Goryaev, M. I., Tolstikov, G. A., *Zh. Obshch. Khim.* **32**, 310 (1962).
(205) Goryaev, M. I., Tolstikov, G. A., Ignatova, L. A., Dembitskii, A. D., *Dok. Akad. Nauk SSSR* **146**, 1331 (1962).
(206) Goryaev, M. I., Yurima, R. A., *Zh. Prikl. Khim.* **36**, 1112 (1963).
(207) Gottlieb, O. R., Fineberg, M., Guimaraes, M. L., Magahaes, M. T., Maravalhas, N., *Perfumery Essent. Oil Record* **55**, 253 (1964).
(208) Gottlieb, O. R., Fineberg, M., Magalhaes, M. T., *Ibid.*, **53**, 299 (1962).
(209) Graf, E., Hoppe, W., *Deut. Apothek. Ztg.* **104**, 287 (1964).
(210) Graf, E., Hoppe, W., *Mitt. Deut. Pharm. Ges.* **34** (4), 53 (1964).
(211) Granger, R., Passet, J., Verdier, R., *France Parfums* **6**, 225 (1963).
(212) Grant, D. W., *J. Chromatog.* **10**, 511 (1963).
(213) Grant, P. K., Carman, R. M., *J. Chem. Soc.* **1962**, p. 3740.
(214) Griffiths, L. A., *J. Exptl. Botany* **13**, 169 (1962).
(215) Guenther, E., *Perfumery Essent. Oil Record* **54**, 645 (1963).
(216) *Ibid.*, **55**, 89 (1964).
(217) Guenther, E., Kulka, K., Rogers, J. A., ANAL. CHEM. **35**, 39R (1963).
(218) Gulati, B. C., Singh, P., Dimri, B. P., *Indian Perfumer* **7** (1), 17 (1963).
(219) Gupta, A. S., Dev, S., *J. Chromatog.* **12**, 189 (1963).
(220) Gupta, B. K., Vishwa, P., Handa, K. L., *Indian J. Chem.* **1**, 188 (1963).
(221) Gupta, G. N., Talwar, Y. P.. Nigam, M. C., Handa, K. L., *Soap, Perfumery Cosmetics* **37**, 45 (1964).
(222) Gutnikov, G., Schenk, G., ANAL. CHEM. **34**, 1316 (1962).
(223) Guven, K. C., *Folia Pharm. (Istanbul)* **5**, 586 (1963).
(224) Guzman, A., Manjarrez, M., *Bol. Inst. Quim. Univ. Nacl. Autonoma Mex.* **14**, 48 (1962).
(225) Habermehl, G., *Angew. Chem.* **76**, 271 (1964).
(226) Halpaap, H., *Chem.-Ingr.-Tech.* **35**, 488 (1963).
(227) Handa, K. L., Chopra, M. M., Nigam, M. C., *Perfumery Essent. Oil Record* **54**, 372 (1963).
(228) Handa, K. L., Nigam, M. C., Sharma, M. L., *Parfuem. Kosmetik* **44**, 261 (1963).
(229) Handa, K. L., Nigam, M. C., Sharma, M. L., *Riechstoffe Aromen* **13**, 181 (1963).
(230) Handa, K. L., Sharma, M. L., Nigram, M. C., *Parfuem. Kosmetik* **44**, 233 (1963).
(231) Handa, K. L., Smith, D. M., Levi, L., *Perfumery Essent. Oil Record* **53**, 607 (1962).
(232) Harispe, M., Mea, D., Horeau, A., *Bull. Soc. Chim. France* **1964**, p. 1035.
(233) Hartsuk, J. A., Paul, I. C., *Chem. Ind. (London)* **1964**, p. 977.
(234) Hashmi, M. H., Elahi, M., Huda, K. Q., *Pakistan J. Sci. Res.* **15** (1), 4 (1963).
(235) Haslam, J., Jeffs, A. R., *Analyst* **87**, 658 (1962).
(236) Hayashi, S., Yano, K., Matsuura, T., *Bull. Chem. Soc. Japan* **37**, 680 (1964).
(237) Hayden, A. L., Sammul, O. R., Selzer, G. B., Carol, J., *J. Assoc. Offic. Agr. Chemists* **45**, 797 (1962).
(238) Hefendehl, F. W., *Naturwissenschaften* **51**, 138 (1964).
(239) Heitler, C., *Talanta* **11**, 1081 (1964).
(240) Hellyer, R. O., Bick, I. R. C., Nicholls, R. G., Rottendorf, H., *Australian J. Chem.* **16**, 703 (1963).
(241) Hellyer, R. O., Keyzer, H., McKern, H. H. G., *Ibid.*, **17**, 283 (1964).
(242) Hellyer, R. O., McKern, H. H. G., *Ibid.*, **16**, 515 (1963).
(243) Hendrickson, J. B., *Tetrahedron* **19**, 1387 (1963).
(244) Hendrickson, J. B., Bogard, T. L., *J. Chem. Soc.* **1962**, p. 1678.
(245) Hendrickson, J. B., Rees, R., *Chem. Ind. (London)* **1962**, 1424.
(246) Herz, W., *J. Am. Chem. Soc.* **84**, 4043 (1962).
(247) Herz, W., Hoegenauer, G., *J. Org. Chem.* **27**, 905 (1962).
(248) Herz, W., Imayama, S., *Tetrahedron* **20**, 341 (1964).
(249) Herz, W., Mitra, R. B., Rabindran, K., Viswanathan, N., *J. Org. Chem.* **27**, 4041 (1962).
(250) Herz, W., Ueda, K., Imayama, S., *Tetrahedron* **19**, 483 (1963).
(251) Herz, W., Vivar, A. R. de, Romo, J., Viswanathan, N., *J. Am. Chem. Soc.* **85**, 19 (1963).
(252) Herz, W., Watanabe, H., Miyazaki, M., Kishida, Y., *Ibid.*, **84**, 2601 (1962).
(253) Hikino, H., Hikino, Y., Kato, H., Takeshita, Y., Takemoto, T., *Yakugaku Zasshi* **83**, 219 (1963).
(254) Hikino, H., Hikino, Y., Takemoto, T., *Chem. Pharm. Bull. (Tokyo)* **11**, 1210 (1963).
(255) Hikino, H., Hikino, Y., Takeshita, Y., Isurugi, Y., Takemoto, T., *Yakugaku Zasshi* **83**, 555 (1963).
(256) Hikino, H., Hikino, Y., Takeshita, Y., Meguro, K., Takemoto, T., *Chem. Pharm. Bull. (Tokyo)* **11**, 1207 (1963).
(257) Hikino, H., Hikino, Y., Takeshita, Y., Shirata, K., Takemoto, T., *Ibid.*, p. 547.
(258) *Ibid.*, p. 952.
(259) Hikino, H., Meguro, K., Kusano, G., Takemoto, T., *Ibid.*, **12**, 632 (1964).
(260) Hirota, N., Hiroi, M., *Koryo* **70**, 23 (1963).
(261) Hirsjarvi, P., Pirila, L., *Suomen Kemistilehti* **B37** (1), 13 (1964).
(262) Hochmannova, J., Novotny, L., Herout, V., *Collection Czech. Chem. Commun.* **27**, 2711 (1962).
(263) *Ibid.*, p. 1870.
(264) Hoerhammer, L., Richter, G., Wagner, H., *J. Chromatog.* **10**, 108 (1963).
(265) Hoerhammer, L., Wagner, H., *Deut. Apotheker-Ztg.* **103**, 1737 (1963).
(266) Hoff, J. E., Feit, E. D., ANAL. CHEM. **35**, 1298 (1963).
(267) *Ibid.*, **36**, 1002 (1964).
(268) Holub, M., Herout, V., *Collection Czech. Chem. Commun.* **27**, 2980–1 (1962).
(269) Holub, M., Popa, D. P., Herout, V., Sorm, F., *Ibid.*, **29**, 938 (1964).
(270) Horacek, J., Pechanec, V., *Ibid.*, p. 1500.
(271) Houlihan, W. J., ANAL. CHEM. **34**, 1846 (1962).
(272) Houlihan, W. J., *Perfumery Essent. Oil Record* **55**, 261 (1964).
(273) Howard, G. E., *Analyst* **88**, 633 (1963).
(274) Huffman, J. W., *J. Org. Chem.* **28**, 601 (1963).
(275) Hunter, G. L. K., *J. Assoc. Offic. Agr. Chemists* **46**, 838 (1963).
(276) Hunter, G. L. K., Brogden, W. B., Jr., ANAL. CHEM. **36**, 1122 (1964).
(277) Hunter, G. L. K., Brogden, W. B., Jr., *J. Org. Chem.* **29**, 498 (1964).
(278) *Ibid.*, p. 982.
(279) Hunter, G. L. K., Parks, G. L., *J. Food Sci.* **29**, 25[illegible]1964).
(280) Hunter, G. L. K., Veldhuis, M. K., *J. Chromatog.* **11**, 11 (1963).
(281) Ikeda, R. M., Rolle, L. A., Vannier, S. H., Stanley, W. L., *J. Agr. Food Chem.* **10**, 98 (1962).
(282) Ikeda, R. M., Spitler, E. M., *Ibid.*, **12**, 114 (1964).
(283) Ikeda, R. M., Stanley, W. L., Rolle, L. A., Vannier, S. H., *J. Food Sci.* **27**, 593 (1962).
(284) Ikeda, R. M., Stanley, W. L., Vannier, S. H., Spitler, E. M., *Ibid.*, p. 455.
(285) Indo, M., *Koryo* **65**, 31 (1961).
(286) Ireland, R. E., Newbould, J., *J. Org. Chem.* **27**, 1931 (1962).
(287) Ishikawa, M., Tsuchiya, T., *Shika Zairyo Kenkyusho Hokoku* **2**, 401 (1962).
(288) Ito, S., Endo, K., Nozoe, T., *Chem. Pharm. Bull. (Tokyo)* **11**, 132 (1963).
(289) Ito, S., Kodama, M., Nozoe, T., Hikino, H., Hikino, Y., Takeshita, Y., Takemoto, T., *Tetrahedron Letters* **1963**, p. 1787.
(290) Jacobsen, J. V., Bernhard, R. A., Mann, L. K., Saghir, A. R., *Arch. Biochem. Biophys.* **104**, 473 (1964).
(291) Jahnsen, V. J., *J. Inst. Brewing* **69**, 460 (1963).
(292) Jahnsen, V. J., *Nature* **196**, 474 (1962).
(293) Jain, M. K., Mishra, R. K., *Indian J. Applied Chem.* **26**, 127 (1964).
(294) Jain, T. C., Maheshwari, M. L., Bhattacharyya, S. C., *Perfumery Essent. Oil Record* **53**, 294 (1962).
(295) Jain, T. C., Varma, K. R., Bhattacharyya, S. C., *Ibid.*, p. 678.
(296) Janackovic, B., *Arhiv. Farm. (Belgrade)* **12**, 229 (1962).
(297) Jansen, H., Van der Holk, H., *Med. Sci. Law* **3**, 77 (1963).
(298) Jaspersen-Schib, R., Flueck, H., *Boll. Chim. Farm.* **101**, 512 (1962).
(299) Jaspersen-Schib, R., Flueck, H., *Congr. Sci. Farm. Conf. Commun., 21°, Pisa* **1961**, 608; *C.A.* **60**, 1533*f*.
(300) Jaureguiberry, G., Wolff, R., *Bull. Soc. Chim. France* **1962**, p. 1985.

(301) Johnston, V. D., *Am. Perfumer Cosmet.* **78** (11), 24 (1963).

(302) *Ibid.*, **79** (6), 63 (1964).

(303) Jordan, D. E., *Anal. Chim. Acta* **30**, 297 (1964).

(304) Kahan, S., Fitelson, J., *J. Assoc. Offic. Agr. Chemists* **47**, 551 (1964).

(305) Kalsi, P. S., Chakravarti, K. K., Bhattacharyya, S. C., *Tetrahedron* **18**, 1165 (1962).

(306) *Ibid.*, **19**, 1073 (1963).

(307) Kapadia, V. H., Nagasampagi, B. A., Naik, V. G., Dev, S., *Tetrahedron Letters* **1963**, p. 1933.

(308) Karawya, M. S., Wahba, S. K., *Bull. Fac. Pharm. (Cairo Univ.)* **1**, 125 (1961–62).

(309) Karawya, M. S., Wahba, S. K., *J. Pharm. Pharmacol.* **14**, 611 (1962).

(310) Karpov, O. N., *Tr. Komis. po Analit. Khim., Akad. Nauk SSSR, Inst. Geokhim. i Analit. Khim.* **13**, 132 (1963).

(311) Kartha, A. R. S., *Indian J. Chem.* **2**, 79 (1964).

(312) Kartha, A. R. S., Duggal, S. P., *Ibid.*, **1**, 405 (1963).

(313) Kartha, A. R. S., Misra, R. C., *Ibid.*, p. 457.

(314) Kartha, C. C., Kalsi, P. S., Shaligram, A. M., Chakravarti, K. K., Bhattacharyya, S. C., *Tetrahedron* **19**, 241 (1963).

(315) Katague, D. B., Kirch, E. R., *J. Pharm. Sci.* **52**, 252 (1963).

(316) Katsuhara, J., Hashimoto, K., Kobayashi, M., *Kogio Kagaku Zasshi* **67**, 821 (1964).

(317) Katsuhara, J., Kobayashi, M., *Nippon Kagaku Zasshi* **83**, 607 (1962).

(318) Kenney, R. L., Fisher, G. S., *J. Gas Chromatog.* **1** (8), 19 (1963).

(319) Kergomard, A., Pigeret, F., Renard, M., *Bull. Soc. Chim. France* **1962**, p. 1648.

(320) Kesterson, J. W., Hendrickson, R., *Am. Perfumer Cosmet.* **77** (12), 21 (1962).

(321) *Ibid.*, **78** (5), 32 (1963).

(322) *Ibid.*, **79**, No. 1, 34–6 (1964).

(323) Kharchenko, L. N., *Maslob.-Zhir. Prom.* **30** (3), 14 (1964).

(324) Kheifits, L. A., Moldovanskaya, G. I., Shulov, L. M., *Zh. Analit. Khim.* **18**, 267 (1963).

(325) Kimura, H., *Yakugaku Zasshi* **82**, 214 (1962).

(326) Kiseleva, E. N., Voitkevich, S. A., *Tr. Vses. Nauchn.-Issled. Inst. Sintetich. i Natural'n. Dushistykh Veshchestv* **1961** (5), p. 116.

(327) Kishore, I., *Soap, Perfumery Cosmetics* **35**, 446 (1962).

(328) Kishore, I., Lal, J. B., Kushwaha, S. C., *J. Oil Technologists Assoc. India* **17**, Pt. 1, 1 (1962).

(329) Kitahara, Y., Yoshikoshi, A., *Tetrahedron Letters* **1964**, p. 1755.

(330) *Ibid.*, p. 1771.

(331) Klein, E., Farnow, H., Rojahn, W., *Ibid.*, **1963**, p. 1109.

(332) Klimova, V. A., Zabrodina, K. S., *Zh. Analit. Khim.* **18**, 109 (1963).

(333) Klouwen, M. H., Heide, R. ter, *Gas Chromatog. Intern. Symp. 1961* **3**, 485 (pub. 1962); *C.A.* **58**, 3873*g*.

(334) Klouwen, M. H., Heide, R. ter, *J. Chromatog.* **7**, 297 (1962).

(335) Klouwen, M. H., Heide, R. ter, *Parfuem. Kosmetik* **43**, 195 (1962).

(336) Klouwen, M. H., Heide, R. ter, *Soap Perfumery Cosmetics* **35**, 1082 (1962).

(337) Klouwen, M. H., Heide, R. ter, Kok, J. G. J., *Fette, Seifen, Anstrichmittel* **65**, 414 (1963).

(338) Knappe, E., Peteri, D., *Z. Anal. Chem.* **190**, 386 (1962).

(339) Kneutter, S., Pohloudek-Fabini, R., *Pharmazie* **17**, 456 (1962).

(340) Kobayashi, H., Akiyoshi, S., *Bull. Chem. Soc. Japan* **35**, 1044 (1962).

(341) *Ibid.*, **36**, 823 (1963).

(342) Kobayashi, H., Nagahama, S., Akiyoshi, S., *Ibid.*, **34**, 1123 (1961).

(343) Kobayashi, M., Yoshigi, H., Kasuhara, J., *Koryo* **60**, 27 (1960).

(344) Kohlmuenzer, S., *Dissertationes Pharm.* **12**, 143 (1960).

(345) Kolsek, J., Maticic, M., *J. Chromatog.* **14**, 331 (1964).

(346) Korte, F., Vogel, J., *Ibid.*, **9**, 381 (1962).

(347) Kovats, E., *Helv. Chim. Acta* **46**, 2705 (1963).

(348) Krasch, H., Suchy, M., Herout, V., Sorm, F., *Collection Czech. Chem. Commun.* **28**, 1618, 1715 (1963).

(349) Krepinsky, J., Herout, V., *Ibid.*, **27**, 2459 (1962).

(350) Krepinsky, J., Romanuk, M., Herout, V., Sorm, F., *Ibid.*, p. 2338.

(351) Krepinsky, J., Romanuk, M., Herout, V., Sorm, F., *Tetrahedron Letters* **1962**, p. 169.

(352) Krepinsky, J., Romanuk, M., Herout, V., Sorm, F., Hoehne, E., *Collection Czech. Chem. Commun.* **28**, 3122 (1963).

(353) Kronrad, L., Panek, K., *Z. Anal. Chem.* **191**, 199 (1962).

(354) Kubrak, M. N., *Tr. po Khim. Prirodn. Soedin., Kishinevsk. Gos. Univ.* **1961** (4), p. 93.

(355) Kucera, J., *Collection Czech. Chem. Commun.* **28**, 1341 (1963).

(356) Kugler, E., Kovats, E. sz., *Helv. Chim. Acta* **46**, 1480 (1963).

(357) Kulesza, J., Cybulska, H., Gora, J., *Zeszyty Nauk. Politech. Lodz. Chem. Spozywcza* **1961** (5), p. 23.

(358) Kulesza, J., Roth, Z., Jaworski, T., *Ibid.*, p. 49.

(359) Kulka, K., *Soap Chem. Specialties* **39** (1), 89, 91, 94, 96 (1963).

(360) Kulkarni, G. H., Bawdekar, A. S., Rao, A. S., Kelkar, G. R., Bhattacharyya, S. C., *Perfumery Essent. Oil Record* **54**, 303 (1963).

(361) Kulkarni, K. S., Paknikar, S. K., Bhattacharyya, S. C., *Tetrahedron* **20**, 1289 (1964).

(362) Kulkarni, K. S., Paknikar, S. K., Vaidya, A. S., Kelkar, G. R., Bates, R. B., Bhattacharyya, S. C., *Tetrahedron Letters* **1963**, p. 505.

(363) Kumar, R. N., Bhat, G. N., Kuloor, N. R., *Current Sci. (India)* **31**, 491 (1962).

(364) Kunth, H. B., Shukla, V. S., Rao, P. R., *Indian Oil Soap J.* **29**, 75 (1963).

(365) Kurko, V. I., Kel'man, L. F., *Myasnaya Ind. SSSR* **34** (6), 50 (1963).

(366) La Bruto, G., *Essence Deriv. Agrumari* **32**, 20 (1962).

(367) *Ibid.*, p. 236.

(368) La Bruto, G., Calvarano, M., *Ibid.*, **29**, 182 (1959).

(369) Laidlaw, R. A., Morgan, J. W. W., *J. Chem. Soc.* **1963**, p. 644.

(370) Lal, J. B., Kushwaha, S. C., Kishore, I., *J. Oil Technologists Assoc. India* **17**, 61 (1962).

(371) Lal, J. B., Patwardhan, V. M., Lal, R. N., *Ibid.*, **16**, 9 (1961).

(372) Lal, R. N., Lal, J. B., *Ibid.*, p. 32.

(373) Lamparsky, D., *Helv. Chim. Acta* **46**, 185 (1963).

(374) Laughton, P. M., Skakum, W., Levi, L., *J. Agr. Food Chem.* **10**, 49 (1962).

(375) Lee, G. D., Kauffman, F. L., Harlan, J. W., Niezabitowski, W., *Gas Chromatog. Intern. Symp. 1961* **3**, 475 (pub. 1962); *C. A.* **58**, 4974*b*.

(376) Levand, O., *Dissertation Abstr.* **24**, 3544 (1964).

(377) Lewe, W. A., *Pharm. Ztg. Ver. Apotheker-Ztg.* **109**, 256 (1964).

(378) Likens, S. T., Nicherson, G. B., *Am. Brewer* **96**, 50 (1963).

(379) Lin, C.-H., Lei, M.-H., Hsieh, C.-C., Yeh, P.-Y., *Chemistry (Taipei)* **1962**, p. 5.

(380) Lipina, T. G., *Tr. po Khim. i Khim. Tekhnol.* **1962** (2), p. 424.

(381) Lippmaa, E., *Tr. Tallinsk. Politekhn. Inst., Ser. A.* **1962** (195), p. 35.

(382) Liu, K.-S., *Yao Hsueh Hsueh Pao* **10** (3), 147 (1963).

(383) Loder, J. W., *Australian J. Chem.* **15**, 389 (1962).

(384) Lucas, R. A., Rovinski, S., Kiesel, R. J., Dorfman, L., MacPhillamy, H. B., *J. Org. Chem.* **29**, 1549 (1964).

(385) Luedde, K. H., *Parfuem. Kosmetik* **43**, 451 (1962).

(386) Lukes, V., Komers, R., *Collection Czech. Chem. Commun.* **29**, 1598 (1964).

(387) Lukic, P., Savin, K., Gorunovic, M., *Arhiv. Farm. (Belgrade)* 12 (2), 69 (1962).

(388) McCarthy, W. C., Bhat, K. V., Roscoe, C. W., Fisher, L., *J. Pharm. Sci.* **52**, 1005 (1963).

(389) McConnell, J. F., Mathieson, A. McI., Shoenborn, B. P., *Tetrahedron Letters* **1962**, p. 445.

(390) McPhail, A. T., Reed, R. I., Sim, G. A., *Chem. Ind. (London)* **1964**, p. 976.

(391) Maheshwari, M. L., Jain, T. C., Bates, R. B., Bhattacharyya, S. C., *Tetrahedron* **19**, 1079 (1963).

(392) Maheshwari, M. L., Varma, K. R., Bhattacharyya, S. C., *Ibid.*, p. 1519.

(393) Makarova, G. M., Borisyuk, Y. G., *Farmatsevt. Zh. (Kiev)* **14** (3), 43 (1959).

(394) Malik, M. N., Khan, F. W., *Pakistan J. Forestry* **14** (1), 81 (1964).

(395) Malla, S., Nigam, M. C., Handa, K. L., *Riechstoffe Aromen* **12**, 221 (1962).

(396) Malla, S., Shukla, V. S., Nigam, M. C., Handa, K. L., *Indian Oil Soap J.* **27**, 173 (1962).

(397) Manfredini, T. A. A., Mizrahi, I., Montes, A. L., *Anales Asoc. Quim. Arg.* **50**, 297 (1962).

(398) Manfredini, T. A. A., Montes, A. L., *Ibid.*, p. 198.

(399) Manjarrez, A., Rios, T., Guzman, A., *Tetrahedron* **20**, 333 (1964).

(400) Mann, C., *Am. Perfumer Cosmet.* **78** (11), 15 (1963).

(401) Marciszewski, H., Trojanowska, Z., Spychala, S., Pogorzelska, J., *Chem. Anal. (Warsaw)* **8**, 939 (1963).

(402) Martin, G. E., Feeny, F. J., Scaringeli, F. P., *J. Assoc. Offic. Agr. Chemists* **47**, 561 (1964).

(403) Martinez Nadal, N. G., Casillas Chapel, C. M., Lecumberry, C., *Am. Perfumer Cosmet.* 79 (4), 43 (1964).

(404) Martin-Smith, M., Smith, S. J., Stenlake, J. B., Williams, W. D., *Tetrahedron Letters* **1963**, p. 1639.

(405) Mathew, C. P., Verghese, J., *Indian J. Chem.* **1**, 229 (1963).

(406) Mathews, W. S., Pickering, G. B., Umoh, A. T., *Chem. Ind. (London)* **1963**, p. 122.

(407) Mathis, C., Ourisson, G., *Phytochemistry* **3** (1), 115 (1964).

(408) Matsuura, T., Komae, H., Aratani, T., Hayashi, S., *Kogyo Kagaku Zasshi* **63**, 1761 (1960).

(409) *Ibid.*, **64**, 791 (1961).

(410) Mattick, L., Robinson, W. B., Weiss, L. D., Barry, D. L., *J. Agr. Food Chem.* **11**, 334 (1963).

(411) Maurel, A., *Compt. Rend. Acad. Agr. France* **48**, 837 (1962).

(412) Mayes, J. A., Kuchar, E. J., Siggia, S., ANAL. CHEM. **36**, 934 (1964).

(413) Mehlitz, A., Minas, T., *Ind. Obst. Gemueseverwert.* 49 (8), 215 (1964).

(414) Mehlitz, A., Minas, T., *Riechstoffe Aromen* **14,** 172 (1964).
(415) Merchant, J. R., Naik, R. M., Hirwe, S. N., *J. Indian Chem. Soc.* **39,** 411 (1962).
(416) Michel, G., *Bull. Soc. Chim. Belges* **72,** 125 (1963).
(417) Mignat, S., Farnow, H., Porsch, F., *Dragoco Rept.* **8,** 290 (1961).
(418) Mignat, S., Porsch, F., *Ibid.*, p. 232, 267.
(419) Minato, H., *Tetrahedron* **18,** 365 (1962).
(420) Minutilli, F., *Rass. Chim.* **15** (3), 105 (1963).
(421) Mirov, N. T., Zaharin, E., Bicho, J. G., *J. Pharm. Sci.* **51,** 1131 (1962).
(422) Mitsuhashi, H., Itoh, T., *Chem. Pharm. Bull.* (*Tokyo*) **10,** 511 (1962).
(423) Mitsuhashi, H., Muramatsu, T., Nagai, V., Nakano, T., Ueno, K., *Ibid.*, **11,** 1317 (1963).
(424) Mitzner, B. M., ANAL. CHEM. **36,** 242 (1964).
(425) Mohan, V., *Indian Oil Soap J.* **28,** 100 (1962).
(426) *Ibid.*, **28,** 191 (1963).
(427) *Ibid.*, p. 296.
(428) Mellan, T. R. M., *Perfumery Essent. Oil Record* **52,** 411 (1961).
(429) Monselise, J. J., *Bull. Res. Council Israel Sect. C* **10** (3), 137 (1961).
(430) Montes, A. L., *Anales Asoc. Quim. Arg.* **50,** 65 (1962).
(431) *Ibid.*, p. 111.
(432) Montes, A. L., *Soc. Arg. Investigacion Productos Aromaticos Boletin* (*Buenos Aires*) **1963** (4), p. 4.
(433) *Ibid.*, (5), 6.
(434) Moryashchev, A. K., Voronin, V. G., *Zh. Analit. Khim.* **18,** 401 (1963).
(435) Motl, O., Bucharov, V. G., Herout, V., Sorm, F., *Chem. Ind.* (*London*) **1963,** p. 1759.
(436) Motl, O., Lukes, V., *Collection Czech. Chem. Commun.* **27,** 987 (1962).
(437) Motl, O., Trivedi, B., Herout, V., Sorm, F., *Chem. Ind.* (*London*) **1963,** p. 1284.
(438) Mukherjee, S. K., Bose, A. N., *J. Proc. Inst. Chemists* (*India*) **34,** 233 (1962).
(439) Nakajima, T., *Yakugaku Zasshi* **82,** 1278 (1962).
(440) *Ibid.*, p. 1323.
(441) Nakazaki, M., *Bull. Chem. Soc. Japan* **35,** 1387 (1962).
(442) *Ibid.*, p. 1904.
(443) Nano, G. M., Sancin, P., *Ann. Chim.* (*Rome*) **53,** 677 (1963).
(444) Naryanan, C. S., Kulkarni, K. S., Vaidya, A. S., Kanthamani, S., Laksmi Kumari, G., Badat, B. V., Paknikar, S. K., Kulkarni, S. N., Kelkar, G. R., Bhattacharyya, S. C., *Tetrahedron* **20,** 963 (1964).
(445) Nathorst-Westfelt, L., *Acta Chem. Scand.* **18,** 572 (1964).
(446) Naves, Y.-R., *Bull. Soc. Chim. France* **1963,** p. 681.
(447) *Ibid.*, p. 1195.
(448) Naves, Y.-R., *Helv. Chim. Acta* **45,** 1598 (1962).
(449) *Ibid.*, **46,** 2139 (1963).
(450) *Ibid.*, **47,** 308 (1964).
(451) Naves, Y.-R., *J. Soc. Cosmetic Chemists* **14,** 29 (1963).
(452) Naves, Y.-R., *Parfuem. Kosmetik* **43,** 461 (1962).
(453) Naves, Y.-R., Alves, H. M., Arndt, V. H., Gottlieb, O. R., Magalhaes, M. T., *Helv. Chim. Acta* **46,** 1056 (1963).
(454) Naves, Y.-R., Ardizio, P., *Ibid.*, **47,** 111 (1964).
(455) Naves, Y.-R., Frei, C., *Ibid.*, **46,** 2551 (1963).
(456) Naves, Y.-R., Grampoloff, A. V., *Ibid.*, **45,** 1161 (1962).
(457) Naves, Y.-R., Grampoloff, A. V., *Ibid.*, p. 1955.
(458) Naves, Y.-R., Grampoloff, A. V., Demole, E., *Ibid.*, **46,** 1006 (1963).
(459) Naves, Y.-R., Ochsner, P., *Ibid.*, **47,** 51 (1964).
(460) Naves, Y.-R., Ochsner, P., Thomas, A. F., Lamparsky, D., *Bull. Soc. Chim. France* **1963,** p. 1608.
(461) Naves, Y.-R., Tullen, P., *Helv. Chim. Acta* **43,** 2150 (1960).
(462) Nayak, U. R., Dev, S., *Tetrahedron Letters* **1963,** p. 243.
(463) Nettesheim, G., *Z. Anal. Chem.* **191,** 45 (1962).
(464) Newhall, W. F., Kesterson, J. W., *Proc. Florida State Hort. Soc.* **74,** 239 (1961).
(465) Nickon, A., McGuire, F. J., Mahajan, J. R., Umezawa, B., Narang, S. A., *J. Amer. Chem. Soc.* **86,** 1437 (1964).
(466) Nigam, I. C., Levi, L., ANAL. CHEM. **35,** 1087 (1963).
(467) Nigam, I. C., Levi, L., *Can. J. Chem.* **41,** 1726 (1963).
(468) *Ibid.*, **40,** 2083 (1962).
(469) Nigam, I. C., Levi, L., *Perfumery Essent. Oil Record* **54,** 814 (1963).
(470) Nigam, I. C., Nigam, M. C., Dhingra, D. R., *Ibid.*, **53,** 302 (1962).
(471) Nigam, I. C., Sahasrabudhe, M., Levi, L., *Can. J. Chem.* **41,** 1535 (1963).
(472) Nigam, I. C., Sahasrabudhe, M., McConnell Davis, T. W., Bartlet, J. C., Levi, L., *Perfumery Essent. Oil Record* **53,** 614 (1962).
(473) Nigam, I. C., Skakum, W., Levi, L., *Ibid.*, **54,** 25 (1963).
(474) Nigam, M. C., Handa, K. L., *Indian Perfumer* **5,** Pt. 2, 119 (1961).
(475) Nigam, M. C., Kapoor, L. D., Handa, K. L., *Soap, Perfumery, Cosmet.* **36,** 686 (1963).
(476) Nigam, S. S., Kumari, G. L., *Perfumery Essent. Oil Record* **53,** 529 (1962).
(477) *Ibid.*, p. 752.
(478) Nigam, S. S., Kumari, G. L., *Riechstoffe Aromen* **13,** 1 (1963).
(479) Nigam, S. S., Purohit, R. M., *Indian Perfumer* **5,** Pt. 1, 3 (1961).
(480) Nigam, S. S., Purohit, R. M., *Riechstoffe Aromen* **12,** 185 (1962).
(481) Nigam, S. S., Radhakrishnan, C., *Perfumery Essent. Oil Record* **54,** 87 (1963).
(482) Nigam, S. S., Radhakrishnan, C., *Riechstoffe Aromen* **12,** 357 (1962).
(483) *Ibid.*, **13,** 293 (1963).
(484) *Ibid.*, p. 357.
(485) Nilov, V. V., *Vinodelie i Vinogradarstvo SSSR* **22** (4), 28 (1962).
(486) Nishimura, K., Hirose, Y., *Nippon Kagaku Zasshi* **83,** 767 (1962).
(487) Norin, T., *Acta Chem. Scand.* **15,** 1676 (1961).
(488) *Ibid.*, **16,** 640 (1962).
(489) *Ibid.*, **17,** 738 (1963).
(490) Norin, T., *Arkiv. Kemi* **22** (10), 123 (1964).
(491) *Ibid.*, (11), p. 129.
(492) *Ibid.*, (12), p. 137.
(493) Novikova, E. N., Plyushchevskii, N. I., Makarevich, N. I., *Dokl. Akad. Nauk Belorussk. SSR* **4,** 514 (1960).
(494) Novotny, L., Herout, V., *Collection Czech. Chem. Commun.* **27,** 1508 (1962).
(495) *Ibid.*, p. 2462.
(496) Novotny, L., Herout, V., Sorm, F., *Ibid.*, p. 1400.
(497) Novotny, L., Jizba, J., Herout, V., Sorm, F., *Ibid.*, p. 1393.
(498) Novotny, L., Jizba, J., Herout, V., Sorm, F., *Tetrahedron* **19,** 1101 (1963).
(499) Obata, S., Hayashi, K., Ichikawa, T., Matsui, K., Nagasaka, M., *Shokuhin Eiseigaku Zasshi* **4,** 375 (1963).
(500) Obtemperanskaya, S. I., Volodz'ko, V. E., *Zh. Analit. Khim.* **18,** 1483 (1963).
(501) Ognyanov, I., Mkhailov, M., Burogudzkiev, Z., *Compt. Rend. Acad. Bulgare Sci.* **16,** 517 (1963).
(502) Ognyanov, I., Vlahov, R., *Ibid.*, **15,** 395 (1962).
(503) Ohloff, G., Osiecki, J., Djerassi, C., *Ber.* **95,** 1400 (1962).
(504) Okamoto, T., Onaka, T., *Chem. Pharm. Bull.* (*Tokyo*) **11,** 1086 (1963).
(505) Okamura, H., Noriishi, S., Tanaka, T., *Koryo* **67,** 27 (1962).
(506) Okazawa, T., *Ibid.*, **60,** 11 (1960).
(507) Okuda, T., *Chem. Pharm. Bull.* (*Tokyo*) **10,** 96 (1962).
(508) Olechnowicz-Stepien, W., *Dissertationes Pharm.* **15** (3), 301 (1963).
(509) Omboly, C., *Acta Pharm. Hung.* **33,** 25 (1963).
(510) Onoda, R., *Nippon Kagaku Zasshi* **82,** 1721 (1961).
(511) Onogaki, T., Urishido, H., *Koryo* **62,** 36 (1961).
(512) Os, F. H. L. van, *Congr. Sci. Farm. Conf. Commun. 21°, Pisa* **1961,** p. 754; *C. A.* 59, 3713*e*.
(513) Oswięcimska, M., *Dissertationes Pharm.* **14,** 539 (1962).
(514) Ota, Y., Nishimura, K., Hirose, Y., *Agr. Biol. Chem.* (*Tokyo*) **28,** 5 (1964).
(515) Paknikar, S. K., Bhattacharyya, S. C., *Tetrahedron* **18,** 1509 (1962).
(516) Palmade, M., Pesnelle, P., Streith, J., Ourisson, G., *Bull. Soc. Chim. France* **1963,** p. 1950.
(517) Parczewski, A., *Dissertationes Pharm.* **15,** 35 (1963).
(518) Parczewski, A., *Riv. Ital. Essenze-Profumi, Piante Offic. Oli. Vegetali-Saponi* **44,** 454 (1962).
(519) Parczewski, A., Rajkowski, Z., *Dissertationes Pharm.* **14,** 83 (1962).
(520) Parekh, C. M., Pruthi, J. S., Jain, N. L., Tandon, G. L., Lal, G., *Food Sci.* (*Mysore*) **10,** 358 (1961).
(521) Parekh, C. M., Pruthi, J. S., Lal, G., Subrahmanyan, V., *Indian Std. Conv., Papers Session T-7, Latest Tech. Chem. Anal. Part II, Hyderbad* **1959-1960,** *Indian Standards Institution, Doc.; CA* **32,** 8 pp.; *C.A.* **57,** 7693*d*.
(522) Paris, R., Godon, M., *Recherches* (*Paris*) **1963** (13), p. 48.
(523) Park, G. J., *Dissertation Abstr.* **23,** 1517 (1962).
(524) Parker, B. A., Geissman, T. A., *J. Org. Chem.* **27,** 4127 (1962).
(525) Pascual Teresa, J. de, Sanchez Bellido, H., Sanchez Bellido, I., *Anales Real Soc. Espan. Fis. Quim.* (*Madrid*) *Ser. B* **58,** 339 (1962).
(526) Pascual Teresa, J. de, Sanchez Bellido, I., *Ibid.*, p. 351.
(527) Patton, S., *Microchem, J. Symp. Ser.* **2,** 757 (1962).
(528) Perel'son, M. E., *Zh. Obshch. Khim.* **33,** 952 (1963).
(529) Pertsev, I. M., Pivnenko, G. P., *Farmatsevt. Zh.* (*Kiev*) **17** (1), 21 (1962).
(530) *Ibid.*, (2), p. 35.
(531) Pesnelle, P., Ourisson, G., *Bull. Soc. Chim. France* **1963,** p. 912.
(532) Petrova, L. N., Skvortsova, A. B., Novikova, E. N., *Zh. Analit. Khim.* **18,** 131 (1963).
(533) Petrowitz, J., *Reichstoffe Aromen* **13,** 273 (1963).
(534) Peyron, L., *Compt. Rend.* **255,** 2981 (1962).
(535) Peyron, L., *Parfum., Cosmet., Savons* **5,** 270 (1962).
(536) Pichler, H., Schulz, H., Firnhaber, B., Rao, R. B., *Erdoel Kohle* **16,** 577 (1963).
(537) Pigulevskii, G. V., Belova, N. V., *Zh. Obshch. Khim.* **34,** 1345 (1964).
(538) Pigulevskii, G. V., Borovkov, A. V., *Ibid.*, **32,** 3106 (1962).
(539) Pigulevskii, G. V., Borovkov, A. V., *Zh. Prikl. Khim.* **36,** 831, 926, 929 (1963).

(540) Pigulevskii, G. V., Kovaleva, V. I., *Zh. Obshch. Khim.* **33**, 705 (1963).
(541) Pigulevskii, G. V., Motskus, D. V., *Ibid.*, **32**, 2365 (1962).
(542) Pigulevskii, G. V., Motskus, D. V., *Zh. Prikl. Khim.* **35**, 1355–60 (1962).
(543) Pigulevskii, G. V., Motskus, D. V., Rodina, L. L., *Zh. Obsch. Khim.* **32**, (1962) 656.
(544) Pigulevskii, G. V., Motskus, D. V., Rodina, L. L., *Zh. Prikl. Khim.* **35**, 1143 (1962).
(545) Pinder, A. R., Williams, R. A., *Chem. Ind.* (*London*) **1961**, p. 1714.
(546) Pohloudek-Fabini, R., Goeckeritz, D., *Nahrung* **7** (2), 122 (1963).
(547) Pohloudek-Fabini, Munchow, P., *Pharmazie* **18**, 734 (1963).
(548) Polyakov, A. F., Falina, L. A., *Izv. Vysshikn. Uchebn. Zavendenii, Pishchevaya Tekhnol.* **1962** (6), 139.
(549) Porcaro, P. J., *J. Gas Chromatog.* **1** (6), 17 (1963).
(550) Porcaro, P. J., Johnston, V. D., ANAL. CHEM. **34**, 1071 (1962).
(551) Porsch, F., Farnow, H., *Dragoco Report* **9**, 163 (1962).
(552) *Ibid.*, p. 194.
(553) Potapov, V. M., Moiseeva, G. P., Terent'ev, A. P., *Vestn. Mosk. Univ., Ser. II, Khim.* **18** (4), 28 (1963).
(554) Potapov, V. M., Moiseeva, G. P., Terent'ev, A. P., *Zh. Analit. Khim.* **18**, 275 (1963).
(555) Potapov, V. M., Terent'ev, A. P., Avakyan, V. G., *Ibid.*, p. 116.
(556) Prabucki, A. L., Lenz, F., *Helv. Chim. Acta* **45**, 2012 (1962).
(557) Prokopenko, A. P., Tarasenko, O. O., *Farmatsevt. Zh.* (*Kiev*) **17** (6), 18 (1962).
(558) Pruthi, J. S., Parekh, C. M., Jain, N. L., Lal, G., Subrahmanyan, V., *Food Sci.* (*Mysore*) **10**, 363 (1961).
(559) Rajkowski, Z., *Acta Polon. Pharm.* **19**, 383 (1962).
(560) Ralls, J. W., ANAL. CHEM. **36**, 946 (1964).
(561) Ramaswami, S. K., Bhattacharyya, S. C., *Tetrahedron* **18**, 575 (1962).
(562) Ramshaw, E. H., *J. Chromatog.* **10**, 303 (1963).
(563) Rao, A. A., Surve, K. L., Chakravarti, K. K., Bhattacharyya, S. C., *Tetrahedron* **19**, 233 (1963).
(564) Rao, G. S. K., Nigam, I. C., Bartlet, J. C., Levi, L., *Proc. Sci. Sec. Toilet Goods Assoc.* **38**, 5 (1962).
(565) Rao, P. S., Sood, V. K., *Parfuem. Kosmetik* **45**, 63 (1964).
(566) Rao, P. S., Sood, V. K., *Soap Perfumery Cosmetics* **35**, 527 (1962).
(567) Rashkes, Y. V., Melkanovitskaya, S. G., *Zh. Analit. Khim.* **17**, 751 (1962).
(568) Rath, G., *Instr. Meas., Chem. Anal., Elec. Quant., Nucleonics Process Control, Proc. Intern. Conf., Stockholm,* **1960** (1), 498 (pub. 1961); *C. A.* **57**, 6588*b*.
(569) Retamar, J. A., Mazzola, E. B., *Rev. Fac. Ing. Quim., Univ. Nacl. Litoral, Santa Fe, Arg.* **31**, 71 (1962).
(570) Retamar, J. A., Ricciardi, A. I. A., *Ibid.*, **29**, 55 (1960).
(571) Ricciardi, A. I. A., Burgos, J. L., Cassano, A. E., *Ibid.*, **30**, 37 (1961).
(572) Ricciardi, A. I. A., Cassano, A. E., *Ibid.*, **31**, 49 (1962).
(573) Ricciardi, A. I. A., Cassano, A. E., Burgos, J. L., *Ibid.*, **30**, 27 (1961).
(574) *Ibid.*, **31**, 43 (1962).
(575) Richter, G., Muscholl, P., *J. Chromatog.* **14**, 439 (1964).
(576) Rillaers, G., Verzele, M., *Bull. Soc. Soc. Chim. Belges* **71**, 438 (1962).
(577) Rispoli, G., DiGiacomo, A., Crupi, F., *Riv. Ital. Essenze-Profumi, Piante Offic. Oli Vegetali-Saponi* **44**, 314 (1962).
(578) Rispoli, G., DiGiacomo, A., Tracuzzi, M. L., *Riv. Ital. Essenze-Profumi, Piante Offic.-Aromi-Saponi-Cosmet.* **45**, 62 (1963).
(579) *Ibid.*, p. 445.
(580) Roberts, D. L., Rowland, R. L., *J. Org. Chem.* **27**, 3989 (1962).
(581) Roberts, J. B., *J. Inst. Brewing* **69**, 343 (1963).
(582) Robinson, J. W., *Anal. Chim. Acta* **27**, 377 (1962).
(583) Rogers, J. A., *Am. Perfumer Cosmet.* **78** (11), 17 (1963).
(584) Rogozinski, M., *J. Gas Chromatog.* **2** (4), 136 (1964).
(585) Romanuk, M., Herout, V., Sorm, F., Naves, Y.-R., Tullen, P., Bates, R. B., Sigel, C. W., *Collection Czech. Chem. Commun.* **29**, 1048 (1964).
(586) Romo, J., Joseph-Nathan, P., Diaz, F., *Chem. Ind.* (*London*) **1963**, p. 1839.
(587) Romo, J., Joseph-Nathan, P., Diaz, F., *Tetrahedron* **20**, 79 (1964).
(588) Romo, J., Romo de Vivar, A., Herz, W., *Ibid.*, **19**, 2317 (1963).
(589) Rothe, H., *Intern. Fruchtsaft-Union, Ber. Wiss.-Tech. Komm.* **1959** (2), p. 44.
(590) Royals, E. E., Leffingwell, J. C., *J. Am. Chem. Soc.* **86**, 2067 (1964).
(591) Ruch'eva, N. I., Fillippov, M. P., *Zh. Analit. Khim.* **19**, 386 (1964).
(592) Rudloff, E. von, *Can. J. Chem.* **41**, 1 (1963).
(593) *Ibid.*, p. 1737.
(594) *Ibid.*, p. 2876.
(595) *Ibid.*, **42**, 635 (1964).
(596) *Ibid.*, p. 1057.
(597) Rudloff, E. von, *Phytochemistry* **1**, 195 (1962).
(598) Rudloff, E. von, *Tappi* **45**, 181 (1962).
(599) Rudloff, E. von, Erdtman, H., *Tetrahedron* **18**, 1315 (1962).
(600) Rudloff, E. von, Nair, G. V., *Can. J. Chem.* **42**, 421 (1964).
(601) Rudman, P., *Chem. Ind.* (*London*) **1964**, p. 808.
(602) Rybalko, K. S., *Zh. Obshch. Khim.* **33**, 2734 (1963).
(603) Rybalko, K. S., Abbsaov, R. M., *Ibid.*, p. 1700.
(604) Rybalko, K. S., Ban'kovskaya, A. N., Evstratova, R. I., *Med. Prom. SSSR* **26** (3), 13 (1962).
(605) Rybalko, K. S., Ban'kovskii, A. I., Kibalchich, P. N., *Zh. Obshch. Khim.* **34**, 1358 (1964).
(606) Ryhage, R., Von Sydow, E., *Acta Chem. Scand.* **17**, 2025 (1963).
(607) Sadgopal, D., *France Parfums* **5**, 313 (1962).
(608) *Ibid.*, **7**, 40 (1964).
(609) Sadgopal, D., *Soap, Perfumery Cosmetics* **32**, 801 (1959).
(610) Safina, G., *Perfumery Essent. Oil Record* **53**, 81 (1962).
(611) Sakai, T., Nishimura, K., Chikamatsu, H., Hirose, Y., *Bull. Chem. Soc. Japan* **36**, 1261 (1963).
(612) Sakai, T., Nishimura, K., Hirose, Y., *Nippon Kagaku Zasshi* **82**, 1716 (1961).
(613) *Ibid.*, **83**, 745 (1962).
(614) Sakai, T., Nishimura, K., Hirose, Y., *Tetrahedron Letters* **1963**, p. 1171.
(615) Sanchez-Viesca, F., Romo, J., *Tetrahedron* **19**, 1285 (1963).
(616) Sandermann, W., Bruns, K., *Tetrahedron Letters* **1962**, p. 261.
(617) Sandermann, W., Schweers, W., *Ibid.*, p. 257.
(618) *Ibid.*, p. 259.
(619) Sawicki, E., Stanley, T. W., Elbert, W., *Microchem. J. Symp. Ser.* **2**, 633 (1962).
(620) Saxby, M. J., ANAL. CHEM. **36**, 1145 (1964).
(621) Schmidt, H., Bernhardt, D., Muehlstaedt, M., *Arch. Pharm.* **296**, 544 (1963).
(622) Schole, J., *Z. Anal. Chem.* **193**, 321 (1963).
(623) Schroeder, H., *Pharmazie* **17**, 555 (1962).
(624) Schultz, O. E., Eckstein, K., *Arzneimittel-Forsch.* **12**, 1005 (1962).
(625) Schwartz, D. P., Parks, O. W., *Microchem. J.* **7**, 6, 403 (1963).
(626) Schwenker, G., Kloehn, W., *Arch. Pharm.* **296**, 844 (1963).
(627) Seed, V. K., Karnek, M. G., *Perfumery Essent. Oil Record* **54**, 576 (1963).
(628) Seher, A., *J. Soc. Cosmetic Chemists* **13**, 385 (1962).
(629) Seidel, C. F., Stoll, M., *Chimia* (*Switz*). **15**, 311 (1961).
(630) Self, R., Land, D. G., Casey, J. C., *J. Sci. Food Agr.* **14**, 209 (1963).
(631) Senic, R. N., *Glasnik Sumarskog. Fak. Univ. Beogradu* **17**, 82 pp. (1959).
(632) Shaligram, A. M., Rao, A. S., Bhattacharyya, S. C., *Tetrahedron* **18**, 969 (1962).
(633) Shaposhnikov, Y. K., Vedeneev, K. P., Vodzinskii, Y. V., Lazareva, N. K., *Gidrolizn. i Lsokhim. Prom.* **15** (6), 22 (1962).
(634) Sharma, M. L., Handa, K. L., *Indian Oil, Soap J.* **29**, 134 (1963).
(635) *Ibid.*, p. 160.
(636) Sharma, M. L., Nigam, M. C., Handa, K. L., *Parfuem. Kosmetik* **43**, 505 (1962).
(637) Sharma, M. L., Nigam, M. C., Handa, K. L., *Perfumery Essent. Oil Record* **54**, 579 (1963).
(638) Sharma, M. L., Nigam, M. C., Handa, K. L., *Riechstoffe Aromen* **13**, 33 (1963).
(639) Shelley, R. N., Salwin, H., Horwitz, W., *J. Assoc. Offic. Agr. Chemists* **46**, 486 (1963).
(640) Sherma, J. A., Locke, D., Bassett, D., *J. Chromatog.* **7**, 273 (1962).
(641) Shigematsu, N., Kitazawa, Y., *Jozo Kagaku Kenkyu Hokoku* **8**, 23 (1962).
(642) Shimizu, S., Ikeda, N., *Agr. Biol. Chem.* (*Tokyo*) **26**, 543 (1962).
(643) Shimizu, S., Ikeda, N., *Nippon Nogeikagaku Kaishi* **36**, 907 (1962).
(644) Shukla, V. S., Nigam, M. C., Handa, K. L., *Parfuem. Kosmetik* **43**, 243 (1962).
(645) Shulgin, A. T., *Nature* **197**, 379 (1963).
(646) Sidorov, R. I., Baboshin, B. K., Rudakov, G. A., *Gidrolizn. i Lesokhim. Prom.* **16** (2), 12 (1963).
(647) Singh, K. P., Gupta, G. N., *Indian Perfumer* **5**, Pt. 1, 23 (1961).
(648) Skvortsova, A. B., Petrova, L. N., Novikova, E. N., *Zh. Analit. Khim.* **17**, 896 (1962).
(649) Slater, C. A., *J. Sci. Food Agr.* **14**, 58 (1963).
(650) Smith, D. M., Skakum, W., Levi, L., *J. Agr. Food Chem.* **11**, 268 (1963).
(651) Smith, H. E., Gordon, A. W., *J. Am. Chem. Soc.* **84**, 2840 (1962).
(652) Smith, R. H., *Phytochemistry* **3**, 259 (1964).
(653) Smith, R. H., *Science* **143**, 1337 (1964).
(654) Soffer, M. D., Gunay, G. E., Korman, O., Adams, M. B., *Tetrahedron Letters* **1963**, p. 389.
(655) Solodovnichenko, N. M., *Tr. Khar'kovsk. Farmatsevt. Inst.* **1962** (2), p. 30.
(656) Solodovnichenko, N. M., Borisyuk, Y. G., *Farmatsevt. Zh.* (*Kiev*) **17** (2), 44 (1962).
(657) Sood, V. K., *Indian J. Chem.* **2**, 170 (1964).
(658) Soucek, M., *Collection Czech. Chem. Commun.* **27**, 2929 (1962).
(659) Soucek, M., Vlad, P., *Chem. Ind.* (*London*) **1962**, p. 1946.
(660) Spetzig, L. O., *Acta Chem. Scand.* **16**, 1586 (1962).
(661) Sprechler, E., *Planta Med.* **11**, 119 (1963).

(662) Sprecker, E., Strackenbrock, K. H., *Z. Naturforsch.* **18b**, 495 (1963).

(663) Srivas, S. R., Pruthi, J. S., Siddappa, G. S., *Food Sci.* (*Mysore*) **12**, 340 (1963).

(664) Stanley, W. L., *Intern. Fruchtsaft-Union, Ber. Wiss.-Tech. Komm.* **4**, 91 (1962).

(665) Starkovsky, N. A., *J. Org. Chem.* **27**, 3733 (1962).

(666) Stedman, R. L., Miller, R. L., *J. Chromatog.* **11**, 409 (1963).

(667) Stevens, R., *J. Chem. Soc.* **1964**, p. 952.

(668) Stevens, R., Wright, D., *Ibid.*, **1963**, p. 1763.

(669) Stevenson, R., *Chem. Ind.* (*London*) **1962**, p. 270.

(670) Stoker, J. R., Bellis, D. M., *J. Biol. Chem.* **237**, 2303 (1962).

(671) Streith, J., Pesnelle, P., Ourisson, G., *Bull. Soc. Chim. France* **1963**, p. 518.

(672) Streith, J., Pesnelle, P., Ourisson, G., *Tetrahedron Letters* **1962**, p. 677.

(673) Suchy, M., *Collection Czech. Chem. Commun.* **27**, 2925 (1962).

(674) Suchy, M., Herout, V., Sorm, F., *Ibid.*, p. 1905.

(675) *Ibid.*, **28**, 2257 (1963).

(676) Suga, K., Watanabe, S., *Chiba Daigaku Kogakuba Kenkyu Hokoku* **13** (23), 9 (1962).

(677) Suga, K., Watanabe, S., *Koryo* **62**, 44 (1961).

(678) Sugathan, K. K., Verghese, J., *J. Sci. Ind. Res.* (*India*) **21B**, 192 (1962).

(679) Sully, B. D., *Analyst* **87**, 940 (1962).

(680) Sundt, E., Willhalm, B., Stoll, M., *Helv. Chim. Acta* **47**, 408 (1964).

(681) Sutherland, M. D., *Australian J. Chem.* **17**, 75 (1964).

(682) Sutherland, M. D., Waters, O. J., *Ibid.*, **14**, 596 (1961).

(683) Suzuki, Y., Takeuchi, T., Maruta, S., *Kogyo Kagaku Zasshi* **67**, 301 (1964).

(684) Sviderskaya, Z. I., *Ref. Zh. Khim.* **1963**, Abstr. No. 17 R, 301.

(685) Swaleh, M., Bhushan, B., Sidhu, G. S., *Perfumery Essent. Oil Record* **54**, 295 (1963).

(686) Swaleh, M., Srivastava, L. M., Bhushan, B., *Ibid.*, p. 375.

(687) Tabacik-Wlotzka, C., *Bull. Soc. Chim. France* **1964**, p. 618.

(688) Tabacik-Wlotzka, C., Mousseron, M., Chafai, A., *Ibid.*, **1963**, p. 2299.

(689) Taga, J., *Chem. Pharm. Bull.* (*Tokyo*) **12**, 389 (1964).

(690) Takeda, K., Ikuta, M., *Tetrahedron Letters* **1964**, p. 277.

(691) Takeda, K., Minato, H., Horibe, I., *Tetrahedron* **19**, 2307 (1963).

(692) Talwar, Y. P., Handa, K. L., *Parfuem. Kosmetik* **45**, 127 (1964).

(693) Talwar, Y. P., Handa, K. L., *Perfumery Essent. Oil Record* **55**, 178, 183 (1964).

(694) *Ibid.*, p. 391.

(695) Talwar, Y. P., Handa, K. L., *Soap, Perfumery Cosmetics* **37**, 421 (1964).

(696) Talwar, Y. P., Nigam, M. C., Dutta, A. K., Handa, K. L., *Indian Perfumer* **5**, Pt. 2, 111 (1961).

(697) Talwar, Y. P., Nigam, M. C., Handa, K. L., *Indian Oil Soap J.* **28**, 154 (1962).

(698) *Ibid.*, **28**, 249 (1963).

(699) *Ibid.*, p. 274.

(700) Talwar, Y. P., Nigam, M. C., Handa, K. L., *Parfuem. Kosmetik* **44**, 93 (1963).

(701) Talwar, Y. P., Nigam, M. C., Handa, K. L., *Perfumery Essent. Oil Record* **53**, 822 (1962).

(702) Talwar, Y. P., Nigam, M. C., Handa, K. L., *Riechstoffe Aromen* **12**, 285 (1962).

(703) Talwar, Y. P., Nigam, M. C., Handa, K. L., Kapoor, D. L., *Ibid.*, **14**, 1 (1964).

(704) Tanaka, Y., *Kinki Daigaku Yakugakkubu Kuyo* **4**, 27 (1963).

(705) Tanker, M., *Eczacilik Bulteni* **4**, 195 (1962).

(706) Taylor, D. A. H., *Chem. Ind.* (*London*) **1961**, p. 1712.

(707) Terada, H., Tsuda, S., Shono, T., *Kogyo Kagaku Zasshi* **65**, 1569 (1962).

(708) Teranishi, R., Schultz, T. H., McFadden, W. H., Lundin, R. E., Black, D. R., *J. Food Sci.* **28**, 541 (1963).

(709) Teshima, T., *Koryo* **61**, 51 (1961).

(710) Tetenyl, P., Tyihak, E., Mathe, I., Svab, J., *Pharmazie* **17**, 463 (1962).

(711) Thiele, F. C., Dean, D. E., Suffis, R., *Am. Perfumer Cosmet.* **77** (10), Sec. 2, 43 (1962).

(712) Theimer, E. T., Somerville, W. T., Mitzner, B. M., Lemberg, S., *Ibid.*, **78** (8), 29 (1963).

(713) Thomas, A. F., Willhalm, B., *Helv. Chim. Acta* **47**, 475 (1964).

(714) Tomlinson, C., *Talanta* **9**, 1065 (1962).

(715) Topalov, P. G., Ivanov, A., Georgiev, E. V., *Nauchn. Tr. Vissh. Inst. po Khraniteliva i Vkusova Prom. Plovdiv* **9**, 87 (1962).

(716) Trabaud, L., *France Parfums* **7**, 141 (1964).

(717) Trivedi, B., Notl, O., Smolikova, J., Sorm, F., *Tetrahedron Letters* **1964**, p. 1197.

(718) Tsonev, I., Chenikov, V., *Bulgar. Tyutyun* **7** (7-8), 37 (1962).

(719) *Ibid.*, (9), p. 17.

(720) Tucakov, J., *Archiv. Farm.* (*Belgrade*) **12** (2), 83 (1962).

(721) Tyihak, E., Vagujfalvi, D., Hagony, P. L., *J. Chromatog.* **11**, 45 (1963).

(722) Ueda, T., *Nippon Kagaku Zasshi* **83**, 338 (1962).

(723) Ueda, T., Fujita, Y., *Chem. Ind.* (*London*) **1962**, p. 1618.

(724) Valkanas, G., Iconomou, N., *J. Chromatog.* **12**, 536 (1963).

(725) Varma, K. R., Jain, T. C., Bhattacharyya, S. C., *Tetrahedron* **18**, 979 (1962).

(726) Vashist, V. N., Handa, K. L., *Soap Perfumery Cosmetics* **37**, 135 (1964).

(727) Vashist, V. N., Nath, Y., Nazir, B. N., Handa, K. L., *Indian Oil Soap J.* **27**, 229 (1962).

(728) Vashist, V. N., Nigam, M. C., Handa, K. L., *Indian Perfumer* **6**, Pt. 1-2, 13 (1962).

(729) Vashist, V. N., Nigam, M. C., Handa, K. L., *Parfuem. Kosmetik* **44**, 43 (1963).

(730) Vashist, V. N., Nigam, M. C., Handa, K. L., Gupta, G. N., *Indian Oil Soap J.* **29**, 45 (1963).

(731) Vashist, V. N., Nigam, M. C., Handa, K. L., Gupta, G. N., *Riechstoffe Aromen* **13**, 61 (1962).

(732) Vashist, V. N., Nigam, M. C., Handa, K. L., Kapoor, L. D., *Proc. Natl. Acad. Sci. India* Sect. A **33**, Pt. 3, 317 (1963).

(733) Venkataraghavan, R., Rao, C. N. R., *Chemist Analyst* **51**, 48 (1962).

(734) Vlad, P., Soucek, M., *Collection Czech. Chem. Commun.* **27**, 1726 (1962).

(735) Von Sydow, E., *Acta Chem. Scand.* **17**, 2504 (1963).

(736) Vrkoc, J., Herout, V., Sorm, F., *Collection Czech. Chem. Commun.* **28**, 1084 (1963).

(737) Vrkoc, J., Jonas, J., Herout, V., Sorm, F., *Ibid.*, **29**, 539 (1964).

(738) Vrkoc, J., Krepinsky, J., Herout, V., Sorm, F., *Ibid.*, p. 795.

(739) Vrkoc, J., Krepinsky, J., Herout, V., Sorm, F., *Tetrahedron Letters* **1963**, p. 225.

(740) Vul'fson, N. S., Zaretsky, V. I., Zaikin, V. G., *Izvest. Akad. Nauk SSSR, Seriya Khim.* **12**, 2215 (1963).

(741) Waginaire, L., Guillot, B., *Perfumery Essent. Oil Record* **54**, 241 (1963).

(742) Wahid, I. M. A., Ikram, M., *Pakistan J. Sci. Ind. Res.* **4** (1), 40 (1961).

(743) Wang, K. T., Weinstein, B., *Experientia* **19**, 519 (1963).

(744) Wang, L. S., Bekker, A., Yang, C. C., Nesmeyanov, A. N., *Izv. Vysshikh Uchebn. Zavendenii, Khim. i Khim. Tekhnol.* **6**, 597 (1963).

(745) Wasicky, R., *Rev. Fac. Farm. Bioquim. Univ. Sao Paulo* **1** (1), 77 (1963).

(746) Wellendorf, M., *Dansk Tidskr. Farm.* **37** (7), 145 (1963).

(747) Weiss, U., Ziffer, H., Charney, E., *Chem. Ind.* (*London*) **1962**, p. 1286.

(748) Wendt, A. S., *J. Assoc. Offic. Agr. Chemists* **46**, 337 (1963).

(749) White, E. H., Winter, R. E. K., *Tetrahedron Letters* **1963**, p. 137.

(750) Wickberg, B., *J. Org. Chem.* **27**, 4652 (1962).

(751) Willhalm, B., Thomas, A. F., Gautschi, F., *Tetrahedron* **20**, 1185 (1964).

(752) Winter, M., Malet, G., Pfeiffer, M., Demole, E., *Helv. Chim. Acta* **45**, 1250 (1962).

(753) Witek, S., Bukala, M., *Chem. Stosowana* **6**, 295 (1962).

(754) *Ibid.*, p. 511.

(755) Wolff, R. E., Ma, J. C. N., Lukas, G., *Compt. Rend.* **257**, 1784 (1963).

(756) Wolford, R. W., Alberding, G. E., Attaway, J. A., *J. Agr. Food Chem.* **10**, 297 (1962).

(757) Wolford, R. W., Attaway, J. A., *Gas Chromatog. Intern. Symp. 1961*, **3**, 457 (pub. 1962).

(758) Wolford, R. W., Attaway, J. A., Alberding, G. E., Atkins, C. D., *J. Food Sci.* **28**, 320 (1963).

(759) Wrolstad, R. E., Jennings, W. G., *Food Technol.* **16** (8), 107 (1962).

(760) Wu, J., *Hua Hsueh Hsueh Pao* **29** (1), 54 (1963).

(761) Yakunina, T. G., *Lekarstv. Syr'evye Resursy Irkutskoi Obl., Irkutsk* 1961 *1961* (3), p. 84.

(762) Yamaguchi, M., Yamaguchi, S., Sakai, K., *Kogyo Kagaku Zasshi* **65**, 1960 (1962).

(763) Yamanishi, T., Kiribuchi, T., Sakai, M., Fujita, N., Ikeda, Y., Sasa, K., *Agr. Biol. Chem.* (*Tokyo*) **27**, 193 (1963).

(764) Yeh, P., *Perfumery Essent. Oil Record* **53**, 371 (1962).

(765) *Ibid.*, p. 454.

(766) Yeh, P., *J. Chinese Chem. Soc.* (*Taiwan*) **11** (1), 41 (1964).

(767) Yeh, P. H., *Chemistry* (*Taipei*) **1963** (1), p. 15.

(768) Yoshida, T., Komatsu, A., Indo, M., *Agr. Biol. Chem.* (*Tokyo*) **27**, 433 (1963).

(769) Yoshida, T., Nakamura, N., *Bunseki Kagaku* **11**, 669 (1962).

(770) Yoshimura, K., *Ibid.*, pp. 678, 682.

(771) Yudovich, E. A., *Tr. Tashkentsk. Farmatsevt. Inst.* **3**, 145 (1962).

(772) Zalkow, L. H., Park, M. K., Ellis, J. W., *Perfumery Essent. Oil Record* **55**, 507 (1963).

(773) Zalkow, V. B., Shaligram, A. M., Zalkow, L. H., *Ibid.*, **1964**, p. 194.

(774) Zalkow, L. H., Zalkow, V. B., Brannon, D. R., *Chem. Ind.* (*London*) **1963**, p. 38.

(775) Ziffer, H., Batterham, T. J., Weiss, U., Rudloff, E. von, *Tetrahedron* **20**, 67 (1964).

(776) Ziffer, H., Charney, E., Weiss, U., *J. Am. Chem. Soc.* **84**, 2961 (1962).

(777) Zozulya, A. P., Novikova, E. V., *Zavodsk. Lab.* **29**, 543 (1963).

(778) Zweifel, G., Ayyangar, N. R., Brown, H. C., *J. Am. Chem. Soc.* **84**, 4342 (1962).

Fertilizers

Charles W. Gehrke and James P. Ussary,

Department of Agricultural Chemistry, University of Missouri, Columbia, Mo.

THIS review covers the literature reported from September 1, 1962, to December 1, 1964, and includes procedures recorded in readily available journals, in *Chemical Abstracts*, and in *Analytical Abstracts*. Some selectivity has been exercised to include only those procedures especially pertinent to, or which, in the author's judgment, could be adapted easily to, fertilizer analytical problems.

A number of papers presented at the 148th annual meeting of the American Chemical Society at Chicago (1964) related to fertilizer technology and analysis covering subjects of sampling; nitrates; water; determination of N, P, and K; reactivity rates; dissolution rates; and various phases of manufacturing technology.

Considerable information of importance to laboratories engaged in fertilizer analyses was obtained in a broad study by 12 state control and 11 industry laboratories. This study was supported by the Association of American Fertilizer Control Officials (AAFCO), the National Plant Food Institute (NPFI), and industry members of the Chemical Control Committee of the NPFI. A summary report on this study was made by Quackenbush at the AAFCO meetings in August 1964 at Poland Springs, Maine; and to the Magruder Committee, meeting in Washington, D. C., October 1964. A manuscript is in preparation for publication in the *Journal of the Association of Official Agricultural Chemists*. The objective of this study was to determine the normal variations of analytical results within and among laboratories. The samples were sent to the respective laboratories with no efforts made to produce a uniform mix, and with particular emphasis that each laboratory did not know that this was a special study. The standard deviation within laboratories was about 0.3 and much larger than the usual Magruder within-laboratory standard deviations. The real among-laboratory variation was about one half of the within-laboratory variation. It was also concluded that the reduction and mixing of nonuniform samples by the 23 laboratories were performed in a satisfactory way; however, there is a real need for standardization of reporting procedures.

OFFICIAL METHODS

The Association of Official Agricultural Chemists (AOAC) gave "official" status to the gravimetric quinolinium phosphomolybdate method based on the "quimociac" reagent for total, water-soluble, citrate-insoluble, and available phosphorus. Also, a quinolinium phosphomolybdate method, without acetone, previously applicable only to total and citrate-insoluble phosphorus was given official status for available phosphorus. A method involving EDTA titration of acid- and water-soluble magnesium in fertilizers was adopted as official. The AOAC removed from official status a volumetric quinolinium phosphomolybdate method for phosphorus and an air-flow method for free water (*3*, *4*).

WATER

Studies were conducted on the moisture content of fertilizers by the vacuum oven method at 60° C., the official 100° C. oven method, and the Cenco balance method (*53*). No significant differences were noted for raw materials, with the exception of superphosphates, urea, and other similar products with low decomposition temperatures. Results on mixed fertilizers were 35% lower by the vacuum oven method and varied widely depending on the total amount of moisture present, amount and type of solution used as a source of nitrogen, and type of fertilizer. Results by the Cenco balance method fell between the two oven methods.

Ruggedness tests revealed that the only experimental variable to which results in the vacuum oven method were sensitive was the oven temperature (*14*). The allowable temperature range within the oven chamber was less than 5° C. Collaborative results showed that the vacuum oven method was as precise as the AOAC official vacuum desiccator method.

A method was proposed for free water in fertilizers, which involves extraction with *p*-dioxane and titration of the extract with Karl Fischer reagent (*13*). The values obtained compared favorably with the official vacuum desiccator method.

The need for collaborative tests of the two AOAC official methods (100° C. oven and vacuum desiccator) for water in fertilizers has been pointed out (*15*). A collaborative study showed that the analyst must be very empirical in his manipulation of the methods and that the difference between total water and free water is much too great for urea-containing fertilizers (*12*).

NITROGEN

The AOAC continued studies on methods for determining nitrogen in fertilizers. A collaborative study (*18*) of the AOAC reduced iron method (2.039), the Gehrke (*22*) modified reduced iron method, a chromous solution reduction method, and a chromium powder method indicated that all of the methods gave equally reliable results. In a critical evaluation of the reduced iron method for the reduction of nitrates it was concluded that hydrogen-reduced iron was a more efficient reductant than electrolytic reduced iron and that 250-mesh is the largest particle size that should be used (*11*). The AOAC official reduced iron method has certain limitations mainly due to imprecision of results. The modified reduced iron method has corrected many of these. Further research is being conducted on these methods by the AOAC and various laboratories.

In a study of several Raney catalyst powders for the reduction of nitrates the most efficient reductant in acid medium was an Al and Ni alloy; others, in decreasing order of reducing efficiency, were Cu-Al, Ni-Cr-Al, Co-Al, and Fe-Al (*10*). In the reduction, nascent hydrogen, generated by the reaction of Al with the acid, was adsorbed on the alloying metal and there reduced the nitrate. A relatively small amount of the alloy was required, and the resulting salts did not interfere with the subsequent Kjeldahl digestion and distillation. The method was applied successfully to mixed fertilizers containing nitrates and to mixtures of potassium nitrate with organic materials. The method is suitable for solid and liquid fertilizers that contain a considerable quantity of chloride.

A method was proposed for separating the various forms of nitrogen in fertilizers by cation and anion exchange (*46*). The method was accurate, rapid, and direct, and gave results not now attainable by AOAC methods.

A simple and rapid nitrogen method using Devarda's alloy has been proposed (*49*). Reduction of nitrates and distillation of ammonia were completed in 45 seconds. The method is applicable to samples containing nonvolatile amides. It was automated so that up to 50 samples per hour could be handled (*61*). This method could be a valuable tool to the laboratory analyzing a large number of inorganic fertilizers.

In a comparison of the Kjeldahl, Nessler, and formaldehyde titration methods it was found that the Kjeldahl method was more reliable than the other methods and the Nessler method more reliable than formaldehyde titration (*27*). The formaldehyde titration yielded low results when the sample was not finely ground.

Nitrogen was determined by the Kjeldahl method without distillation (*16*). An aliquot of the digestate was neutralized with NaOH, a known excess of standard NaOH added, the solution boiled for one hour to drive off the ammonia, and the excess NaOH back-titrated with standard H_2SO_4. Ten per cent accuracy was achieved.

A method using a modification of the Pregl-Dumas method was found to give accurate results (*1*). The sample was placed in a small quartz test tube, which was then placed in a larger quartz tube packed with precalcined NiO or CuO and burned at 850° to 900° C. Carbon dioxide was used to flush the combustion products into the azotometer.

Sauchelli (*50*) edited a comprehensive book providing a single source of reference to the latest research and technology of synthetic ammonia and its derivatives, especially as applied to the fertilizer industry.

PHOSPHORUS

The need of a separate acid hydrolysis step to convert all phosphates to the ortho form was required in the determination of water-soluble phosphorus by the AOAC official volumetric method but not in the quinolinium phosphomolybdate method. In the direct determination of "available" phosphorus complete precipitation of phosphorus as quinolinium phosphomolybdate was obtained by carefully controlling the phosphorus and ammonium citrate concentrations (*30*). The quinolinium phosphomolybdate method was simplified by the use of a single precipitating solution (*31*) which contains quinoline, sodium molybdate, citric acid, and acetone, and has been designated the quimociac reagent. Acetone eliminated interference from ammonium ions in the precipitation and made the method suitable for the determination of available phosphorus in fertilizers.

A study was made to determine the reliability of the AOAC volumetric ammonium phosphomolybdate method for P_2O_5 (*26*). It was found that the method was reliable if more explicit directions were made for each step. End point detection and solution standardization required careful control.

A study was made on the effect of extraction time on the citrate-soluble P_2O_5 content of fertilizer (*24*). The data show that for mixed fertilizers and straight materials, all water- and citrate-soluble phosphorus was extracted in 60 minutes. Two exceptions were noted: Further solubilization of citrate-insoluble P_2O_5 occurred in samples of tricalcium phosphate and ammoniated triple superphosphate for extraction times of 2 to 3 hours.

An automated spectrophotometric determination of total phosphorus in fertilizer based on the formation of the yellow phosphomolybdovanadate was reported (*20*). The average automated results agreed closely with quimociac results, but precision with the automated method was not so good as with the quimociac method.

Results of field tests of ammoniated superphosphate fertilizers correlated well with availabilities determined by an alkaline ammonium citrate extraction, but showed no relation to availabilities determined by the official AOAC neutral ammonium citrate extraction procedure. It was recommended that the alkaline ammonium citrate extraction method be considered for determination of the availability of phosphate fertilizers (*9*).

Water-soluble phosphorus was determined by neutralizing the sample solution using bromocresol green as indicator, then titrating with $0.1N$ NaOH to a phenolphthalein end point. The P_2O_5 content was calculated from the difference of the volumes used in titrating with the two indicators, bromocresol green and phenolphthalein (*36*).

POTASSIUM

During the past two years the flame photometric determination of potassium has undergone further study and refinement. An automated method which gave accurate and precise results with a significant saving in analytical time was reported. Anion exchange cleanup was found to be unnecessary for samples containing less than 16% K_2O (*25*). The halogen acids and their ammonium salts decreased the emission intensities of alkali metal lines in flame spectrophotometry (*52*).

The effects of HCl, H_2SO_4, Na, Ca, Mg, and Al on the determination of potassium by atomic absorption spectrophotometry were studied (*58*). All except H_2SO_4 suppressed the absorption by potassium. This influence could be detected down to 10 p.p.m. of potassium.

Potassium was determined in the presence of sodium and magnesium by converting the chlorides to hydroxides with Ag_2O, and titrating the potassium conductometrically in ethanol with an ethanol solution of chloroplatinic acid (*43, 44*). The method is claimed to be superior to that using titration with an ethanol solution of perchloric acid.

A precision comparison study was made between the perchlorate method and the titrimetric sodium tetraphenylborate method (*47*). Standard deviations of 0.12 and 0.095, respectively, were obtained on analyses of 15 routine samples.

Potassium was determined by precipitating with a measured excess of sodium cobaltinitrite, filtering, and titrating the excess cobalt with disodium EDTA (*34*).

Dugger (*19*) recommended a substitution of lithium for sodium as added base in the Lindo Gladding method. Complete drying of the chloroplatinate precipitate was suggested as preferable to drying to a mush, provided enough sodium or lithium was present to ensure that upon drying all excess chloroplatinic acid was converted to sodium or lithium chloroplatinate.

Small amounts of potassium have been determined by precipitating the potassium with sodium tetraphenylborate, dissolving the precipitate in acetone, and determining the boron in the precipitate with carminic acid (*8*). The accuracy was ±1% relative.

Potassium was determined by precipitating, in an ethanol solution, with a measured excess of tartaric acid, filtering, and titrating the excess tartaric acid with $0.1N$ sodium hydroxide (*36*). One standard deviation was less than 0.08.

SECONDARY AND MICRONUTRIENTS

Complexometric titration of calcium and magnesium with EDTA continues to attract fertilizer chemists.

In a collaborative study it was shown that calcium and magnesium can be determined by EDTA titration equally as accurately and more precisely than by the official AOAC gravimetric methods (*56*). The EDTA method for calcium and magnesium in limestone was modified to give more accurate results for samples containing 2 to 4% of magnesium (*23*). The effect of phosphate on the EDTA titration of calcium and magnesium was also investigated. Complete recovery of calcium and magnesium was obtained at all levels of added phosphate, but at the higher levels the time required to complete the titration increased. Interference of phosphate was completely avoided by using an anion exchange resin column.

Aluminon was used as an indicator for calcium, magnesium, or calcium and magnesium at pH 8.5 to 9.9. When methylene blue was added to the aluminon, the color change was wine red to green (*35*). Interferences can be masked with NaCN in NaOH, and triethanolamine (*60*).

Atomic absorption has received some attention in the determination of calcium and magnesium. Silicon interference was obviated by removal as SiF_4 (*7*). Lanthanum was used to buffer the interference due to aluminum.

Millet (*40*) and Barker (*5*) in studies of methods for determining sulfur in fertilizer recommended gravimetric precipitation of $BaSO_4$ as one of the best methods. These investigators also reported on the following methods. Sulfate can be titrated directly with $Pb(NO_3)_2$ using dithizone indicator; $BaCl_2$ with sodium rhodizonate indicator; or $Ba(ClO_4)_2$ with thorin or sodium alizarin-sulfonate indicator. The last indicator was preferred, but it was found necessary to remove phosphate. Data were presented on the precipitation of sulfate with excess barium ion and titration of the excess with EDTA and Eriochrome Black T indicator. Reduction of H_2SO_4 to H_2S with HI and H_3PO_4 in a special apparatus followed by titration of the H_2S with $Hg(OAc)_2$ was found to be rapid. These procedures had to be modified for various fertilizers.

An indirect spectrophotometric method for the determination of boron with carminic acid was reported (*32*). The method obeys Beer's law up to 8 p.p.m., and is sensitive and relatively free from chemical interferences. A simple and sensitive complexometric method was reported by Heyes and Metcalfe (*29*) using the boron-curcumin complex in a nonaqueous medium. Fluoride and nitrate interfere but can be overcome. Hiiro (*33*) used alizarin S for the spectrophotometric determination of boron in an aqueous solution instead of in concentrated sulfuric acid. Iron and aluminum interfere but can be tied up with EDTA. Protocatechuic acid (3,4-dihydroxybenzoic acid) forms a 1 to 1 complex with boron at pH 8 and can be read with an ultraviolet spectrophotometer at 302 mμ (*33*).

Atomic absorption spectrophotometry has been applied by Morgan (*41*) in the determination of copper. Low levels were determined by extracting copper from an HCl solution into methyl isobutyl ketone with ammonium pyrrolidine dithiocarbamate by the method of Allan (*2*). When iron and a mixed acid were added, a slightly smaller slope was observed (*57*). The sensitivity of the determination was increased by analyzing the exhaust gases of the flame (*62*) or using high pressure atomization (*28*).

A gravimetric method for copper using resacetophenone phenylhydrazone as the precipitant was reported (*59*). In the absence of cadmium, copper could be accurately determined up to 64 mg., whereas in the presence of cadmium the method was good to 32 mg. Copper and cobalt have been titrated with EDTA using bis(carboxymethylaminomethyl) dichlorofluorescein as a metallofluorochromic indicator (*6*).

The addition method in flame photometry has been applied to the determination of copper (*42*). Special attention was paid to the use of variable apparent blank readings for different concentration ranges on a calibration curve with a maximum of 200 p.p.m. of copper.

Sirois (*55*) reported quantitative results for iron, copper, and zinc by plasma jet spectroscopy under noninterference conditions in multielement environments. Results were precise and accurate, and the method appeared to be widely applicable.

Zinc has been determined by EDTA titration in the presence of nickel and cobalt, and was based on the selective decomposition of the cyano complex of zinc by formaldehyde (*37*). By using a photometric end point detection system and a weight buret, precision of 0.1% absolute or better for the EDTA determination of zinc was obtained (*51*). Mekada, Yamaguchi, and Ueno (*39*) used dimercaptosuccinic acid as a masking agent of cadmium, copper, and mercury in the determination of zinc.

It was reported by Fuwa *et al.* (*21*) that an atomic absorption spectroscopy method for zinc was 10 to 100 times more sensitive than the dithizone method, required only one tenth as much time, and gave more precise results. The presence of HCl or HNO_3 influenced the results for zinc, but when the concentration of acid was changed from 0.3*N* to 1.2*N* little further change in absorbance values was observed (*48*).

Cobalt was determined spectrophotometrically with 4-(2-pyridylazo)resorcinal, but iron and nickel interfered even on addition of EDTA or KCN (*54*).

Molybdate was reduced to Mo(V) by the stannous chloride–perchlorate reaction and determined spectrophotometrically as tricaprylmethylammonium oxytetrathiocyanatomolybdate(V) (*38*).

The Association of Official Agricultural Chemists conducted a collaborative study on colorimetric determinations of aluminum, iron, manganese, phosphorus, and titanium in liming materials (*17*). Aluminum was determined with ammonium aurintricarboxylate (aluminon); iron with 2,4,6-tripyridyl-*s*-triazine (TPTZ); manganese by oxidation to permanganate with KIO_4; phosphorus by a heteropoly blue method; and titanium with disodium-1,2 - dihydroxybenzene - 3,5 - disulfonate (Tiron). Sample solutions were prepared by perchloric acid digestion or sodium hydroxide fusion. The results were precise and accurate, and no significant differences were observed for the two methods of sample solution preparation.

Yields of *Aspergillus niger* mycelia were used to determine magnesium, zinc, manganese, copper, and molybdenum in fertilizers, plants, and soils, their ashed residues, or water extracts. The error was 4 to 8% and the time required was 2 to 5 days (*45*).

PESTICIDES

Techniques that are well suited to handling an analytical problem such as that posed by residues of pesticides are gas-liquid (GLC) and thin-layer chromatography (TLC) supplemented with confirmation by infrared. These chromatographic methods offer the obvious advantages inherent in the refined separations that are possible: very small sample size, speed, accuracy, and simplicity. Gas-liquid chromatography with electron capture and microcoulometric detection systems have been used most widely and successfully for the determination of most pesticides. Thin-layer chromatography has been used for the separation of pesticides (qualitative and semiquantitative) and identification of degradation products.

This paper does not discuss methods for pesticide residues in fertilizers, since they are covered thoroughly in another review.

LITERATURE CITED

(1) Abramyan, A. A., Kocharyan, A. A., Karapetyan, A. G., *Izv. Akad. Nauk Arm. SSR, Khim. Nauki* **15,** 225–30 (1962).
(2) Allan, J. E., *Spectrochim. Acta* **17,** 467 (1961).
(3) Assoc. Offic. Agr. Chemists, *J. Assoc. Offic. Agr. Chemists* **46** (1), 127 (1963).
(4) *Ibid.*, **47** (1), (1964).
(5) Barker, J. E., *Ibid.*, **47** (3), 436 (1964).
(6) Bermejo-Martinez, F., Margalet, A., *Chemist-Analyst* **53** (2), 45 (1964).
(7) Billings, G. K., Adams, J. A. S., *Atomic Absorption Newsletter* (Perkin-Elmer Corp.), No. **23** (August 1964).
(8) Bovalini, E., Casini, A., *Ann. Chim.* (*Rome*) **52,** 482 (1962).
(9) Brabson, J. A., Burch, W. G., Jr., *J. Assoc. Offic. Agr. Chemists* **47** (3), 439 (1964).
(10) *Ibid.*, p. 1035.
(11) Brabson, J. A., Burch, W. G., Jr., Woodis, T. C., Jr., *Ibid.*, **46** (4), 599 (1963).
(12) Caro, J. H., *Ibid.*, **47** (3), 443 (1964).
(13) *Ibid.*, p. 626.
(14) Caro, J. H., Heinly, N. A., *Ibid.*, **47** (6), 1040 (1964).
(15) Caro, J. H., Rexroad, P. R., *Ibid.*, **46** (4), 582 (1963).

(16) Chang, K., *Hua Hsueh Tung Pao* **6,** 3 (1960).
(17) Chichilo, P., *J. Assoc. Offic. Agr. Chemists* **47** (6), 1019 (1964).
(18) Davis, H. A., Durgin, O. B., *Ibid.*, **46** (4), 595 (1963).
(19) Dugger, J. F., Jr., *Ibid.*, **46** (4), 773 (1963).
(20) Ferretti, R. J., Hoffman, W. M., *Ibid.*, **45** (4), 993 (1962).
(21) Fuwa, K., Pulido, P., McKay, R., Vallee, B., ANAL. CHEM. **36,** 2407 (1964).
(22) Gehrke, C. W., Beal, B. M., Johnson, F. J., *J. Assoc. Offic. Agr. Chemists* **44,** 239 (1961).
(23) Gehrke, C. W., Johnson, F. J., *Ibid.*, **46** (4), 611 (1963).
(24) Gehrke, C. W., Kramer, G. H., Jr., *Ibid.*, **47** (3), 457 (1964).
(25) Gehrke, C. W., Ussary, J. P., Kramer, G. H., Jr., *Ibid.*, **47** (3), 459 (1964).
(26) Harwell, A. O., Parks, K. L., Harwood, W. M., Baker, J. E., *Ibid.*, **47** (3), 428 (1964).
(27) Hecht, H., Fritz, A., *Brauwissenschaft* **15** (11), 347 (1962).
(28) Herrmann, R., Lang, W., *Colloq. Spectros. Intern., 9th, Lyons,* **1961, 3,** 291 (Pub. 1962).
(29) Heyes, M. R., Metcalfe, J., U. K. Atomic Energy Authority, Prod. Group, P. G. Rept. **251** (5) (1963).
(30) Hoffman, W. M., Ferretti, R. J., Breen, H. J., *J. Assoc. Offic. Agr. Chemists* **46** (4), 570 (1963).
(31) Hoffman, W. M., Wiles, C. C., *Ibid.*, **46** (4), 579 (1963).
(32) Hiiro, K., *Bunseki Kagaku* **12** (8), 703 (1963).
(33) Hiiro, K., *Nippon Kagaku Zasshi* **83,** 711 (1962).
(34) Kuan, Tyu-Yuan, *Tu Jang Tung Pao* **5,** 46 (1963).
(35) Kundu, P. C., *Z. Anal. Chem.* **196,** 246 (1963).
(36) Lasiewicz, K., *Chem. Anal.* (*Warsaw*) **7,** 937 (1962).
(37) Liteann, C., Marinescu, L., *Studia Univ. Babes-Bolyai, Ser. Chem.* **7** (2), 127 (1962).
(38) McFarland, O. K., Wilson, A. M., ANAL. CHEM. **36,** 2488 (1964).
(39) Mekada, T., Yamaguchi, K., Ueno, K., *Talanta* **11,** 1459 (1964).
(40) Millet, R., *Chim. Anal.* (*Paris*) **45,** 174 (1963).
(41) Morgan, M. E., *Atomic Absorption Newsletter* (Perkin-Elmer Corp.), No. **21** (July 1964).
(42) Mosqueda-Suarey, A., Capacho-Delgado, L., Ramirey-Munoz, J., *Rev. Univ. Ind. Santander* **5,** 396 (1963).
(43) Novak, V., Dolejs, V., *Collection Czech. Chem. Commun.* **27,** 2006 (1962).
(44) Novak, V., Koyak, P., *Sb. Ved. Praci, Vysoka Skola Chem. Technol. Pardubice* **41,** 7 (1–2) (1962).
(45) Nowosielski, R., *Roczniki Nauk Rolniczych,* Ser. **A87,** 201 (1963).
(46) O'Neal, J. M., Clark, K. G., *J. Assoc. Offic. Agr. Chemists* **47** (6), 1054 (1964).
(47) Ordonez, M., *Rev. Inst. Invest. Tecnol.* (*Bogota*) **3** (9), 31 (1961).
(48) Parker, H. E., *Atomic Absorption Newsletter* (Perkin-Elmer Corp.), No. **13** (May 1963).
(49) Potrafke, K. A., Kroll, M., Blom, L., *Anal. Chim. Acta* **31,** 128 (1964).
(50) Sauchelli, V., "Fertilizer Nitrogen. Its Chemistry and Technology," Reinhold, New York, 1964.
(51) Scarfe, B., *Analyst* **88,** 618 (1963).
(52) Shaevich, A. B., Nauchu, T., *Issled. Inst. Redkikh Metal* **11,** 37 (1963).
(53) Shannon, R. D., *J. Assoc. Offic. Agr. Chemists* **46** (4), 592 (1963).
(54) Shijo, Y., Tangio, T., *Bunseki Kagaku* **13** (6), 536 (1964).
(55) Sirois, E. H., ANAL. CHEM. **36,** 2394 (1964).
(56) Smith, J. B., Manning, P. B., *J. Assoc. Offic. Agr. Chemists* **47** (3), 450 (1964).
(57) Sprague, S., Slavin, W., *Atomic Absorbance Newsletter* (Perkin-Elmer Corp.), No. **23** (August 1964).
(58) Takeuchi, T., Suzuki, M., Yasuda, K., *Kogyo Kagaku Zasshi* **65,** 1025 (1962).
(59) Umapathy, P., Raju, N. A., *Indian J. Chem.* **1,** 272 (1963).
(60) Venturini, A., *Ann. Univ. Ferrara, Sey.* **52** (3), 25 (1961).
(61) Willemsen, L. M., Wijenberg, J. G., Stornebrink, P. J., *Anal. Chim. Acta* **31,** 139 (1964).
(62) Zelyukova, Y. J., Poluektov, N. S., *Zh. Analit. Khim.* **18** (4), 435 (1963).

CONTRIBUTION from the Missouri Agricultural Experiment Station, Journal Series No. 2873. Approved by the Director.

Food

Katherine G. Sloman and Emanuel Borker

General Foods Technical Center, White Plains, N. Y.

THIS REVIEW covers advances in food analysis since the 1963 review (*6P*) as reported in publications available to the authors through early October 1964. Once again, pesticide residue methods are omitted since they are covered in another review. In reviewing the literature, it is readily apparent that parallel studies are being made in laboratories in many nations. In these cases, the authors have generally elected to cite the work reported in American journals while recognizing that there is excellent work reported in other journals

Several new compendia of standard methods have appeared in this period. "Food Chemical Codex" (*12P*) is similar in organization to the U. S. Pharmacopoeia. However, there are some differences in the methods used even for chemicals common to both. The "Official, Standardized, and Recommended Methods of Analysis" of the Society for Analytical Chemistry (*27P*) is, in many ways, a British counterpart of the A.O.A.C. book of methods whose 10th edition will appear this fall. The seventh edition of "Cereal Laboratory Methods" (*4P*) has appeared. Gunderson's guide to food standards in the United States (*13P*) will be of interest to food chemists, as will the new Journal of the Association of Public Analysts. This journal is available through the County Agent's Department, Maidstone, Kent, England.

ADDITIVES

Great interest continues in the determination of protective additives, of preservatives, of noncaloric additives, and in improved methods for determining one food added to another. General procedures for food additives are included in Vol. I of a four-volume series edited by Zweig (*65A*). The Association of Public Analysts has issued a report (*2A*) on the methods required for the determination of antioxidants in foods. Hamence (*18A*) has discussed the role of analytical chemistry in antioxidant control in the United Kingdom. A general method of evaluating antioxidants using the polarograph has been reported (*19A*). A general method for antioxidants reacting with iron has been described (*4A*). Davidek has discussed the separation of gallic acid esters by both thin layer chromatography (*9A*) and centrifugal chromatography (*10A*). Gas chromatographic methods for BHA and BHT in cereals have been discussed (*1A*, *52A*, *58A*). Two colorimetric methods for BHT have been proposed; the reaction with *o*-dianisidine (*57A*) and the reaction with diazotized *p*-nitroaniline (*44A*).

Microbiological methods for the determination of preservatives have been proposed (*3A*, *39A*). Ultraviolet spectrophotometry after paper chromatography has been described for *p*-hydroxybenzoates and sorbic acid determination (*22A*). Spectrophotometric determination of preservatives in foods after distillation has been reported (*38A*). A general chromatographic method for ether-soluble preservatives in wine has been reported by Romani, *et al.* (*48A*). Brominated preservatives have been determined (*25A*) by determining the bromine content of wines and calculating the bromine to chlorine ratio. Mergenthaler (*42A*) determines bromoacetic acid in wines and beverages by chromotropic acid after separation on an ion exchange column. Chloracetic acid has been detected in wine and cider by the use of paper chromatography with ammonia and ninhydrin as the chromogenic agents (*13A*). Extraction and distillation followed by chlorine determination is used to determine p-chlorobenzoic acid in wines (*12A*). The same preservative was determined by its reaction with sulfuric acid and potassium nitrate followed by paper chromatography (*6A*). Chloropicrin has been determined colorimetrically after extraction with heptane (*46A*). Meikle (*41A*) has achieved the separation of 2-halogenated carboxylic acids by paper chromatography and the use of 4-dimethylaminoazobenzene as an indicator. Esters of p-hydroxybenzoic acids have been separated and determined by paper chromatography (*11A*) and by colorimetry (*35A*). A simple titrimetric method for sorbic acid using the reaction with potassium iodide has been described (*47A*). Two methods of determining sorbic acid with thiobarbituric acid have been described for sorbic acid in wine (*20A*) and for sorbic acid and sorbate in foodstuffs (*26A*). The reaction of sorbic acid with resorcinol is used to determine sorbic acid in jam and other foodstuffs (*36A*), and the reaction of sorbic acid with p-hydroxybenzaldehyde and other compounds is described by Salo (*49A*).

The Monier-Williams procedure for sulfur dioxide has been further modified and the results of collaborative analyses reported (*60A*). Sulfur dioxide in gelatin has been determined polarographically (*55A*). A special variation of the Monier-Williams principle has been used to determine sulfur dioxide in cider (*7A*). The sulfur dioxide is collected in iodine–potassium iodide solution. Borginon (*5A*) has applied the malachite green test to the direct determination of sulfite in gelatin. A special desorption and trapping method, followed by colorimetric or iodimetric determination of the sulfur dioxide is described by Lloyd *et al.* (*37A*).

The lactic acid content of lactic acid modified fatty glycerides was determined (*56A*) using saponification and extraction. The 1-monoglycerides have been identified in ice cream after periodate reaction by the chromotropic acid formaldehyde test (*51A*). Kröller has published a series of papers on the detection of various emulsifiers in foods. These include monostearylesters of glyceride free "ring acids" (*30A*), glycerides of tartaric and acetyltartaric acids by paper chromatography (*31A*), sugar fatty acid esters (*32A*), and citric acid glycerides and tartaric acid polyglycerides (*33A*).

Synthetic sweeteners, sodium cyclamate, dulcin, and sodium saccharin have been separated by paper chromatography (*23A*) and by thin layer chromatography (*50A*).

Several different methods for the determination of the sugar alcohols have been reported. The conditions required for their reaction with anthrone have been studied by Graham (*15A*). Chromatography on paper impregnated with ammonium molybdate has been described (*16A*). Polarimetric determination of sorbitol and mannitol as well as malic and tartaric acids in the presence and absence of molybdate ion has been reported (*29A*). Total polyhydric alcohols in soya sauce have been estimated after ion exchange clean-up (*45A*). Smith and Carlsson (*53A*) report gas chromatographic analysis of glycerol, mannitol and other polyhydric compounds after conversion to the trimethylsilyl ethers. Optimum conditions for the micro determination of glycerol were determined by Ghimicescu, *et al.* (*14A*). An enzymatic determination of glycerol in grape juice and wine has been described (*40A*). Sorbitol has been determined in chocolate (*61A*) and in foodstuffs (*24A*) by polarimetry before and after molybdate treatment. Hause *et al.* (*21A*) have converted sorbitol to its hexaacetate for determination by gas chromatography.

Ethyl pyrocarbonate is determined (*34A*) by its reaction with p-bromoaniline, and by gas chromatography (*27A*); Kielhofer and Wurdig (*28A*) have studied the decomposition of diethylpyrocarbonate in wine. Stearyl tartrate in bread has been determined by thin layer chromatography of the unsaponifiable matter (*63A*). Wurziger *et al.* (*64A*) have determined polyglycerol fatty acid esters in edible fats by extraction and paper chromatography. A method for the determination of Polysorbate 80 in baking products has been reported (*17A*).

A chemical method for the determination of truffle in pork-meat has been suggested (*62A*). Milk protein in meat is determined by the isolation of the milk phosphoprotein and determination of the phosphorus (*59A*). Nonfat dry milk solids content of cereal bars has been determined by paper chromatography for the lactose (*8A*). Egg products in various foods have been determined by their cholesterol contents (*43A*), and egg in pasta is determined by methyl ester analysis of the fat (*54A*).

ADULTERATION, CONTAMINATION, AND DECOMPOSITION

The means and the indices for the determination of adulteration, contamination, and decomposition have expanded markedly in the past two years, therefore, a separate section has been added to this review to include the information on identity and purity of foods subject to adulteration. A great spurt in information on the chemical detection of mycotoxins has occurred in the past two years. Aflatoxin has been the mycotoxin which has received most of the analysts' attention. Coomes *et al.* (*13B*) have reported an extraction and paper chromatographic method for the analysis of peanuts for aflatoxin. A more sensitive thin layer chromatographic method has been reported by Broadbent, *et al.* (*9B*). Modifications of these basic methods have been prepared by Genest, *et al.* (*26B*), and a more rapid procedure by Nesheim, *et al.* (*48B*, *49B*). An ultraviolet method for assaying meals containing 1 p.p.m. or more of aflatoxin has been proposed (*47B*). Confirmatory tests for aflatoxin B_1 by forming fluorescent reaction products of the aflatoxin have also been suggested (*3B*).

The presence of residual antibiotics in foods has been shown by microbiological techniques, for instance, a plate diffusion method for nisin (*75B*), and growth-time curves in whey are used to detect antibiotics in milk (*33B*). Penicillin in milk is determined by inhibition zones on *Bacillus subtilis* plates (*44B*). Sulfonamide residues are separated on Celite columns and determined colorimetrically (*71B*). Radio-isotope assay has been used (*35B*) to determine neomycins in beer after conversion to their N-[14C] acetates. Furaltadone and nitrofurazone have been determined in milk by reading at 430 mμ after careful extraction and clarification (*14B*).

The detection of foreign coloring matter has been accomplished for carotinoids in orange juice by thin layer chromatography (*7B*), and also by the use of ultraviolet curves (*30B*). The addition of caramel color to fruit juices, etc., has been detected by the determination of 5-hydroxy-methyl-2-furaldehyde (*8B*).

Investigations continue on the problems of determining one fat in another. Gallardo *et al.* (*24B*) have used the presence of arachidonic acid determined by ultraviolet spectrometry as a means of detecting 5% of animal fat in vegetable oil. A method of separating sterols and sterol acetate by thin layer chromatography has been proposed (*55B*) as a means of analyzing mixtures of animal and vegetable oils. Sterol identification by gas chromatography is reviewed and has been used by Recourt *et al.* (*61B*) to detect 2 to 5% animal fat in vegetable fat. The Bömer value may be used to detect beef tallow in lard (*74B*). However, transesterification may cause erroneous Bömer values to be obtained (*73B*). Paper chromatography of fatty acids has been used to determine butyric acid as an approximate measure of butter content in mixtures (*62B*). Adulteration of butter with an interestified fat may be detected by gas chromatography, spectrophotometry and dielectric constant measurements, according to Luck *et al.* (*41B*). Adulteration of butter with 10% of vegetable oils can be determined by analysis for tocopherol (*42B*). Hardened fats above 5% in cacao butter have been determined by comparing the extinction of the sample with that of a standard cacao butter (*57B*). Foreign fats in cacao butter may be determined by a special dilatation method (*34B*). Spectrophotometric examination in the region 250 to 300 mμ has been used to indicate the presence of extracted cacao fat in chocolate (*28B*), if vanillin is absent (*29B*). Differential infrared spectroscopy is used to detect adulteration in cacao butter extracted from chocolate (*12B*). A method for the determination of dimeric acids in oils has been proposed (*65B*), and is believed to be more sensitive than the official method for polymerized constituents in heat-treated oils (*66B*). A study of thermally oxidized fats has been made by Wurziger *et al.* (*87B*). A method for the detection of a small amount of acetone in oils uses the hydroxylamine hydrochloride test (*46B*). Castor oil is detected in vegetable oils (*38B*) by turbidity in the presence of ammonium molybdate. Tritolyl phosphate is identified in edible oils by its color reaction with diazotized sulfanilic acid or Gibb's Reagent (*25B*). The analysis of edible oils contaminated with synthetic ester lubricants is described by Crump (*16B*), and a scheme for analyzing mixtures of glycerides with synthetic oils is presented. Adulteration of butter fat with vegetable fat could be detected by gas chromatographic triglyceride analysis (*37B*). Rancidity in fats as detected by peroxide determination has been determined by the use of triphenylphosphine (*72B*), by heating with potassium hydroxide in isopropyl alcohol (*60B*), and by the use of resorcinol for colorimetric evaluation (*77B*). The thiobarbituric acid test for oxidative rancidity has been applied to fish oils (*36B*), and both thiobarbituric acid and benzidine procedures have been applied to vegetable oils (*68B*). Micro detection of peroxides in fats using spectrophotometric procedures (*78B*) or spotting on paper (*79B*) has been proposed by Vioque *et al.* An investigation of the determination of malonaldehyde as a measure of lipid oxidation using ultraviolet spectrophotometry has been proposed (*39B, 40B*).

For the determination of filth in foods, Cox (*15B*) has suggested the use of an improved test paper for the detection of mammalian urine residues, and an enzymatic ultraviolet method has been proposed for the determination of uric acid in flour (*23B*). An x-ray method has been described for the detection of insects in dried peas (*32B*). Filth and decomposition in peanuts and peanut products has been investigated by Ponder (*58B*). Stability of caramel to storage has been followed by ultraviolet measurement (*51B*). The determination of 5-hydroxymethyl-2-furaldehyde in jam and wine has been used to investigate sugar containing foods which have been overheated (*18B*). A review of chemical methods for assessing spoilage of fish and meat has been published by Pearson (*54B*). Oxidative rancidity and browning have been followed by gas chromatography of the food vapors (*11B*). The color changes in black fish-roe products caused by changes in the added dye can be used as a criterion or freshness (*86B*). The damaged starch in flour has been determined by the reaction with β-amylase (*5B*), and by a modification of the ferricyanide method for maltose using Rhozyme 33 (*20B*). A positive correlation between glutamic acid decarboxylase activity and the percentage of germination has been obtained, and may be used as a measure of damage in dried corn (*6B*).

Surface active substances have been determined in sugar by a polarographic procedure (*69B*), in beverages by the reaction with Orange II and sodium carbonate (*63B*), and in olive oils by reaction with methylene blue (*43B*). Trace concentrations of surface active agents have been determined by their suppressive effect on the polarographic wave for oxygen (*19B*). Nonionic polyoxyethylene surfactants in low concentrations have been determined colorimetrically by reaction with barium chloride and tungstophosphoric acid (*56B*). Alkylbenzenesulfonates in foods have been determined by ultraviolet spectrophotometry (*83B*). A scheme for quantitative microdetermination and identification of surface-active compounds has been presented by Burger (*10B*). A rapid test for anionic detergents in drinking water proposed by Abbott (*1B*) uses extraction into methyl green in chloroform. The reaction of cobalt with (ethylenedinitrilo)tetraacetic acid (EDTA) is used as a means of determining this compound in foodstuffs (*80B*). Quaternary ammonium compounds in dairy products have been determined spectrophotometrically after extraction (*81B*).

Studies of the determination of formaldehyde in maple sirup include a discussion of the method (*85B*) and a collaborative study (*76B*). Mineral oil detection in dried fruit is carried out by thin layer chromatography (*59B*) and in raisins by column chromatography through silica gel (*67B*). Methods for the detection of the adulteration of dried egg-yolk are discussed by Acker *et al.* (*2B*), including interpretation of results. The ASTM method for the extractability of flexible materials by foods or simulated food solvents has been described by Rosser (*64B*). The ASTM cell has been modified so that it can be irradiated to measure extractables in irradiated materials (*45B*). A special all-glass apparatus for the determination of small amounts of alcohol in sour milk has been described (*4B*). Parks *et al.* (*53B*) have identified *o*-aminoacetophenone as a flavor compound in stale dry milk by gas chromatography. Paper-strip indicators have been successfully used to detect glucose in adulterated milk (*21B*). Other methods of determining milk adulteration include electrical conductivity (*52B*), and calculation from the measurement of density, fat, and solids (*27B*). Adulteration of pepper with palm-kernel meal has been detected by polarization-microscopy (*17B*). Differences in rate of gelatinization may be used to detect rice flour in wheat flour (*70B*). Malt treated with glucose syrup is detected by the determination of the reducing power of a water extract of the sample (*82B*). Methods of examining brandy and liquors for added wine have been described by Neumann (*50B*). "Sand" in mill products is determined by carbon tetrachloride isolation and screening (*22B*). Methods of determining urea, thiourea, and some of their substitution products by reaction with *p*-dimethylaminobenzaldehyde, have been described (*31B*). "Medicinal" or "phenol-like" taste in beer appears to be due to phenols and chlorophenols and a method for the determination of these compounds has been described (*84B*).

CARBOHYDRATES

In the field of carbohydrate analysis stress continues to be placed on rapid methods, improved separation techniques and the use of instrumental procedures. A test paper for small amounts of glucose using a glucose oxidase preparation has been patented by Scott (*51C*). The use of Tris buffer to prevent side effects from hydrolysis of sucrose by sucrase in commercial glucose oxidase has been recommended for small amounts of glucose in sucrose (*49C*), for glucose in starch conversion products (*36C*), and for the determination of glucose in the presence of maltose and isomaltose (*17C*). Rapid determination of glucose using automatic measurements of slopes of rates of reaction has been described by Pardue (*41C*). Specific enzymatic microdetermination of glucose, fructose, glycerol, and malic acid in wine has been described (*20C*). A modified Fehling's solution has been used for the determination of invert sugar in the presence of large amounts of sucrose (*31C*), and a reduction in the amount of Fehling's reagent required has been used to improve the speed of the determination (*4C*). An electrometric method for reducing sugars has been reported (*73C*). The fluorometric determination of carbohydrates with *o*-phenylenediamine has been discussed (*61C*). A new color reaction of monoaldoses utilizing their reaction with ethyl malonate at 90° C. has been suggested (*26C*). Thiobarbituric acid has been used (*42C*) to determine fructose and fructofuranosides. The reducing and total sugars in sake, beer, and wine have been determined with 3,6-dinitrophthallic acid (*38C*) as well as lactose and sucrose in milk and condensed milk (*37C*). A dead stop method for lactose in milk has been described (*55C*).

Gas chromatography may become the new tool for the separation of sugars, Nishio (*40C*) has separated pentoses and hexoses after the formation of acetyl derivatives. Bentley *et al.* (*8C*) have determined sugars by gas chromatography after formation of the trimethylsilyl derivatives, and expanded the procedure to include the determination of tetrasaccharides and complex glycosides.

An automatic system for monitoring column eluants for sugars in microgram amounts using anthrone has been described (*57C*). Mannose and picose have been separated by chromatography on heated carbon-celite columns and determined by paper chromatography or electrophoresis during molasses formation (*10C*). Similarly a quantitative separation of reducing sugars and maltotriose and maltotetrose from starch conversion products was obtained (*35C*). Partition chromatography is used (*25C*) to separate monosaccharides of the pentose and hexose series. Thin layer chromatography (TLC) has shown its expected usefulness in sugar analysis. Schweiger (*50C*) has suggested TLC on cellulose for some monosaccharides. Paper chromatography using a small amount of benzeneboronic acid in the solvents has been shown (*12C*) to give good separation of acyclic polyhydric compounds from corresponding aldoses or ketoses.

Polarization methods still continue to be useful. Rasper (*43C*) has used a double polarization method for the determination of sucrose, starch syrup, and invert sugar in the presence of each other, and direct polarization is used (*60C*) to determine sucrose in the presence of invert sugar. Polarization in the presence of borate has been used (*28C*) for the determination of sucrose in impure cane sugar. A new and more accurate international table of refractive indices of sugar solutions has been suggested by Hill and Orchard (*29C*). The reaction of sucrose with sulfuric acid has been used (*6C*) for the determination of sucrose in condenser waters. Hadorn (*24C*) summarizes the results of a study of these different methods for the determination of sugar in chocolate by 18 laboratories. The complexometric method gave the most reproducible results.

Paper chromatography for raffinose in beet juice has been accomplished by Kato *et al.* (*33C*), in cane sugar by Gross *et al.* (*23C*), and for raffinose and kestose in sugar-beet roots by Bichsel *et al.* (*9C*). Extraction in 2*N* HCl has been used (*71C*) to determine pentosans in wheat. Hydrolysis and chromatography were used to determine polysaccharides in sucrose (*62C*). Polarographic determination of furfuraldehyde in the distillate for pentosans in cereals speeds up this procedure according to Davidek *et al.* (*15C*). The difference between acid and enzyme hydrolysis is used to determine dextrin in beer (*48C*). An iodimetric procedure has been used (*63C*) to determine starch in sucrose. Zelenka *et al.* (*74C*) have carried out an investigation of the effect of clarifying agents on the polarimetric method for the determination of starch. Carrez reagent appears to be the reagent of choice for this procedure.

A microdetermination of the formic acid generated by the periodate oxidation of starch fractions has been used to help in the determination of degree of polymerization and of the structure of amylose and amylopectin (*30C*). Amperometric titration has been used (*16C*) to study the sorption of iodine by starch. Paper chromatographic studies on starch fractions have been carried out by several authors (*46C, 47C, 58C*). Column chromatography using aluminum oxide was used to follow changes in starch fractions during baking (*64C*). Kolbach and Rinke (*34C*) have proposed a direct method for the determination of starch in husks, and Reeve (*44C*) proposes a separation and iodine staining technique for the determination of extra-cellular starch in dehydrated potatoes. A means of measuring starch gelatinization by its susceptibility to β-amylolysis has been suggested by Sullivan *et al.* (*56C*). Starches have been differentiated by proper staining with dyes (*52C*). Infrared spectrometric studies of starch derivatives such as ethers and esters have been carried out by van der Bij and Vogel (*65C*). Wolfram (*72C*) has isolated cellulose from green coffee beans. A study of the nonfermentable reducing sugars in the analysis of preserves and fruit cordials by paper chromatography indicates that from high concentrations of sugars the yield of nonfermentables may be as high as 0.2% (*59C*). Verhaart *et al.* (*67C*) have analyzed the alcoholic precipitates in sugar-factory juices for polysaccharides and protein by a combination of methods. Pectins in sugar-beet liquor are determined nephelometrically (*1C*). Colorimetric methods for the determination of the uronic acid contents of polysaccharides have been reported to be useful only for rapid screening (*2C*). In another report (*3C*) the same authors present a method using distillation from hydriodic acid for the simultaneous detection of uronic acid and alkoxyl groups in polysaccharides. Methods for uronic acids based on Tollens reaction are described by Wagner (*68C, 69C*). A qualitative scheme for the detection of pectins and alginic acid in foodstuffs is reported by Bock (*11C*). The optimum conditions for the reaction of food gums with anthrone have been determined (*21C*). The carbazole reaction after acid hydrolysis has been used to determine pectins in sugar beet (*18C*). Thickening materials in meat products have been determined by thin layer chromatography after hydrolysis and the reactions of thickening agents as well as of substrates used in sausages are reported (*22C*). Anionic groups in water soluble polymers are determined (*54C*) by their reaction with cetyl pyridinium chloride. Cuzzoni and Lissi (*13C*) describe paper electrophoretic conditions for the separation of polysaccharide gums. Acidic polysaccharides from gum acacia and other gums have been separated on DEAE cellulose (*32C*). Paper chromatography and electrophoresis in combination have been used to identify many types of gums and thickeners (*53C*). Microscopic detection of gums is described by Czaja (*14C*). Infrared spectra of pectic substances have been reported (*45C*), and an improved method of making films for infrared analysis is described (*39C*).

A procedure for the determination of saponin in sugar-beet products has been described (*66C*). Analysis of the fatty-acid esters of sucrose for mono-and di-esters, free sucrose, etc., has been described by Bares (*5C*). The different isomeric forms of sugars in solution were investigated by gas chromatography after glycoside formation and methylation (*7C*). Thin layer chromatography has been used in the study of sucrose esters (*19C*), in the study of *O*-methyl sugars and sugar acids (*27C*), and for the separation of sugars and sugar alcohols (*70C*).

COLOR

Color of specific foods has been determined in several ways. Values obtained using a color-difference meter have been used to measure discoloration in fresh beef (*33D*). Color has been determined in capsicum by transmission measurements after ethanol extraction using potassium dichromate–cobaltous chloride standards (*31D*). A simple photometer fitted with four interference filters is suggested by Naudet *et al.* (*23D*) for the color measurement of fats and oils. An objective method for the color of potato chips requires careful grinding before reading in a filter photometer (*15D*). An alcohol extraction and color reading at 440 mμ is used (*24D*) to determine the color of dried fruits as a means of quality determination. An improved clarification of opalescent worts before color reading has been suggested (*42D*).

The study of natural pigments continues. Thin layer chromatography is used (*38D*) to characterize anthocyanin coloring matters. The analysis of the red coloring matters in wines uses paper chromatography after extraction (*9D*). The anthocyanin pigments of Cabernet Sauvignon grapes have been identified (*34D*). McFarlane *et al.* (*20D*) have critically examined methods for the anthocyanogens of beer and an improved method is suggested (*19D*). Beet pigments have been separated by paper electrophoresis (*26D*). The development of chromophores during nonenzymic browning has been followed (*5D*). Gel filtration has been used to separate caramel color from salts and sugar (*36D*). A sharp separation of carotenes from free xanthophylls has been obtained (*4D*) using column chromatography. Thin layer chromatography and spectrophotometry have been used to detect carotenoids other than β-carotene in pasta (*28D*). A column chromatographic method is suggested by Rother (*29D*) as a means of separating carotenes from carotenoids in orange juice. Curl (*8D*) has determined the carotenoids of Italian prunes. Experimental conditions and mathematical procedures have been developed for the determination of chlorophylls, chlorophyllides, pheophytins, and pheophorbides in plant material (*43D*). Ion exchange resin has been used to convert chlorophylls to pheophytins (*44D*). Changes in the coloring matter of wine are followed spectrophotometrically (*6D*). Hesperidin has been determined in the dry matter of citrus tissues by ultraviolet spectrophotometry (*13D*). Lycopene in tomatoes has been determined (*14D*) by actone extraction. Two carbonyl carotenoids have been identified in tangerine peel (*7D*). Tannins and dyestuffs in dry wines have been determined by permanganate or potassium ferricyanide oxidation (*17D*).

The determination of artificial color remains a problem for the color analysts. Column chromatography (*46D*) and paper chromatography (*45D*) are suggested by Yanuka *et al.* for extraction and identification of food dyes. Added colors in foodstuff are detected by wool-dyeing and by paper chromatography (*21D*). Paper chromatographic procedures have been suggested (*2D*) for Italian permitted artificial colors. The results of paper chromatography of 600 commercial dyes are tabulated (*12D*). The R_f values of 13 food dyes have been reported (*30D*). Stanley, *et al.* (*35D*) have published a systematic identification of artificial food colors including separation techniques and column and paper chromatography. A systematic method for the identification of food and other dyes using paper chromatography has been prepared by Pla-Delfina (*25D*). A review of methods of extraction and identification of dyes in foods has been given by Simonetti (*32D*). The First Report of the Trace Materials (Colours) Committee (*39D*) covers the extraction and identification of permitted food colors. Artificial dyes in wines are detected by extraction with isobutyl alcohol after chemical preparation of the sample (*40D*). A method for the chromatographic identification of synthetic colors in macaroni has been described (*22D*). Methods for the oil soluble dyes include thin layer chromatography (*11D*), paper chromatography (*3D*), and paper chromatography and spectrophotometric detection (*37D*). Thin-layer chromatography on silica gel has been reported (*1D*) for 15 dyes. Koch (*16D*) has reported a method for separating subsidiary dyes in D&C Red Nos. 6 and 7. Link *et al.* (*18D*) have presented an ion exchange paper, x-ray emission procedure for determining mercury in inorganic pigments used in foods, etc. A paper chromatographic procedure for Orange RN and Orange II in meat has been described (*10D*). The detection of Sunset yellow FCF and Orange GGN in table jellies by paper chromatography has been reported (*27D*). The characteristics of some prohibited water-insoluble colors have been tabulated by Villanua, *et al.* (*41D*).

ENZYMES

A method for the determination of enzyme molecular weights, by Sephadex filtration, has been described (*1E*). Many methods for specific enzyme determinations in foodstuffs have been reported in the last two years. Saletan *et al.* (*19E*) have described some new procedures for chill-proofing enzymes in beer using the binding by unhydrolyzed casein of sodium sulfobromophthalein. Fluorometric determination of lipase, acylase, alpha- and gamma-chymotrypsin has been reported (*7E*). Thiobarbituric acid determination of free and bound *N*-acetylneuraminic acid was used to compare the action of heat and rennin on casein (*4E*). A method of determining amylase activity has been investigated by Feller *et al.* (*5E*) on germinated rye; and the effect of metal ions on α-amylase and malt diastatic power has been determined (*3E*). The deleterious effect of copper in water used for diastatic power determination has been shown (*6E*). Lactase evaluation by paper chromatography (*16E*), and by manometric measurement of carbon dioxide produced by the enzymatically hydrolyzed lactose of yeast (*15E*) has been reported. Several analytical methods for lactase have been discussed by Pomeranz (*14E*).

Fluorescein esters are used as a substrate for lipase activity determination (*12E*). Glucose oxidase has been determined electrochemically (*8E*), and after fractionation on DEAE cellulose, by measuring the rate of utilization of hydrogen peroxide in the formation of tetraguaiacol from guaiacol (*22E*). Thin layer chromatography on DEAE cellulose and Sephadex has been used (*21E*) to separate lactic acid dehydrogenase isozymes using gradient development of the chromatoplates. A method for the determination of phosphatase in milk has been reported (*20E*) with the note that storage conditions of the milk must be known before the results of the test can be properly interpreted.

Seasonal changes in pectinesterase activity of Valencia oranges have been determined (*18E*). Purr *et al.* (*17E*) have determined the enzyme spectra of cocoa beans, particularly the enzyme polyphenoloxidase. Tests for polyphenoloxidase using colorimetric procedures (*10E*) and polarographic methods (*11E*) have been critically examined by Heimann and Andler. Papain in meat tissue has been measured (*13E*) using a test based on the biuret reaction

Alkaline phosphatase and peroxidase in butter were determined (*2E*) as a means of determining correct pasteurization of the initial cream. Hadorn and Zurcher (*9E*) has compared three methods of determining the saccharase activity of honey and recommends that of Duisberg and Gebelein.

FATS, OILS, AND FATTY ACIDS

The study of methods for the determination of fats in foods continued to be of interest in the last two years. Wren *et al.* (*85F*) have proposed hydrolysis in boiling hydrochloric acid, chloroform extraction, and silicic acid chromatography as a highly selective and precise means of determining "hydrolysate lipids." It is suggested that this procedure be used as a reference method for fats in foods. A refractometric method for the rapid determination of fats in cornstarch industry products has been reported (*71F*). The Gerber test has been applied to the determination of fat in meat (*13F*). The use of labeled carbon or sulfur is proposed by Williamson (*83F*) for the determination of fat and water in emulsions such as milk.

Several new and improved methods for the determination of the various fat constants have been proposed. Baker (*6F*) has suggested a rapid colorimetric procedure for the determination of free fatty acids with cupric acetate. Fatty acids have been separated from other lipids by silicic acid chromatography

(*42F*). A new procedure for the determination of hydroxyl groups has been reported (*67F*) using stearic anhydride. The acetic anhydride–pyridine procedure for hydroxyl value has been modified to provide a colorimetric microdetermination of this fat constant using hydroxylamine (*78F*), and by the introduction of a special acetylating reagent (*31F*). Chernobai *et al.* (*9F*) have made a study of various methods of determining iodine values in vegetable oils and conclude that a reagent consisting of bromine in chloroform gives more rapid and stable results. Iodine chloride in hydrochloric acid (*5F*) has been used as the halogenating agent for the determination of iodine value of fatty acids and alchohols. Pokorny (*59F*) has investigated the analysis of fatty acid peroxides by paper chromatography, and the use of titanium tetrachloride for the colorimetric determination of fat peroxides (*57F, 58F*). Jacobsen *et al.* (*26F*) have modified the thiobarbituric acid test for the determination of certain components of oxidized fat. The leuco-base of 2,6-dichlorophenolindophenol has been used in the colorimetric determination of the peroxides of fatty acid glycerides (*24F*). Pohle *et al.* (*55F*) have compared several analytical techniques for the prediction of relative stability of fats and oils, and propose a rapid oxygen bomb method for evaluating the stability of fats and shortenings (*56F*). Micro determinations of neutralization equivalents of higher fatty acids in nonaqueous media have been described (*70F*). Methanolic barium hydroxide has been used as a saponification reagent and found to give maximum saponification values (*4F*). Nuclear magnetic resonance has been proposed by Johnson and Shoolery (*28F*) as a means of determining unsaturation and average molecular weight of natural fats. Wide-line nuclear magnetic resonance determinations of the liquid solid content of fats have been proposed by Ferren *et al.* (*17F*), and Taylor *et al.* (*69F*). The use of solvents and low temperatures for the quantitative separation of liquid and solid fatty acids has been reported with results which are comparable to the lead salts methods (*43F*).

Interest continues to be concentrated on the determination of fatty acids and fatty acid esters by various means. James (*27F*) has published a review of methods of separation of long-chain unsaturated acids. Williams and Reiser (*82F*) have reviewed chemical, biological and physical methods for essential fatty acids. The applications of mass spectrometry in fatty acid and lipid chemistry are reviewed by O'Connor (*51F*). The analysis of fatty acid mixtures by EA (electronenanlagerung) mass spectrometry appears to be a possible method for mixtures with a large number of components (*80F*). Fatty acids and related long-chain compounds, nitriles, amides, etc., have been chromatographed on phosphoric acid treated columns (*45F*). Ackman (*1F*) has investigated the relationship between structure and retention time of unsaturated acids and studied separation factors leading to systematic identification of the methyl esters of unsaturated acids (*3F*). Addition of formic acid to the carrier gas improves the resolution of volatile fatty acids (*2F*). Behenic acid has been used as the stationary phase in the analysis of lower-molecular weight fatty acids from formic to hexanoic (*21F*). Short-chain fatty acids have been gas chromatographed by Craig *et al.* (*11F*) as their butyl, phenacyl, and decyl esters. The latter is the most satisfactory. A multiunit microsteam distillation apparatus useful for preparing samples for gas chromatography has been described (*75F*). Programmed flow gas chromatography has been applied to the analysis of volatile fatty acids (*47F*), and to the analysis of the fatty acids of butter (*74F*). Linolenic, arachidic, and eicosenoic acids are separated as their methyl esters on Chromasorb W containing 20% Craig polyester succinate at 218° C. (*8F*), and on an improved two-column system (*23F*).

Napier (*48F*) suggests the use of 2-methylalkanoic acids as internal standards for the gas chromatographic analysis of fatty acids. A silver salt-alkyl halide esterification has been used for the quantitative preparation of methyl esters for gas chromatography and applied to milk fat (*22F*). Pascaud (*53F*) has used tagged (C^{14}) methyl palmitate to demonstrate the degree of transesterification that can occur during the gas chromatographic separation of methyl esters. The way in which a mass spectrometer may be used as a detector for effluents from gas chromatographic columns is described by Ryhage (*60F*). Geometric isomers of methyl linoleate have been quantitatively determined (*40F*) by gas chromatography on two capillary columns. Glycerol and fatty acids have been determined simultaneously (*25F*) by hydrogenolysis and acetylation, and by determining the fatty acids as methyl esters and the glycerol as isopropylidine glycerol (*44F*). Adsorption chromatography on silica impregnated with silver nitrate has been used by De Vries (*14F*) to separate milligram quantities of higher fatty acid methyl esters.

The adherents of paper chromatography for fatty acids continue to improve their technique. Saturated and unsaturated higher fatty acids have been detected with fuchsin dyes (*39F*). Impregnation of paper with ceresin in acetic acid has been used (*10F*) to achieve separation of fatty acids up to C_{26}. Paper chromatography followed by conversion of the acids on the paper to lead salts has been used for the detection of several fatty acids (*87F*). Hydrogenation on the paper has been used (*86F*) to determine the fatty acid content of wheat-germ oil. Qualitative and quantitative determinations of hydroxy and keto fatty acids have been carried out by Kaufmann and Ko (*30F*) on papers impregnated with a petroleum fraction. Rapid separation of fatty acids was achieved using circular paper chromatography (*50F*), and reversed phase circular paper chromatography has been applied to the determination of higher fatty acids (*79F*).

Thin layer chromatography has been used for the separation of bromo- and hydroxy derivatives of stearic acid (*64F*). Configurational isomers were separated by this technique. Two-dimensional thin layer chromatography on silica gel has been used to separate methyl esters of structural isomers of mono-unsaturated fatty acids (*7F*). The separation of all naturally occurring polyunsaturated fatty acids has been achieved by thin layer chromatography of their mercurial adducts (*81F*).

Partition chromatography on an alumina column has been used by Kuemmel (*36F*) to separate the mercury derivatives of unsaturated fatty acid esters. Mixtures of hydroxy fatty acids, their methyl esters and reduced fatty acid hydroxides have been separated by column chromatography (*20F*). Infrared spectroscopy has been used (*49F*) to determine the ethylenic bonds in trans-form in fatty materials, and to determine the trans-fatty acids in lard (*41F*). A better method for the determination of erucic acid in rapeseed oil is proposed by Sesbes (*63F*). Soyabean oil fatty acids have been fractionated by crystallization as "acid" sodium soaps (*73F*). The use of an internal standard simplifies and aids quantitation in the determination of higher fatty acid esters from alcoholic beverages (*15F*). The ferric hydroxamic acid reaction has been adapted to the determination of long chain fatty acids (*54F*).

The use of a silver nitrate–methanol solvent in counter current distribution has served to separate cis- and trans- monounsaturated esters (*61F*). Esterified fatty acids in glycerides, cholesteryl esters and phosphatides have been determined by a modified hydroxylamine reaction (*65F*). Fatty esters have been separated by thin layer chromatography, and in conjunction with gas chromatography, a rapid analysis of the fatty acid composition of seed oils may be obtained (*77F*).

Methods for the determination of the

glyceride composition of fats continue to be developed and improved. Vander Wal (*76F*) has published a review of the various methods for the determination of glyceride structure. Chromatography on a silicic acid–silver nitrate column followed by gas chromatography has been used (*66F*) to evaluate the glyceride types in natural fats. The structure of ethanol-insoluble high melting glycerides has been studied by Wolf and Dugan (*84F*) using pancreatic lipase to determine the position of the fatty acids in triglycerides. Silver nitrate impregnated plates are used for initial fractionation, then a reversed phase system provides further separation of triglycerides (*34F*). Gas chromatographic separation of triglycerides of butterfat has been achieved by Kuksis *et al.* (*38F*) and the molar proportions of triglycerides can be calculated from the response of the hydrogen flame ionization detector. Separation of triglycerides by number of carbon atoms has been obtained on lightly loaded silicone SE-30 columns (*37F*).

Lipids differing in number of double bonds and in configuration may be separated by thin layer chromatography with silver nitrate impregnated silica gel (*16F*). The lipids of the coffee bean have been studied by Kaufmann and Sen Gupta; cafestol and kahweol have been separated by thin layer chromatography (*32F*), and triterpenes and hydrocarbons have been isolated (*33F*). Paper chromatography has been used to quantitatively determine neutral glyceride glycerol and phosphoglyceride glycerol (*12F*).

Firestone (*19F*) has published a review on the determination of polymers in fats and oils. A column chromatographic method has been used to separate saturated hydrocarbons in mixtures with saturated monohydric alcohols (*72F*). Methyl ketones in fats have been isolated and chromatographed on paper as their 2,4-dinitrophenylhydrazones (*68F*). Free monocarbonyl compounds in fats and oils have been determined by conversion to dinitrophenylhydrazones by passage of a solution of the sample over an alumina dinitrophenylhydrazine reaction column (*35F*), and by conversion to 2,4-dinitrophenylhydrazones using special conditions (*62F*). A three-layer column followed by gas chromatography has been used by Parks *et al.* (*52F*) to determine bound aldehydes in butter oil.

The gas chromatographic separation of long-chain acid amides has been reported (*46F*). Paper chromatography is proposed as a method of determining the methyl esters of fatty acids in fats (*29F*). Fett (*18F*) has separated the components of lactylated emulsifiers by chromatography on silicic acid.

FLAVORS AND VOLATILE COMPOUNDS

The study of aromas and flavors in foods has relied heavily on gas chromatography in the past two years. Among the new techniques suggested were use of lightly loaded Nichrome helices as columns for separating high boiling compounds such as maltol and xanthine from natural products (*24G*) and a solid sampling device for direct gas chromatographic analysis of spices (*60G*). The use of capillary units has been described by Self (*58G*) and many others. The use of headspace analysis for flavor has been described (*21G*, *50G*) and its pitfalls discussed (*46G*). Sulfur components have been found by use of a dual unit with both electron capture and flame ionization detectors (*47G*). Functional group analysis in gas chromatography by syringe reactions has been described (*12G*). Compounds have been identified by a combination of pyrolysis and gas chromatography techniques (*7G*, *8G*). Infrared spectrometry has been used in differentiation of C_3 to C_{16} *n*-alkanal dinitrophenylhydrazone derivatives (*26G*). A review of flavor research includes aroma sampling and concentration, effects of enrichment on composition of volatiles, separation techniques, and component identification (*25G*).

The Quartermaster group at Natick has continued its work using high vacuum low temperature fractionation, mass spectrometry, and gas chromatography to study food volatiles. Reports have been made on beef (*43G*), coffee (*41G*), and meat, fish, and coffee (*42G*). A similar fractionation scheme separates neutral carbonyl compounds for subsequent identification by spectral means and has been applied to examination of fresh and stored fish (*9G*). The volatiles of lamb (*13G*, *17G*) and cured hams (*48G*) have been studied. Hornstein and Crowe have reviewed the analysis of meat flavors (*14G*). An abstract reports that staling of coffee has been examined by quantitative gas chromatography (*55G*). Vacuum distillation and infrared analysis have been used to distinguish between chocolate materials (*54G*). A gas chromatographic examination of the essential oil of tea leaves was reported in a series of papers on the flavor of green tea (*72G*). The isolation and identification of the volatiles of celery (*10G*) and cooked rutabaga (*11G*) have been described. A survery was reported of low boiling volatiles of green and root vegetables (*57G*). A gel filtration separation scheme was used for peanut flavor precursors (*38G*). A review on maltol includes methods of analysis (*3G*). Seven monohydrophenols in vanilla extract have been separated by paper chromatography (*1G*). Vanillin and ethyl vanillin have been determined by gas chromatography using an internal standard (*35G*)

An extensive review has been made of concentrations and gas chromatography techniques for studying fruit aromas (*40G*). Analyses have been reported for apple juice and wine (*28G*, *61G*), bananas (*16G*, *29G*, *30G*), Florida orange juice (*70G*), Bartlett pears (*18G*, *19G*), maple syrup (*66G*) and honey (*44G*, *62G*). The Western Utilization Research Laboratory has continued its studies with sophisticated instrumentation (*31G*, *63G*) and reported on strawberry volatiles (*64G*) and the hydrocarbons from oranges (*65G*). A method was described for recovery of citrus juice volatiles by reverse phase, liquid-liquid extraction and distillation (*59G*). Methyl anthranilate in grape juice has been determined by gas chromatography using an electron affinity detector (*39G*). The study of beer produced methods for esters (*20G*), diacetyl (*49G*), phenethyl alcohol (*71G*) and other volatiles (*67G*). Wine and distilled spirits have been analyzed for their methanol (*33G*), higher alcohol or fusel oil (*34G*, *53G*, *68G*), carbonyl compounds (*36G*, *37G*) and amine (*15G*) content.

Work has continued on the volatiles of dairy products. Gas chromatography has been used in the study of fresh and stored milk (*2G*, *32G*), evaporated milk (*45G*), and cheese (*6G*, *23G*, *27G*, *51G*, *52G*). Vanillin was isolated and identified in heated milks by gas and thin layer chromatography (*4G*). The carbonyls of whole milk powder (*5G*) and butter (*69G*) were separated by partition chromatography of their dinitrophenylhydrazones. The methyl ketones in blue cheese have been quantitatively estimated (*56G*). The volatile sulfur content of soft cheese was determined by steam distillation, combustion and absorption of SO_3 (*22G*).

IDENTITY

Accompanying the interest in analyzing for possible adulteration or decomposition in foods is the need to know the analytical picture of the fresh authentic food. For this reason a section on identity and purity has been added to this Review to help provide the necessary background for adulteration and analyses, and to show the techniques that have been used to obtain this information.

Among the cereal foods, gas chromatography has been used to determine the fatty acid composition of rice bran oil (*57H*), of rice lipids (*45H*), and of the triglyceride composition of oil extracted from maize (*38H*). Lipids from hard and soft wheats have been differentiated by infrared spectrophotometry (*13H*). The proteins of wheat gluten have been examined by zone electrophoresis on polyacrylamide gels (*44H*), and the flour proteins from various varieties of wheat have been examined by moving-boundary electrophoresis (*42H*). Bell and Simmonds (*9H*) have studied the protein composition of different flours and its relationship to baking quality. The organic acids of rice and other cereal seeds have been determined (*34H*).

Coffee composition has been quite thoroughly investigated, both as green and roasted bean and as beverage. Thaler, *et al.* (*72H*) has determined the composition of the protein of green coffees. Complete analyses of 143 samples of Robusta coffee have been reported (*6H*). Chemical changes occurring during roasting are reported by Skarka *et al.* (*69H*) and Bressani *et al.* (*12H*). The soluble substances and aromatic constituents of roasted coffee extract have been determined (*71H*), as well as the nitrogeneous substances of these extracts (*73H*). The effect of pretreatment of raw coffee before roasting has been reported (*1H*). The ageing of roasted coffee has been followed (*59H*) by the determination of volatile reducing substances.

Chemicaland organoleptic data have been compiled on thawed and unthawed fish fillets (*31H*). A new electrical resistance measurement is said to permit the determination of the degree of freshness of whole fish (*25H*). Disk electrophoresis has been used (*49H*) as a means of identifying fish species from protein patterns. Water soluble flavor and odor precursors in beef have been studied qualitatively (*47H*) and quantitatively (*48H*).

Fruits and fruit juices have also been studied extensively. The fruit acids in apple and pear juice have been determined by paper chromatography (*77H*). A study of the composition of black currant juices and the use of the data to evaluate juice in syrups, etc., has been reported (*3H, 4H, 7H*). Spectrophotometric readings have been used by Rice (*62H*) to identify grape varieties. A series of reports discusses the characterization of lemon juice by amino acid and L-malic acid content (*74H*), by polyphenolic content (*75H*), and the computer treatment of the data obtained (*64H*). Clements has determined organic acids in citrus fruits (*15H*), and in varieties of oranges (*16H*). Primo *et al.* (*60H*) have determined acids in orange juice by thin layer and gas chromatography. Various criteria for determining orange juice in beverages are reported (*65H*). The chemical composition of natural and processed orange juices is reported (*66H*). Components of citrus and tomato juices produced in Israel are listed (*76H*). Fruit juices may be characterized by acid profiles obtained by ion exchange chromatography (*37H*).

The change in the electrophoretic pattern of milk proteins with various treatments has been shown by Bagnulo *et al.* (*5H*). The proteins of milk serum have been separated and quantitatively evaluated by electrophoresis on cellulose acetate membranes (*51H*). Studies of milk fat include the variation of conjugated-acid content with the season (*19H*), the fatty acid composition variation with the feed (*39H*), and the fatty acid composition of a large number of samples showing seasonal trends (*36H*). Gas chromatography has been used extensively in the study of butter. Boniforti (*10H*) uses the chromatograms of the ethyl esters for the study of butter produced in Italy and in other countries. The ratios of various fatty acids may be used as an indication of the purity of butter (*22H*). A method for studying butters with abnormal contents of soluble and insoluble volatile acids uses esterification with methanol and gas chromatography (*58H*). Significant fatty acids and fatty acid ratios for genuine butter are reported (*8H*). Programmed temperature gas chromatography is used to analyze butter and volatile fats (*56H*). The polyunsaturated acid content of margarines has been determined (*63H*).

Aged, but not rancid, soya bean oil has been examined for monocarbonyl content by column purification and identification techniques (*55H*). Thin layer chromatography and gas chromatography have been used to establish the purity of cacao butter (*40H*). Normal samples of cacao butter contain measurable amounts of caffeine and some theobromine (*24H*). Thin layer chromatographic techniques were used (*61H*) to investigate the composition of egg lipids. Various fats and oils have been tested by thin layer chromatography for identity and purity (*2H*). By applying gas chromatography to the unsaponifiable portion of several oils Eisner and Firestone (*23H*) have found a means to identify these oils when present alone. On the problem of the purity of olive oils, squalene index apappears to be of little value (*27H*). The ultraviolet absorption characteristics of olive oils may prove a better index. The absorption in isooctane (*35H*) and the use of alumina clarification (*18H, 43H, 53H*) appear to provide a better means of classification. Some confusion may be introduced, if the olive oil is of the "deep-green" variety (*26H*). The measurement of extinction at 270 mμ has been proposed as a criterion for the rapid study of changes in stored olive oil (*70H*). The ratio of extinctions at 220 and 232 mμ has also been considered as an index of quality (*14H*). Limits for four types of virgin olive oils are proposed, to be calculated from ultraviolet extinction data (*17H*). Fatty acids of olive oils from different regions have been determined by gas chromatography (*54H*). Total fluorescence measurements are reported to provide a means of distinguishing between olive oils (*20H*).

A method of determining methyl anthranilate in orange-flower honey provides a means of determining the authenticity of a sample of this type of honey (*21H*). The formol number, pH, free acid, and lactone content of 65 honeys are tabulated by Hadorn *et al.* (*28H*). The presence of anthocyanins in jams and marmalade is a positive quality criterion, and a method of detecting these compounds is given (*30H*). A study of chemical changes occurring during the storage of tomato paste and tomato products includes methods for the determination of browning, lycopene, and hydroxymethyl-furfural (*67H*). Storage stability of tomato paste is discussed by Luh *et al.* (*46H*). Hamdy and Gould (*29H*) have investigated the α-keto acids, α-amino compounds, and citric acid in tomato varieties before and after processing. Acids in different varieties of tomatoes have been determined (*11H*). Complete analyses of commercial sauerkraut samples have been reported (*50H*). A clear analytical distinction between canned processed and canned garden peas has been shown (*33H*). The rum content of rum butter has been determined (*68H*). The almond content of marzipan (*52H*) may be determined by determination of oil and protein, or by determination of oil alone (*32H*). The organic acids in a variety of foodstuffs have been determined (*41H*) for identification purposes.

INORGANIC CONSTITUENTS

A continuing nuclear test ban should ultimately make radionuclides in our food supply of little concern. However, the earlier fall-out motivated development of improved techniques for concentrating the nuclides in milk and water for analysis (*4J*) and of rapid or improved methods for the radioisotopes of caesium (*10J, 23J, 39J*), iodine (*3J, 29J*), and strontium (*11J, 22J*). X-ray fluorescence techniques have been reported for strontium (*35J*) and for strontium and calcium (*20J*) in milk. Trace iron, manganese, molybdenum, and zinc in milk have been determined spectrographically after clean-up by ashing, chelation, and solvent extract of the chelates (*36J*). A colorimetric method for manganese at the 1- to 5-μg. level in milk and other foods is based on its catalytic effect on the $NaIO_4$ oxidation of tetramethyldiaminodiphenylmethane (*24J*). A scheme for the rapid determination of copper, iron, manganese, and zinc in plant material uses separate aliquots from a single digest for estimate of zinc by atomic absorption spectroscopy and of the other elements colorimetrically (*5J*). Atomic absorption spectroscopy has been applied in determining lead, copper, and zinc in beer (*40J*) and magnesium in plant material (*38J*). Trace metals in beer are apparently important as shown by use of the spectrograph for aluminum, iron, and tin (*30J*), the polarograph for tin (*28J*), colorimetry for aluminum (*32J*), zinc (*31J*) and copper (*8J*), and neutron activation for copper (*8J*), which was superior to three spectral methods. Copper in fats has been separated by boiling the fat with 5 to 10*N* sulfuric acid, removing fat with chloroform, and extraction by chelation for determination (*19J*). A rapid, selective, one-color dithizone method for zinc in foods has been described (*37J*). A microdetermination of mercury is based on wet digestion, reduction of mercury salts, steam distillation of mercury into $KMnO_4$, and a dinaphthyldithiocarbazone estimation (*34J*). Selenium in plant material, oxidized by Schoniger combustion, has been determined fluorimetrically (*13J*, *17J*); however, isotopic dilution technique is reported necessary for accuracy (*13J*). Anthocyanin interference in determination of calcium with EDTA was eliminated by using hydroxylammonium chloride or hydrazine sulfate to decolorize the pigments (*7J*). Alkali metals and alkaline earths in molasses have been separated by ion exchange chromatography for determination by titration methods (*12J*).

Perchlorate at trace levels in plant material can be separated with a quaternary ammonium exchange resin and determined by infrared spectrometry (*25J*). Bromates in bread were determined colorimetrically through a brominated rosaniline derivative (*14J*). The common inorganic flour-bleaching agents have been separated and identified by a scheme using three paper chromatographic runs (*2J*). Nitrate and nitrite determination in meat products is still being studied with colorimetric (*1J*, *6J*, *15J*, *18J*) and polarographic (*16J*) methods reported. A study of six titrimetric and polarographic methods for iodine in salt has been made (*26J*) and an automatic reaction rate method has been reported (*21J*).

An automatic sampling device for the head space gas of cans and bottles on a filling line has been described (*33J*).

Gas chromatography was used for analysis of hydrogen, nitrogen, and oxygen in beer head space (*9J*). Carbon dioxide in cheese was estimated by nitrogen sweep of a slurry and absorption in alkali (*27J*).

MOISTURE

Although few specific applications to food products were reported, the abstracts of a Symposium on Humidity and Moisture, its Measurement and Control, are a very useful source for the food analyst in developing or choosing techniques for moisture measurement (*3K*). The complete proceedings of this symposium are expected to be published. Fraade (*5K*, *6K*) in discussions of methods for moisture in liquids and solids paid particular attention to an electrolytic hygrometer. A near infrared method has been reported for analysis of fruits and vegetables over a wide range of moistures (*7K*). NMR calibration curves suitable for moisture determination of several types of wheat and flour have been established (*11K*). The pitfalls in using commercial moisture meters for wheat are discussed by Christensen and Linko (*4K*). Several workers found refractometric methods more reliable than other techniques for estimating solids in wheat extract, malt extract or beers (*1K*, *17K*). Use of four different electrical units has been reported in automatic control of moisture in dairy products (*16K*). Karl Fischer techniques are recommended for accuracy and rapidity in estimating water in butter (*8K*), cheese (*10K*), and dried vegetables (*15K*). A gas chromatographic method is reported as suitable for routine control analysis of moisture in foods (*14K*). A rapid method for moisture in sugar is based on the color produced with fuchsine (*9K*). Amdur discussed several types of sensors available for humidity measurement (*2K*). Nemitz reported a simple method for determining sorption isotherms and gave equilibrium moisture contents for a number of foods (*12K*, *13K*).

ORGANIC ACIDS

Differential thermal analysis, reported for 25 organic acids (*32L*), should be an aid in identification studies. Use of bromophenol blue in a developing solvent for nonvolatile organic acids gives an automatically developed chromatogram (*2L*). A butanol–pyridine–water system performs similarly since the acids appear dark blue on a light blue background when viewed under ultraviolet light (*3L*). Identification of dicarboxylic acids by paper chromatography of their dinitrobenzyl esters was suitable for those acids with an even number of carbon atoms (*5L*). Thin layer chromatography procedures have been reported for the dicarboxylic acid series from oxalic to sebacic (*9L*) and for cis-trans isomeric acids (*16L*). Ion exchange chromatography has been used for determination of the acids of beet juice or molasses (*34L*), blueberries (*11L*), and fruits (*7L*). It has been used for separation and determination of lactic, malic, and tartaric acids (*6L*). A scheme for the isolation and concentration of 0.5 to 100 μg. of organic acids in small samples with determination by gas chromatography has been described (*20L*). Methyl esters of the acids were prepared on injection into a gas chromatograph by pyrolysis of their tetramethylammonium salts (*21L*). Gas chromatographic methods have been used for quantitative determination of formic, acetic, propionic and butyric acids (*25L*), identification of the Krebs cycle acids (*10L*), and determination of the α-acids of hops (*1L*).

A specific color reaction of formic acid with anisole and lead nitrate has been reported (*17L*). Citric acid has been determined spectrophotometrically by molybdate method (*12L*) and a pyridine–acetic anhydride procedure (*4L*). Malic acid in wine was determined by an enzymatic method (*13L*); lactic and tartaric acids colorimetrically (*19L*). Procedures have been described for determining laevulic acid in bagasse (*30L*), naphthylacetic acid in pineapple (*33L*), and oxalic acid in food (*35L*).

Herrmann has prepared a review on the naturally occurring tannins, their food sources and methods of determination (*8L*). A quantitative paper chromatographic method for phenolic acids has been described (*14L*). The phenolic acids of barley have been identified by thin layer chromatography on polyamides (*31L*). A procedure has been claimed for obtaining good infrared spectra of traces of phenolic acids eluted after separation by paper chromatography (*24L*). The acidity of coffee, its determination, and significance has been discussed (*18L*). The use of a paper chromatographic method for the chlorogenic acids of coffee to control roasting has been suggested (*27L*). Elution of chlorogenic acid from paper chromatograms and spectral measurement has been reported (*29L*). An extraction and concentration procedure for small amounts of chlorogenic acid in plant material has been described as well as its identification in oranges and castor beans (*26L*). Pyrocatechol has been determined colorimetrically from its reaction in solutions of sodium tungstate and nitrite (*15L*). Additional papers by the late E. A. H. Roberts on the determination of phenolic substances of tea and their significance have appeared (*22L*, *23L*). A scheme has been presented for the analysis of the phenolic and volatile acids of smoked foods (*28L*).

PROTEINS, AMINO ACIDS, AND NITROGEN

Smyth and Elliot reviewed some analytical problems in determining protein and peptide structure (*41M*). The Society for Analytical Chemistry recommended an average nitrogen factor of 3.55 for beef (*42M*) and factors varying from 3.6 to 3.9 for different types of chicken meat (*43M*). The use of iodic acid–phosphoric acid in decomposing biological material is claimed to permit rapid, accurate nitrogen determination (*48M*). Good correlation with the Kjeldahl estimate of protein in meats was found for a biuret method and Orange G dye binding but not for an Amido Black dye binding (*52M*). Fluorometry was found to be a rapid means for determining protein in milk directly (*11M*); however, temperature must be rigidly controlled (*14M*). An improved method for the "protein reducing substance" value of milk can be used to detect admixtures of raw, pasteurized, and reconstituted milk (*30M*). The problems in separation of milk proteins on DEAE cellulose were discussed by Tarassuk and Yaguchi (*49M*). Electrophoretic techniques were described for separating wheat (*12M*) and other cereal proteins (*29M*). Gel filtration was used for separating the proteins and polyphenols of grape juice (*55M*). Protein molecular weights can be estimated by gel filtration techniques (*1M*, *53M*). Bell has studied the methods for determining the nonprotein nitrogen fractions of several foods (*2M*, *3M*).

In hydrolyzing tissue for amino acid analysis, phenol in the digest mixture prevented the formation of a dark insoluble precipitate (*10M*). Conditions have been described for hydrolysis of DNP protein by Dowex 50 catalysis, which is considered more reliable than strong acid hydrolysis for terminal residues (*46M*). A mass spectrometric method was reported for quantitative analysis of amino acid mixtures (*19M*). Amino acids have been differentiated by gas chromatography of their pyrolysis products (*54M*), and of their *N*-acetyl (*39M*), several *N*-trifluoracetyl (*9M*, *17M*, *58M*) and phenylthiohydantoin and DNP derivatives (*31M*). High sensitivity for DNP derivatives was obtained with an electron capture detector (*22M*). Anion exchange resins in gas chromatographic columns were used to generate and separate the free base forms of the methyl esters of amino acid salts (*27M*). Complex peptide mixtures in hydrolyzates were separated by chromatographing on a cation exchanger and then on an anion exchange resin (*36M*). Hamilton has described a single column, high resolution, fully automatic ion exchange system for quantitative determination of 10^{-8} mole of amino acids (*15M*). More stable buffer and ninhydrin reagents have been reported for use in automatic amino acid analysis (*34M*). The elution behavior of ninhydrin positive compounds, such as amino acids and related compounds, during ion exchange chromatography has been studied in analysis of nonprotein fractions of natural materials (*57M*). The chromatographic behavior and ninhydrin reactions of carbonyl compounds and saccharides suggest caution in interpretation of amino acid analyses (*35M*). Two dimensional thin layer chromatography has been used for amino acid separation (*13M*). Procedures have been described for determining the amino acids of coffee protein (*50M*, *51M*) and the nitrogeneous compounds of molasses (*28M*, *44M*). Amino acid esters have been determined as hydroxamates after chromatographic separation (*6M*, *7M*).

A paper chromatographic method has been reported for determining the basic amino acids in plant extracts (*47M*). Sulfhydryl groups in milk protein have been determined amperometrically (*56M*) and fluorimetrically in a method based on reduction of thiamine disulfide (*20M*). The sulfhydryl groups of wheat flour were determined through their reaction with 2,2′-dihydroxy-6,6′-binaphthyldisulfide (*24M*). Moore has improved the method for determining cystine and cysteine as cysteic acid and of methionine as the *S*-dioxide (*25M*). Methionine has been determined by infrared spectrometry from the methane obtained on reduction with Raney nickel; cystine and cysteine do not interfere and are determined from the difference between total and methionine sulfur (*18M*). Cysteic acid after separation on ion exchange paper has been determined densitometrically or spectrometrically after elution (*16M*). Cystine in sodium glutamate was detected polarographically (*37M*). Glutamic acid in soup preparations was estimated by ascending chromatography with butanol–acetic acid–water as the developing solvent (*45M*). Several improved methods for determining hydroxyproline have been reported (*4M*, *5M*). Available lysine in oilseed protein was determined after dinitrophenol reaction, acid hydrolysis, and ion exchange chromatography (*33M*). In a study of lysine methods for cereals, microbiological and ion exchange chromatographic methods were found reliable but enzymatic paper chromatographic procedures were not (*32M*). Kofranyi has reported a method for tryptophan in food using a barium hydroxide digest (*21M*). A method for tryptophan might be based on its effect on the color produced in the anthrone–carbohydrate reaction (*26M*). A colorimetric method for tyrosine in gelatin is based on the α-nitroso-β-naphthol reaction (*8M*). An enzymatic method for ribonucleotides in foods has been reported (*38M*). Lento *et al.* used an exchange chromatographic method for separation and estimation of five 5′-nucleotides (*23M*). A sensitive method for estimation of frost injury to plant tissue is based on the release of amino acids and other ninhydrin reacting compounds from the frozen cells (*40M*).

VITAMINS

The Society for Analytical Chemistry has recommended methods for water and fat soluble vitamins in feeds (*30N*, *31N*). Separation of vitamins by chromatography in a centrifugal field (*2N*, *7N*) and on ion exchange treated paper (*20N*) have been reported. Separation of vitamin A and related compounds have been obtained by gas (*10N*), thin layer (*37N*), column (*4N*, *28N*) and paper chromatography (*21N*). The low potency 9-cis-isomers of vitamin A have been determined spectrometrically by the reaction of their retinols with opsin (*19N*). Wilkie has recommended a new method of background correction in vitamin A spectrophotometry (*38N*). Trifluoroacetic acid in reaction with vitamin A produces a spectrally identical species to that formed with antimony trichloride; however, this new reagent is more convenient (*9N*). Polarography in nonaqueous media has been used for vitamin A estimation (*34N*). Vitamin D has been separated and determined by several chromatographic techniques (*24N*, *29N*). The tocopherols have been determined by gas chromatography (*26N*), separated from milk by silicic acid chromatography (*11N*), isolated from margarine by column and paper chromatography (*23N*) and estimated in plants, such as spinach, after saponification, and chromatographic separation by spectral means before and after oxidization (*8N*).

Thin layer chromatography of thiamine has been reported (*6N*). Thiamine has been determined polarographically with reported advantage from use of low concentrations of aluminum sulfate (*36N*) and starch (*35N*) in the electrolyte. A rapid extraction procedure for riboflavine in enriched cereals has been established (*1N*). A paper chromatographic method for nicotinic acid in coffee was used in a study of the vitamin level at different roasts (*3N*). After a collaborative study it was concluded that acid hydrolysis is preferred in determination of nicotinic acid in cereals by the Association of Vitamin Chemists method (*13N*). The B_6 vitamins were separated on sulfonic acid exchange resin and determined fluorimetrically (*15N*). Vitamin B_{12} has been separated by thin layer chromatography (*5N*) and with an ion exchange resin; however, tracer study was needed to compensate for losses (*27N*). Polarographic studies of ascorbic acid assay have continued (*25N*, *39N*). Paper chromatography was used to determine ascorbic acid and related compounds in cooked products

(*16N, 17N, 18N*). A rapid thin layer procedure was used to study ascorbic acid changes in potatoes under irradiation (*14N*). Carbon dioxide sweeping was found the most suitable method for sulfur dioxide removal before determining ascorbic acid (*22N*). Cysteine was recommended for quantitative reduction of dehydroascorbic to ascorbic acid (*32N*). In determination of ascorbic acid as the dinitrophenylhydrazone, chromatographic separation from sugar derivative is made before spectral estimation (*33N*). A simple titration method for determining ascorbic acid in highly colored solutions such as fruit juices (*12N*) has been reported.

MISCELLANEOUS

Johnson has discussed the problems of sampling and sampling devices in food processing (*18P*). An extensive discussion of thin layer chromatography covered many applications to the separation of food constituents (*28P*). Reviews have been prepared on application of microchemistry (*11P*) and mass spectrometry (*10P*) in food analysis. Several surveys on brewing processes include sections on analytical methods (*14P, 22P, 23P*). By international agreements, the specific gravity and alcohol content are now defined at 20° C. instead of 15°; new temperature correction tables have been prepared to cover this change (*16P*).

In the analysis of coffee, the determination of caffeine has continued to receive attention (*1P, 2P, 5P, 31P*). In the spectral methods, the interference by 5-hydroxymethyl furfural (*25P, 36P*) is not usually eliminated. In methods using nitrogen as a caffeine index, the interference of polypeptides extracted by the solvents, particularly with decaffeinated products (*36P*), is not corrected. To eliminate these problems, Smith and Rees use aluminum amalgam reduction in a spectral method (*26P*) while Yeransian, *et al.* use a dual column chromatographic purification of caffeine or spectral estimation (*36P*). Borker and Sloman recommended another dual column method to the A.O.A.C. for decaffeinated products (*7P*). A method for caffeine in beverages is based on color obtained by its reaction with a malonic acid–acetic anhydride reagent (*17P*). A sublimation method has been applied to caffeine in tea (*24P*). Other coffee analytical methods reported include fractionation of flavor and color components with Sephadex (*29P*), correlation of degree of roasting with absorbance of an alkaline extract (*21P*), and estimation of the yield of roasted coffee in a soluble coffee powder from potassium or mineral content or the weight of a precipitate from treatment of an extract with alcohol (*35P*). The estimation of tea constituents and their influence on quality have been discussed in a number of papers (*20P, 30P, 32P, 33P, 34P*).

The acid soluble organic phosphates of milk were obtained by trichloracetic acid extraction or dialysis and separated by ion exchange chromatography (*15P*). Gas chromatography (*8P*) and paper chromatography followed by fluorescence study (*9P*) has been used in analysis of coumarin in natural products. The estimation of the maturity of peanuts has been attempted by calculation from light absorption measurement on nut halves (*19P*). A photo densitometer has been used as an objective method for marbling in meat (*3P*).

LITERATURE CITED

Additives

(1A) Anderson, R. H., Nelson, J. P., *Food Technol.* **17**(7), 95 (1963).
(2A) Association of Public Analysts, *Special Rept.* 1 (1963).
(3A) Baum, F., Lamm, H., *Ernahrungsforschung* **8**, 355 (1963).
(4A) Biefer, K. W., *Mitt. Gebiete Lebensm. Hyg.* **53**, 243 (1962); *Anal. Abstr.* **10**, 812 (1963).
(5A) Borginon, H., *Phot. Korr.* **99**, 26 (1963).
(6A) Brouwer, T., *Chem. Weekblad* **59**, 12 (1963).
(7A) Burroughs, L. F., Sparks, A. H., *Analyst* **88**, 304 (1963).
(8A) Coleman, C. H., Johnson, W. D., Pleticka, W. J., Butnis, C., *J. Agr. Food Chem.* **10**, 293 (1962).
(9A) Davidek, J., *J. Chromatog.* **9**, 363 (1962).
(10A) Davidek, J., *Ibid.*, **11**, 419 (1963).
(11A) Fellegiova, M., *Z. Lebensm.-Untersuch.-Forsch.* **120**, 17 (1963).
(12A) Fischer, R., Benkendorf, F. G., *Ibid.*, **117**, 400 (1962); *Anal. Abstr.* **10**, 371 (1963).
(13A) Garcia Fernandez, J. C., Lanucura, E. N., *Rev. Asoc. Bioquim. Argentina* **28**, 179 (1963); *Anal. Abstr.* **10**, 5395 (1963).
(14A) Ghimicescu, G., Ghimicescu, C., Dumbrava, E., *Acad. Rep. Populare Romine, Baza Cercetari Stiint Timisoara, Studii Cercetari, Stiinte Chim.* **11**, 103 (1963); *Anal. Abstr.* **11**, 1322 (1964).
(15A) Graham, H. D., *J. Food Sci.* **28**, 440 (1963).
(16A) Guerra Salazar, G. E., *Anales Asoc. Quim. Arg.* **50**, 59 (1962); *Anal. Abstr.* **10**, 2333 (1963).
(17A) Hall, M. J., *J. Assoc. Offic. Agr. Chem.* **47**, 685 (1964).
(18A) Hamence, J. H., ANAL. CHEM. **35**(1), 24A (1963).
(19A) Hamilton, J. W., Tappel, A. L., *J. Am. Oil Chem. Soc.* **40**, 52 (1963).
(20A) Handshack, W., *Nahrung* **7**, 155 (1963); *Anal. Abstr.* **10**, 5397 (1963).
(21A) Hause, J. A., Hubicki, J. A., Hazen, G. G., ANAL. CHEM. **34**, 1567 (1962).
(22A) Hoyem, T., *J. Assoc. Offic. Agr. Chem.* **45**, 902 (1962).
(23A) Ichibangase, H., Kojima, S., *J. Pharm. Soc. Japan* **82**, 1616 (1962); *Anal. Abstr.* **10**, 5391 (1963).
(24A) Invernizzi, I., Sampietro, C., *Boll. Lab. Chim. Provinciali (Bologna)* **14**, 127 (1963).
(25A) Jaulmes, P., Brun, S., Cabanis, J. C., *Chim. Anal. (Paris)* **44**, 327 (1962); *Anal. Abstr.* **10**, 2493 (1963).
(26A) Jensen, P. T., *Nord. Veterinarmed.* **15**, 171 (1963); *Anal. Abstr.* **11**, 1499 (1964).
(27A) Kielhofer, E., Wurdig, G., *Deut. Lebensm. Rundschau* **59**, 197 (1963).
(28A) *Ibid.*, p. 224.
(29A) Kirsten, W. J., Nilsson, S. K., *Anal. Chim. Acta* **27**, 345 (1962).
(30A) Kröller, E., *Fette, Seifen, Anstrichmittel* **64**, 85 (1962).
(31A) *Ibid.*, p. 602; *Anal. Abstr.* **10**, 2025 (1963).
(32A) Kroller, E., *Ibid.*, **65**, 482 (1963).
(33A) Kroller, E., *Ibid.*, **66**, 456 (1964).
(34A) Larrouquere, J., *Bull. Soc. Chim. (France)* **1963**, 1026.
(35A) Lemieszek-Chodorowska, K., *Roczniki Panstwowego Zakladu Hig.* **13**, 133 (1962); *Anal. Abstr.* **9**, 5441 (1962).
(36A) Lemieszek-Chodorowska, K., *Ibid.*, **14**, 231 (1963); *Anal. Abstr.* **11**, 2815 (1964).
(37A) Lloyd, W. J. W., Cowle, B. C., *Analyst* **88**, 394 (1963).
(38A) Lorenzen, W., Sieh, R., *Z. Lebensm.-Untersuch.-Forsch.* **118**, 223 (1962).
(39A) Luthi, H., Bezzegh, T., *Mitt. Gebiete Lebensm. Hyg.* **53**, 259 (1962).
(40A) Mayer, K., Busch, I., *Ibid.*, **54**, 297 (1963).
(41A) Meikle, R. W., *Nature* **196**, 61 (1962).
(42A) Mergenthaler, E., *Z. Lebensm.-Untersuch.-Forsch.* **119**, 144 (1963).
(43A) Mil'skii, A. V., Gaidukovich, A. Y., *Khlebopek. i Konditer. Prom.* **1962**, 5; *Anal. Abstr.* **10**, 3443 (1963).
(44A) Nakamura, N., Yoshida, T., Kusumoto, S., *Japan Analyst* **11**, 978 (1962).
(45A) Okuhara, A., Yokotsuka, T., *J. Agr. Chem. Soc. Japan* **36**, 320 (1962).
(46A) Palladini, F., *Boll. Lab. Chim. Provinciali (Bologna)* **13**, 114 (1962); *Anal. Abstr.* **9**, 5448 (1962).
(47A) Polyanskii, N. G., Tsekhmister, E. F., *Zh. Analit. Khim.* **18**, 888 (1963).
(48A) Romani, B., Bastianutti, J., *Boll. Lab. Chim. Provinciali (Bologna)* **14**, 119 (1963).
(49A) Salo, T., *Suomen Kemistilenti B* **36**, 1 (1963); *Anal. Abstr.* **10**, 5396 (1963).
(50A) Salo, T., Airo, E., Salminen, K., *Z. Lebensm.-Untersuch.-Forsch.* **125**, 20 (1964).
(51A) Schmidt, H. E., *Fette, Seifen, Anstrichmittel* **65**, 488 (1963).
(52A) Schwecke, W. M., Nelson, J. H., *J. Agr. Food Chem.* **12**, 86 (1964).
(53A) Smith, B., Carlsson, O., *Acta Chem. Scand.* **17**, 455 (1963).
(54A) Spiteri, J., Castang, J., Solere, M., *Ann. Fals. Expert. Chim.* **56**, 652 (1963).
(55A) Stefan, V., *Chem. Prumysl* **12**, 652 (1962); *Anal. Abstr.* **10**, 4255 (1963).
(56A) Stetzler, R. S., Andress, T. B., *J. Am. Oil Chem. Soc.* **39**, 509 (1962).
(57A) Szalkowski, C. R., Garber, J. B., *J. Agr. Food Chem.* **10**, 490 (1962).
(58A) Takahashi, D. M., *J. Assoc. Offic. Agr. Chem.* **47**, 367 (1964).
(59A) Thalacker, R., *Deut. Lebensm. Rundschau.* **59**, 111 (1963).
(60A) Thrasher, J. J., *J. Assoc. Offic. Agr. Chem.* **45**, 905 (1962).
(61A) Turner, A., *Analyst* **89**, 115 (1964).
(62A) Volff, R., Barraud, C., *Ann. Fals. Expert Chim.* **55**, 309 (1962); *Anal. Abstr.* **10**, 3897 (1963).
(63A) Williams, E. I., *Analyst* **89**, 289 (1964).
(64A) Wurziger, J., Gebauer, W., *Brot. Gebaeck* **16**, 209 (1962); *Anal. Abstr.* **10**, 4417 (1963).
(65A) Zweig, G., "Analytical Methods for Pesticides, Plant Growth Regulators, and Food Additives," Vol. 1, Academic Press, New York, 1963.

Adulteration, Contamination, and Decomposition

(1B) Abbott, D. C., *Analyst* **88,** 240 (1963).
(2B) Acker, L., Greve, H., Beutler, H. O., *Deut. Lebensm.-Rundschau* **59,** 231 (1963).
(3B) Andrellos, P. J., Reid, G. R., *J. Assoc. Offic. Agr. Chem.* **47,** 801 (1964).
(4B) Andrews, R. E. S., Cooper, P. J., *Analyst* **89,** 272 (1964).
(5B) Apling, E. C., Kent-Jones, D. W., Amos, A. J., *J. Sci. Food Agr.* **13,** 516 (1962).
(6B) Bautista, G. M., Linko, P., *Cereal Chem.* **39,** 455 (1962).
(7B) Benk, E., Wolff, I., Treiber, H., *Deut. Lebensm.-Rundschau.* **59,** 39 (1963); *Anal. Abstr.* **10,** 5401 (1963).
(8B) Benk, E., *Riechstoffe Aromen* **12,** 205 (1962); *Anal. Abstr.* **10,** 365 (1963).
(9B) Broadbent, J. H., Cornelius, J. A., Shone, G., *Analyst* **88,** 214 (1963).
(10B) Burger, K., *Z. Anal. Chem.* **196,** 15, 22 (1963).
(11B) Buttery, R. G., Teranishi, R., *J. Agr. Food Chem.* **11,** 504 (1963).
(12B) Cleverley, B., Dawes, S. N., *New Zealand J. Sci.* **6,** 555 (1963).
(13B) Coomes, T. J., Sanders, J. C., *Analyst* **88,** 209 (1963).
(14B) Cox, P. L., Heotis, J. P., *J. Agr. Food Chem.* **10,** 402 (1962).
(15B) Cox, W. S., *J. Assoc. Offic. Agr. Chem.* **45,** 655 (1962).
(16B) Crump, G. B., *Analyst* **88,** 456 (1963).
(17B) Czaja, A. T., *Z. Lebensm.-Untersuch.-Forsch.* **118,** 32 (1962).
(18B) De Francesco, F., Margheri, G., *Boll. Lab. Chim. Provinciali* (*Bologna*) **13,** 466 (1962); *Anal. Abstr.* **10,** 2896 (1963).
(19B) Dolezil, M., Kopanica, M., *Chemist-Analyst* **52,** 76 (1963).
(20B) Donelson, J. R., Yamazaki, W. T., *Cereal Chem.* **39,** 460 (1962).
(21B) Dorier, P., Verelle, L., *Ann. Fals. Expert. Chim.* **55,** 177 (1962); *Anal. Abstr.* **10,** 804 (1963).
(22B) Dvorak, J., Bryndac, J., Hronikova, M., *Prumysl Potravin* **13,** 428 (1962); *Anal. Abstr.* **10,** 1576 (1963).
(23B) Farn, G., Smith, D. M., *J. Assoc. Offic. Agr. Chem.* **46,** 522 (1963).
(24B) Gallardo, I., Sameh, I., *Rev. Arg. Grasas Aceites* **3,** 105 (1962); *Anal. Abstr.* **10,** 1591 (1963).
(25B) Gamoty, L., Maljournal, –., *Ann. Fals. Expert. Chim.* **55,** 217 (1962); *Anal. Abstr.* **10,** 820 (1963).
(26B) Genest, C., Smith, D. M., *J. Assoc. Offic. Agr. Chemists* **46,** 817 (1963).
(27B) Goded y Mur, A., *Rev. Univ. Ind. Santander* **5,** 477 (1963).
(28B) Hennig, W., *Deut. Lebensm.-Rundschau* **58,** 251 (1962); *Anal. Abstr.* **10,** 2474 (1963).
(29B) *Ibid.*, p. 363; *Anal. Abstr.* **10,** 4386 (1963).
(30B) Higby, W. K., *Food Technol.* **17,** 331 (1963).
(31B) Hoseney, R. C., Finney, K. F., Anal. Chem. **36,** 2145 (1964).
(32B) Hurlock, E. T., *Food Manuf.* **38,** 367 (1963).
(33B) Jaquet, J., Charton, F., *Compt. Rend. Acad. Agr. France* **49,** 373 (1963).
(34B) Janicek, G., Pokorny, J., Pokorna, V., *Ind. Aliment. Agr.* (*Paris*) **79,** 183 (1962); *Anal. Abstr.* **10,** 5404 (1963).
(35B) Kaiser, D. G., Anal. Chem. **35,** 552 (1963).
(36B) Koning de, A. J., Silk, M. H., *J. Am. Oil Chem. Soc.* **40,** 165 (1963).
(37B) Kuksis, A., McCarthy, M. J., *Ibid.*, **41,** 17 (1964).
(38B) Kumar, R., *Ibid.*, **40,** 80 (1963).
(39B) Kwon, T.-W., *Dissertation Abstr.* **24,** 4623 (1964).
(40B) Kwon, T.-W., Watts, B. M., *J. Food Sci.* **28,** 627 (1963).
(41B) Luck, H., Kohn, R., *Fette, Seifen, Anstrichmittel* **65,** 314 (1963).
(42B) Markuze, Z., *Roczniki Panstwowego Zakladu Hig. Warsaw* **13,** 255 (1962); *Anal. Abstr.* **10,** 1197 (1963).
(43B) Martinez Moreno, J. M., Ruiz Cruz, J., *Grasas Aceites* (*Seville, Spain*) **14,** 257 (1963).
(44B) Mei, H. M., Clegg, L. F. L., *Nature* **196,** 691 (1962).
(45B) Maturi, V. F., Garlock, E., *J. Assoc. Offic. Agr. Chem.* **47,** 382 (1964).
(46B) Mikhlin, E. D., Gurvich, A. I., Chukaeva, V. N., *Zh. Khim.* **8,** 20N222 (1962); *Anal. Abstr.* **10,** 2496 (1963).
(47B) Nabney, J., Nesbitt, B. F., *Nature* **203,** 862 (1964).
(48B) Nesheim, S., *J. Assoc. Offic. Agr. Chem.* **47,** 1010 (1964).
(49B) Nesheim, S., Banes, D., Stoloff, L., Campbell, A. D., *Ibid.*, **47,** 586 (1964).
(50B) Neumann, K., *Deut. Lebensm.-Rundschau* **58,** 360 (1962); *Anal. Abstr.* **10,** 4411 (1963).
(51B) Nikiforova, V. N., Evstigneev, V. B., *Khlebopekar. i. Konditer. Prom.* **1961,** 14; *Zh. Khim.* **8,** 12N38 (1962); *Anal. Abstr.* **10,** 1182 (1963).
(52B) Pal, R. N., *Indian J. Dairy Sci.* **16,** 92 (1963).
(53B) Parks, O. W., Schwartz, D. P., Keeney, M., *Nature* **202,** 185 (1964).
(54B) Pearson, D., *Lab. Pract.* **11,** 538 (1962); *Anal. Abstr.* **10,** 1188 (1963).
(55B) Peereboom, J. W. C., Beekes, H. W., *J. Chromatog.* **9,** 316 (1962).
(56B) Pitter, P., *Chem. Ind.* (*London*) **1962,** 1832; *Anal. Abstr.* **10,** 3339 (1963).
(57B) Pokorny, J., Smisek, J., Kakac, B., Janicek, G., *Sb. Vysoke Skoly Chem.-Technol. Praze, Oddil Fak. Potravinareske Technol.* **4,** 313 (1960) [1962]; *Anal. Abstr.* **10,** 2003 (1963).
(58B) Ponder, C., *J. Assoc. Offic. Agr. Chem.* **45,** 664 (1962).
(59B) Radler, F., Grncarevic, M. V., *J. Agr. Food Chem.* **12,** 266 (1964).
(60B) Ramsey, C. B., Kemp, J. D., Grainger, R. B., *Food Technol.* **18,** 105 (1964).
(61B) Recourt, J. H., Beerthuis, R. K., *Fette, Seifen, Anstrichmittel* **65,** 619 (1963).
(62B) Rego, del C., M., Garcia-Olmedo, R., *Anales Bromatol.* (*Madrid*) **15,** 53 (1963).
(63B) Renault, J., Giraud, J. G., *Ann. Fals. Expert. Chim.* **56,** 105 (1963).
(64B) Rosser, C. M., *Modern Packaging* **37,** 131 (1963).
(65B) Rost, H. E., *Fette, Seifen, Anstrichmittel* **64,** 427 (1962).
(66B) Rost, H. E., *Ibid.*, **65,** 463 (1963).
(67B) Rotsch, A., *Brot. Gebaeck* **17,** 129 (1963).
(68B) Rzhekhin, V. P., Pogonkina, N. I., Voronova, E. K., Solov'eva, I. A., *Zh. Khim.* **8,** 3N348 (1962); *Anal. Abstr.* **10,** 822 (1963).
(69B) Schiweck, H., *Zucker* **16,** 80 (1963).
(70B) Seidemann, J., *Deut. Lebensm.-Rundschau* **58,** 168 (1962); *Anal. Abstr.* **10,** 341 (1963).
(71B) Selzer, G. B., Banes, D., *J. Assoc. Offic. Agr. Chem.* **46,** 703 (1963).
(72B) Stein, R. A., Slawson, V., Anal. Chem. **35,** 1008 (1963).
(73B) Taufel, K., Sehrt, I., Franzke, C., *Z. Lebensm.-Untersuch.-Forsch.* **116,** 503 (1962).
(74B) Tollenaar, F. D., van Gend, H., Hoekman, H., *Fette, Seifen, Anstrichmittel* **64,** 303 (1962).
(75B) Tramer, J., Fowler, G. G., *J. Sci. Food Agr.* **15,** 522 (1964).
(76B) Underwood, J. C., *J. Assoc. Offic. Agr. Chem.* **47,** 548 (1964).
(77B) Valentinis, G., *Boll. Lab. Chim. Provinciali* (*Bologna*) **13,** 336 (1962); *Anal. Abstr.* **10,** 1214 (1963).
(78B) Vioque, A., Vioque, E., *Grasas Aceites* (*Seville, Spain*) **13,** 203 (1962); *Anal. Abstr.* **10,** 3460 (1963).
(79B) *Ibid.*, p. 211; *Anal. Abstr.* **10,** 3459 (1963).
(80B) Vogel, J., Deshusses, J., *Mitt Gebiete Lebensm. Hyg. Bern* **53,** 175 (1962); *Anal. Abstr.* **9,** 5447 (1962).
(81B) *Ibid.*, p. 179; *Anal. Abstr.* **9,** 5433 (1962).
(82B) Waller, H., *Monatsh. Brauerie* **15,** 58 (1962); *Anal. Abstr.* **10,** 2033 (1963).
(83B) Weber, W. J., Jr., Morris, J. C., Stumm, W., Anal. Chem. **34,** 1844 (1962).
(84B) West, D. B., Lautenbach, A. F., Brumsted, D. D., *Am. Soc. Brewing Chemists Proc.* **1963,** 194.
(85B) Willits, C. O., Costilow, R. N., Underwood, J. C., *J. Assoc. Offic. Agr. Chem.* **46,** 331 (1963).
(86B) Wurziger, J., *Deut. Lebensm.-Rundschau* **58,** 357 (1962); *Anal. Abstr.* **10,** 4391 (1963).
(87B) Wurziger, J., Junker, M., *Ibid.*, **59,** 133 (1963).

Carbohydrates

(1C) Aimukhamedova, G. B., Shelukhina, N. P., Maslinkovskaya, Z. A., *Zh. Khim* **8,** 6N336 (1962) (abstract); *Anal. Abstr.* **10,** 1226 (1963).
(2C) Anderson, D. M. W., Garbutt, S., *Anal. Chim. Acta* **29,** 31 (1963).
(3C) Anderson, D. M. W., Garbutt, S., Zaidi, S. S. H., *Ibid.*, **29,** 39 (1963).
(4C) Arkhipovich, N. A., Voloshanenko, G. P., *Sakharn. Prom.* **1963,** (3) 21.
(5C) Bares, M., *Prumysl Potravin* **14,** 161 (1963).
(6C) Bartholomae, F., *Zucker* **16,** 144 (1963).
(7C) Bayer, E., Widder, R., Anal. Chem. **36,** 1452 (1964).
(8C) Bentley, R., Sweeley, C. C., Makita, M., Wells, J. W., *Biochem. Biophys. Res. Commun.* **11,** 14 (1963).
(9C) Bichsel, S. E., Johnson, J. R., *J. Am. Soc. Sugar Beet Technol.* **12,** 449 (1963).
(10C) Binkley, W. W., Roberts, E. J., Jackson, J. T., Martin, L. F., *Intern. Sugar J.* **65,** 169 (1963).
(11C) Bock, W., *Ernahrungsforschung* **7,** 561 (1963).
(12C) Bourne, E. J., Lees, E. M., Weigel, H., *J. Chromatog.* **11,** 253 (1963).
(13C) Cuzzoni, M. T., Lissi, T. P., *Farmaco* (*Pavia*) *Ed. Pract.* **16,** 416 (1961); *C.A.* **56,** 7564*a* (1962).
(14C) Czaja, A. T., *Z. Lebensm.-Untersuch.-Forsch.* **117,** 499 (1962).
(15C) Davidek, J., Hampl, J., Vejrova, M., *Getreide Mehl* **13,** 73 (1963).
(16C) Deulin, V. I., Nazarov, V. I., *Zh. Anal. Khim.* **18,** 1016 (1963).
(17C) Fleming, I. D., Pegler, H. F., *Analyst* **88,** 967 (1963).
(18C) Gaponenkov, T. K., Protsenko, Z. I., *Sakharn. Prom.* **1962,** 27; *Zh. Khim.* **19R,** 9R295 (1963); *Anal. Abstr.* **11,** 788 (1964).
(19C) Gee, M., *J. Chromatog.* **9,** 278 (1962).
(20C) Garoglio, P.-G., *Ann. Fals. Expert. Chim.* **56,** 243 (1963).
(21C) Graham, H. D., Mitchell, G., *J. Food Sci.* **28,** 546 (1963).
(22C) Grau, R., Schweiger, A., *Z. Lebensm.-Untersuch.-Forsch.* **119,** 210 (1963).
(23C) Gross, D., Gardiner, F. J., Butters, R. W., *Intern. Sugar J.* **64,** 69 (1962).

(24C) Hadorn, H., *Mitt. Gebiete Lebensm. Hyg.* (*Bern*) **54,** 179 (1963).
(25C) Hall, R. H., *Anal. Biochem.* **4,** 395 (1962).
(26C) Harada, T., *Biochim. Biophys. Acta* **63,** 334 (1962).
(27C) Hay, G. W., Lewis, B. A., Smith, F., *J. Chromatog.* **11,** 479 (1963).
(28C) Hernandez, J. A. L., *Intern. Sugar J.* **65,** 46 (1963); Reprinted from *Arch. Farm. Bioquim., Tucuman.* **9,** 125 (1961).
(29C) Hill, S., Orchard, W. J. H., *Intern. Sugar J.* **64,** 100 (1962).
(30C) Hollo, J., Laszlo, E., Gantner, G. S., Hoschke, A., Szejtli, J., *Nahrung* **7,** 33 (1963); *Anal. Abstr.* **10,** 5381 (1963).
(31C) Hwang, M.-W., *J. Chinese Chem. Soc., Taiwan* **9,** 298 (1962); *Anal. Abstr.* **10,** 4726 (1963).
(32C) Jermyn, M. A., *Australian J. Biol. Sci.* **15,** 787 (1962); *Anal. Abstr.* **10,** 3381 (1963).
(33C) Kato, K., Matsui, S., Sawae, S., *Proc. Res. Soc. Japan Sugar Refineries Technol.* **11,** 20 (1962).
(34C) Kolbach, P., Rinke, W., *Monatsh. Brauerie* **15,** 89 (1962); *Anal. Abstr.* **10,** 4912 (1963).
(35C) Mansford, K. R. L., *Analyst* **88,** 649 (1963).
(36C) Mansford, K. R. L., Opie, R. K., *Ibid.*, **88,** 646 (1963).
(37C) Momose, T., Mukai, Y., *J. Pharm. Soc. Japan* **81,** 227 (1961); *Anal. Abstr.* **9,** 4930 (1962).
(38C) Momose, T., Mukai, Y., Kawabe, S., Suzuki, J., Yamamoto, K., *Japan Analyst* **11,** 959 (1962).
(39C) Morris, W. W., Jr., Salmon, W., Anal. Chem. **35,** 600 (1963).
(40C) Nishio, T., *Proc. Res. Soc. Japan Sugar Refineries Technol.* **13,** 17 (1963).
(41C) Pardue, H. L., Anal. Chem. **36,** 1110 (1964).
(42C) Percheron, F., *Compt. Rend.* **255,** 2521 (1963).
(43C) Rasper, V., *Listy Cukrovar* **78,** 277 (1962); *Anal. Abstr.* **10,** 4381 (1963).
(44C) Reeve, R. M., *J. Food Sci.* **28,** 198 (1963).
(45C) Reintjes, M., Musco, D. D., Joseph, G. H., *Ibid.*, **27,** 441 (1962).
(46C) Richter, M., *Staerke* **14,** 337 (1962).
(47C) Richter, M., Stroh, H.-H., *Ibid.*, **14,** 415 (1962).
(48C) Schild, E., Weyh, H., *Brauwissenschaft* **16,** 72 (1963).
(49C) Schiweck, H., *Zucker* **16,** 170 (1963).
(50C) Schweiger, A., *J. Chromatog.* **9,** 374 (1962).
(51C) Scott, D., U. S. Pat. **3,104,209** (1960)
(52C) Seidemann, J., *Staerke* **14,** 348 (1962).
(53C) Stoll, S., Prat, Y., *Ann. Fals. Expert. Chim.* **55,** 159 (1962); *Anal. Abstr.* **10,** 810 (1963).
(54C) Stoloff, L., Blethen, J., Anal. Chem. **36,** 2192 (1964).
(55C) Sudheendranath, C. S., Rao, D. S., Anantakrishnan, C. P., *Indian J. Dairy Sci.* **16,** 41 (1963).
(56C) Sullivan, J. W., Johnson, J. A., *Cereal Chem.* **41,** 73 (1964).
(57C) Syamananda, R., Staples, R. C., Block, R. J., *Contrib. Boyce Thompson Inst.* **21,** 363 (1962).
(58C) Taki, M., *Agr. Biol. Chem.* (*Tokyo*) **26,** 1 (1962); *Anal. Abstr.* **9,** 4917 (1962).
(59C) Taufel, K., Grunert, M., Grunert, S., *Z. Lebensm.-Untersuch.-Forsch.* **119,** 1 (1962).
(60C) Ticha, B., Friml, M., *Listy Cukrovar* **79,** 57 (1963); *Anal. Abstr.* **11,** 1477 (1964).
(61C) Towne, J. C., Spikner, J. E., Anal. Chem. **35,** 211 (1963).
(62C) Tu, C. C., Okamoto, R. H., *J. Agr. Food Chem.* **11,** 330 (1963).
(63C) *Ibid.*, p. 331.
(64C) Ulmann, M., *Ernaehrungsforschung* **7,** 113 (1962); *C.A.* **57,** 3833*h* (1962).
(65C) van der Bij, J. R., Vogel, W. F., *Staerke* **14,** 113 (1962).
(66C) van Duuren, A. J., *Mededel. Inst. Rationele Suikerprod.* **33** (1963).
(67C) Verhaart, M. L. A., de Visser, N. H. M., *Z. Zuckerind.* **12,** 657 (1962); *Anal. Abstr.* **10,** 4382 (1963).
(68C) Wagner, W., *Anal. Chim. Acta* **29,** 182 (1963).
(69C) *Ibid.*, p. 227.
(70C) Wassermann, L., Hanus, H., *Naturwissenschaften* **50,** 351 (1963).
(71C) Weswig, P. H., Foote, W. H., Drusts, G. R., *Cereal Chem.* **40,** 169 (1963).
(72C) Wolfrom, M. L., Patin, D. L., *J. Agr. Food Chem.* **12,** 376 (1964).
(73C) Yamauchi, T., Takebe, T., *Proc. Res. Soc. Japan Sugar Refineries Technol.* **12,** 1 (1963).
(74C) Zelenka, S., Svoboda, A., Kopriva, B., Hampl, J., *Sb. Vysoke Skoly Chem. Technol. Praze. Oddil Fak. Potravinareske Technol.* **1958** [1961], 315; *Anal. Abstr.* **9,** 4918 (1962).

Color

(1D) Barrett, J. F., Ryan, A. J., *Nature* **199,** 372 (1963).
(2D) Bastianutti, J., *Boll. Lab. Chim. Provinciali* (*Bologna*) **13,** 179 (1962); *Anal. Abstr.* **9,** 5438 (1962).
(3D) Bastianutti, J., *Ibid.*, **14,** 16 (1963); *Anal. Abstr.* **10,** 5406 (1963).
(4D) Blessin, C. W., *Cereal Chem.* **39,** 236 (1962).
(5D) Burton, H. S., McWeeny, D. J., Biltcliffe, D. O., *J. Food Sci.* **28,** 631 (1963).
(6D) Cortes, I. M., Diez de Bethencourt, C., *Chim. Anal.* (*Paris*) **44,** 527 (1962); *Anal. Abstr.* **10,** 4408 (1963).
(7D) Curl, A. L., *J. Food Sci.* **27,** 537 (1962).
(8D) Curl, A. L., *Ibid.*, **28,** 623 (1963).
(9D) Diemair, W., Postel, W., Sengewald, H., *Z. Lebensm.-Untersuch.-Forsch.* **120,** 173 (1963).
(10D) Dutt, M. C., *Analyst* **89,** 142 (1964).
(11D) Fujii, S., Kamikura, M., *J. Food Hyg. Soc. Japan* **4,** 96 (1963).
(12D) Gaspovic, J., Gemzova-Taborska, I., *Collection Czech. Chem. Commun.* **27,** 2996 (1962); *Chem. Ind.* (*London*) **1963,** 319.
(13D) Goren, R., Monselise, S. P., *J. Assoc. Offic. Agr. Chem.* **47,** 677 (1964).
(14D) Holland, G. J., *Analyst* **87,** 797 (1962).
(15D) Isleib, D. R., *Am. Potato J.* **40,** 58 (1963).
(16D) Koch, L., *J. Assoc. Offic. Agr. Chem.* **46,** 344 (1963).
(17D) Korablev, A. I., Trofimenko, B. S., *Zh. Khim.* **8,** 12N264 (1962); *Anal. Abstr.* **10,** 1210 (1963).
(18D) Link, W. B., Heine, K. S., Jr., Jones, J. H., Wattlington, P., *J. Assoc. Offic. Agr. Chem.* **47,** 391 (1964).
(19D) McFarlane, W. D., Sword, P. T., *J. Inst. Brewing* **68,** 344 (1962); *Anal. Abstr.* **10,** 1207 (1963).
(20D) McFarlane, W. D., Vader, M. J., *Ibid.*, **68,** 254 (1962); *Anal. Abstr.* **10,** 367 (1963).
(21D) Mitra, S. N., Sengupta, P. N., Roy, B. R., Mathew, T. V., Mallik, A. K., *J. Inst. Chem., India* **33,** 280 (1961); *Anal. Abstr.* **9,** 5437 (1962).
(22D) Muntoni, F., Tassi-Micco, C., *R. C. Ist. Sup. Sanit.* **25,** 567, (1962); *Anal. Abstr.* **10,** 2484 (1963).
(23D) Naudet, M., Sambuc, E., *Fette, Seifen, Anstrichmittel* **64,** 441 (1962).
(24D) Nury, F. S., Brekke, J. E., *J. Food Sci.* **28,** 95 (1963).
(25D) Pla-Delfina, J. M., *J. Soc. Cosmet. Chem.* **13,** 214 (1962); *Anal. Abstr.* **10,** 1873 (1963).
(26D) Powrie, W. D., Fennema, O., *J. Food Sci.* **28,** 214 (1963).
(27D) Reed, R. H., Heinekey, D. M., *Analyst* **87,** 391 (1962).
(28D) Rispoli, G., di Giacomo, A., *Boll. Lab. Chim. Provinciali* (*Bologna*) **13,** 587 (1962); *Anal. Abstr.* **10,** 3907 (1963).
(29D) Rother, H., *Riechstoffe Aromen* **12,** 87 (1962); *Anal. Abstr.* **10,** 364 (1963).
(30D) Sadini, V., *J. Chromatog.* **10,** D8 (1963).
(31D) Sancho, J., Serna, A., Navarro, F., *Anales Bromatol.* (*Madrid*) **14,** 35 (1962); *Anal. Abstr.* **9,** 4933 (1962).
(32D) Simonetti, E. R., *Anales Fac. Quim. Farm., Univ. Chile* **13,** 118 (1961); *C.A.* **58,** 4965*g* (1963).
(33D) Snyder, H. E., *J. Food Sci.* **29,** 535 (1964).
(34D) Somaatmadja, D., Powers, J. J., *Ibid.*, **28,** 617 (1963).
(35D) Stanley, R. L., Kirk, P. L., *J. Agr. Food Chem.* **11,** 492 (1963).
(36D) Stinson, E. E., Willits, C. O., *J. Assoc. Offic. Agr. Chem.* **46,** 329 (1963).
(37D) Szokolay, A., *Z. Lebensm.-Untersuch.-Forsch.* **120,** 295 (1963).
(38D) Tanner, H., Rentschler, H., Senn, G., *Mitt.* (*Klosterneuburg*), *Ser. A, Rebe Wein* **13,** 156 (1963); *Anal. Abstr.* **11,** 1507 (1964).
(39D) Trace Materials (Colours) Committee, First Rept., British Food Manufacturing Industries Research Assoc., *Analyst* **88,** 864 (1963).
(40D) Valcher, S., *Boll. Lab. Chim. Provinciali* (*Bologna*) **13,** 530 (1962); *Anal. Abstr.* **10,** 2915 (1963).
(41D) Villanua, L., Carbolido, A., Garcia Olmedo, R., Valdehita, M. T., *Anales Bromatol.* (*Madrid*) **14,** 51 (1962); *Anal. Abstr.* **9,** 4934 (1962).
(42D) Weith, L., *Brauwelt* **1963,** 1309.
(43D) White, R. C., Jones, I. D., Gibbs, E., *J. Food Sci.* **28,** 431 (1963).
(44D) Wilson, J. R., Nutting, M.-D., Anal. Chem. **35,** 144 (1963).
(45D) Yanuka, B., Shalon, Y., Weissenberg, E., Nir-Grosfeld, I., *Analyst* **87,** 791 (1962).
(46D) Yanuka, Y., Shalon, Y., Weissenberg, E., Nir-Grosfeld, I., *Ibid.*, **88,** 872 (1963).

Enzymes

(1E) Auricchio, F., Bruni, C. B., *Biochem. Zeitschrift* **340,** 321 (1964).
(2E) Avakyan, A. O., *Vopr. Pitaniya* **22,** 82 (1963).
(3E) Dawson, G. V., Olson, W. J., *Am. Soc. Brewing Chemists Proc.* **1962,** 54.
(4E) de Koning, P. J., Jenness, R., Wijnand, H. P., *Neth. Milk Dairy J.* **17,** 352 (1963).
(5E) Feller, K., Richter, M., Ruttloff, C., *Ernahrungsforschung* **7,** 575 (1963).
(6E) Fosnot, R. H., Haman, R. W., *Am. Soc. Brewing Chemists Proc.* **1962,** 70.
(7E) Guibault, G. G., Kramer, D. N., Anal. Chem. **36,** 409 (1964).
(8E) Guilbault, G. G., Tyson, B. C., Jr., Kramer, D. N., Cannon, P. L., Jr., *Ibid.*, **35,** 582 (1963).
(9E) Hadorn, H., Zurcher, K., *Mitt. Gebiete Lebensm. Hyg.* **53,** 6 (1962); *Anal. Abstr.* **10,** 801 (1963).
(10E) Heimann, W., Andler, S., *Z. Lebensm.-Untersuch.-Forsch.* **117,** 121 (1962).

(11E) *Ibid.*, p. 203.
(12E) Kramer, D. N., Guilbault, G. G., ANAL. CHEM. **35**, 588 (1963).
(13E) Moreau, J. R., Jankus, E. E., *Food Technol.* **17**, 1046 (1963).
(14E) Pomeranz, Y., *Ibid.*, **18**(5), 96 (1964).
(15E) Pomeranz, Y., Miller, B. S., *J. Agr. Food Chem.* **11**, 19 (1963).
(16E) Pomeranz, Y., Robinson, R. J., Shellenberger, J. A., *Enzymologia* **25**, 157 (1963); *Anal. Abstr.* **10**, 4900 (1963).
(17E) Purr, A., Morcinek, H., Springer, R., *Z. Lebensm.-Untersuch.-Forsch.* **123**, 341 (1963).
(18E) Rouse, A. H., *J. Food Sci.* **27**, 419 (1962).
(19E) Saletan, L. T., Gantz, C. S., Gray, P. P., *Am. Soc. Brewing Chemists Proc.* **1963**, 74.
(20E) Schwarz, G., Ludwig, J., Fricker, A., *Nahrung* **6**, 688 (1962); *Anal. Abstr.* **10**, 2906 (1963).
(21E) Wieland, T., Determann, H., *Experientia* **18**, 431 (1962); *Anal. Abstr.* **10**, 3509 (1963).
(22E) Wilder, C. J., *J. Food Sci.* **27**, 567 (1962).

Fats, Oils, and Fatty Acids

(1F) Ackman, R. G., *Nature* **194**, 970 (1962).
(2F) Ackman, R. G., Burgher, R. D., ANAL. CHEM. **35**, 647 (1963).
(3F) Ackman, R. G., Burgher, R. D., *J. Chromatog.* **11**, 185 (1963).
(4F) Andre, E., Henry, J., *Compt. Rend.* **257**, 143 (1963).
(5F) Babaev, V. I., Dan'shina, N. M., *Maslob.-Zhir. Prom.* **27**, 30 (1961); *Zh. Khim.* **8**, 17N297 (1962); *Anal. Abstr.* **10**, 1217 (1963).
(6F) Baker, D., *J. Am. Oil Chemists Soc.* **41**, 21 (1964).
(7F) Bergel'son, L. D., Dyatlovitskaya, E. V., Voronkova, V. V., *Izv. Akad. Nauk SSSR, Otd. Khim. Nauk* **1963**, 954; *Anal. Abstr.* **11**, 2194 (1964).
(8F) Buoncristiani, D., Taponeco, G., Salvadorini, R., *Boll. Lab. Chim. Provinciali (Bologna)* **13**, 218 (1962); *Anal. Abstr.* **9**, 5460 (1962).
(9F) Chernobai, A. V., Lavygin, I. A., Klevanskii, M. L., *Maslob.-Zhir. Prom.* **1962**, 10; *Zh. Khim.* **19R**, IR330 (1963); *Anal. Abstr.* **10**, 3458 (1963).
(10F) Chowdhury, D. K., Muckherjea, R. N., *Nahrung* **7**, 111 (1963); *Anal. Abstr.* **10**, 5407 (1963).
(11F) Craig, B. M., Tulloch, A. P., Murty, N. L., *J. Am. Chem. Soc.* **40**, 61 (1963).
(12F) DeFreitas, A. S. W., *Can. J. Biochem.* **42**, 195 (1964).
(13F) de Langen, H., *New Zealand J. Agr. Res.* **6**, 452 (1963).
(14F) De Vries, B., *J. Am. Oil Chemists Soc.* **40**, 184 (1963).
(15F) de Vries, M. J., *S. African J. Agr. Sci.* **5**, 395 (1962); *Anal. Abstr.* **10**, 2912 (1963).
(16F) de Vries, B., Jurriens, G., *Fette, Seifen, Anstrichmittel* **65**, 725 (1963).
(17F) Ferren, W. P., Morse, R. E., *Food Technol.* **17**, 1066 (1963).
(18F) Fett, H. M., *J. Am. Oil Chemists Soc.* **40**, 81 (1963).
(19F) Firestone, D., *Ibid.*, **40**, 247 (1963).
(20F) Frankel, E. N., McConnell, D. G., Evans, C. D., *Ibid.*, **39**, 297 (1962).
(21F) Fukui, K., Nagatomi, H., Murata, S., *Japan Analyst* **11**, 432 (1962).
(22F) Gehrke, C. W., Goerlitz, D. F., ANAL. CHEM. **35**, 76 (1963).
(23F) Grieco, D., *Olearia* **16**, 11 (1962).
(24F) Hautfenne, A., Jacqmain, D., *Rev. Ferment. Ind. Aliment.* **17**, 107 (1962); *Anal. Abstr.* **10**, 2497 (1963).
(25F) Horrocks, L. A., Cornwell, D. G., *J. Lipid Res.* **3**, 165 (1962).
(26F) Jacobsen, G. A., Kirkpatrick, J. A., Goff, H. E., Jr., *J. Am. Oil Chemists Soc.* **41**, 124 (1964).
(27F) James, A. T., *Analyst* **88**, 572 (1963).
(28F) Johnson, L. F., Shoolery, J. N., ANAL. CHEM. **34**, 1136 (1962).
(29F) Kaufmann, H. P., Grothues, B., *Fette, Seifen, Anstrichmittel* **64**, 805 (1962).
(30F) Kaufmann, H. P., Ko, Y. S., *Ibid.*, **64**, 434 (1962).
(31F) Kaufmann, H. P., Schmulling, E., *Ibid.*, **64**, 319 (1962).
(32F) Kaufmann, H. P., Sen Gupta, A. K., *Ibid.*, **65**, 529 (1963).
(33F) Kaufmann, H. P., Sen Gupta, A. K., *Ibid.*, **66**, 461 (1964).
(34F) Kaufmann, H. P. K., Wessels, H., *Ibid.*, **66**, 81 (1964).
(35F) Keith, R. W., Day, E. A., *J. Am. Oil Chemists Soc.* **40**, 121 (1963).
(36F) Kuemmel, D. F., ANAL. CHEM. **34**, 1003 (1962).
(37F) Kuksis, A., McCarthy, M. J., *Can. J. Biochem. Physiol.* **40**, 679 (1962); *Anal. Abstr.* **9**, 5459 (1962).
(38F) Kuksis, A., McCarthy, M. J., Beveridge, J. M. R., *J. Am. Oil Chemists Soc.* **40**, 530 (1963).
(39F) Kwapniewski, Z., Sliwiok, J., *Chem. Anal., Warsaw* **8**, 433 (1963).
(40F) Litchfield, C., Isbell, A. F., Reiser, R., *J. Am. Oil Chemists Soc.* **39**, 330 (1962).
(41F) Luck, H., Saumweber, W., Kohn, R., *Z. Lebensm.-Untersuch.-Forsch.* **120**, 286 (1963).
(42F) McCarthy, R. D., Duthie, A. H., *J. Lipid Res.* **3**, 117 (1962).
(43F) Martinenghi, I. G. B., *Olearia* **17**, 47 (1963).
(44F) Mason, M. E., Eager, M. E., Waller, G. R., ANAL. CHEM. **36**, 587 (1963).
(45F) Metcalfe, L. D., *J. Gas Chromatog.* **1**, 7 (1963).
(46F) Metcalfe, L. D., Germanos, G. A., Schmidt, A. A., *Ibid.*, **1**, 32 (1963).
(47F) Morgantini, M., *Boll. Lab. Chim. Provinciali (Bologna)* **13**, 545 (1962); *Anal. Abstr.* **10**, 3992 (1963).
(48F) Napier, E. A., Jr., ANAL. CHEM. **35**, 1294 (1963).
(49F) Naudet, M., *Bull. Soc. Chim. France* **1962**, 664; *Anal. Abstr.* **9**, 5452 (1962).
(50F) Noda, M., Hirayama, O., *J. Japan Oil Chemists Soc.* **10**, 24 (1961); *Anal. Abstr.* **9**, 4943 (1962).
(51F) O'Connor, R. T., *J. Am. Oil Chemists Soc.* **41**, 4 (1964).
(52F) Parks, O. W., Keeney, M., Schwartz, D. P., *J. Dairy Sci.* **44**, 1940 (1961).
(53F) Pascaud, M., *J. Chromatog.* **10**, 125 (1963).
(54F) Pilz, W., *Z. Anal. Chem.* **193**, 338 (1963).
(55F) Pohle, W. D., Gregory, R. L., Taylor, J. R., *J. Am. Oil Chemists Soc.* **39**, 226 (1962).
(56F) Pohle, W. D., Gregory, R. L., Van Giessen, B., *Ibid.*, **40**, 603 (1963).
(57F) Pokorny, J., Cmolik, J., Janicek. G., *Sb. Vysoke Skoly Chem. Technol, Praze Oddil Fak. Potravinareske Technol.* **4**, 197 (1960) [1962]; *Anal. Abstr.* **9**, 4947 (1962).
(58F) Pokorny, J., Cmolik, J., Janicek, G., *Ibid.*, **4**, Part 2, 211 (1960).
(59F) Pokorny, J., *Riv. Ital. Sostanze Grasse* **38**, 484 (1961); *Anal. Abstr.* **9**, 4945 (1962).
(60F) Ryhage, R., ANAL. CHEM. **36**, 759 (1964).
(61F) Scholfield, C. R., Jones, E. P., Butterfield, R. O., Dutton, H. J., *Ibid.*, **35**, 1588 (1963).
(62F) Schwartz, D. P., Haller, H. S., Keeney, M., *Ibid.*, **35**, 2191 (1963).
(63F) Sesbes, T. A., *Chim. Anal. (Paris)* **45**, 351 (1963).
(64F) Sgoutas, D., Kummerow, F. A., *J. Am. Oil Chemists Soc.* **40**, 138 (1963).
(65F) Skidmore, W. D., Entenman, C., *J. Lipid Res.* **3**, 356 (1962).
(66F) Subbaram, M. R., Youngs, C. G., *J. Am. Oil Chemists Soc.* **41**, 445 (1964).
(67F) Sully, B. D., *Analyst* **87**, 940 (1962).
(68F) Taufel, K., Schmidt, K., Franzke, C., *Fette, Seifen, Anstrichmittel* **64**, 957 (1962).
(69F) Taylor, J. R., Pohle, W. D., Gregory, R. L., *J. Am. Oil Chemists Soc.* **41**, 177 (1964).
(70F) Tiwari, R. D., Sharma, J. P., *Z. Anal. Chem.* **195**, 267 (1963).
(71F) Trukhacheva, A. A., Tanklaeva, R. A., *Sakharn. Prom.* **1963**, 56; *Zh. Khim.* **19R**, 24R310 (1963).
(72F) Turgel', E. O., Balukov, R. V., Boyarinova, L. A., *Zh. Khim.* **2**, 23D167 (1962); *Anal. Abstr.* **10**, 3291 (1963).
(73F) Uksila, E., Mattila, I., Roine, P., *Suomen Kemistilehti B* **36**, 84 (1963).
(74F) Valussi, S., Cofleri, G., *Riv. Ital. Sostanze Grasse* **39**, 617 (1962); *Anal. Abstr.* **10**, 4398 (1963).
(75F) Vandenheuvel, F. A., ANAL. CHEM. **36**, 1930 (1964).
(76F) Vander Wal, R. J., *J. Am. Oil Chemists Soc.* **40**, 242 (1963).
(77F) Vioque, E., Holman, R. T., *J. Am. Chem. Soc.* **39**, 63 (1962).
(78F) Vioque, E., Maza, M. P., *Grasas Aceites (Seville, Spain)* **13**, 207 (1962); *Anal. Abstr.* **10**, 3286 (1963).
(79F) Viswanathan, C. V., Meera Bai, B., *J. Chromatog.* **7**, 507 (1962).
(80F) von Ardenne, M., Steinfelder, K., Tummler, R., *Seifen-Oele-Fette-Wachse* **88**, 415 (1962); *Anal. Abstr.* **10**, 1588 (1963).
(81F) Wagner, H., Pohl, P., *Biochem. Z.* **340**, 337 (1964).
(82F) Williams, M. C., Reiser, R., *J. Am. Oil Chemists Soc.* **40**, 237 (1963).
(83F) Williamson, M. B., U. S. Patent **3,050,628** (1957).
(84F) Wolf, D. P., Dugan, L. R., Jr., *J. Am. Oil Chemists Soc.* **41**, 139 (1964).
(85F) Wren, J. J., Wojtczak, P. P., *Analyst* **89**, 122 (1964).
(86F) Yamamoto, N., Miyakawa, T., *J. Japan Oil Chemists Soc.* **10**, 200 (1961); *Anal. Abstr.* **9**, 4941 (1962).
(87F) Zajic, J., Forman, L., *Sb. Vysoke Skoly Chem.-Technol. Praze Oddil Fak. Potravinareske Technol.* **5**, 195 (1961); *Anal. Abstr.* **11**, 2390 (1964).

Flavor and Volatile Compounds

(1G) Anwar, M. H., ANAL. CHEM. **35**, 1974 (1963).
(2G) Bassette, R., Ozeris, S., Whitnah, C. H., *J. Food Sci.* **28**, 84 (1963).
(3G) Beitter, H., *Brot Geback* **17**, 132 (1963); *J. Sci. Food Agr.* **15**, 35i (1964).
(4G) Cobb, W. Y., Patton, S., Grill, H., *J. Dairy Sci.* **46**, 566 (1963).
(5G) Corbin, E. A., ANAL. CHEM. **34**, 1244 (1962).
(6G) Day, E. A., Libbey, L. M., *J. Food Sci.* **29**, 583 (1964).
(7G) Dhont, J. H., *Analyst* **89**, 71 (1964).
(8G) Dhont, J. H., *Nature* **200**, 882 (1963).
(9G) Diemair, W., Schams, F., *Z. Anal. Chem.* **189**, 149 (1962); *Anal. Abstr.* **10**, 1179 (1963).
(10G) Gold, H. J., Wilson, C. W., III, *J. Food Sci.* **28**, 484 (1963).

(11G) Hing, F. S., Weckel, K. G., *Ibid.*, **29**, 149 (1964).
(12G) Hoff, J. E., Feit, E. D., ANAL. CHEM. **36**, 1002 (1964).
(13G) Hornstein, I., Crowe, P. F., *J. Agr. Food Chem.* **11**, 147 (1963).
(14G) Hornstein, I., Crowe, P. T., *J. Gas Chromatog.* **2**, 128 (1964).
(15G) Hrdlicka, J., Kubicek, J., *Kvasny Prumysl* **9**, 35 (1963); *Anal. Abstr.* **10**, 1115 (1963).
(16G) Issenberg, P., Wick, E. L., *J. Agr. Food Chem.* **11**, 2 (1963).
(17G) Jacobson, M., Koehler, H. H., *Ibid.*, **11**, 336 (1963).
(18G) Jennings, W. G., Creveling, R. K., *J. Food Sci.* **28**, 91 (1963).
(19G) Jennings, W. G., Sevenants, M. R., *Ibid.*, **29**, 158 (1964).
(20G) Kepner, R. E., Maarse, H., Strating, J., ANAL. CHEM. **36**, 77 (1964).
(21G) Kepner, R. E., Strating, J., Weurman, C., *J. Inst. Brewing* **69**, 399 (1963).
(22G) Kiermeier, F., Jarczynski, R., *Z. Lebensm.-Untersuch.-Forsch.* **117**, 306 (1962).
(23G) Kroger, M., Patton, S., *J. Dairy Sci.* **47**, 296 (1964).
(24G) Kung, J.-F. T., Romognoli, R. J., ANAL. CHEM. **36**, 1161 (1964).
(25G) Lea, C. H., *Chem. Ind.* (*London*) **1963**, 1406.
(26G) Lento, H. G., Ford, J. A., ANAL. CHEM. **35**, 1418 (1963).
(27G) Libbey, L. M., Bills, D. D., Day, E. A., *J. Food Sci.* **28**, 329 (1963).
(28G) MacGregor, D. R., Sugisawa, H., Matthews, J. S., *Ibid.*, **29**, 448 (1964).
(29G) McCarthy, A. I., Palmer, J. K., Shaw, C. P., Anderson, E. E., *Ibid.*, **28**, 379 (1963).
(30G) McCarthy, A. I., Wyman, H., Palmer, J. K., *J. Gas Chromatog.* **2**, 121 (1964).
(31G) McFadden, W. H., Teranishi, R., Black, D. R., Day, J. C., *J. Food Sci.* **28**, 316 (1963).
(32G) Mabbitt, L. A., McKinnon, G., *J. Dairy Res.* **30**, 359 (1963).
(33G) Martin, G. E., Caggiano, G., Beck, J. E., *J. Assoc. Offic. Agr. Chem.* **46**, 297 (1962).
(34G) Martin, G. E., Caggiano, G., Schlesinger, H., *Ibid.*, **46**, 294 (1963).
(35G) Martin, G. E., Feeny, F. J., Scaringelli, F. P., *Ibid.*, **47**, 561 (1964).
(36G) Maruta, S., Suzuki, Y., *J. Chem. Soc. Japan, Ind. Chem. Sect.* **64**, 176 (1961); *Anal. Abstr.* **10**, 1586 (1963).
(37G) *Ibid.*, p. 2129; *J. Sci. Food Agr.* **14**, *i*-318 (1963).
(38G) Mason, M. E., Waller, G. R., *J. Agr. Food Chem.* **12**, 274 (1964).
(39G) Mattick, L. R., Robinson, W. G., Weirs, L. D., Barry, D. L., *Ibid.*, **11**, 334 (1963).
(40G) Mehlitz, A., Gierschner, K., *Reichstoffe Aromen* **13**, 202 (1963).
(41G) Merritt, C., Jr., Bazinet, M. L., Sullivan, J. H., Robertson, D. H., *J. Agr. Food Chem.* **11**, 152 (1963).
(42G) Merritt, C., Jr., Walsh, J. T., ANAL. CHEM. **35**, 110 (1963).
(43G) Merritt, C., Jr., Walsh, J. T., Forss, D. A., Angelini, P., Swift, S. M., *Ibid.*, **36**, 1502 (1964).
(44G) Merz, J. H., *J. Apicult Res.* **2**, 13 (1962); *C.A.* **60**, 9827*c* (1964).
(45G) Muck, G. A., Tobias, J., Whitney, R. McL., *J. Dairy Sci.* **46**, 774 (1963).
(46G) Nawar, W. W., Fagerson, I. S., *Food Technol.* **16**, 107 (Nov. 1962).
(47G) Oaks, D. M., Hartmann, H., Dimick, K. P., ANAL. CHEM. **36**, 1560 (1964).
(48G) Ockerman, H. W., Blumer, T. N., Craig, H. B., *J. Food Sci.* **29**, 123 (1964).
(49G) Owades, J. L., Jakovac, J. A., *Proc. Amer. Soc. Brew. Chem.* **1963**, 22.
(50G) Ozeris, S., Bassette, R., ANAL. CHEM. **35**, 1091 (1963).
(51G) Patton, S., *J. Dairy Sci.* **46**, 856 (1963).
(52G) Patton, S., *Ibid.*, **47**, 817 (1964).
(53G) Pfenninger, H., *Z. Lebensm.-Untersuch-Forsch* **120**, 100 (1963).
(54G) Ramachandran, K. S., Gould, I. A., Lindamood, J. B., *J. Dairy Sci.* **47**, 458 (1964).
(55G) Reymond, D., Chavan, F., Egli, R. H., *Abstr. 1st Int. Congr. Fd. Sci. Technology* **1962**, 82; *J. Sci. Food Agr.* **13**, 211-*ii* (1962).
(56G) Schwartz, D. P., *J. Dairy Sci.* **46**, 989 (1963).
(57G) Self, R., Casey, J. C., Swain, T., *Chem. Ind.* (*London*) **1963**, 863.
(58G) Self, R., Land, D. G., Casey, J. C., *J. Sci. Food Agr.* **14**, 209 (1963).
(59G) Senn, V. J., *J. Food Sci.* **28**, 531 (1963).
(60G) Stahl, W. H., Sullivan, J. H., Voelker, W. A., *J. Assoc. Offic. Agr. Chem.* **46**, 819 (1963).
(61G) Sugisawa, H., Matthews, S. S., MacGregor, D. R., *J. Food Sci.* **27**, 435 (1962).
(62G) ten Hoopen, H. J. G., *Z. Lebensm.-Untersuch.-Forsch* **119**, 478 (1963).
(63G) Teranishi, R., Buttery, R. G., Lundin, R. E., ANAL. CHEM. **34**, 1033 (1962).
(64G) Teranishi, R., Corse, J. W., McFadden, W. H., Black, D. R., Morgan, A. I., Jr., *J. Food Sci.* **28**, 478 (1963).
(65G) Teranishi, R., Schultz, T. H., McFadden, W. H., Lundin, R. E., Black, D. R., *Ibid.*, **28**, 541 (1963).
(66G) Underwood, J. C., Filipic, V. J., *J. Assoc. Offic. Agr. Chem.* **46**, 334 (1963).
(67G) von Szilvinyi, A., Puspok, J., *Brauwissenschaft* **16**, 204 (1963).
(68G) Webb, A. D., Ribereau-Gayon, P., Boidron, J. N., *Compt. Rend. Acad. Agric. France* **49**, 115 (1963).
(69G) Winter, M., Stoll, M., Wernhoff, F. W., Greuter, F., Buchi, G., *J. Food Sci.* **28**, 554 (1963).
(70G) Wolford, R. W., Attaway, J. A., Alberding, G. E., Atkins, C. D., *Ibid.*, **28**, 320 (1963).
(71G) Van der Kloot, A. P., Wilcox, F. A., *Proc. Amer. Soc. Brew. Chem.* **1963**, 93.
(72G) Yamanishi, T., Kiribuchi, T., Sakai, M., Fujita, N., Ikeda, Y., Sasa, K., *Agr. Biol. Chem., Japan* **27**, 193 (1963); *Anal. Abstr.* **11**, 789 (1964).

Identity

(1H) Albanese, F., *Cafe, Cacao, The* **7**, 321 (1963); *C.A.* **60**, 15061*a* (1964).
(2H) Anker, L., Sonanini, D., *Pharm. Acta Helv.* **37**, 360 (1962); *Anal. Abstr.* **10**, 795 (1963).
(3H) Ayres, A. D., Charley, V. L. S., Swindells, R., *Food Process. Packaging* **30**, 413, 425 (1961).
(4H) Ayres, A. D., Charley, V. L. S., Swindells, R., *Ibid.*, **31**, 14 (1962).
(5H) Bagnulo, R., Criscuolo, A., Pane, D., *Igiene San. Pubbl.* **17**, 469 (1961); *Anal. Abstr.* **10**, 1579 (1963).
(6H) Baiao, A., *Cafe, Cacao, The* **7**, 200 (1963); *C.A.* **60**, 11293*a* (1964).
(7H) Barker, A. G., Billington, A. E., Charley, V. L. S., *Food Process. Packaging* **30**, 325 (1961).
(8H) Bartolomeo, D., Gabucci, G., *Boll. Lab. Chim. Provinciali* (*Bologna*) **14**, 3 (1963); *C.A.* **59**, 12086*f* (1963).
(9H) Bell, P. M., Simmonds, D. H., *Cereal Chem.* **40**, 121 (1963).
(10H) Boniforti, L., *Ann. Fals. Expert. Chim.* **55**, 255 (1962); *Anal. Abstr.* **10**, 1195 (1963).
(11H) Bradley, D. B., *J. Agr. Food Chem.* **12**, 213 (1964).
(12H) Bressani, R., Gomez-Grenes, R., Conde, R., *Arch. Venezolanos Nutr.* **12**, 93 (1962); *C.A.* **58**, 4974*g* (1963).
(13H) Brogioni, M., Franconi, U., *Boll. Lab. Chim. Provinciali* (*Bologna*) **14**, 135 (1963).
(14H) Cabada, L., Carballido, A., Villanua, L., Valdehita, M. T., *Anales Bromatol.* (*Madrid*) **15**, 27 (1963).
(15H) Clements, R. L., *J. Food Sci.* **29**, 276 (1964).
(16H) *Ibid.*, p. 281.
(17H) Comm. Interministerials, Min. Agric. e Foreste., *Riv. Ital. Sostanze Grasse* **40**, 108 (1963).
(18H) Corbi, D., Cicero, L., *Olearia* **17**, 148 (1963).
(19H) Davidov, R. V., Zalashko, M. V., *Zh. Khim.* **19R**, 1R105 (1963); *Anal. Abstr.* **10**, 3448 (1963).
(20H) De'Gori, R., Grandi, F., *Boll. Lab. Chim. Provinciali* (*Bologna*) **13**, 175 (1962); *Anal. Abstr.* **9**, 5456 (1962).
(21H) Deshusses, J., Gabbai, A., *Mitt. Gebiete Lebensm. Hyg.* **53**, 408 (1962); *Anal. Abstr.* **10**, 2473 (1963).
(22H) Doro, B., Gabucci, G., *Boll. Lab. Chim. Provinciali* (*Bologna*) **14**, 3 (1963); *Anal. Abstr.* **10**, 5387 (1963).
(23H) Eisner, J., Firestone, D., *J. Assoc. Offic. Agr. Chem.* **46**, 542 (1963).
(24H) Fincke, A., *Fette, Seifen, Anstrichmittel* **65**, 647 (1963).
(25H) German Federal Republic's Institute of Fishery, *Food Eng.* **35**, 48 (1963).
(26H) Giancaspro, B., Florio, I., *Riv. Ital. Sostanze Grasse* **40**, 121 (1963).
(27H) Gracian Tous, J., Arevalo, G., Martel, J., *Grasas Aceites* (*Seville, Span*) **14**, 101 (1963).
(28H) Hadorn, H., Zurcher, K., *Mitt. Gebiete Lebensm. Hyg.* **54**, 304 (1963).
(29H) Hamdy, M. M., Gould, W. A., *J. Agr. Food Chem.* **10**, 499 (1962).
(30H) Herrmann, K., *Deut. Lebensm.-Rundschau* **58**, 228 (1962); *Anal. Abstr.* **10**, 2472 (1963).
(31H) Hillig, F., Shelton, L. R., Jr., Yates, W., Bethea, S., Campbell, C. M., *J. Assoc. Offic. Agr. Chem.* **46**, 493 (1963).
(32H) Houlbrooke, A., *J. Assoc. Publ. Analysts* **1**, 33 (1963).
(33H) *Ibid.*, p. 16.
(34H) Houston, D. F., Hill, B. E., Garrett, V. H., Kester, E. B., *J. Agr. Food Chem.* **11**, 512 (1963).
(35H) Jaforte, A., *Riv. Ital. Sostanze Grasse* **40**, 374 (1963).
(36H) Jensen, R. G., Gander, G. W., Sampugna, J., *J. Dairy Sci.* **45**, 329 (1962).
(37H) Jorysch, D., *J. Assoc. Offic. Agr. Chem.* **46**, 365 (1963).
(38H) Kaderavek, G., Gay, G., *Olearia* **17**, 145 (1963).
(39H) Kaufmann, H. P., Mankel, G., *Fette, Seifen, Anstrichmittel* **65**, 295 (1963).
(40H) Kaufmann, H. P., Wessels, H., Das, B., *Ibid.*, **64**, 723 (1962).
(41H) Kihara, Y., Takeuchi, E., *Eiyo to Shokuryo* **13**, 253 (1960); *C.A.* **59**, 12077*f* (1963).
(42H) Kelley, J. J., Koenig, V. L., *J. Sci. Food Agr.* **14**, 29 (1963).
(43H) Laporta, L., *Boll. Lab. Chim. Provinciali* (*Bologna*) **13**, 498 (1962).
(44H) Lee, J. W., *Biochim. Biophys. Acta* **69**, 159 (1963).
(45H) Lugay, J. C., Juliano, B. O., *J. Am. Oil Chemists Soc.* **41**, 273 (1964).
(46H) Luh, B. S., Chichester, C. O., Co, H., Leonard, S. J., *Food Technol.* **18**, 159 (1964).

(47H) Macy, R. L., Jr., Naumann, H. D., Bailey, M. E., *J. Food Sci.* **29,** 136 (1964).
(48H) *Ibid.*, p. 142.
(49H) Mancuso,. V. M., *J. Assoc. Offic. Agr. Chemists* **47,** 841 (1964).
(50H) Mehlitz, A., Gelbrich, D., *Deut. Lebensm.-Rundschau* **58,** 284 (1962); *Anal. Abstr.* **10,** 3453 (1963).
(51H) Mhatre, N. S., Leeder, J. G., Wogan, G. N., *J. Dairy Sci.* **45,** 717 (1962).
(52H) Monk, H. E., *J. Assoc. Publ. Analysts* **1,** 20 (1963).
(53H) Montefredine, A., Laporta, L., *Boll. Lab. Chim. Provinciali* (*Bologna*) **13,** 498 (1962); *Anal. Abstr.* **10,** 2924 (1963).
(54H) Montefredine, A., Laporta, L., *Riv. Ital. Sostanze Grasse* **40,** 379 (1963).
(55H) Mookherjee, E. D., Chang, S. S., *J. Am. Oil Chemists Soc.* **40,** 232 (1963).
(56H) Morgantini, M., *Riv. Ital. Sostanze Grasse* **40,** 49 (1963).
(57H) Morita, S., Higashiya, H., *Japan Analyst* **11,** 282 (1962).
(58H) Olivari, L., Benassi, R., *Boll. Lab. Chim. Provinciali* (*Bologna*) **14,** 304 (1963).
(59H) Pekkarinen, L., Porkka, E., *Z. Lebensm.-Untersuch.-Forsch* **120,** 277 (1963).
(60H) Primo, E., Sanchez, J., Alberola, J., *Rev. Agroquim. Tecnol. Alimentos* **3,** 349 (1963); *Anal. Abstr.* **11,** 2365 (1964).
(61H) Privett, O. S., Blank, M. L., Schmit, J. A., *J. Food Sci.* **27,** 463 (1962).
(62H) Rice, A. C., *J. Assoc. Offic. Agr. Chem.* **47,** 671 (1964).
(63H) Rice, E. E., *J. Am. Dietetics Assoc.* **41,** 319 (1962).
(64H) Rolle, L. A., Vandercook, C. E., *J. Assoc. Offic. Agr. Chem.* **46,** 362 (1963).
(65H) Rojas, M. D., Garcia-Olmedo, R., *Anales Bromatol.* (*Madrid*) **15,** 81 (1963).
(66H) Sawyer, R., *J. Sci. Food Agr.* **14,** 302 (1963).
(67H) Schormuller, J., Lange, H.-J., *Z. Lebensm.-Untersuch.-Forsch.* **118,** 214 (1962).
(68H) Sherratt, J. G., Sinar, R., *J. Assoc. Publ. Analysts* **1,** 28 (1963).
(69H) Skarka, B., Telepcak, M., *Prumsyl Potravin* **15,** 188 (1964); *C.A.* **61,** 6283*h* (1964).
(70H) Spiteri, J., *Deut. Lebensm.-Rundschau* **58,** 155 (1962); *Anal. Abstr.* **10,** 1589 (1963).
(71H) Thaler, H., *Cafe, Cacao, The* **7,** 240 (1963); *C.A.* **60,** 12589*h* (1964).
(72H) Thaler, H., Gaigl, R., *Z. Lebensm.-Untersuch.-Forsch.* **119,** 10 (1962).
(73H) Thaler, H., Gaigl, R., *Ibid.*, **120,** 449 (1963).
(74H) Vandercook, C. E., *J. Assoc. Offic. Agr. Chem.* **46,** 353 (1963).
(75H) Vandercook, C. E., Rolle, L. A., *Ibid.*, **46,** 359 (1963).
(76H) Wagner, D., Monselise, J. J., *Israel J. Technol.* **1,** 33 (1963).
(77H) Wucherpfennig, K., Bretthauer, G., *Deut. Lebensm.-Rundschau* **58,** 190 (1962); *Anal. Abstr.* **10,** 2029 (1963).

Inorganic Constituents

(1J) Barakat, M. Z., Sadek, I., *Food Technol.* **18,** 242 (1964).
(2J) Bastianutti, J., *Boll. Lab. Chim. Provinciali* (*Bologna*) **14,** 296 (1963).
(3J) Boni, A. L., *Analyst* **88,** 64 (1963).
(4J) Boni, A. L., *U. S. Atomic Energy Comm., Rept.* **DPSPU-63-30-6** (1963).
(5J) Bradfield, E. G., *J. Sci. Food Agr.* **15,** 469 (1964).
(6J) Braunsdorf, K., *Nahrung* **7,** 166 (1963).
(7J) Brekke, J. E., Taylor, D. H., Stanley, W. L., *J. Agr. Food Chem.* **11,** 260 (1963).
(8J) Brenner, M. W., Mayer, M. J., Blick, S. R., *Proc. Amer. Soc. Brew. Chem.* **1963,** 165.
(9J) Brenner, M. W., Vigilante, C., Arthurs, M. J., *Ibid.*, **1963,** 158.
(10J) Broadbank, R. W. C., Hands, J. D., Harding, R. D., *Analyst* **88,** 43 (1963).
(11J) Butler, F. E., ANAL. CHEM. **35,** 2069 (1963).
(12J) Chen, S. Y., *Taiwan Sugar* **9**(3), 33 (1962); *Anal. Abstr.* **10,** 2894 (1963).
(13J) Cukor, P., Walzcyk, J., Lott, P. F., *Anal. Chim. Acta* **30,** 473 (1964).
(14J) Dangoumau, A., Ducos, R., *Chim. Anal.* (*Paris*) **44,** 292 (1962); *Anal. Abstr.* **10,** 1186 (1963).
(15J) Davidek, J., Klein, S., Zackova, A., *Z. Lebensm.-Untersuch.-Forsch.* **119,** 342 (1963).
(16J) Davidek, J., Zackova, A., *Z. Anal. Chem.* **189,** 320 (1962); *Anal. Abstr.* **10,** 1190 (1963).
(17J) Dye, W. B., Bretthauer, E., Seim, H. J., Blincoe, C., ANAL. CHEM. **35,** 1687 (1963).
(18J) Follett, M. J., Ratcliff, P. W., *J. Sci. Food Agr.* **14,** 138 (1963).
(19J) Gaigl, R., *Z. Lebensm.-Untersuch.-Forsch.* **119,** 506 (1963).
(20J) Gloyna, E. F., Bhagat, S. K., Felsing, W. A., Jr., *J. Water Pollut. Control Fed.* **35**(7), 893 (1963).
(21J) Hadjiioannou, T. P., *Anal. Chim. Acta* **30,** 537 (1964).
(22J) Harley, J. H., *J. Assoc. Offic. Agr. Chem.* **46,** 371 (1963).
(23J) *Ibid.*, p. 380.
(24J) Kiermeier, F., Johannsmann, H., *Z. Lebensm.-Untersuch.-Forsch.* **119,** 497 (1963).
(25J) Loach, K. W., *Nature* **196,** 754 (1962).
(26J) Pliska, V., Likar, C., Reisenauer, R., *Z. Lebensm.-Untersuch.-Forsch.* **118,** 429 (1962).
(27J) Robertson, P. S., *J. Dairy Res.* **29,** 321 (1962).
(28J) Rooney, R. C., *Analyst* **88,** 959 (1963).
(29J) Smith, H., Whitehead, E. L., *Nature* **199,** 503 (1963).
(30J) Steiner, R. L., Oliver, R. T., *Proc. Amer. Soc. Brew. Chem.* **1963,** 111.
(31J) Stone, I., *Ibid.*, **1962,** 64.
(32J) Stone, I., Gantz, C. S., Saletan, L. T., *Ibid.*, **1963,** 149.
(33J) Stutler, J. R., Dakin, P. E., *Ibid.*, **1963,** 26.
(34J) Truhaut, R., Boudene, C., *Ann. Fals. Expert. Chim.* **56,** 225 (1963).
(35J) Visapaa, A., *Tekn. Kem. Aikl.* **20,** 146 (1963).
(36J) Voth, J. L., ANAL. CHEM. **35,** 1957 (1963).
(37J) Westoo, G., *Analyst* **88,** 287 (1963).
(38J) Wunsch, A., Teicher, K., *Z. Pfl-Ernahr. Dung.* **97,** 101 (1962); *Anal. Abstr.* **10,** 257 (1963).
(39J) Yakabe, H. M., Dimitroff, J. M., Laug, E. P., *J. Assoc. Offic. Agr. Chem.* **45,** (3), 775 (1962).
(40J) Zeeman, P. B., Butler, P. R. P., *Appl. Spectry.* **16,** 120 (1962).

Moisture

(1K) Abragam, D. R., *Tr. Tsentr. Issled. Inst. Krakhmalopatoch. Prom.* **1963** (5) 47; *Anal. Abstr.* **11,** 1910 (1964).
(2K) Amdur, E. J., *Instr. Control Systems* **36,** (6) 93–7 (1963).
(3K) Amer. Soc. Heat. Refrig., Air-Cond. Eng.; *Abstracts, 1963 Symposium on Humidity and Moisture,* New York, N. Y.
(4K) Christensen, C. M., Linko, P., *Cereal Chem.* **40,** 129 (1963).
(5K) Fraade, D. J., *Instr. Control Systems* **36,** (2) 99 (1963).
(6K) Fraade, D. J., *Ibid.*, **36,** (4) 100–5 (1963).
(7K) Gold, H. J., *Food Technol.* **18,** 586 (1964).
(8K) King, L. F. *Australian J. Dairy Technol.* **17,** 50 (1962).
(9K) Lysanskii, E. B., *Sakharn. Prom.* **1961** (12) 40–41; *Anal. Abstr.* **10,** 1181 (1963).
(10K) Martelli, L., *Ann. Fals. Exp. Chim.* **55,** 211 (1962).
(11K) Miller, B. S., Kaslow, H. D., *Food Technol.* **17,** (5), 142 (1963).
(12K) Nemitz, G. Z., *Lebensm. Untersuch. Forsch.* **123,** 1 (1963).
(13K) Nemitz, G., *Starke* **14,** 276 (1962).
(14K) Schwecke, W. M., Nelson, J. H., ANAL. CHEM. **36,** 689 (1964).
(15K) Thung, S. B., *J. Sci. Food Agric.* **15,** 236 (1964).
(16K) Vainberg, V. Y., *Tr. Vses Nauch-Issled Inst. Moloch Prom* **1962,** (22) 71; *Anal. Abstr.* **11,** 2357 (1964).
(17K) Weinfurtner, F., Wullinger, Piendl, A., *Brauwelt* (**1963**), 897; *Anal. Abstr.* **11,** 4548 (1964).

Organic Acids

(1L) Aunstrup, K., Djurtoft, R., *Brygmesteren* **17,** 137 (1960); *Anal. Abstr.* **10,** 815 (1963).
(2L) Blundstone, H. A. W., *Nature* **197,** 377 (1963).
(3L) Cerbulis, J., Taylor, M. W., ANAL. CHEM. **35,** 114 (1963).
(4L) Choy, T. K., Quattrone, J. J., Jr., Elefant, M., *Anal. Chim. Acta* **29,** 114 (1963).
(5L) Churacek, J., Vanek, J., *Sb. Vysoke Skoly Chem.-Technol. Praze* **1,** 61 (1963); *Anal. Abstr.* **11,** 3732 (1964).
(6L) Courtoisier, A. J., Ribereau-Gayon, J., *Bull. Soc. Chim. France* **1963,** 350.
(7L) Goudie, A. J., Rieman, W., III, *Anal. Chim. Acta* **26,** 419 (1962).
(8L) Herrmann, K., *Deut. Lebensm.-Rundschau* **59,** 309 (1963).
(9L) Knappe, E., Peteri, D., *Z. Anal. Chem.* **188,** 184 (1962).
(10L) Luke, H. H., Freeman, T. E., Kier, L. B., ANAL. CHEM. **35,** 1916 (1963).
(11L) Markakis, P., Jarczyk, A., Krishna, S. P., *J. Agr. Food Chem.* **11,** 8 (1963).
(12L) Matulis, R. M., Guyon, J. C., ANAL. CHEM. **36,** 118 (1964).
(13L) Mayer, K., Busch, I., *Mitt. Gebiete Lebensm. Hyg.* **54,** 60 (1963).
(14L) Metche, M., Jacquin, F., Nguyen, Q. H., Urion, E., *Bull. Soc. Chim. France* **1962,** 1763.
(15L) Nair, P. M., Vaidyanathan, C. S., *Anal. Biochem.* **7,** 315 (1964).
(16L) Pastuska, G., Petrowitz, H.-J., *J. Chromatog.* **10,** 517 (1963).
(17L) Qureshi, M., Husain, W., Rawat, J. P., ANAL. CHEM. **35,** 1592 (1963).
(18L) Raisch, M., Becker, G., *Deut. Lebensm. Rundschau* **60,** 151 (1964).
(19L) Rebelein, H., *Ibid.*, **59,** 129 (1963).
(20L) Riddle, V. M., ANAL. CHEM. **35,** 853 (1963).
(21L) Robb, E. W., Westbrook, III, *Ibid.*, **35,** 1644 (1963).
(22L) Roberts, E. A. H., *J. Sci. Food Agr.* **14,** 700 (1963).
(23L) Roberts, E. A. H., Smith, R. F., *Ibid.*, **14,** 689 (1963).
(24L) Seligson, H., Kramer, B., Seligson, D., Balthrush, H., *Anal. Biochem.* **6,** 362 (1963).
(25L) Shelley, R. N., Salwin, H., Horwitz, W., *J. Assoc. Offic. Agr. Chem.* **46,** 486 (1963).
(26L) Siddiqi, A. I., Freedman, S. O., *Can. J. Biochem. Physiol.* **41,** 947 (1963).

(27L) Smith, R. F., *Cafe, Cacao, The* **7**, 245 (1963); *C.A.* **60**, 11292*d* (1964).
(28L) Spanyar, P., Szeredy, I., *Z. Lebensm.-Untersuch.-Forsch.* **118**, 293 (1962).
(29L) Taufel, K., Voigt, J., *Ibid.*, **118**, 481 (1962).
(30L) Torres, J. R., Goodman, S., Roman, F., de Carballo, N. F., *Rev. Col. Quim., Puerto Rico* **20**, 38 (1962); *Anal. Abstr.* **10**, 5379 (1963).
(31L) Urion, E., Metche, M., Haluk, J.-P., *Brauwissenschaft* **16**, 211 (1963).
(32L) Wendlandt, W. W., Hoiberg, J. A., *Anal. Chim. Acta* **28**, 506 (1963).
(33L) Young, H. Y., Shimabukuro, S., Aono, L., *J. Agr. Food Chem.* **11**, 132 (1963).
(34L) Zagrodzki, S., Szwajcowska, K., *Gaz Cukr.* **71**, 120 (1963); *Anal. Abstr.* **11**, 3341 (1964).
(35L) Zarembski, P. M., Hodgkinson, A., *Analyst* **87**, 698 (1962).

Proteins, Amino Acids, and Nitrogen

(1M) Andrews, P., *Biochem. J.* **91**, 222 (1964).
(2M) Bell, P. M., *Anal. Biochem.* **5**, 443 (1963).
(3M) Bell, P. M., *J. Sci. Food Agr.* **14**, 133 (1963).
(4M) Bergman, I., Loxley, R., Anal. Chem. **35**, 1961 (1963).
(5M) Bloomfield, J., Farrar, J. F., *Ibid.*, **36**, 950 (1964).
(6M) Botvinik, M. M., Troshko, E. V., *Zh. Obshch. Khim.* **32**, 1389 (1962); *Anal. Abstr.* **10**, 296 (1963).
(7M) Botvinik, M. M., Troshko, E. V., Gorshkova, T. A., *Ibid.*, **32**, 1382 (1962); *Anal. Abstr.* **10**, 295 (1963).
(8M) Corbett, W. G., Kenchington, A. W., Ward, A. G., *Biochem. J.* **84**, 468 (1962).
(9M) Cruickshank, P. A., Sheehan, J. C., Anal. Chem. **36**, 1191 (1964).
(10M) Drawert, F., Reuther, K.-H., *Angew. Chem. Int. Ed.* **2**, 101 (1963).
(11M) D'yachenko, P., *Moloch. Prom.* **1963**, 7; *Zh. Khim.* **19R**, 21R114 (1963); *Anal. Abstr.* **11**, 2361 (1964).
(12M) Elton, G. A. H., Ewart, J. A. D., *J. Sci. Food Agr.* **14**, 175 (1963).
(13M) Fahmy, A. R., *Helv. Chim. Acta* **44**, 2022 (1961).
(14M) Fox, K. K., Holsinger, V. H., Pallansch, M. J., *J. Dairy Sci.* **46**, 302 (1963).
(15M) Hamilton, P. B., Anal. Chem. **35**, 2055 (1963).
(16M) Hartel, J., Pleumeekers, A. J. G., *Ibid.*, **36**, 1021 (1964).
(17M) Ikekawa, N., *J. Biochem., Tokyo* **54**, 279 (1963).
(18M) Ivanov, C. P., Alexiev, B. V., Krsteva, M., Yordanov, B., *Anal. Chim. Acta* **30**, 549 (1964).
(19M) Junk, G., Svec, H., *Ibid.*, **28**, 164 (1963).
(20M) Kiermeier, F., Hamed, M. G. E., *Nahrung* **6**, 639 (1962).
(21M) Kofranyi, E., *Z. Physiol. Chem.* **335**, 161 (1964).
(22M) Landowne, R. A., Lipsky, S. R., *Nature* **199**, 141 (1963).
(23M) Lento, H. G., Fods, J. A., Denton, A. E., *J. Food Sci.* **29**, 435 (1964).
(24M) Linko, P., *Suomen Kem.* **35**, 77 (1962); *Anal. Abstr.* **9**, 4920 (1962).
(25M) Moore, S., *J. Biol. Chem.* **238**, 235 (1963).
(26M) Nakamura, Y., *J. Pharm. Soc. Japan* **82**, 689 (1962); *Anal. Abstr.* **10**, 5206 (1963).
(27M) Nicholls, C. H., Makisumi, S., Saroff, H. A., *J. Chromatog.* **11**, 327 (1963).
(28M) Onda, F., *Proc. Res. Soc. Japan Sugar Refineries Technol.* **12**, 67 (1963); *Anal. Abstr.* **11**, 3339 (1964).
(29M) Padmoyo, M., Hogl, O., *Mitt. Gebiete Lebensm. Hyg.* **53**, 290 (1962).
(30M) Peters, I. I., *J. Dairy Sci.* **45**, 1075 (1962).
(31M) Pisano, J. J., Vanden Heuvel, W. J. A., Horning, E. C., *Biochem. Biophys. Research Commun.* **7**, 82 (1962); *C.A.* **57**, 1213*a* (1962).
(32M) Pomeranz, Y., Miller, B. S., *J. Assoc. Offic. Agr. Chem.* **46**, 399 (1963).
(33M) Rao, S. R., Carter, F. L., Frampton, V. L., Anal. Chem. **35**, 1927 (1963).
(34M) Rosen, H., Berard, C. W., Levenson, S. M., *Anal. Biochem.* **4**, 213 (1962).
(35M) Schilling, E. D., Burchill, P. I., Clayton, R. A., *Ibid.*, **5**, 1 (1963).
(36M) Schroeder, W. A., Jones, R. T., Cormick, J., McCalla, K., Anal. Chem. **34**, 1570 (1962).
(37M) Seto, J., *Japan Analyst* **10**, 133 (1961); *Anal. Abstr.* **10**, 2425 (1963).
(38M) Shimazono, H., *Food Technol.* **18**, 294 (1964).
(39M) Shlyapnikov, S. V., Karpeiskii, M. Y., Litvin, E. F., *Biokhimiya* **28**, 664 (1963); *Anal. Abstr.* **11**, 3269 (1964).
(40M) Siminovitch, D., Therrien, H., Wilner, J., Gfeller, F., *Can. J. Botany* **40**, 1267 (1962).
(41M) Smyth, D. G., Elliot, D. F., *Analyst* **89**, 81 (1964).
(42M) Soc. Anal. Chem., *Analyst* **88**, 422 (1963).
(43M) *Ibid.*, p. 583.
(44M) Stark, J. B., *Anal. Biochem.* **4**, 103 (1962).
(45M) Staub, M., Romann, E., *Mitt. Lebensmitt. Hyg., Bern* **53**, 272 (1962).
(46M) Steven, F. S., *Anal. Biochem.* **4**, 316 (1962).
(47M) Stewart, I., *J. Chromatog.* **10**, 404 (1963).
(48M) Takagi, T., Honda, T., *Japan Analyst* **11**, 286 (1962).
(49M) Tarassuk, N. P., Yaguchi, M., *J. Dairy Sci.* **45**, 253 (1962).
(50M) Thaler, H., Gaigl, R., *Z. Lebensm.-Untersuch.-Forsch.* **118**, 22 (1962).
(51M) Thaler, H., Gaigl, R., *Ibid.*, **119**, 10 (1962).
(52M) Torten, J., Whitaker, J. R., *J. Food Sci.* **29**, 168 (1964).
(53M) Whitaker, J. R., Anal. Chem. **35**, 1950 (1963).
(54M) Winter, L. N., Albro, P. W., *J. Gas. Chromatog.* **2**, 1 (1964).
(55M) Wucherpfennig, K., Francke, I., *Z. Lebensm.-Untersuch.-Forsch.* **124**, 22 (1963).
(56M) Yoshino, U., Wilson, H. K., Herreid, E. O., *J. Dairy Sci.* **45**, 1459 (1962).
(57M) Zacharius, R. M., Talley, E. A., Anal. Chem. **34**, 1551 (1962).
(58M) Zomzely, C., Marco, G., Emery, E., *Ibid.*, **34**, 1414 (1962).

Vitamins

(1N) Anderson, R. H., Moran, D. F., *Cereal Chem.* **39**, 463 (1962).
(2N) Blattna, J., Deyl, Z., Rosmus, J., *Mikrochem. Acta* **1962**, 868.
(3N) Boddeker, H., Mishkin, A. R., Anal. Chem. **35**, 1662 (1963).
(4N) Chen, P. S., Jr., Terepka, A. R., Remsen, N., *Ibid.*, **35**, 2030 (1963).
(5N) Cima, L., Mantovan, R., *Farmaco. Ed. Prat.* **17**, 473 (1962); *Anal. Abstr.* **10**, 2039 (1963).
(6N) David, S., Hirshfeld, H., *Bull. Soc. Chim. France* **1963**, 1011.
(7N) Deyl, Z., Rosmus, J., *J. Chromatog.* **8**, 537 (1962).
(8N) Dilley, R. A., Crane, F. L., *Anal. Biochem.* **5**, 531 (1963).
(9N) Dugan, R. E., Frigerio, N. A., Siebert, J. M., Anal. Chem. **36**, 114 (1964).
(10N) Dunagin, P. E., Jr., Olson, J. A., *Ibid.*, **36**, 756 (1964).
(11N) Erickson, D. R., Dunkley, W. L., *Ibid.*, **36**, 1055 (1964).
(12N) Fischer, R., Benkendorf, F. G., Kochan, C., *Dtsch. Apoth Ztg.* **103**, 301 (1963); *Anal. Abstr.* **11**, 1492 (1964).
(13N) Gassmann, B., Janiki, J., *Ernahrungsforschung* **8**, 377 (1963).
(14N) Hasselquist, H., Jaarma, M., *Acta Chem. Scand.* **17**, 529 (1963).
(15N) Hedin, P. A., *J. Agr. Food Chem.* **11**, 343 (1963).
(16N) Herrmann, J., Zobel, M., *Z. Lebensm.-Untersuch.-Forsch.* **116**, 477 (1962).
(17N) Herrmann, J., Zobel, M., *Ibid.*, **117**, 1 (1962).
(18N) Herrmann, J., Zobel, M., *Ibid.*, **117**, 189 (1962).
(19N) Herting, D. C., Drury, E.-J. E., Harris, P. I., *Anal. Biochem.* **4**, 459 (1962).
(20N) Huttenrauch, R., Klotz, L., *Experientia* **19**, 95 (1963).
(21N) Jungalwala, F. B., Cama, H. R., *J. Chromatog.* **8**, 535 (1962).
(22N) Krauze, S., Bozyk, Z., Korzen, K., *Chem. Anal., Warsaw* **8**, 179 (1963); *Anal. Abstr.* **11**, 1493 (1964).
(23N) Lambertsen, G., Myklestad, H., Braekkan, P. R., *J. Food Sci.* **29**, 164 (1964).
(24N) Leemann, H. G., Stich, K., *Helv. Chim. Acta* **45**, 1275 (1962).
(25N) Lento, H. G., Daugherty, C. E., Denton, A. E., *J. Agr. Food Chem.* **11**, 22 (1963).
(26N) Libby, D. A., Sheppard, A. J., *J. Assoc. Offic. Agr. Chem.* **47**, 371, (1964).
(27N) Monnier, D., Ghaliounghi, Y., *Anal. Chim. Acta* **28**, 30 (1963).
(28N) Murray, T. K., Anal. Chem. **34**, 1241 (1962).
(29N) Norman, A. W., DeLuca, H. F., *Ibid.*, **35**, 1247 (1963).
(30N) Soc. Anal. Chem., *Analyst* **89**, 1 (1964).
(31N) *Ibid.*, p. 7.
(32N) Spanyar, P., Kevei, E., Blazovich, M., *Z. Anal. Chem.* **195**, 268 (1963).
(33N) Strohecker, R., Jr., Pies, H., *Z. Lebensm.-Untersuch.-Forsch.* **118**, 394 (1962).
(34N) Takahashi, R., Tachi, I., *Agr. Biol. Chem., Japan* **26**, 771 (1962); *Anal. Abstr.* **10**, 3928 (1963).
(35N) Tikhomirova, G. P., Belen'kaya, S. L., *Ukr. Khim. Zh.* **29**, 97 (1963); *Anal. Abstr.* **10**, 4921 (1963).
(36N) Tikhomirova, G. P., Belen'kaya, S. L., *Zh. Anal. Khim.* **17**, 767 (1962); *Anal. Abstr.* **10**, 2925 (1963).
(37N) Varma, T. N. R., Panalaks, T., Murray, T. K., Anal. Chem. **36**, 1864 (1964).
(38N) Wilkie, J. B., *Ibid.*, **36**, 896 (1964).
(39N) Woggon, H., Kohler, U., *Mitt. Gebiete Lebensm. Hyg.* **54**, 95 (1963).

Miscellaneous

(1P) Albanese, F., *Deut. Lebensm. Rundschau.* **59**, 260 (1963).
(2P) Albanese, F., *Z. Lebensm.-Untersuch.-Forsch.* **120**, 364 (1963).
(3P) Allen, E., Cook, C. F., Bray, R. W., *J. Animal Sci.* **21**, 977 (1962).
(4P) American Association of Cereal Chemists, Cereal Laboratory Method, St. Paul, Minn. (1962).
(5P) Barbera, C. E., *Z. Lebensm.-Untersuch.-Forsch.* **117**, 483 (1962).

(6P) Borker, E., Sloman, K. G., ANAL. CHEM. **35,** 62R (1963).
(7P) Borker, E., Sloman, K. G., *J. Assoc. Offic. Agr. Chem.* **46,** 319 (1963); Assoc. Offic. Agr. Chemist Meeting Absts. (1964).
(8P) Brown, S. A., Shyluk, J. P., ANAL. CHEM. **34,** 1058 (1962).
(9P) Crosby, D. G., *Anal. Biochem.* **4,** 349 (1962).
(10P) Deffner, G., *Z. Lebensm.-Untersuch.-Forsch.* **120,** 300 (1963).
(11P) Gorbach, G., *Nahrung* **7,** 261 (1963).
(12P) Food Chemicals Codex. Part I. National Academy of Sciences—National Research Council *Publication* **1143** (1963).
(13P) Gunderson, F., "Food Standards and Definitions in the United States," Academic Press, New York, 1963.
(14P) Hilderson, H., *Petit J. Brass.* **71,** 346 (1963).
(15P) Hoff, J. E., Wick, E. L., *J. Food Sci.* **28,** 510 (1963).
(16P) Jaulmes, P., Brun, S., *Ann. Fals. Expert. Chim.* **56,** 143 (1963).
(17P) Jayaraman, K. S., Ramanujam, S., Vijayaraghavan, P. K., *Curr. Sci.* **31,** 282 (1962); *Anal. Abstr.* **10,** 3455 (1963).
(18P) Johnson, N. L., *Food Technol.* **17,** 1516 (1963).
(19P) Kramer, H. A., Gates, J. E., Damaree, K. D., Sidwell, A. P., *Ibid.*, **17,** 1044 (1963).
(20P) Nakagawa, M., Torii, H., *Agr. Biol. Chem.* (*Tokyo*) **28,** 160 (1964); *C. A.* **60,** 15061*g* (1964).
(21P) Pekkarinen, L., Porkka, E., *Z. Lebensm.-Untersuch.-Forsch.* **120,** 20 (1963).
(22P) Preece, I. A., "A Literature Survey," Institute of Brewing, 28 pp., (1962); *Anal. Abstr.* **10,** 3457 (1963).
(23P) *Ibid.*, 32 pp., (1963); *Anal. Abstr.* **11,** 3359 (1964).
(24P) Shibata, E., Nakano, T., Saito, S., Brit. Patent **915,305** (3/13/59); *Anal. Abstr.* **10,** 2069 (1963).
(25P) Smith, R. F., *Analyst* **89,** 146 (1964).
(26P) Smith, R. F. Rees, D. I., *Ibid.*, **88,** 310 (1963).
(27P) Soc. Anal. Chem., "Official Standardized and Recommended Methods of Analysis," S. C. Jolly, ed., W. Heffer & Sons, Cambridge, England, 1963.
(28P) Stahl, E., *Angew. Chem.* **73,** 646 (1962).
(29P) Streuli, H., *Chimia* **16,** 371 (1962); *Anal. Abstr.* **10,** 3916 (1963).
(30P) Subrahmanyan, V., *et al.*, *Indian J. Technol.* **1,** 244 (1963); *J. Sci. Food Agr.* **15,** 105*i* (1964).
(31P) Vercillo, A., Manzone, A. M., *Rend. Ist. Super. Sanita* **25,** 574–80 (1962); *Anal. Abstr.* **10,** 2486 (1963).
(32P) Wood, D. J., *et al.*, *J. Sci. Food Agr.* **15,** 8 (1964).
(33P) Wood, D. J , *et al.*, *Ibid.*, **15,** 14 (1964).
(34P) Wood, D. J., Roberts, E. A. H., *Ibid.*, **15,** 19 (1964).
(35P) Wurziger, J., *Cafe, Cacao, The* **7,** 216 (1963); *C.A.* **60,** 11292*h* (1964).
(36P) Yeransian, J. A., Kadin, H., Borker, E., Stefanucci, A., *J. Assoc. Offic. Agr. Chem.* **46,** 315 (1963).

Ferrous Metallurgy

H. F. Beeghly and L. C. Pasztor

Jones & Laughlin Steel Corp., Pittsburgh, Pa. 15227

THE TREND, noted in our last review, continued toward automation with use of computers and programming to utilize analytical data more effectively in controlling steelmaking processes. A number of the proposed systems are sufficiently proprietary at this time that a fair evaluation of their merits must await publication of supporting data. As a general statement, the value of the systems is contingent on the ability to identify the critical variables of the process and on the ability to deliver samples representing these variables for analysis on a continuous basis. Much need for improvement is evident in these two phases of process control.

This review draws extensively from abstracts, is intended to be selective, and is not a complete compilation of references to published work in the interval from June 1962 to September 1964.

Emission and x-ray spectrometric methods (an increasing number use vacuum or controlled atmospheres to extend the sensitivity range and increase the number of elements that can be handled) with quick sample delivery and reporting equipment continue to handle the major proportion of applied analyses in production laboratories.

Interest continues in perfecting gauges or meters for the continuous measurement of oxygen in high temperature gas streams and in liquid metals. Continuous meters for measuring oxygen in molten ferrous metals would be valuable and their successful development, on which some progress has been made (*140a, 156*), would be a significant contribution to improving process control.

Work on laboratory-type neutron sources has progressed to the stage that several evaluations of the method for measuring the oxygen content of iron base alloys have brought this method nearer to the category of an applied method for metals analysis (*58, 69, 78, 89, 152*). When laboratory neutron sources with sufficient energy and reliability can be obtained for general use, analysis of metals for constituents at a very low level will become feasible and more commonplace in works laboratories. A number of procedures for this purpose were described (*7, 116, 138, 141*).

Valuable reference books (*32, 39, 40, 44, 54a, 91, 115a, 116a, 144, 162, 182, 196, 228*), reviews (*55, 70, 113, 176a, 180, 186, 205*), standard method compilations (*2*), and symposia proceedings were published (*51, 146, 229*). These include discussion of the problematical value of the average chemical analysis and the statistical evaluation of analytical data (*137*).

Metallurgical sampling techniques, especially for molten metals, continue to be in a generally unsatisfactory state despite a number of efforts to improve them (*15, 26, 28, 35, 53, 129, 135, 153, 160, 184, 192*).

ALUMINUM

Procedures for determination of aluminum continue to require separation from interfering elements prior to measurement of the aluminum. Atomic absorption (*155*) and vacuum x-ray spectroscopy (*224*), the latter for aluminum in the 2 to 16% concentration range, were used successfully for determining aluminum.

ANTIMONY

Antimony was determined after extraction from a hydrochloric acid solution with di-isopropyl ether, as the rhodamine-B complex (*106*), and as the triiodide complex after reduction of the iron with ascorbic acid (*168*).

ARSENIC

Separation from interfering elements is necessary in most procedures for determining arsenic. Distillation seems to be the most frequently used (*130*), solvent extraction continues to be investigated (*136*), and x-ray fluorescence was capable of determining concentrations as low as 10 parts per million (*227*).

BISMUTH

Extraction of the bismuth tetraiodate complex into dithiozine (*24*) and ion exchange (*122*) were used to separate bismuth from interferences. The bismuth content was then determined colorimetrically (*24*) or polarographically (*122*).

BORON

A review compared methods for determining boron in steel (*233*). A number of modifications of the methylene blue method were evaluated

(*45, 179, 211*). The different known reagents which do not require sulfuric acid to form color complexes with boron were studied (*62*).

CADMIUM

Polarography was used for the determination of cadmium in stainless steels after a thioacetamide separation followed by extraction to remove copper with Neocuproine in chloroform (*95*).

CALCIUM

Prior to determination of calcium by EDTA titration (*10*) or flame photometry (*60*), iron was extracted as ferric chloride into butyl acetate, amyl acetate, or methyl isobutyl ketone.

CARBON

Carbon analyses continue to be made by direct oxidation at high temperature; chromatographic methods and equipment for measuring the evolved carbon dioxide have come into widespread use in the past two years (*133, 206, 226*) and probably are second only to the vacuum emission direct reading spectrograph for this purpose. Thermal conductivity (*127*), infrared (*217*), and conductometric methods (*175*) are used to a lesser extent. Effects of heat treatment of the specimen on determination of carbon with the vacuum spectrometer were evaluated (*102, 145*).

CHROMIUM

Atomic absorption was recommended for determination of chromium in low alloy steels (*108*). For trace amounts (0.002 to 0.030%) the diphenylcarbazide color reaction was studied (*187*). X-ray spectroscopic methods were evaluated for measuring the chromium in alloy steels (*81–2*).

COBALT

Procedures useful for the separation and determination of cobalt were discussed in a brief review of the analytical reactions of cobalt (*218*). Solvent extraction is one of the most frequently used methods for separating cobalt from interfering elements (*142, 149*). Atomic absorption spectroscopy was found useful in determining cobalt at concentrations as low as 0.001% (*143*).

COPPER

The copper-*N*-acetylanabasine-thiocyanate complex was extracted into chloroform and its color intensity was measured at 410 mμ (*212*). Atomic absorption was found to be an acceptable method for determining minor amounts of copper in steel (*107*).

GASES AND NONMETALLIC COMPOUNDS

Gases. With the demand for closer control of steel properties and commercial use of vacuum steelmaking methods, there has come a greatly increased recognition of the value of accurate methods for determining the gas content of steel.

Vacuum fusion remains the major method for determining the contents of oxygen, hydrogen, and nitrogen in a single sample (*46, 56, 59, 120, 148, 177*). More generally, in practice, a different method is used for determination of each gas. Probably more determinations of hydrogen are made with the hot extraction method, of oxygen by the inert (carrier gas) method, and of nitrogen by the wet (Kjeldahl) method than are made by vacuum fusion determinations.

Chromatography has become very commonly used to trap and measure the gases liberated by heating metal in vacuum (vacuum fusion) (*50, 177*) or in an inert gas (carrier gas) (*17, 77, 86*).

Sampling problems pose a serious limitation on the value of analyses of bath samples from the molten steel bath and work has continued to devise better sampling methods (*77*).

Hydrogen. Hot extraction of hydrogen in vacuum and by means of an inert carrier gas remain the principal methods used for determination of hydrogen. Chromatography, frequently with a thermal conductivity detector, appears to have gained on other methods for measuring the hydrogen liberated from the sample (*13, 183, 238*).

A number of evaluations of methods for determining hydrogen were reported (*1, 98, 139, 180, 232, 240*).

Sampling and storage of samples to be analyzed for hydrogen poses a special problem because of the relatively rapid loss of abnormal hydrogen contents from steels on standing at room temperature (*1, 11, 23, 34, 134, 214*).

Nitrogen. Of the steps in determining nitrogen in metals, the decomposition step is the source of most problems. Studies intended to improve this step were reported for both the chemical (*12, 96, 115, 140, 165*) and hot extraction (*47, 49, 96, 140, 163, 165, 207*) procedures. Complete recovery of nitrogen from high silicon (electrical) steels seems to be especially difficult.

Oxygen. An abstract-bibliography on determination of oxygen in metals was published (*126*). Vacuum fusion methods continue to be the subject for study and modification. Most modifications have been in the methods for recovering and measuring the evolved gas (*19, 37, 84, 185, 215*). Direct current arc heating in an argon atmosphere was used to liberate oxygen from samples as carbon monoxide which was then measured by means of its ultraviolet absorption (*84*).

Neutron activation, based on the $O^{16}(n,p)N^{16}$ reaction, has continued to show the promise indicated in previous reviews as a potentially valuable method for determining oxygen. Ultimately, the value of this method for works laboratory and plant use will depend on the success with which neutron sources can be provided with the dependability and output necessary for service under production conditions. Encouraging progress has been made.

Electrochemical methods, well established for determining oxygen in water and low temperature gases, are being used successfully for determining oxygen in gases at high temperature and are being re-examined for direct, continuous measurement of oxygen in liquid metals (*156*). For the latter use, they have not reached the applied stage.

Nonmetallic Compounds. Summaries outlining the techniques and state-of-the-art of work on isolation of nonmetallic compounds from steel (*29, 31, 79, 167, 234*) reveal few new techniques. Applications, with re-discovery and improvement of established methods, have increased.

Mechanical methods for removing segregated areas from metals for analysis were used (*189, 221–2*).

Displacement (*72, 109*), aqueous iodine (*71*), aqueous hydrochloric acid, electrolytic (*41, 63, 101, 109–12, 154, 190–1, 197*) and chlorination (*159*) were investigated for isolating different nonmetallic compounds.

Differential thermal analysis was used to identify compounds isolated from steels (*5*).

Activation analysis (*31*) and tracer techniques (*29, 174*) were used in studying nonmetallic compounds.

HAFNIUM

A colorimetric method using xylenol orange was evaluated for determining hafnium in steel (*150*).

LEAD

Comparison of the ASTM and British Standard 1121 procedures for determining lead was made (*27*). Careful control of pH was found to be essential to quantitative precipitation of lead as the sulfide.

MAGNESIUM

The magnesium–titan yellow and magnesium–eriochrome Black T color complexes were made the basis for colorimetric determination (*3, 188*). The x-ray spectrometer was used for determining the magnesium in nodular cast iron (*161*).

MANGANESE

Methods for determining manganese were evaluated critically (*203*). X-ray spectroscopy was used for determination of manganese in ferromanganese (*83*).

MOLYBDENUM

Molybdenum in ferro-molybdenum was determined by EDTA titration (*43, 99, 118*).

NIOBIUM

Niobium, after separation from interfering elements by precipitation with phenylarsonic acid, was determined by means of the niobium–thiocyanate color complex (*231*). The color reaction of niobium with quinol was the basis for two other color methods that were studied (*14, 100*). After separation from iron by hydrolysis, niobium was determined polarographically (*121*).

PHOSPHORUS

Extraction of the ammonium phosphomolybdate into a mixture of ethyl and butyl acetates was basis for a rapid phosphorus determination method (*67*). The gravimetric determination of phosphorus as the quinoline molybdate was said to give reproducible results (*125*). Phosphates on steel surfaces were determined by use of the molybdenum blue reaction with a cellophane printing technique (*164*).

RARE EARTHS

Cerium was determined colorimetrically as the yellow citric acid complex (*236*), the red *o*-dianisidine complex (*216*), or the *o*-tolidine complex (*151*) after separation from interferences.

Spectroscopic methods were used for determination of cerium and other rare earths (*202, 209–10*). The iron-arc spectrum lines of rare earth elements in the 2679- to 4550-A. region were compiled (*92*).

SILICON

The conditions necessary for the accurate determination of silicon by titration of the potassium silicofluoride complex were studied (*172*). A method for separation of silicon from interferences in the sample by diffusion was described (*88*). X-ray spectrometry was used for the determination of silicon (*237*). Thermoelectric potential measurements were evaluated for determination of silicon (*4, 119, 219*).

SPECTROMETRY

Emission. Emission spectrometry has held its position as the mainstay in works laboratories for analyses and production control (*74, 85, 230*). At present, its major competitor is x-ray spectrometry. Vacuum and controlled atmosphere equipment and other apparatus refinements have strengthened the position of both by extending to higher concentration levels the applicability of emission equipment and by enabling x-ray equipment to handle lower concentration ranges and lighter elements.

Wide variation in composition and physical form of the matrix and large differences in the concentration range of major alloying constituents of alloys are sources of the principal problems in quantitative emission spectrometry.

Much of the work reported has been concerned with efforts to avoid or circumvent these limitations (*16, 22, 33, 35, 75, 147*). The many specialized applications of emission spectroscopy include analysis of high purity iron (*128*), detection and determination of elements in segregated areas (*18*), determination of trace constituents (*128*), determination of gases in metals (*8, 21, 68, 73, 178, 194–5, 199*), and analysis of ceramics (*158*).

Mass. The mass spectrometer provides a highly sensitive method for determining the constituents of metal samples (*80, 114*). Further refinements in excitation and simplification will be necessary before the method will be generally used in works laboratories. Possibly vacuum steelmaking could be considered its first use in production control.

X-Ray. In many laboratories, x-ray spectrometry is supplementing or, occasionally, replacing emission spectrometry for determination of major alloying constituents. One of the necessary steps generally is development of working curves to correct for inter element effects (*30, 83, 176, 193, 208*).

Methods of sample preparation have been developed for different types of samples (*48, 131, 200*).

SULFUR

Methods designed to yield a stoichiometric recovery of sulfur by the combustion method were proposed (*65, 87*). Use of sulfur-35 to check recovery of sulfur by the combustion method was evaluated (*42*). Variations of the evolution method in which the sample was dissolved in hydrochloric acid and sulfur evolved as hydrogen sulfide were tried (*103, 204, 220*).

Several techniques were studied for determination of small amounts of sulfur (*20, 54, 104, 204*). The gravimetric method was re-evaluated in an effort to devise an absolute method for determining sulfur (*9, 170*).

TANTALUM

Tantalum was determined in ferroalloys (*198*) and in steel (*138*) by neutron activation.

TELLURIUM

Tellurium was determined by means of its color reaction with thiourea after separation of iron by extraction into butyl acetate and further separation of tellurium by extraction into isobutyl methyl ketone from a $5N$ hydrochloric acid solution (*223*).

TIN

Tin was determined by titration with thorium nitrate after co-precipitation with beryllium and complexing with EDTA (*225*). In ferro-tungsten (*93*) and in steel (*166*), tin was determined by means of its color reaction with phenylfluorone. Neutron activation was investigated for determination of tin (*76*).

TITANIUM

Iron and aluminum were complexed with triethanolamine and titanium precipitated and was determined as the oxide or by EDTA titration (*171*).

Chromotropic acid (*201*), quinol (*235*); diantipyrinylmethane (*169, 213*), 3,4,5-trihydroxybenzonilide (*123*), 3,6-dichlorochromotropic acid (*6*), and 5,6-dihydroxybenzene-1,3-disulfonate (*105*) were used in the colorimetric determination of titanium.

After separation from iron, titanium was determined polarographically (*57, 97*). Ion exchange was used to separate titanium, tantalum, zirconium, tungsten, niobium, and molybdenum (*36*).

TUNGSTEN

After precipitation of tungsten by addition of excess lead nitrate, the excess lead was back titrated with EDTA (*117*) to determine tungsten in steels and ferro tungsten. Tungsten, extracted with 8-hydroxyquinoline in chloroform (*38, 132*) or with thiocyanate–methyl isobutyl ketone (*94*) was determined colorimetrically.

URANIUM

A survey outlining different methods for determining uranium in iron base alloys was published (*90*). After separation by ion exchange (*52*) or solvent extraction (*66*), uranium was determined by means of the uranium–peroxide color complex.

VANADIUM

The color reactions of vanadium with N - benzoyl - N - phenyl - hydroxylamine (*61*) and with diphenylamine (*239*) were used for its determination.

ZINC

Iron was separated from zinc in a $2M$ hydrochloric acid solution by ion exchange and the zinc was determined polarographically (*157*) or by titration with EDTA (*173*). X-ray spectrometry was used to determine zinc in powder samples (*25*).

ZIRCONIUM

X-ray spectrometry (*181*) and the color reaction of zirconium with catechol violet were used for its determination (*64, 124*).

LITERATURE CITED

(1) Acosta, R. S., *Inst. Hierro Acero* **16,** 173 (1963).

(2) Am. Soc. Testing and Materials, Comm. E-3 on Chemical Analysis of Metals, ASTM, Philadelphia, Pa., 1964.

(3) Babko, A. K., Lutokhina, N. V., *Ukr. Khim. Zh.* **28,** 389 (1962).

(4) Baecklund, J., *Advan. X-Ray Anal.* **6,** 328 (1963).

(5) Bandi, W. R., Karp, H. S., Straub, W. A., Melnick, L. M., *Talanta* **11,** 1327 (1964).

(6) Basargin, N. N., Tkachenko, A. N., Stupa, L. R., Borodaevskaya, L. N., *Zavodsk. Lab.* **28,** 1311 (1962).

(7) Benson, P. A., Gleit, C. E., ANAL. CHEM. **35,** 1029 (1963).

(8) Berneron, R., *Centre Doc. Sider. Circ.* **20,** 743 (1963).

(9) Bertoldi, S., Grosso, O., Casalvolone, F., *Chim. Anal.* (*Paris*) **45,** 560 (1963).

(10) Bezuglyi, D. V., Amsheeva, A. A., *Zh. Analit. Khim.* **17,** 1045 (1962).

(11) Blake, P. D., Pumphrey, W. I., *Brit. Welding J.* **9,** 594 (1962).

(12) Blazejak-Ditges, D., *Radex Rundschau* **1,** 335 (1963).

(13) Boenisch, E., *Materialpruefung* **4,** 247 (1962).

(14) Brhacek, L., Kurzova, K., *Hutnicke Listy* **18,** 594 (1963).

(15) Brown, E. E., Cline, J. C., *J. Metals* **15,** 912 (1963).

(16) Brown, J. R., Wilkinson, D., *Spectr. Met. Ind., Papers Buxton Symp., 2nd, Derbyshire, Eng., 1962,* 55 (1963).

(17) Bryan, F. R., Bonfiglio, S., *J. Gas Chromatog.* **2,** 97 (1964).

(18) Bulat, N. L., Korotkov, V. F., Faivilevich, G. A., Zhurenkov, P. M., *Sb. Tr. Tsentr. Nauch.-Issled. Inst. Chernoi Met.* **31,** 34 (1963).

(19) Burden, J. P., Masson, C. R., *J. Iron Steel Inst.* (*London*) **202,** 28 (1964).

(20) Burke, K. E., Davis, C. M., ANAL. CHEM. **34,** 1747 (1962).

(21) Buyanov, N. V., Fedorova, L. M., *Sb. Tr. Tsentr. Nauch.-Issled. Inst. Chernoi Met.* **24,** 105 (1962).

(22) Calker, van, J., Tappe, W., *Arch. Eisenhuttenw.* **34,** 679 (1963).

(23) Calmettes, J., Glenat, L., Bertin, J., *Centre Doc. Sid. Circ.* **20,** 749 (1963).

(24) Carlström, C. G., Pälvärinne, V., *Jernkontorets Ann.* **146,** 453 (1962).

(25) Carr-Brion, K. G., *Analyst* **89,** 346 (1964).

(26) Cavalier, G., *Rev. Met.* (*Paris*) **61,** 221 (1964).

(27) Claassen, A., Hastings, L., *Analyst* **88,** 67 (1963).

(28) Clarke, W. E., *BCIRA J.* **11,** 231 (1963).

(29) Cochran, A. A., Miller, V. R., *U.S. Bur. Mines, Rept. Invest.* **6256** (1963).

(30) Creton, F., Moschin, B., *Met. Ital.* **55,** 425 (1963).

(31) Crussard, C., *Berg-Huettenmaenn. Monatsh. Montan. Hochschule Leoben* **107,** 1 (1962).

(32) Dal Nogare, S., Juvet, R., "Gas-Liquid Chromatography: Theory and Practice," Interscience, New York, 1962.

(33) DeLeo, V., *Met. Ital.* **54,** 355 (1962).

(34) Dickens, P., *Stahl Eisen* **84,** 776 (1964).

(35) Dickens, P., Koenig, P., Dippel, T., *Arch. Eisenhuttenw.* **34,** 243 (1963).

(36) Dixon, E. J., Headridge, J. B., *Analyst* **89,** 185 (1964).

(37) Donovan, P. D., Evans, J. L., Bush, G. H., *Analyst* **88,** 771 (1963).

(38) Eberle, A. R., ANAL. CHEM. **35,** 669 (1963).

(39) Elving, P. J., Kolthoff, I. M., eds., "Chemical Analysis," Vol. XVIII, Interscience, New York, 1963.

(40) Elwell, W. T., Wood, D. F., "The Analytical Chemistry of Niobium and Tantalum," Imperial Chemical Industries, Birmingham, England, 1962.

(41) Emel'yanenko, Yu. G., Prokhorenko, K. K., Tyutina, E., *Vopr. Proizv. Stali, Akad. Nauk Ukr. SSR, Otd. Tekhn. Nauk* **9,** 73 (1963).

(42) Endo, T., *Nippon KoKan Tech. Rept.,* May, 111 (1963).

(43) Endo, Y., Higashimori, T., *Japan Analyst* **11,** 1310 (1962).

(44) Erdey, Laszlo, "Gravimetric Analysis," Vol. I, Macmillan, New York, 1963.

(45) Eremin, Y. G., Romanov, P. N., *Zavodsk. Lab.* **29,** 420 (1963).

(46) Etterich, O., Taxhet, H., Tomich, W., *Arch. Eisenhuttenw.* **35,** 613 (1964).

(47) Evens, F. M., Fassel, V. A., ANAL. CHEM. **35,** 1444 (1963).

(48) Fahlbusch, W. A., *Appl. Spectr.* **17,** 72 (1963).

(49) Fassel, V. A., Evens, F. M., Hill, C. C., ANAL. CHEM. **36,** 2115 (1964).

(50) Feichtinger, H., Bachtold, H., Brauner, K., *Schweiz. Arch. Angew. Wiss. Tech.* **29,** 125 (1962).

(51) Ferraro, J. R., Ziomek, J. S., eds., "Developments in Applied Spectroscopy," Proceedings of 13th Symposium of Spectroscopy, Plenum Press, New York, 1963.

(52) Ferraro, T. A., Czyrklis, W. F., *Watertown Arsenal, Tech. Rept.* **WAL-TR-823/3,** 1962.

(53) Fiala, A., *Hutnicke Listy* **17,** 581 (1962).

(54) Fischer, W., Bastius, H., Mehlhorn, R., *Neue Huette* **8,** 35 (1963).

(54a) Freiser, Henry, Fernando, Quintus, "Ionic Equilibria in Analytical Chemistry," Wiley, New York, 1963.

(55) Frost, B. R. T., *J. Nucl. Mater.* **7** (2) 109 (1962).

(56) Fuchs, A., Reinhard, H., Niebuhr, J., Beck, R., *Arch. Eisenhuttenw.* **34,** 361 (1963).

(57) Fukui, S., Arita, T., *Japan Analyst* **11,** 295 (1962).

(58) Gilmore, J. T., Hull, D. E., ANAL. CHEM. **35,** 1623 (1963).

(59) Gokcen, G. A., Tankins, E. S., *J. Metals* **14,** 584 (1962).

(60) Goto, H., Ikeda, S., Kimura, J., *Sci. Rept. Res. Inst., Tohoku Univ., Ser. A.* **15A,** 155 (1963).

(61) Goto, H., Kakita, Y., *Japan Analyst* **10,** 904 (1961).

(62) Goward, G. W., Wiederkehr, V. R., ANAL. CHEM. **35,** 1542 (1963).

(63) Gras, W. D., Wittmoser, A., *Arch. Eisenhuttenw.* **34,** 527 (1963).

(64) Green, H., *BCIRA J.* **10,** 586 (1962).

(65) *Ibid.,* **11,** 76 (1963).

(66) *Ibid.,* **12,** 632 (1964).

(67) Gregorczyk, S., *Hutnik* **29,** 369 (1962).

(68) Grigorova, V. S., Lindrstrem, I. S., Sventitskii, N. S., Sukhenko, K. A., *Izv. Akad. Nauk SSSR, Ser. Fiz.* **26,** 924 (1962).

(69) Gruverman, I. J., Henninger, W. A., ANAL. CHEM. **34,** 1680 (1962).

(70) Guenther, K. H., *Stahl Eisen* **83,** 1513 (1963).

(71) Gurevich, A. B., Kirzhnev, O. M., Sandler, N. I., Murav'ev, V. N., *Zavodsk. Lab.* **29,** 283 (1963).

(72) Gurevich, A. B., Turubiner, L. M., *Zavodsk. Lab.* **29,** 280 (1963).

(73) Gwilliam, L., *Spectr. Met. Ind., Papers Buxton Symp., 2nd, Derbyshire, Eng., 1962,* 32 (1963).

(74) Hagenah, W. D., Laqua, K., *Z. Anal. Chem.* **198,** 88 (1963).

(75) Hagihara, T., Harada, M., *Nisshin Seiko Giho* **7,** 52 (1962).

(76) Hamaguchi, H., Kawabuchi, K., Onuma, N., Kuroda, R., *Anal. Chim. Acta* **30,** 335 (1964).

(77) Hanin, M., Jaudon, E., *Chim. Anal.* **45,** 623 (1963).

(78) Harris, W. F., *Talanta* **11,** 1376 (1964).

(79) Hasegawa, M., Uchiyama, I., *Tetsu To Hagane* **3,** 342 (1963).

(80) Hintenberger, H., *Arch. Eisenhuttenw.* **33,** 355 (1962).

(81) Hirokawa, K., Goto, H., *Sci. Rept. Res. Inst., Tohoku Univ., Ser. A* **14,** 278 (1962).

(82) *Ibid.,* p. 294.

(83) Hirokawa, K., Shimanuki, T., Goto, H., *Sci. Rept. Res. Inst., Tohoku Univ., Ser. A* **15,** 124 (1963).

(84) Höller, P., *Arch. Eisenhuttenw.* **34,** 425 (1963).

(85) Höller, P., Dick, W., *Stahl Eisen* **83,** 1490 (1963).

(86) Holt, B. D., Goodspeed, H. T., ANAL. CHEM. **35,** 1510 (1963).

(87) Horn, H., Kendler, E., *Neue Huette* **8,** 31 (1963).

(88) Householder, R., Russell, R. G., ANAL. CHEM. **36,** 2279 (1964).

(89) Houseman, D. H., *Steel and Coal* **186,** 896 (1963).

(90) Ingles, J. C., Zimmermann, J. B., Horwood, J. L., *Can. Dept. Mines Tech. Surv. Res. Rept.* **R. 95,** April, 259 (1962).

(91) International Union of Pure and Applied Chemistry, "Commission on Spectrochemical and Other Optical Procedures, I. Tables of Spectrophotometric Absorption Data of Compounds Used for the Colorimetric Determination of Elements," Butterworth, London, 1963.

(92) Intonti, R., Cecchetti, G., *Met. Ital.* **55,** 378 (1963).

(93) Jankovsky, J., *Hutnicke, Listy* **18,** 276 (1963).

(94) Kakita, Y., Goto, H., *Sci. Rept. Res. Inst., Tohoku Univ., Ser. A* **15,** 1 (1963).

(95) Karp, H. S., Krapf, G., *Talanta* **11,** 1365 (1964).

(96) Karp, H. S., Lewis, L. L., Melnick, L. M., *J. Iron Steel Inst.* (*London*) **200,** 1032 (1962).

(97) Kawahata, M., Mochizuki, H., Kajiyama, R., Irikura, K., *Japan Analyst* **11,** 312 (1962).

(98) Kawahata, M., Mochizuki, H., Kajiyama, R., Ishii, M., *Ibid.,* **12,** 296 (1963).

(99) *Ibid.,* **11,** 748 (1962).

(100) Kawahata, M., Mochizuki, H., Misaki, T., *Ibid.,* p. 188.

(101) Kawamura, K., Iwamoto, M., *Tech. Rep. Fuji Iron Steel Co.,* **12,** 186 (1963).

(102) Kawamura, K., Kitayama, M., Ichijima, I., *Ibid.,* March, 67 (1963).

(103) Kaya, A., Kakita, Y., *J. Japan Inst. Metals* (*Sendai*) **26,** 522 (1962).

(104) *Ibid.,* **27,** 28 (1963).

(105) Kenna, B. T., Conrad, F. J., ANAL. CHEM. **35,** 1255 (1963).

(106) Kidman, L., Waite, C. B., *Metallurgia* **66,** 143 (1962).

(107) Kinson, K., Belcher, C. B., *Anal. Chim. Acta* **30,** 180 (1963).

(108) Kinson, K., Hodges, R. J., Belcher, C. B., *Ibid.,* **29,** 134 (1963).

(109) Klyachko, Yu. A., Shapiro, M. M., Yakovleva, E. F., *Sb. Tr. Tsentr. Nauchn.-Issled. Inst. Chernoi Met.* **24**, 75 (1962).

(110) *Ibid.*, p. 45.

(111) Klyachko, Yu. A., Yakovleva, E. F., *Ibid.*, p. 30.

(112) Klyachko, Yu. A., Yakovleva, E. F., *Ibid.*, **31**, 135 (1963).

(113) Koch, O. G., *Stahl Eisen* **83**, 456 (1963).

(114) Koch, W., Sauer, K. H., *Arch. Eisenhuttenw.* **35**, 861 (1964).

(115) Koch, W., Sauer, K. H., *Z. Anal. Chem.* **190**, 81 (1962).

(115a) Kodama, Kazunobu, "Methods of Quantitative Inorganic Analysis" Wiley, New York, 1963.

(116) Kohn, A., *Chim. Anal.* **44**, 425 (1962).

(116a) Kolthoff, I. M., Elving, P. J., eds. "Treatise on Analytical Chemistry," Part I, Vol. 3 (1963), Vol. 4 (1964), Part II, Vol. 6 (1964), Vol. 8 (1963), Wiley, New York.

(117) Konkin, V. D., Zhikhareva, V. I., *Sb. Tr. Ukr. Nauchn.-Issled. Inst. Met.* **8**, 338 (1962).

(118) Konkin, V. D., Zhikhareva, V. I., *Zavodsk. Lab.* **29**, 791 (1963).

(119) Krajina, A., Dolezal, J., *Talanta*, **11**, 1127 (1964).

(120) Kraus, T., Winkler, O., *Rev. Met.* **61**, 87 (1964).

(121) Kurbatov, D. I., Skorynina, I. S., *Zavodsk. Lab.* **28**, 1061 (1962).

(122) Kurobe, M., Tereda, H., Tajima, N., *Japan Analyst* **12**, 4 (1963).

(123) Lapin, N. N., Efimenko, A. G., *Sb. Nauch. Tr. Zhdanovsk. Met. Inst.* **9**, 103 (1963).

(124) Leeb, A. J., Hecht, F., *Radex Rundschau* **1**, 360 (1963).

(125) Lench, A., Anal. Chem. **35**, 1695 (1963).

(126) LePape, M., Platzer, R., *Comm. Energie At.* (*France*), *Ser. Bibl.* **No. 23** (1962).

(127) Lewis, L. L., Nardozzi, M. J., Anal. Chem. **36**, 1329 (1964).

(128) Lifshits, E. V., Mosova, L. N., *Zovodsk. Lab.* **28**, 1329 (1962).

(129) Loader, J., Webb, C., *Foundry Trade J.* **114**, 63 (1963).

(130) Ludemann, K. F., Zimmermann, R., Wemme, H., *Neue Huette* **8**, 418 (1963).

(131) Luke, C. L., Anal. Chem. **35**, 56 (1963).

(132) Luke, C. L., *Ibid.*, **36**, 1327 (1964).

(133) Lysyj, I., Offner, H. G., Bedwell, V. E., *J. Chromatog.* **11**, 320 (1963).

(134) Maekawa, S., Nakagawa, Y., Matsumi, S., *Imono* **35**, 20 (1963).

(135) *Ibid.*, p. 482.

(136) Maekawa, S., Yoneyama, Y., Fujimori, E., *Japan Analyst* **11**, 493 (1962).

(137) Malissa, M., Swoboda, K., *Radex Rundschau* 494 (1963).

(138) Marchart, H., Hecht, F., Wetternick, L., Plöckinger, E., *Ibid.*, 55 (1964).

(139) Martin, J. F., Takacs, R. C., Rapp, R., Melnick, L. M., *AIME Trans.* **230**, 107 (1964).

(140) Masson, C. R., Pearce, M. L., *Ibid.*, **224**, 1134 (1962).

(140a) Matsushita, Yukio, Goto, Kazuhiro, *Tetsu To Hagane Overseas* **4**, 128 (1964).

(141) May, S., Pinte, G., *Bull. Soc. Chim. France* **2**, 287 (1962).

(142) McClellan, B. E., Benson, V. M. Anal. Chem. **36**, 1985 (1964).

(143) McPherson, G. L., Price, J. W., Scaife, P. H., *Nature* **199**, 371 (1963).

(144) Meites, L., "Handbook of Analytical Chemistry," McGraw-Hill, New York, 1963.

(145) Mints, I. M., *Zavodsk. Lab.* **29**, 555 (1963).

(146) Mueller, W. M., Fae, M., eds., "Advances in X-ray Analysis," Vol. 6, Proc. of XI (1962) Conference, Plenum Press, New York, 1963.

(147) Muir, S., Ambrose, A. D., Swingler, D. W., *Metallurgia* **67**, 251 (1963).

(148) Muramatsu, K., *Trans. Japan Inst. Metals* **3**, 137 (1962).

(149) Namiki, M., Kakita, Y., Goto, H., *Sci. Rept. Res. Inst., Tohoku Univ., Ser. A* **14**, 309 (1962).

(150) Narita, K., Nesu, S., *Tetsu To Hagane* **49**, 631 (1963).

(151) Nechaeva, E. A., Kholodkova, T. M., *Zavodsk. Lab.* **29**, 803 (1963).

(152) Nickel, H., Rottmann, J., Stocker, H. J., Koster-Pflugmacher, A., Frohberg, M. G., *Arch. Eisenhuttenw.* **35**, 637 (1964).

(153) Nicoloiu, E., Fruchter, S., *Metalurgia* **15**, 690 (1963).

(154) Nicoloiu, E., Fruchter, S., *Met. Constructia Masini* **14**, 1125 (1962).

(155) Nikolaev, G. I., Aleskovskii, V. B., *Zh. Analit. Khim.* **18**, 816 (1963).

(156) Ohtani, M., Sanbongi, K., *Tetsu To Hagane* **49**, 22 (1963).

(157) Okubo, T., Uehara, F., *Japan Analyst* **11**, 761 (1962).

(158) O'Neil, R. L., Mattarella, S. P., *Ceramic Age* **79** (6) 33 (1963).

(159) Opravil, O., Borovsky, M., Svatik, I., Pazitny, J., *Hutnicke Listy* **18**, 52 (1963).

(160) Padget, G., *Steel Coal* **187**, 648 (1963).

(161) Papp, L., Váralljay, L., *Kohász. Lapok, Öntöde* **95**, 278 (1962).

(162) Parr, N. L., ed., "Laboratory Handbook," George Newnes Ltd., London, 1963.

(163) Parsons, M. L., Pennington, S. N., Walker, J. M., Anal. Chem. **35**, 842 (1963).

(164) Pasztor, L. C., *Metal Finishing* **61** (9), 72 (1963).

(165) Pearce, M. L., *AIME Trans.* **227**, 1393 (1963).

(166) Picasso, G., Pizzimenti, A., *Met. Ital.* **55**, 361 (1963).

(167) Pickering, F. B., paper from "Clean Steel," Iron and Steel Institute, London, 1962.

(168) Ploum, H., *Arch. Eisenhuttenw.* **33**, 601 (1962).

(169) Polyak, L. Ya., *Zh. Analit. Khim.* **17**, 206 (1962).

(170) Ponomarev, A. I., Astanina, A. A., *Tr. Inst. Met. Akad. Nauk. SSSR* **10**, 231 (1962).

(171) Pribil, R., Vesely, V., *Talanta* **11**, 1197 (1964).

(172) Prumbaum, R., *Giesserei* **49**, 855 (1962).

(173) Répás, P., Sajó, I., *Kohász. Lapok* **7**, 326 (1963).

(174) Richardson, H. M., *I.S.I. Spec. Rept.* **77**, 57 (1963).

(175) Rickard, E. F., *Analyst* **89**, 235 (1964).

(176) Rickenbach, J. R., *Advan. X-Ray Anal.* **6**, 352 (1963).

(176a) Reilley, C. N., ed., "Advances in Analytical Chemistry and Instrumentation," Vol. 2 (1963), Vol. 3 (1964), Wiley, New York.

(177) Roff, J. A., *Chem. Ind.* **13**, 537 (1963).

(178) Romand, M. J., Berneron, M. R., *Publ. Group Avan. Methodes Spectrog.* **1963**, 327.

(179) Rosotte, R., *Chim. Anal.* **44**, 208 (1962).

(180) Rothmann, H., Keil, W., Richter, H., *Z. Erzbergbau Metallhuettenw.* **16**, 239 (1963).

(181) Rudolph, J. S., *Talanta* **10**, 215 (1963).

(182) Samuelson, O., "Ion Exchange Separations in Analytical Chemistry," Wiley, New York, 1963.

(183) Sandrinelli, R., Guarinieri, E., *Met. Ital.* **55**, 152 (1963).

(184) Sato, S., *Denki Seiko* **34**, 217 (1963).

(185) Schenck, H., Frohberg, M., Schmitz, K., *Stahl Eisen* **83**, 162 (1963).

(186) Schoefmann, E., *Ibid.*, p. 605.

(187) Scholes, P. H., Smith, D. V., *Metallurgia* **67**, 153 (1963).

(188) Schwarz, H., *Radex Rundschau* 606 (1963).

(189) Segawa, K., Futamura, E., Shimada, H., Oba, N., Yokooji, T., *Tetsu To Hagane* **48**, 1297 (1962).

(190) Segawa, H., Shimada, H., *Ibid.*, p. 1562.

(191) *Ibid.*, p. 1679.

(192) Senior, B. H., *Metal Progr.* **83**, 108 (1963).

(193) Sermin, D. F., *Steel & Coal* **187**, 116 (1963).

(194) Shubina, S. B., Shaevich, A. B., Dement'eva, V. G., *Zavodsk. Lab.* **29**, 552 (1963).

(195) Shubina, S. B., Shaevich, A. B., Kilina, S. I., Mel'nikov, S. I., Bazanova, L. A., *Ibid.*, **28**, 942 (1962).

(196) Shumilovskii, N. N., Mel'hser, L. V., "Radioactive Isotopes in Instrumentation and Control," Pergamon Press, Oxford, England, 1964.

(197) Sicha, M., *Hutnicke Listy* **17**, 537 (1962).

(198) Simkova, M., *Ibid.*, **18**, 357 (1963).

(199) Skotnikov, S. A., *Tr. Inst. Met., Akad. Nauk SSSR* **15**, 43 (1963).

(200) Smith, G. S., *Chem. Ind., London,* **1963**, 907.

(201) Sommer, L., *Collection Czech. Chem. Commun.* **27**, 2212 (1962).

(202) Sorokina, N. N., Golubera, V. M., *Sb. Tr. Tsentr. Nauchn.-Issled. Inst. Chern. Met.* **31**, 41 (1963).

(203) Spauszus, S., *Neue Huette* **8**, 108 (1963).

(204) Spaszus, S., Schwarz, C., *Ibid.*, p. 40.

(205) Stricker, F., *Stahl Eisen* **83** (16) 990–2 (1963).

(206) Stuckey, W. K., Walker, J. M., Anal. Chem. **35**, 2015 (1963).

(207) Subat, G., *Schweiz. Arch.* **29**, 338 (1963).

(208) Sugimoto, M., *Japan Analyst* **11**, 1273 (1962).

(209) Takao, Z., Kawaguchi, T., Kudo, Y., Matsumura, T., *Tetsu To Hagane* **48**, 1511 (1962).

(210) Takao, Z., Kawaguchi, T., Kudo, Y., Matsumura, T., *Ibid.*, **49**, 1513 (1963).

(211) Takeyama, S., Goto, H., *Sci. Rept. Res. Inst., Tohoku Univ., Ser. A* **15**, 144 (1963).

(212) Talipov, Sh. T., Nigai, K. G., Abramova, E. L., *Zavodsk. Lab.* **29**, 804 (1963).

(213) Tananaiko, M. M., Nebylitskaya, S. L., *Ibid.*, **28**, 263 (1962).

(214) Taxhet, H., Thomich, W., Winterfeld, F., *Stahl Eisen* **82**, 1561 (1962).

(215) Teske, K., Hundrieser, A., *Neue Huette* **8**, 370 (1963).

(216) Thomann, H. J., Junghans, U., *Ibid.*, **7**, 421 (1962).

(217) Tipler, G. A., *Analyst* **88**, 272 (1963).

(218) Tombu, C., *Cobalt* **1963**, 103.

(219) Tumanov, A. K., Chabanenko, N. I., *Zavodsk. Lab.* **29**, 627 (1963).

(220) Turina, S., Marjanovic-Krajovan, V., *Z. Anal. Chem.* **196**, 32 (1963).

(221) Uchiyama, I., Nomura, M., Ueno, M., *Tetsu To Hagane* **47**, 519 (1963).

(222) Uchiyama, I., Nomura, M., Ueno, M., *Trans. Nat. Res. Inst. Metals* (*Tokyo*) **5**, 1 (1963).

(223) Uzumasa, Y., Hayashi, K., Ito, S., *Bull. Chem. Soc. Japan* **36**, 301 (1963).
(224) Wagner, J. C., Bryan, F. R., *Advan. X-Ray Anal.* **6**, 339 (1963).
(225) Wakamatsu, S., *Japan Analyst* **11**, 1151 (1962).
(226) Walker, J. M., Kuo, C. W., ANAL. CHEM. **35**, 2017 (1963).
(227) Watling, J., *U.K. At. Energy Authority, Res. Group* **AERE-R 3974**, 1962.
(228) Welcher, Frank S., ed., "Standard Methods of Chemical Analysis," 6th ed., Vol. II, Van Nostrand, Princeton, N. J., 1963.
(229) West, Philip W., MacDonald, A. M. G., West, T. S., eds., "Analytical Chemistry 1962," Fritz Fiegl 70th Birthday Symp. Papers, Elsevier, Amsterdam, 1963.
(230) Weszpremy, B., *Kohász. Lapok* **96**, 74 (1963).
(231) White, G., Scholes, P. H., *Metallurgia* **70**, 197 (1964).
(232) Wojcik, A., *Hutnik* **30**, 281 (1963).
(233) Yakovlev, P. Y., Kozina, G. V., *Zavodsk. Lab.* **29**, 920 (1963).
(234) Yakovleva, E. F., Belyaeva, V. A., *Sb. Tr. Tsentr. Nauchn.-Issled. Inst. Chernoi Met.* **31**, 129 (1963).
(235) Yamaguchi, N., Hasegawa, M., *Japan Analyst* **11**, 126 (1962).
(236) Yatsyk, I. E., Orzhekhovskaya, A. I., *Sb. Nauchn.-Tekhn. Tr. Nauchn.-Issled. Inst. Met. Chelyabinsk. Sovnarkhoza* **1961**, 205.
(237) Zeuner, H., *Giesserei* **49**, 858 (1962).
(238) Zielinski, E., *Przeglad Odlewnictwa* **13**, 219 (1963).
(239) Zindel, E., Zeiher, R., *Z. Anal. Chem.* **195**, 27 (1963).
(240) Zitter, H., Schwarz, W., *Arch. Eisenhuttenw.* **35**, 109 (1964).

Nonferrous Metallurgy

I. Light Metals

Fritz Will, III

Alcoa Research Laboratories, Aluminum Co. of America, New Kensington, Pa.

THIS is the tenth review on nonferrous metallurgical analysis and covers the two-year period from September 1962 through August 1964, as documented by *Chemical Abstracts* and *Analytical Abstracts.* Also, the following journals were surveyed directly for the same period: ANALYTICAL CHEMISTRY, *Analytica Chimica Acta*, *The Analyst*, and *Talanta.* Not all of the creditable contributions are mentioned, nor are those that are discussed necessarily the most important.

As mentioned in the last nonferrous metallurgy review (*207*), because of the increasing diversity of publications in the field of nonferrous metallurgy, the scope of this review surveys methodology on analysis of only the light structural metals: aluminum, beryllium, magnesium, and titanium. Fifty-five per cent of the references discussed the analysis of aluminum, 14% beryllium; 11% magnesium, and 20% titanium.

In a breakdown of the references as to percentages of the various techniques of analyses mentioned, the following results were found: 27% colorimetric, 22% spectrochemical, 11% activation analysis, 11% classical, 6% gas determinations, 5% polarographic, 4% chelometric, 3% ion exchange, 3% x-ray, 3% flame photometric, 2% atomic absorption, 3% miscellaneous. As in the past, the colorimetric and spectrochemical methods of analyses are predominant with an increasing number of papers on activation analysis. Even though there were only four references to atomic absorption spectroscopy, more papers in this field are anticipated.

Wood, Marron, and Lambert (*211*) discussed the electron-probe microanalysis of anodic oxide films on aluminum alloys. The oxide films formed by anodizing aluminum alloy in 15% H_2SO_4 were subjected to cleaning or sealing treatments and examined by electron probe for aluminum, sulfur, chromium, and nickel. This was the only paper on electron-probe analysis.

Van Sandt *et al.* (*193*) constructed an automatic, direct-reading spectrograph suitable for the analysis of beryllium samples with a commercially available grating monochromator as its basic unit. Specially designed high-purity graphite electrodes were used.

Ivanova, Kovalenko, and Tsyvenkova (*75*) discussed the spectrographic analysis of an aluminum alloy by fractional exposure. During analysis only one standard was employed and its spectra was photographed for various times. Lemieux (*106*) recommended an improved spectrochemical analysis of alumina by mineralized calcination. A procedure which converted samples to essentially alpha alumina was outlined. Calcination with a small amount of aluminum fluoride eliminated many of the striking differences in physical properties of the samples.

Pocze (*149*) and Brune (*15*) summarized the activation analysis of aluminum. Ross (*156*) described the determination of 62 elemental impurities in beryllium, aluminium, and iron by activation analysis. The gamma activities of radionuclides produced by neutron activation were measured.

Kern (*82*) critically reviewed important methods for determining hydrogen, nitrogen, and oxygen in beryllium, magnesium, and titanium, among others. The methods discussed were vacuum melting, carrier gas methods, chemical, spectrographic by a carbon d.c. arc, intermolecular viscosity, mass spectrometry, and activation analysis.

Four recent books concerning in part the analysis of light metals are noteworthy. "Chemical Analysis of Metals," Part 32 of the American Society for Testing and Materials' 1964 Book of Standards (*3*), contains sections on the chemical analysis of aluminum, magnesium, and titanium and their alloys and the spectrochemical analysis of aluminum and its alloys. "The Chemistry of Beryllium" (*39*) includes chapters on chemical reactions of beryllium, extractive metallurgy of beryllium, and analytical chemistry of beryllium. "Treatise on Analytical Chemistry" (*95*), Part II, Vol. 5, discusses the determination of 24 elements in titanium. "Standard Methods of Chemical Analysis" (*50*), Vol. 1, describes the analysis of aluminum, beryllium, magnesium, and titanium.

As in the previous nonferrous review (*207*), the remaining text of this review is arranged alphabetically according to constituents determined. Tables I and II list analytical procedures according to materials analyzed and constituents determined.

Aluminum. Wood, Marron, and Lambert (*211*) reported the electron-probe microanalysis of anodic oxide films on aluminum alloys. Fedorav and Linkova (*41*, *42*) described the determination of alumina in metallic aluminum by hydrochlorination. A quartz boat containing the aluminum was inserted into a molybdenum glass tube. The aluminum was dissolved in a current of hydrogen chloride and hydrogen and was distilled as the chloride. The alumina which remained was dissolved and determined photometrically with aluminon. Iron, manganese, silicon, <0.5% tin, and <2.5% copper did not interfere.

Munshi and Dey (*127*) proposed a paper chromatographic separation and determination of copper and aluminum in aluminum–bronze alloys. The solution of the sample was applied as spots to a strip of Whatman No. 1 paper, and the chromatogram was developed with a butanol–concentrated HCl–water mixture. The strip was cut in two, each piece was extracted with boiling dilute HCl, and the copper and aluminum were determined colorimetrically. Burke and Davis (*18*) utilized CDTA [(1,2,-cyclohexylenedinitrilo)tetracetic acid] for the chelometric determination of aluminum in aluminum-base alloys. An excess of CDTA was added to the solution, the pH was adjusted to 5.5 to 6 with hexamethylenetetramine, and the solution was titrated with zinc using Xylenol Orange as an indicator.

Tikhonov (*179*) recommended the complexometric determination of aluminum in magnesium alloys by titration of the sum of aluminum and zinc at pH 3 with Complexon III in the presence of PAN and a small amount of copper complexonate. The solution was titrated hot with Complexon III until yellow, boiled again, and the titration was finished. The aluminum concentration was calculated after the zinc content was deducted. A photometric method for the determination of aluminum in magnesium metals was developed using 8-hydroxyquinoline (*188*). Iron was complexed with *o*-phenanthroline at pH 5.6 to 6.0.

Malevannyi (*113*) used (4,4′-bis-3,-4-dihydroxyphenylazo)-2,2′.-stilbenedisulfonic acid for the colorimetric determination of aluminum in titanium dioxide pigments. The reagent formed a stable pink complex with Al^{+3}. Ferric iron interfered and was reduced with thiourea. Tikhonov and Grankina (*180*) investigated the complexometric determination of aluminum in titanium slags and concentrates by titration with Complexon III at pH 3 in the presence of PAN as an indicator and copper complexonate. Potassium, sodium, calcium, magnesium, manganese, or chromium did not interfere. The interference of titanium and iron was eliminated by extraction of their cupferronates. Navyazhskaya and Sporykhina (*129*) determined alumina in titanium oxide by titrating the excess of Trilon B added with $0.05N$ $FeCl_3$ at pH 4.5 to 5.0.

Antimony. Vinogradova and Vasil'eva (*196*) reported the determination of small amounts of tin ($3 \times 10^{-5}\%$), bismuth ($2 \times 10^{-6}\%$), and antimony ($3 \times 10^{-5}\%$) in high-purity aluminum by anodic voltammetry at a stationary mercury electrode. Optimum conditions were developed for the separation of these metals at the electrode, followed by the anodic polarization of the amalgam obtained.

Todd, Cuthbert, and Dickinson (*182*) recommended the determination of antimony and cobalt in magnesium-base alloys by neutron activation analysis. The method involved remote irradiation in a nuclear reactor, radiochemical separation of the resulting antimony and cobalt activities, and comparison of these activities with those isolated from standards. Antimony and cobalt were determined down to 0.05 and 0.01 p.p.m., respectively.

Galliford and Yardly (*52*) studied the colorimetric determination of antimony in titanium dioxide based on the compound developed with sodium hexametaphosphate and Brilliant Green. The colored complex was extracted with toluene and the absorbance was measured at 640 mμ. Yakovlev and Malinina (*212*) investigated the polarographic determination of 0.01 to 0.2% antimony in titanium dioxide. Interference by iron was avoided by reduction with ascorbic acid.

Table I. Methods for Nonferrous Metallurgical Materials

A. Activation
At. Atomic absorption
C. Chemical
Col. Colorimetric
F. Flame photometric
P. Polarographic
S. Spectrographic
X. X-ray

Material	Constituents determined	Methods used	References
Aluminum	Cd, Co, Cu, Fe, Zn	Col. P	(*71*)
Aluminum	Cu, Ni, Zn, Fe, Mn, Sn, Si	C	(*37*)
Aluminum	45 elements	A	(*2*)
Aluminum	Sc, Co, Np, Fe, Sn, Cd, Ag, Rb, Ho, Tb, Lu, Ce, Tm, Cr, La, Nd, Yb, Cs, Sm	A	(*55*)
Aluminum	Fe, Co, Zn, Sc	A	(*91*)
Aluminum	Cu, Mn, Zn, Ga, Co, Sc, Fe	A	(*105*)
Aluminum	Cu, Sb, As, Ga, Sc, Fe, Zn, Cd	A	(*94*)
Aluminum	Cu, Zn, Fe, Mn, Pb, Ni, Ag, Bi, Cd, Cr	S	(*19*)
Aluminum	Fe, Si, Mg, Mn, Ti, Cu, Pb, Sn, Ag, Ni	S	(*59*)
Aluminum	34 elements	S	(*29*)
Aluminum	Cu, Fe, Mg, Mn	S	(*171*)
Aluminum alloys	Na, Li, Mg, Ca	F	(*69*)
Aluminum alloys	Cu, Mn, Ni, Fe, Zn, Ti, Cr	X	(*141*)
Aluminum	Zn, Cu, Fe, Mn, V, Ti	X	(*54*)
Aluminum, beryllium	62 elements	A	(*156*)
Aluminum–uranium alloys, titanium–uranium alloys	Al, Fe, Mo, Nb, V, Zr, Cr, Ti, Ni	Col.	(*66*)
Alluminum alloys	Pb, Mg, Zn, Cd, Co, Ca, Ag, Na	At	(*34*)
Aluminum alloys	Si, Mg, Cu, Fe	C	(*51*)
Aluminum alloys	Si, Cu, Mn, Mg, Cr, Zn	Col.	(*20*)
Aluminum alloys	Cr, W, Ti	S	(*9*)
Aluminum alloys	Cu, Mg, Mn, Fe, Si, Ti, Zn, Be, V, Zr, Cr, Na, Pb, Sn	S	(*111*)
Aluminum alloys	Si, Mn, Mg, Cu, Fe, Zn	S	(*24*)
Aluminum oxide	Si, Ti, P, Fe	Col.	(*157*)
Aluminum oxide	Si, Ca, Mg, Fe, Ti, Na, K	C, Col., F	(*99*)
Aluminum oxide	Ni, Cr, Ca, Mg, Fe, Si, Al	C, Col., S, X	(*190*)
Aluminum oxide	Cr, Co, Cu, Fe, Mn, Ni, V	S	(*142*)
Aluminum oxide	Si, Ni, Te, Ni, Ca, Mg	S	(*185*)
Aluminum oxide	Si, Ti, Fe, Ca, Mg, Cr, Pb, Zn, Mn, V, Cu, B, Na, K, Li	S, F	(*195*)
Aluminum oxide	25 elements	S	(*43*)
Aluminum oxide	Si, Fe, V, Zn, Ti	S	(*97*)
Beryllium	Al, Bi, B, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, Si, Ag, Sn, Ti, V	S	(*107*)
Beryllium	Al, V, Mn, Na, K, Cr, Ta	A	(*146*)
Beryllium	Fe, Ni, Cu, Al, Si, Cr, Mn	Col.	(*151*)
Beryllium	Fe, Mn, Mg, Cr, Cu, B	S	(*124*)
Beryllium	Mn, Na, Cu, Cr, Sc, Fe, Zn, Co, Ta, Ni	A	(*104*)
Beryllium oxide	B, Fe, Al, Mg, Ni, Mn, Cr, Ca, Cu, Pb, Si	S	(*123*)
Beryllium oxide	33 elements	S	(*125*)
Aluminum, magnesium	Cu, Fe, Ni, Pb, V, Zn	Col., P	(*56*)
Magnesium–beryllium alloys	Be, Al, Zr, Ca, Cu, Fe, Ni	S	(*36*)
Magnesium	Ag, Cd, Be, Cu, Li, P, Ga, Si, W, Sb, Ni, Sn, Fe, Mn, Na, Co, Bi, In, Mo, Ba, Zn, V, As, Tl, Cr, Sr, Pb, Al	S	(*28*)
Magnesium alloy	Ce, Nd, Pr, La, Zn, Zr	S	(*199*)
Magnesium alloy	Al, Be, Ca, Fe, Si, Zn	S	(*1*)
Titanium	Ta, Mn, Fe, Ni, Al, Cr, Mg, P, Si	C, Col.	(*130*)
Titanium	B, Ba, Fe, Si, Sr	S	(*206*)
Titanium	Al, Cr, Fe, Mn, Mg, Cu, V, Mo, W, Sn, Bi, Pb	S	(*79*)
Titanium, titanium alloys	Fe, Si, Ni, Cr, Mn	S	(*158*)
Titanium alloys	Al, Mo, Cr, Mn, V	S	(*192*)
Titanium dioxide	Cu, Ca, Cr, Al, V, W, Fe, Si, Mg, Mn, Ni, Nb, Sn, Pb	S	(*47*)
Titanium dioxide	Fe, Si, Nb, Ca, V, Mg, Al, P	S	(*176*)
Titanium sponge	Mg, Fe, Mn, Si	S	(*136*)

Arsenic. Steckel and Hall (*167*) reported a spectrophotometric determination of trace arsenic in aluminum and its alloys using silver diethyldithiocarbamate. The procedure included reducing As(V) to As(III), evolving arsenic as AsH_3 into a pyridine solution of the reagent to form the stable red As(III) complex, and measuring the absorbance at 540 mμ. Copper and antimony interfered.

Mikheeva and Wihitill (*117*) proposed the determination of arsenic, phosphorus, and sulfur in beryllium oxide by radioactivation analysis. The sample was activated by a stream of 10^{13} neutrons per sq. cm. per sec. for 10 minutes and kept for 30 hours. The arsenic was reduced with hydroxylamine hydrochloride, precipitated as the sulfide, and measured by radioactivity.

Beryllium. Kida *et al.* (*86*) described the photometric determination of beryllium in copper–beryllium and beryllium–aluminum alloys with 8-hydroxylquinaldine in chloroform at 380 mμ. Copper and aluminum were masked with KCN and EDTA, respectively, at pH 8.0. Postma and McMurray (*152*) utilized activation analysis for the determination of beryllium in beryllium metal by concentrically surrounding an Sb^{124} pencil and counting neutrons produced in beryllium with fission counter tubes.

Bugaeva and Mironenko (*17*) recommended sulfosalicylic acid for the spectrophotometric determination of beryllium in magnesium alloys. Hydroxylamine hydrochloride was added to mask iron and Trilon B to mask magnesium. The complex was developed at pH 10 to 11 and the absorbance was measured at 320 mμ.

Bismuth. DeAngelis and Gerardi described spectrophotometric determinations of copper and bismuth separately (*26*) and simultaneously (*27*) in aluminum alloys containing lead. In the separate determinations diethyldithiocarbamate was used for copper and the cupferron–KI or the HBr method was used for bismuth. In the simultaneous determinations a nitric acid sample solution was treated with thiosemicarbazide for copper, KI for bismuth, and NaCl to complex lead. Copper and bismuth were measured at 560 and 466 mμ. Vinogradova and Vasil'eva (*196*) reported the determination of small amounts of bismuth, tin, and antimony in aluminum as described under the section on Antimony.

Tumanov and Sidorenko (*187*) studied the determination of bismuth in titanium dioxide based on the formation of the yellow complex iodide which is stabilized with hydrazine sulfate.

Boron. Ichiryu and Hashimoto (*72*) studied the spectrophotometric determination of traces of boron in aluminum with 1,1′-dianthrimide at

Table II. Methods for Elements in

Constituent determined	Material	Reagent or method	References
Ag	Aluminum alloys	Atomic absorption	(*208*)
Al	Aluminum–bronze alloy	Paper chromatography, photometric	(*127*)
	Aluminum–base alloy	Chelometric	(*18*)
	Magnesium alloy	Chelometric	(*179*)
	Magnesium metals	Photometric, oxine	(*188*)
	Titanium dioxide pigment	Photometric	(*113*)
	Titanium slags	Chelometric	(*180*)
Al_2O_3	Aluminum	Aluminon, photometric	(*41, 42*)
	Titanium oxide	Trilon B, $FeCl_3$	(*129*)
As	Aluminum, aluminum alloys	Photometric	(*167*)
	Beryllium oxide	Activation	(*117*)
B	Aluminum	1,1′-Dianthrimide	(*72*)
	Aluminum alloys	Spectrographic	(*101*)
	Aluminum	Photometric	(*132*)
	Aluminum	Acidimetric	(*155*)
	Beryllium oxide	Photometric	(*80*)
	Magnesium alloys	Photometric	(*57*)
	Magnesium	Curcumin	(*189*)
	Titanium	Curcumin	(*35, 67*)
Be	Copper–beryllium, beryllium–aluminum	Photometric	(*86*)
	Beryllium	Activation	(*152*)
	Magnesium alloys	Photometric	(*17*)
Bi	Aluminum alloys	Photometric	(*26, 27*)
	Aluminum	Anodic voltammetry	(*196*)
	Titanium dioxide	Photometric, iodide	(*187*)
C	Beryllium	Activation analysis	(*14*)
	Titanium	Combustion, titration	(*49*)
	Titanium sponge	High-frequency combustion	(*162*)
Ca	Aluminum	Flame photometric	(*92*)
	Titanium dioxide	Photometric, murexide	(*10*)
Cd	Aluminum	Photometric, dithizone	(*65*)
	Aluminum	Anodic voltammetry	(*194*)
	Aluminum, aluminum alloys	Polarographic	(*46*)
	Aluminum alloys	Polarographic	(*70*)
	Copper–aluminum alloys	Polarographic	(*45*)
Ce	Aluminum oxide	Photometric	(*115*)
	Magnesium alloys	Photometric	(*177*)
Cl	Beryllium	X-ray fluorescence	(*83*)
	Titanium dioxide	$HgCl_2$	(*187*)
Co	Magnesium–base alloys	Activation analysis	(*182*)
Cr	Aluminum black	Diphenylcarbazide	(*61*)
Cu	Aluminum alloys	Photometric, diethyldithiocarbamate	(*26*)
	Aluminum alloys	Photometric, thiosemicarbazide	(*27*)
	Aluminum alloys	Extraction, iodimetric	(*173*)
	Aluminum alloys	Iodimetric	(*126*)
	Aluminum–bronze alloy	Paper chromatographic, photometric, Chrome Azurol S	(*127*)
	Aluminum alloys	Spectrographic	(*161, 183, 200*)
	High-purity aluminum	Spectrographic	(*77*)
	Aluminum	Spectrographic	(*137*)
	Aluminum alloys	Electrolytic	(*6*)
	Aluminum alloys	Cation exchange, electrolytic	(*84*)
	Aluminum	Activation analysis	(*89, 100*)
	Aluminum alloys	Square-wave polarographic	(*172*)
Dy	Aluminum	Activation analysis	(*89*)
F	Aluminum	Photometric	(*191*)
Fe	Aluminum	Spectrographic	(*21, 77, 137*)
	Aluminum alloy	Photometric, EDTA, H_2O_2	(*138*)
	Aluminum	Photometric, 1,10-phenanthroline	(*76*)
	Aluminum black	Photometric, haematoxylin	(*61*)
	Aluminum	Activation analysis	(*60*)
	Aluminum alloys	Square-wave polarographic	(*172*)
	Titanium chips	Spectrographic	(*62*)
	Titanium dioxide	Photometric, 1,10-phenanthroline	(*187*)
Ga	High-purity aluminum	Anodic voltammetry	(*194*)
	Aluminum	Photometric	(*170*)
	Aluminum	Activation analysis	(*90*)
	Bauxites	Extraction, spectrographic	(*102*)
H	Aluminum	Mass spectrometric	(*140*)
	Aluminum	Vacuum heating	(*93*)
	Aluminum	Spectral-isotopic	(*139*)

630 mμ. Most of the aluminum was removed on a column of Dowex-50 resin and the boron was eluted with water. The eluate was heated with the reagent in sulfuric acid. There was no interference from <1% chromium and <0.2% vanadium. Kuzovlev and Gusarski (*101*) proposed a spectral method of determination of thousandths of a per cent of boron in aluminum alloys. Nemodruk, Palei, and Hun-I (*132*) described a rapid photometric determination of traces of boron (down to 10^{-4}%) in metallic aluminum with acetylquinalizarin at 620 mμ. Rosenberg (*155*) recommended determining boron present in amounts >100 p.p.m. in aluminum by first removing the aluminum by cation exchange. The H_3BO_3 was converted to its complex with mannitol and determined acidimetrically with α-naphtholphthalein as indicator.

Karalova and Nemodruk (*80*) proposed an extraction and photometric determination of boron in beryllium oxide. The sample was heated with HF and neutralized with urotropine by using Brilliant Green. After extraction with benzene and centrifugation, the complex was measured at 656 mμ.

Pitwell (*148*) investigated the dissolution of samples for determining traces of boron without loss of boron as boranes or H_3BO_3. Inclusion of residual acid in the distillation was avoided. The sample was placed in a distillation flask fitted with a dropping funnel and a condenser which dipped below the surface of a solution of NaOH in aqueous glycerol. Anhydrous methanol was added and H_2SO_4 or H_3PO_4 in methanol was added gradually until the sample was dissolved. The distillate contained methyl borate and any borane included was hydrolyzed by the NaOH. The procedure was applicable to boron in magnesium and aluminum.

Gordievskii and Ustyugov (*57*) described the fluorometric determination of boron in magnesium alloys with Anthraquinone blue SVG. In the presence of boron the reagent produced an intense bright red fluorescence. The photometric determination of boron in magnesium metal has also been accomplished with curcumin (*189*). Elwell and Wood (*35*) and Hayes and Metcalfe (*67*) used curcumin for determining boron in titanium, zirconium, and hafnium with no preliminary separation.

Cadmium. Hashimoto and Tanaka (*65*) determined traces of cadmium in aluminum by extraction with dithizone in chloroform and photometrically at 517 mμ. Hine and Bates described a polarographic determination of 0.1 to 0.35% cadmium in aluminum alloys containing 5% copper by using the derivative curve with 1*M* HCl as the supporting electrolyte. Lithium, iron,

Nonferrous Metallurgical Materials

Constituent determined	Material	Reagent or method	References
H	Aluminum, beryllium	Isotopic dillution	(*38*)
	Magnesium	Pressure, Pd tube	(*5*)
	Titanium	Spectrographic	(*78, 98*)
Hf	Aluminum alloys	Gravimetric, titrimetric	(*165*)
K	Aluminum	Flame photometric	(*92*)
Li	Aluminum	Flame photometric	(*147*)
Mg	Aluminum alloys	Chelometric	(*22, 166*)
	Aluminum alloys	Spectrographic	(*144*)
	Aluminum alloys	Atomic absorption	(*203*)
	Aluminum oxide	Photometric, magneson	(*96*)
	Titanium sponge	Spectrographic	(*184*)
	Titanium dioxide	Photometric, magneson	(*10, 11*)
Mn	Aluminum, aluminum alloys	Potentiometric titration	(*150*)
	Aluminum alloys	Iodometry	(*120*)
	Aluminum	Spectrographic	(*21*)
	Aluminum alloys	Spectrographic	(*103, 145*)
	Aluminum	Activation analysis	(*90*)
	Magnesium	Photometric	(*181*)
Mo	Aluminum alloys	Photometric, thiocyanate	(*168*)
	Beryllium	X-ray absorption	(*81*)
	High-purity titanium	Extraction, photometric	(*164*)
N	Beryllium	Activation analysis	(*14*)
	Titanium	Spectrographic	(*154*)
Na	High-purity aluminum	Activation analysis	(*175*)
	Aluminum, aluminum alloys	Flame photometric	(*92, 114, 119, 204*)
	Aluminum alloys	Spectrographic	(*63*)
Ni	Aluminum, magnesium, their alloys	Photometric, dimethylglyoxime	(*143*)
	Beryllium, beryllium oxide	Ion exchange, photometric, dimethylglyoxime	(*68*)
O	Beryllium	Activation analysis	(*14, 53, 112*)
	Beryllium	Vacuum fusion	(*40*)
	Beryllium oxide	Vacuum fusion, mass spectrometric	(*116*)
	Magnesium	Gas analysis	(*135*)
	Titanium	Hydrogen adsorption	(*110*)
	Titanium	Spectrographic	(*154*)
	Titanium, aluminum	Activation analysis	(*48*)
P	Aluminum–silicon alloys	Photometric, molybdenum blue	(*25*)
	Aluminum–silicon alloys	Photometric, gravimetric	(*118*)
	Aluminum–silicon alloys	Activation analysis	(*8*)
	Beryllium oxide	Activation analysis	(*117*)
Pb	Aluminum alloys	Polarographic	(*169*)
	Aluminum–copper alloys	Polarographic	(*45*)
	Beryllium–copper alloy	Polarographic	(*88*)
Pd	Titanium alloys	Photometric, PAN	(*159*)
	Titanium alloys	Photometric, $SnCl_2$	(*160*)
Re	Titanium alloys	Photometric, thiooxime	(*33*)
S	Beryllium oxide	Combustion, titration	(*13*)
	Beryllium oxide	Adsorption, titration	(*109*)
	Beryllium oxide	Activation analysis	(*117*)
	Titanium	Iodimetric, $BaSO_4$	(*134*)
Sb	Aluminum	Anodic voltammetry	(*196*)
	Magnesium–base alloy	Activation analysis	(*182*)
	Titanium dioxide	Photometric	(*52*)
	Titanium dioxide	Polarographic	(*212*)
Sc	Magnesium alloys	Photometric, Xylenol Orange	(*198*)
Si	High-purity aluminum	Spectrographic	(*77*)
	Aluminum alloys	Elemental silicon	(*133*)
	Aluminum alloys	Titrimetric	(*64*)
	Aluminum–silicon alloy	X-ray fluorescence	(*108*)
	Copper–beryllium alloy	Photometric, molybdate	(*87*)
Sn	High-purity aluminum	Anodic voltammetry	(*196*)
	Aluminum alloys	Photometric, haematoxylin	(*174*)
	Beryllium–copper alloy	Polarographic	(*88*)
Ti	Aluminum	Photometric, salicylate, quinine	(*197*)
	Aluminum	Photometric, phosphotitanomolybdate	(*163*)
	Aluminum alloys	Photometric, isoxanthenone compound	(*4*)
	Aluminum alloys	Photometric, dichlorochromotropic acid	(*16*)
	Aluminum alloys	Polarographic	(*85*)
	Beryllium	Photometric, thymol	(*202*)

(*Continued on page 96R*)

Table II. Methods for Elements in Nonferrous Metallurgical Materials (*Continued*)

Constituent determined	Material	Reagent or method	References
Ti	Titanium–aluminum alloys	Chelometric	*(153)*
	Titanium slag	Titrimetric	*(214)*
U	Aluminum alloy	X-ray fluorescence	*(128)*
	Beryllium oxide–uranium oxide	Titrimetric	*(12)*
V	Aluminum	Activation analysis	*(30)*
	Aluminum powder	Photometric, phosphotungstate	*(7)*
	Titanium	Photometric, phosphotungstate	*(213)*
W	Titanium	Photometric, thiocyanate	*(32)*
Zn	Aluminum alloy	Photometric, Xylenol Orange	*(121)*
	Aluminum alloy	Chelometric	*(31, 73, 201)*
	Aluminum alloy	Ion exchanger, electrolytic	*(84)*
	Copper–aluminum alloys	Polarographic	*(44)*
	Aluminum alloys	Polarographic	*(172)*
	High-purity aluminum	Anodic voltammetry	*(194)*
	Aluminum alloys	Spectrographic	*(58)*
	High-purity aluminum	Spectrographic	*(131)*
	Aluminum alloys	Atomic absorption	*(205)*
	Bauxite, alumina, aluminum	Photometric, dithizone	*(122)*
	Nickel–magnesium alloy	Ion exchange, chelometric	*(74)*
Zr	Magnesium alloys	Photometric, tartrazine	*(209)*
	Magnesium alloys	Photometric, PAN	*(23)*
	Magnesium alloys	Photometric, morin	*(186)*
	Magnesium alloys	Photometric, Alizarin S	*(178)*
	Titanium alloys	Photometric, Alizarin Red S	*(210)*

manganese, titanium, vanadium, and lead did not interfere. Fleury and Capelle *(45)* reported the polarographic determination of lead and 0.001 to 0.25% cadmium in copper–aluminum alloys. Copper was separated by electrodeposition and the lead and cadmium were determined in a chloride medium. In a later paper Fleury and Capelle *(46)* recommended the polarographic determination of cadmium in aluminum and its alloys by dissolving the sample in HCl, separating the metallic copper and SiO_2 by filtration and reducing the ferric iron with ascorbic acid. This method avoids the electrolytic removal of copper, iron, tin, and lead. Vasil'eva and Vinogradova *(194)* studied the determination of very small amounts of gallium, zinc, and cadmium in high-purity aluminum by anodic voltammetry at a stationary mercury electrode with a silver contact. Cadmium (2×10^{-8}%) was determined in an $AlCl_3$ and HCl (pH 2 to 3) medium.

Calcium. Klug and Sajo *(92)* described the flame photometric determination of calcium in analyses in the aluminum industry by the addition of butanol to HCl solutions of the samples.

Bogatyrev, Navyazhskaya, and Sporykhina *(10)* investigated rapid methods for the colorimetric determination of magnesium and calcium in titanium dioxide. Murexide was used for calcium.

Carbon. Bradshaw, Johnson, and Beard *(14)* developed a method for the determination of trace amounts of oxygen, nitrogen, and carbon in beryllium by gamma-ray activation. It is the only method available for the determination of carbon contained in beryllium in the range of 10 to 1000 p.p.m.

Fujishima and Takeuchi *(49)* recommended a rapid combustion (1200° C.) and titration determination of traces of carbon in titanium. An absorber containing a $Ba(OH)_2$ solution and isopentyl alcohol (as a defoaming agent) was fitted on the combustion apparatus. Cresol red and thymol blue were used as the indicators for the final titration. Shimazaki, Nakayama, and Nakamura *(162)* utilized high-frequency combustion for the determination of microamounts of carbon in titanium sponge using a crucible made of material containing Al_2O_3 and SiO_2 in the ratio of 10 to 8.

Cerium. Mendlina *(115)* reported a photometric determination of cerium in aluminum oxide by treating the solution with potassium citrate and hydrogen peroxide at pH 8 to 9. Up to 0.05% of Fe_2O_3, SiO_2, Ca, or MgO offered no interference. In the determination of cerium in magnesium alloys by the citrate method, Tikhonov *(177)* proposed ways of eliminating the effect of rare earths, magnesium, and manganese and developed a method without physical destruction of the sample.

Chlorine. Keys and Rowan *(83)* recommended the determination of chloride in beryllium metal by x-ray fluorescence. Microgram quantities were determined by comparing the secondary fluorescent emission from an unknown sample to that from standard pellets.

Tumanov and Sidorenko *(187)* studied the determination of chloride in titanium dioxide based on the formation of $HgCl_2$.

Chromium. Guerreschi and Romita *(61)* used diphenylcarbazide to determine chromium (5% Cr_2O_3) in aluminum black after the separation of Fe^{+3} by precipitation as hydroxide from a solution buffered with KOH–$(NH_4)_2SO_4$ in the presence of $(NH_4)_2U_2O_7$ as a carrier. Under these conditions aluminum and chromium were not precipitated. Iron was determined in a solution of the precipitate.

Cobalt. Todd, Cuthbert, and Dickinson *(182)* proposed a method for the determination of antimony and cobalt in magnesium alloyed with small amounts of aluminum and manganese by neutron activation analysis. The method is described in the Antimony section.

Copper. DeAngelis and Gerardi recommended spectrophotometric determinations of copper and bismuth separately *(26)* and simultaneously *(27)* in aluminum alloys containing lead. The procedures are summarized in the Bismuth section. Tajima, Kurobe, and Terada *(173)* investigated the extraction of the copper thiocyanate complex into tributyl phosphate and its application to the analysis of aluminum alloys. A 0.2*M* to 0.8*M* thiocyanate solution at pH 1 to 5 was used and the tributyl phosphate was dissolved in 1:1 light petroleum–benzene. Copper was re-extracted into 1*M* NH_4Cl containing NH_3 and determined iodimetrically. Munshi and Dey *(127)* reported a paper chromatographic separation and determination of copper and aluminum in aluminum-bronze alloys. The method is described in the Aluminum section. Copper was determined colorimetrically using Chrome Azurol S. Mukai and Mochizuki *(126)* developed a rapid method for the determination of copper in aluminum alloy containing silicon. The sample was dissolved in nitric acid containing NH_4HF_2. After complete dissolution NH_4HF_2, NH_4OAc and urea were added and the copper was titrated iodimetrically.

Vvedenskii, Shekhobalova, and Novikova *(200)* found that silicon markedly affected the determination of copper in the analysis of aluminum–copper alloys by spark excitation of the spectra. The effect produced by silicon appeared suddenly at a concentration of

2% Si and remained unchanged up to 6% Si. Torok (*183*) utilized a low-voltage spark excitation for the spectrographic determination of 0.1 to 1.0% copper in aluminum alloys. In establishing the spectra below 2300 A., spectral plates were previously sensitized with sodium salicylate in methanol. Kajzer (*77*) reported the spectrochemical analysis of high-purity aluminum for iron, silicon, and copper in the concentration range 10^{-3} to 1.4×10^{-2}% by the intermittent-arc technique. The line Al 2669 A. was used as internal standard, and the lines Si 2516 A., Fe 2599 A., and Cu 3274 A. were used for the determination. Schroeder and Strasheim (*161*) studied the synchronized direct-reading spectrometer-spark system for time-resolved spectra of copper spectral analysis lines used for the determination of copper in aluminum alloys. Oida, Fujita, and Takahashi (*137*) developed a spectrochemical method using a quantorecorder for the determination of 0.0005 to 0.01% silicon, iron, and copper in high-purity aluminum. The direct point-to-plane method with excitation with over damped discharge by low voltage was used.

Bertoldi and Tartari (*6*) reported the electrolytic determination of copper in aluminum alloys in the presence or absence of bismuth from a HNO_3–HBF_4 or HNO_3–HF–HBF_4 solution. Tin was complexed but bismuth was removed by co-deposition with lead at the anode. Kharin and Soroka (*84*) developed an ion exchange–electrolytic determination of zinc and copper in aluminum alloys not containing nickel. Iron, aluminum, manganese, magnesium, chromium, and silicon were complexed with $Na_4P_2O_7$ and zinc and copper were complexed with NH_3. The solution was passed through a cation exchange resin which allowed the pyrophosphate anions to pass through while the zinc and copper cations were adsorbed. After elution of zinc with NaOH and copper with HCl, they were determined by electrolytic separation.

Kiesl, Bildstein, and Hecht (*89*) proposed activation analysis for the determination of copper and dysprosium in aluminum. The copper complex of 2,2′-diquinolyl was extracted by amyl alcohol, re-extracted into the aqueous layer with HNO_3 and precipitated by benzoin oxime. The 0.51-m.e.v. peak of the annihilation radiation was measured. Dysprosium was determined without destruction by measuring the 91-k.e.v. peak after a short time of radiation. Kukula, Slunecko, and Simkova (*100*) recommended the activation analysis of aluminum for copper with Cu^{64} after separation with ammonium reineckate.

Tajima and Kurobe (*172*) investigated the determinationn of copper, zinc, and iron in aluminum alloys by square-wave polarography without interference from other constituents. The polarograms for copper and zinc were recorded from an HCl solution at −0.025 and −1.05 volts, respectively, *vs.* the mercury-pool electrode.

Dysprosium. Kiesl, Bildstein, and Hecht (*89*) proposed activation analysis for the determination of copper and dysprosium in aluminum. The method is discussed in the Copper section.

Fluorine. Valach (*191*) described the photometric determination of fluorine in aluminum based on the considerable stability of the chelates of zirconium with Xylenol Orange type dyes in 1 to 2*N* $HClO_4$, HNO_3, and HCl which decomposed interfering fluoroaluminates.

Gallium. Vasil'eva and Vinogradova (*194*) studied the determination of very small amounts of gallium, zinc, and cadmium in high-purity aluminum by anodic voltammetry at a stationary mercury electrode with a silver contact.

Suzuki (*170*) recommended the spectrophotometric determination of gallium in aluminum with PAN, 1-(2-pyridylazo)-2-naphthol. An HCl solution of the sample was extracted with isopropyl ether (Fe^{+3} was reduced to Fe^{+2} with $TiCl_3$), the ether was removed, and the residue was adjusted to pH 4.3. This solution was extracted with a chloroform solution of the reagent and its absorbance was measured at 550 mμ.

Kiesl, Bildstein, and Sorantin (*90*) utilized activation analysis for the determination of manganese and gallium in aluminum. Manganese was separated from neutron-irradiated aluminum by extraction of MnO_4^- with $(C_6H_5)_4AsCl$ and $CHCl_3$ and measured by counting the 0.85-m.e.v. photopeaks of Mn^{56}. After a cooling period of 17 hours, the Mn^{56} died out, and the 0.85-m.e.v. peak of Ga^{72} was determined directly with the aluminum sample.

Landi (*102*) reported the determination of gallium in bauxites by a combined extraction–spectrographic method which consisted of extracting gallium chloride with ethyl ether, evaporation, hydrolysis with NaOH, dehydration at 700° C., and mixing the oxide with graphite for sparking.

Hafnium. Sikes, Wade, and Yamamura (*165*) discussed procedures for both a titrimetric and gravimetric determination of hafnium in aluminum alloys. The sample was dissolved in aqua regia and any undissolved hafnium was taken into solution by pyrosulfate fusion. The hafnium was separated from the aluminum and contaminants in the aluminum by precipitation as the mandelate. In the gravimetric procedure the hafnium mandelate was heated to the oxide and weighed. In the titrimetric procedure the mandelate was destroyed by acids and the hafnium was determined by back titrating an excess of EDTA with bismuth with Xylenol Orange as indicator.

Hydrogen. Orlovtsev, Krapukhin, and Krestovnikov (*140*) discussed the mass spectrometric determination of hydrogen in aluminum. Klyachko, Kunin, and Chistyakova (*93*) reported the determination of hydrogen in aluminum by vacuum heating.

Orlova and Petrov (*139*) proposed a spectral-isotopic method for the determination of hydrogen in aluminum alloys. The method was utilized at 500° C. on samples 5 to 6 mm. in diameter and 10 to 20 grams in weight. The experimental determined time for isotopic equilibration was 25 to 30 minutes. Evans and Herrington (*38*) recommended the determination of hydrogen in aluminum and beryllium by isotopic dilution with tritium.

Berry and Walker (*5*) developed a method for determining hydrogen in magnesium alloys involving the simultaneous sublimation of magnesium from a graphite crucible and the removal of hydrogen from the furnace system. The amount of hydrogen was determined by collecting the gas in a calibrated volume and measuring the pressure with a McLeod gauge before and after diffusion through a palladium tube.

Kalinin, Kondrashova, Mironov, and Yalymov (*78*) studied the spectral determination of hydrogen in titanium. The optimum conditions for the calibration curve were: inductance, 3 μhenry and slit width, 0.06 mm. A small interelectrode gap was recommended to increase the accuracy. Kovalenko (*98*) investigated the characteristics of the photoamplifying system in the spectral determination of 0.002 to 0.2% hydrogen in titanium.

Kern (*82*) critically discussed methods of determining hydrogen, nitrogen, and oxygen in beryllium, magnesium, and titanium.

Iron. Christ (*21*) utilized the melt-in-rod technique for the spectrochemical determination of iron and manganese in aluminum chips, sheet, or foil when only 5 to 7 grams are available. The sample was melted (in a graphite block) and forced into an aluminum rod having a preformed crater and air outlets. The cool sample was then machined and analyzed. Kajzer (*77*) reported the spectrochemical analysis of high-purity aluminum for iron, silicon, and copper as described in the Copper section. Oida, Fujita, and Takahashi (*137*) developed a spectrochemical method using a quantorecorder for the determination of silicon, iron, and copper as discussed in the Copper section.

Onishi (*138*) recommended a rapid

colorimetric determination of iron in aluminum alloy with EDTA and H_2O_2. Absorbance measurements were made at 517 mμ. Jackson and Phillips (*76*) studied the colorimetric determination of iron in zone-refined aluminum after solvent extraction. Iron was first extracted from the sample solution with isobutyl methyl ketone, which in turn was extracted with a buffer solution containing 1,10-phenanthroline. Guerreschi and Romita (*61*) determined iron in aluminum black (containing chromium) spectrophotometrically with haematoxylin. The procedure was discussed in the Chromium section.

Grossmann and Doege (*60*) described the determination of iron in pure aluminum by activation analysis. The samples are irradiated for 100 hours at a rate of 10^{13} neutrons per sq. cm.-sec. Iron was separated by ion exchange, solvent extraction, and precipitation before counting. Tajima and Kurobe (*172*) investigated the determination of copper, zinc, and iron in aluminum alloys by square-wave polarography without interferences from other constituents. The polarogram for iron was recorded at -0.3 volt *vs.* the mercury-pool electrode after a solvent extraction separation of the iron.

Gusarskii and Kuzovlev (*62*) proposed a spectrochemical determination of iron in titanium chips using the analytical line pair Fe 2599.40 A. and Ti 2555.99 A. Tumanov and Sidorenko (*187*) reported the determination of chloride, iron, and bismuth in titanium dioxide. A photometric method for iron was employed using 1,10-phenanthroline.

Lead. Stross and Clark (*169*) recommended the polarographic determination of lead in aluminum alloys containing molybdenum or up to 5% iron. Fleury and Capelle (*45*) employed a polarographic determination of 0.005 to 0.25% lead and cadmium in copper–aluminum alloys. Copper was separated by electrodeposition and the lead and cadmium were determined in a chloride medium.

Kida *et al.* (*88*) determined tin and lead in a beryllium–copper alloy polarographically with a salt calomel electrode. Tin and lead were coprecipitated with $Be(OH)_2$ and separated from copper, which was redissolved in excess NH_4OH. The polarogram of tin and lead was taken on a portion of the acid solution of the precipitate. A second polarogram (of lead) was taken on a portion of the solution made alkaline. The tin was obtained by difference.

Lithium. Pilgrim and Ford (*147*) reported the determination of lithium in aluminum by an improved flame photometric method. The addition of isopropyl alcohol and acetone to acid solutions containing lithium and aluminum permitted the determination of microgram levels of lithium without separation.

Magnesium. Costin (*22*) developed a rapid complexometric method for the determination of 0.3 to 6% magnesium in aluminum alloys by titrating a solution (buffered at pH 10) with Complexon III using Eriochrome T as indicator. Smart (*166*) preferred Calmagite as an indicator for the complexometric determination of magnesium in aluminum alloys. Also, more careful control of pH at the manganese precipitation stage overcame positive errors.

Peter (*144*) described a spectrochemical determination of magnesium in aluminum alloys by differences in line densities. The determination was carried out under two sets of excitation conditions. Wallace (*203*) recommended atomic absorption spectroscopy for the determination of magnesium in aluminum alloys. The serious interference of aluminum was overcome by the use of 8-hydroxyquinoline. Korenman (*96*) determined trace amounts of magnesium in aluminum oxide with magneson IREA, sodium 5-chloro-2-hydroxy-3-(2-hydroxy-1-naphthylazo)benzenesulfonate. By allowing the reaction to proceed for 1 to 2 days, the sensitivity was increased.

Tsuji, Abe, and Ando (*184*) proposed a spectrographic determination of magnesium in titanium sponge with a simplified reservior-cupped conical electrode. A polyethylene cup was cut and fitted over a graphite electrode so that it could hold the solution to be analyzed. Bogatyrev, Navyazhskaya, and Sporykhina (*10*, *11*) studied rapid methods for the colorimetric determination of magnesium and calcium in titanium dioxide. Magneson was used for magnesium.

Manganese. Pohl (*150*) described the potentiometric titration of manganese in aluminum and its alloys. Mn(II) was titrated with $KMnO_4$ in a pyrophosphate solution at pH 6.5 to form a Mn(III) pyrophosphate complex. Miyajima (*120*) investigated the determination of 0.12 to 1.53% manganese in aluminum alloys by iodometry using EDTA as a masking agent.

Christ (*21*) utilized the melt-in-rod technique for the spectrochemical determination of iron and manganese in aluminum chips, sheet, and foil when only 5 to 7 grams are available. The method is summarized in the Iron section. Laszlo (*103*) and Peter (*145*) reported spectrochemical methods based on the density differences of manganese in aluminum alloys. The density differences of the pairs of Mn lines 3547.79–2798.27A. and 3547.74–2576.10 A. were used to plot a calibration curve. Kiesl, Bildstein, and Sorantin (*90*) employed activation analysis for the determination of manganese and gallium in aluminum as described in the Gallium section.

Tikhonov and Nikitina (*181*) reported the determination of small quantities of manganese in magnesium alloys with rare earths by oxidation with KIO_3 after extraction with a CCl_4 solution of sodium diethyldithiocarbamate.

Molybdenum. Stross and Clark (*168*) used NaSCN and $SnCl_2$ for the spectrophotometric determination of molybdenum in aluminum alloys. Iron was reduced by $SnCl_2$, and copper which precipitated was filtered off. Other usual constituents or impurities had no effect.

Karev and Matyushenko (*81*) recommended x-ray absorption analysis of beryllium for the determination of molybdenum.

Shustova and Nazarenko (*164*) determined traces of molybdenum in high-purity titanium by making a chloroform extraction as the 8-quinolinolate and determining the molybdenum colorimetrically as the salicylfluorene or thiocyanate complex.

Nickel. Penner and Inman (*143*) applied the determination of 0.0005 to 1.0% nickel by spectrophotometric measurement of the chloroform extract of nickel(II) dimethylglyoximate to the analysis of aluminum and magnesium and their alloys. Zinc and copper, which interfered, were complexed with $Na_2S_2O_3$ at pH 4.5 to 5.0 and pH 6.5, respectively. Large amounts of copper were removed electrolytically.

Hibbits and Kallmann (*68*) described the photometric determination of nickel in beryllium and beryllium oxide after its separation by precipitation and ion exchange. Cadmium was added to the sample solution and was precipitated along with nickel from a chloride–sulfate–tartrate medium at pH 8.5 with benzotriazole. After dissolution of the precipitate in HNO_3–$HClO_4$, the nickel was separated by ion exchange before being determined at 465 mμ as the dimethyl–glyoxime complex.

Nitrogen. Bradshaw, Johnson, and Beard (*14*) developed a method for the determination of trace amounts of oxygen, nitrogen, and carbon in high-purity beryllium by gamma-ray activation. The method was used to estimate the nitrogen content when the iron and copper contents of the sample were accurately known.

Romand, Balloffet, and Vodar (*154*) proposed the spectrochemical determination of oxygen and nitrogen in titanium by vacuum sparks. Kern (*82*) critically discussed methods of determining hydrogen, nitrogen, and oxygen in beryllium, magnesium, and titanium.

Oxygen. Gamma-ray activation was employed by Bradshaw, Johnson, and Beard (*14*) for the determination of trace amounts of oxygen (10 to 5000 p.p.m.), nitrogen, and carbon in high-purity beryllium. The technique was used as a standard method for the evaluation of analytical techniques for oxygen. McCrary, Morgan, and Baggerly (*112*) recommended the determination of oxygen in beryllium by neutron activation analysis. The short half-life of N^{16} resulting from the reaction $O^{16}(n,p)N^{16}$ was used to determine oxygen. Irradiation was carried out with 1.8×10^8 neutrons per sq. cm.-sec. Fujii, Muto, and Miyoshi (*48*) also utilized neutron activation analysis in the determination of 0.002 to 0.5% oxygen in titanium and aluminum using the above-mentioned reaction. The neutron intensity in this case was 10^{10} neutrons per sq. cm.-sec. Gilman and Isserow (*53*) discussed both chemical and radioactivation determinations of oxygen in beryllium. Two chemical methods were used: (a) volatilization of beryllium as the chloride and (b) selective dissolution of beryllium in bromine–methanol. In both methods, a residue containing oxygen as BeO was analyzed for beryllium. In activation analysis oxygen was converted to O^{15} by gamma-neutron reaction.

Meyer, Austerman, and Swarthout (*116*) described a method and apparatus for the determination of small isotopic oxygen variations in beryllium oxide. A vacuum fusion apparatus using a platinum flux contained in a graphite crucible at 2200° C. was used to release the oxygen in BeO as CO. The resultant CO was analyzed for CO^{18}–CO^{16} ratio by mass spectrometry.

Everett and Thompson (*40*) studied the determination of oxygen in beryllium by vacuum fusion. Suggestions were made with regard to apparatus design and operating technique. Maintenance of high pumping speeds for high pressures at the crucible and the use of capsules to enclose all samples were the most important factors.

Oda, Norishima, and Kubo (*135*) determined oxygen in magnesium by placing a sample in a graphite boat, the bottom of which was covered with powdered carbon, and heated at 900° C. in an HCl stream. The evolved gas was passed onto a platinum–carbon layer at 1000° C. in a platinized tube, so that the oxygen was reduced to CO. After the removal of HCl, CO was oxidized to CO_2 with HI_3O_3, followed by weighing.

Romand, Balloffet, and Vodar (*154*) proposed the spectrochemical determination of oxygen and nitrogen in titanium by vacuum sparks. Livanov, Bukhanova, and Kolachev (*110*) reported a method for the determination of oxygen in titanium based on the measurement of equilibrium pressure of hydrogen introduced into the sample. The method was claimed to be faster than the conventional vacuum melting method and required simpler apparatus and less skilled personnel.

Palladium. Sawada and Kato proposed two spectrophotometric methods for the determination of palladium in titanium alloys. The first was the PAN, 1-(2 pyridylazo)-2-naphthol, method (*159*) which is applicable to the concentration range 0.001 to 1% Pd. The sample was dissolved in HCl–citric acid–HF and finally in HNO_3–H_3BO_3. EDTA and PAN in methanol were added, the pH was adjusted to 3.0 to 3.5 and the solution was heated to 100° C. After cooling the Pd–PAN complex was extracted with chloroform and the absorbance was read at 675 mμ. All elements usually found in titanium alloys did not interfere.

The second method (*160*) employed $SnCl_2$ and was applicable to 0.05 to 5% Pd in titanium alloys. The procedure for dissolution of the sample was similar to the PAN method except urea was added along with H_3BO_3. The absorbance was measured at 635 mμ after the addition of $SnCl_2$.

Phosphorus. Davey (*25*) described the colorimetric determination of phosphorus in aluminum–silicon alloys by development of the molybdenum-blue complex in isobutyl alcohol. Mills and Hermon (*118*) claimed that a direct photometric determination of phosphorus in hypereutectic aluminum–silicon alloys was not possible because it was first necessary to separate the phosphorus from aluminum and silicon. The apparatus and procedure were described for the separation of phosphorus from aluminum by distillation as PH_3. Also, both gravimetric and photometric procedures were discussed and compared.

Blackburn and Peters (*8*) recommended the determination of 0.001 to 0.01% phosphorus in hypereutectic aluminum–silicon alloys by neutron activation. The sample was irradiated in a flux of 5×10^{11} neutrons per sq. cm.-sec. for 50 hours. After 5 days the irradiated sample was dissolved in HF–HNO_3 and the H_3PO_4 converted into the molybdophosphoric acid complex, which was extracted into isobutyl alcohol and counted for P^{32}. The method was claimed to be free from the disadvantages of the usual spectrophotometric methods.

Mikheeva and Wihitill (*117*) reported the determination of arsenic, phosphorus, and sulfur in beryllium oxide by radioactivation analysis. The sample was irradiated by a stream of 10^{13} neutrons per sq. cm.-sec. for 10 minutes and kept for 30 hours.

Potassium. (See Sodium.)

Rhenium. Egorova and Gurevich (*33*) studied the photometric determination of rhenium in titanium alloys with 8-mercaptoquinoline (thiooxime) by extracting the complex with chloroform from a 9 to 11*N* HCl solution and measuring the absorbance at 438 mμ. Ti(III) was oxidized to Ti(IV) with hydroxylamine hydrochloride in order to eliminate its interference.

Scandium. Volodarskaya and Derevyanko (*198*) proposed the colorimetric determination of scandium in magnesium alloys with Xylenol Orange. Ascorbic acid was added to reduce Fe(III) and Ce(IV).

Silicon. Neverovsky (*133*) described the determination of silicon in aluminum alloys. After the sample was dissolved in acid, the remaining elemental silicon was filtered, washed with water, ethanol, and acetone, dried at 120° to 140° C., and weighed. Hasegawa (*64*) reported a rapid titrimetric determination of silicon in aluminum alloys, which consisted of the formation of K_2SiF_6 and its titration with NaOH. Titanium, tantalum, and zirconium interfered.

Kajzer (*77*) used spectrochemical analysis of high-purity aluminum for iron, silicon, and copper. Details are covered in the Copper section. Lihl and Fischhuber (*108*) utilized x-ray fluorescence analysis in the determination of silicon in aluminum–silicon alloys.

Kida *et al.* (*87*) developed a rapid colorimetric determination of silicon in copper–beryllium alloy using ammonium molybdate. The absorbance was measured at 750 mμ.

Silver. Wilson (*208*) recommended atomic absorption spectroscopy for the determination of 5 to 70 p.p.m. silver in aluminum alloys. A tube current of 5 ma. and slit width of 0.05 mm. were optimum. The sample solution was aspirated into a nonluminous air–CO plus hydrogen flame and the Ag 328.1-mμ emission line was measured.

Sodium. Klug and Sajo (*92*) described the flame photometric determination of sodium, potassium, and calcium in aluminum. Matelli and Attini (*114*) discussed the preparation of a sodium-free aluminum sample in the determination of sodium in aluminum by flame photometry. Milos (*119*) reported a procedure for the flame photometric determination of 0.001 to 0.02% sodium in aluminum and its alloys. Wallace (*204*) investigated the addition of methanol to suppress the effect of aluminum on the emission from sodium in the flame photometric determination of sodium in aluminum.

Gusarskii and Tarasevich (*63*) proposed a spectrographic method for the determination of sodium in aluminum alloys. Solutions of the sample were excited in an a.c. arc and the line at 5889.92 A. was measured to determine sodium contents of 0.0003%.

Teillac (*175*) used a new method of

indirect activation analysis to determine sodium in high-purity aluminum based on the fact that it was possible to produce sodium from aluminum by nuclear reaction and that the impurity itself also produced the radionuclide.

Sulfur. Boyle, Gregory, and Sunderland (*13*) reported a combustion and titration method for the determination of 10 to 2000 p.p.m. of sulfur in beryllium oxide. The sample was burned at 1500°C. in a high-frequency single-tube induction furnace in a stream of oxygen. The sulfur was converted to SO_2, carried by the oxygen stream to an acidified starch–iodide solution, and titrated with KIO_3. Lin (*109*) described the determination of milligram amounts of sulfate in beryllium oxide. The sample was dissolved in HNO_3–HF, and the residue after fuming was dissolved in water. The sulfate was adsorbed on an alumina column, eluted with NH_4OH, passed through a Dowex 50-X8 column (to remove NH_4^+) and the sulfate was determined by precipitating $BaSO_4$ and titrating the excess barium with EDTA.

Mikheeva and Wihitill (*117*) recommended the determination of arsenic, phosphorus, and sulfur in beryllium oxide by radioactivation. The sample was irradiated in a flux of 10^{13} neutrons per sq. cm.-sec. for 10 minutes and kept for 30 hours. The sulfur was determined in $BaSO_4$ precipitates from the sample solution.

Oda and Kubo (*134*) investigated the determination of total sulfur in titanium. The sample was decomposed with HCl and the evolved gases were absorbed in an ammoniacal $CdCl_2$ solution. The sulfur in the precipitate (CdS) and in the solution (SO_2^{-2}) were determined iodimetrically. Sulfate was determined as $BaSO_4$ after the titanium was precipitated with NH_4OH.

Tin. Vinogradova and Vasil'eva (*196*) reported the determination of small amounts of tin, bismuth, and antimony in high-purity aluminum by anodic voltammetry at a stationary mercury electrode as discussed in the Antimony section. Tanaki (*174*) recommended the photometric determination of >0.05% tin in aluminum alloys using oxidized haematoxylin at 570 mμ.

Kida *et al.* (*88*) determined tin and lead in a beryllium–copper alloy polarographically with a salt calomel electrode. More details are given in the Lead section.

Titanium. Volkova, Get'man, and Emtsova (*197*) determined titanium in aluminum photometrically as the titanium - salicylate - quinine complex, which was extracted with chloroform from a pH 3 solution. Shkaravskii (*163*) proposed the photometric determination of small amounts of titanium in aluminum by extraction as phosphotitanomolybdate with butanol and chloroform. The absorbance was measured at 360 mμ. Asmus, Kurzmann, and Wallsdorf (*4*) recommended the use of 2,3,7 - trihydroxy - 9 - (3,4 - dihydroxyphenyl)-6-isoxanthenone for the photometric determination of 0.005 to 1.2% titanium in aluminum alloys at a pH of 1.5. Budanova and Pinaeva (*16*) reported the photometric determination of titanium in aluminum alloys containing vanadium with dichlorochromotropic acid at 490 mμ.

Khasnobis (*85*) utilized polarography for the estimation of titanium in aluminum and its alloys. Titanium oxide was precipitated from the sample solution and dissolved, after filtration, with oxalic acid. The polarogram was obtained between −0.2 and −0.7 volt.

Pribil and Vesely (*153*) applied chelometric methods to the determination of titanium in titanium–aluminum alloys. Sodium salicylate (to mask aluminum) and triethanolamine were added to the sample solution and the pH was adjusted to 1 to 2. Bismuth was used to titrate the excess EDTA added with Xylenol Orange as indicator. When iron was present, titanium was first precipitated with NaOH in the presence of triethanolamine, which kept Fe(III) and Al in solution.

Walkden and Heathfield (*202*) studied the photometric determination of titanium in beryllium by a chloroform–cupferron extraction, evaporation of the organic layer to dryness and fusion of the residue. The titanium in a solution of the residue was determined by a freshly prepared thymol reagent.

Zinchenko, Ershova, and Gertseva (*214*) reported the determination of bi- and trivalent titanium in titanium slag. The sample was decomposed with H_3PO_4 and the titanium was titrated with $FeNH_4(SO_4)_2$ with thiocyanate indicator.

Tungsten. Dymov and Kozel (*32*) described the colorimetric determination of tungsten in titanium based on the extraction of the thiocyanate complex with isobutyl alcohol.

Uranium. Musil (*128*) developed an x-ray fluorescence technique for the rapid determination of 100 to 1000 p.p.m. of uranium in aluminum alloy.

Boyle (*12*) determined uranium in beryllium oxide–uranium oxide mixtures by dissolving the sample in $HClO_4$ and H_2SO_4 overnight, reducing the uranium with Cr(III), and titrating electrometrically with $K_2Cr_2O_7$.

Vanadium. Deyris and Albert (*30*) recommended the determination of vanadium in zone-refined aluminum by activation analysis based on irradiation of vanadium, coprecipitated by adsorption on $Fe(OH)_3$, in a flux of 5×10^{12} neutrons per sq. cm.-sec. The activity of V^{52} was measured after extraction of vanadium cupferrate into CCl_4. Biechler, Jordan, and Leslie (*7*) reported the spectrophotometric determination of vanadium in high-purity aluminum powder by converting the vanadium into the phosphotungstate, which was extracted into *n*-hexanol.

Zinchenko and Barinova (*213*) proposed the photometric determination of vanadium in titanium by extraction of the vanadium phosphotungstate complex with isobutyl alcohol. Fluoride was added to complex the large amount of titanium.

Zinc. Miyajima (*121*) reported the rapid photometric determination of zinc in aluminum alloys with Xylenol Orange at a pH of 0.3. Ions such as calcium were masked with thiourea, ascorbic acid, and NH_4F. Monnier and Prod'hom (*122*) recommended a colorimetric determination of traces of zinc in bauxite, alumina, and refined aluminum with dithizone and a series of $CHCl_3$ and CCl_4 extractions.

Wakamatsu (*201*) proposed a rapid determination of zinc in aluminum alloys by titration with EDTA at pH 5 to 6 with PAN as indicator. The interferences of aluminum and tin were masked by NH_4F. Dimitrova (*31*) studied the chelometric determination of zinc in aluminum alloys by titrating with EDTA at pH 8.5 to 9.5 with Eriochrome Black T as indicator. Ishibashi and Komaki (*73*) developed a method for the determination of zinc in aluminum alloy by using a liquid anion exchanger and EDTA titration with Eriochrome Black T as an indicator at pH 9 to 10. The interfering iron was removed by a methyl isobutyl ketone extraction. Kharin and Soroka (*84*) described an ion exchange–electrolytic determination of zinc and copper in aluminum alloys not containing nickel. More details are in the Copper section.

Fleury (*44*) recommended the polarographic determination of 0.01 to 0.35% zinc in copper–aluminum alloys. Copper was separated by electrodeposition, iron was extracted by isobutyl methyl ketone, and zinc was measured polarographically in a NaOH–EDTA–gelatin mixture. Tajima and Kurobe (*172*) investigated the determination of copper, zinc, and iron in aluminum alloys by square-wave polarography without interference from other constituents. The polarograms for copper and zinc were recorded from an HCl solution at −0.025 and −1.05 volts, respectively, *vs.* the mercury pool electrode. Vasil'eva and Vinogradova (*194*) studied the determination of gallium, zinc, and cadmium in high-purity aluminum by anodic voltammetry at a stationary mercury electrode with a silver contact.

Gornaya (*58*) applied emission spectroscopy to make an approximate determination of 0.2 to 0.8% zinc in aluminum alloys by using an iron electrode. Neeb (*131*) determined small amounts of zinc in high-purity aluminum by vaporization and condensation on a water-cooled finger-condenser, followed by spark spectrography.

Wallace (*205*) employed atomic absorption spectroscopy for the determination of 0.02 to 6.0% zinc in aluminum alloys at 2138 A., slit width 0.3 mm., lamp current 44 ma., and air pressure 12 lb. per sq. in.

Ishibashi and Komaki (*74*) used liquid anion exchangers and EDTA titration for the separation and determination of zinc in nickel-magnesium alloy.

Zirconium. Wolna and Studencki (*209*) determined 0.05 to 1% zirconium in magnesium alloys by precipitating the zirconium with tartrazine and weighing the complex. Crawley (*23*) described the photometric determination of soluble and insoluble zirconium in magnesium alloys with PAN after extraction with trioctylphosphine oxide. Tuma and Kabicky (*186*) reported the photometric determination of zirconium in magnesium alloys with morin. The absorbance was measured at 436 mμ after the complex was held at 20°C. for 20 to 60 minutes. Titanium and fluorides interfere. Tikhonov (*178*) recommended the colorimetric determination of zirconium in magnesium alloys with Alizarin S without weighing the sample. A plexiglas cylinder was glued on the surface of the sample and 2 to 3 drops of concentrated HCl were added. After 5 minutes the solution was transferred to a graduate cylinder and the color was developed.

Wood and McKenna (*210*) proposed the photometric determination of 0.5 to 6% zirconium in titanium alloys with Alizarin Red S at 560 mμ.

LITERATURE CITED

(1) Adell, M. Roca, *Anales Real Soc. Espan. Fis. Quim. (Madrid) Ser. B* **59**, 345 (1963).
(2) Albert, Philippe, Gaittet, Jean, *Radioisotopes Phys. Sci. Ind., Proc. Conf. Use, Copenhagen*, 1960 **2**, 243–59 (Pub. 1962).
(3) Am. Soc. Testing and Materials, "Chemical Analysis of Metals," Part 32, Philadelphia, Pa., 1964.
(4) Asmus, E., Kurzmann, P., Wallsdorf, F., *Z. Anal. Chem.* **197**, 413 (1963).
(5) Berry, R., Walker, J. A. J., *Analyst* **88**, 280 (1963).
(6) Bertoldi, S., Tartàri, A., *Alluminio* **31**, 127 (1962).
(7) Biechler, D. G., Jordan, D. E., Leslie, W. D., ANAL. CHEM. **35**, 1685 (1963).
(8) Blackburn, R., Peters, B. F. G., *Ibid.*, p. 10.
(9) Bobrov, V. A., Chernogus, E. N., Yaroslavova, T. N., *Materialy Tret'ego Ural'sk. Soveshch. po Spektroskopii, Inst. Fiz. Metal., Akad. Nauk SSSR, Komis. po Spektroskopii, Sverdlovsk* **1960**, 66 (Pub. 1962).
(10) Bogatyrev, P. M., Navyazhskaya, E. A., Sporykhina, V. S., *Lakokrasochnye Materialy i ikh Primenenie* **1962** (3), 68.
(11) Bogatyrev, P. M., Navyazhskaya, E. A., Sporykhina, V. S., *Tr. Vses. Nauchn. Issled. Inst. Khim. Reaktivov* **1963** (25), p. 240.
(12) Boyle, Walter G., *U. S. At. Energy Comm. Rept.* **UCRL-6639,** 1–9 (1961).
(13) Boyle, Walter G., Jr., Gregory, Lewis, J., Sunderland, William, *U. S. At. Energy Comm. Rept.* **UCRL-7204,** 9 pp. (1963).
(14) Bradshaw, W., Johnson, R., Beard, D., U. S. Dept. Comm., Office Tech. Serv., **PB 159,872,** 28 pp. (1960).
(15) Brune, Dag, Aktiebolaget Atomenergi, Stockholm **AE-51,** 8 pp. (1961).
(16) Budanova, L. M., Pinaeva, S. N., *Zavodsk. Lab.* **29**, 149 (1963).
(17) Bugaeva, N. I., Mironenko, L. K., *Ibid.*, **30**, 419 (1964).
(18) Burke, Keith E., Davis, C. Manning, ANAL. CHEM. **36**, 172 (1964).
(19) Calrararu, H., *Rev. Chim. (Bucharest)* **14**, 349 (1963).
(20) Cattogio, Jose A., *Publ. Inst. Invest. Microquim., Univ. Nacl. Litoral* **24,** (22), 99 (1958).
(21) Christ, N., ANAL. CHEM. **35,** 775 (1963).
(22) Costin, I., *Metalurgia (Bucharest)* **15,** 511 (1963).
(23) Crawley, R. H. A., *Anal. Chim. Acta* **26,** 281 (1962).
(24) Czajka, Zofia, *Magy. Kem Folyoirat* **68,** 519 (1962).
(25) Davey, M. L., *Metallurgia, Manchr* **65,** 151 (1962).
(26) DeAngelis, Giorgio, Gerardi, Maria, *Ric. Sci. Rend. Sez. A. (2)*, **1** (1), 67 (1961).
(27) *Ibid.*, p. 76.
(28) Degtyareva, O. F., Sinitsyna, L. G., Proskuryakova, A. E., *Zh. Analit. Khim.* **17,** 926 (1962).
(29) *Ibid.*, **18,** 510 (1963).
(30) Deyris, M., Albert, P., *Mem. Sci. Rev. Metall.* **59,** 14 (1962).
(31) Dimitrova, M., *Mashinostroene (Sofia)* **10** (4), 23 (1961).
(32) Dymov, A. M., Kozel, L. Z., *Izv. Vysshikh Uchebn. Zavedenii, Chernaya Met.* **1961** (5), p. 192.
(33) Egorova, K. I., Gurevich, A. N., *Zavodsk. Lab.* **29** 789 (1963).
(34) Eisen, J., *Z. Erzbergbau Metallhuettenw.* **16,** 579 (1963).
(35) Elwell, W. T., Wood, D. F., *Analyst* **88,** 475 (1963).
(36) Erko, V. F., Lifshits', E. V., Konovalov, V. G., Dubins'kii, I. G., Bugaiova, N. I., *Ukr. Fiz. Zhur.* **6,** 837 (1961).
(37) Ershova, K. N., *Tr. Vses. Nauchn. Issled. Inst. Zheleznodor. Transp.* **1962** (227), 4 (1962).
(38) Evans, C., Herrington, J., *Radioisotopes Phys. Sci. Ind., Proc. Conf. Use, Copenhagen* **1960, 2,** 309 (Pub. 1962).
(39) Everest, D. A., "The Chemistry of Beryllium," Elsevier, New York, 1964.
(40) Everett, M. R., Thompson, G. E., *Analyst* **87,** 515 (1962).
(41) Fedorov, A. A., Linkova, F. V., *Zh. Analit. Khim.* **17,** 53 (1962).
(42) Fedorov, A. A., Linkova, F. V., *Sb. Tr. Tsentr. Nauchn. Issled. Inst. Chernoi Met.*, **1962** (24), 172.
(43) Fitzgerald, John J., NASA (Natl. Aeron. Space Admin.), *Doc.* **N63-14839,** 14 pp. (1963).
(44) Fleury, M., *Chim. Anal. (Paris)* **45,** 463 (1963).
(45) Fleury, M., Capelle, R., *Ibid.*, p. 117.
(46) *Ibid.*, p. 357.
(47) Fratkin, Z. G., *Zavodsk. Lab.* **30** (2), 170 (1964).
(48) Fujii, Isao, Muto, Haruo, Miyoshi, Katsuhiko, *Bunseki Kagaku* **13,** 249 (1964).
(49) Fujishima, I., Takeuchi, T., *Ibid.*, **10,** 1221 (1961).
(50) Furman, N. H., ed., "Standard Methods of Chemical Analysis," 6th ed., Vol. **1**, Van Nostrand, Princeton, 1962.
(51) Furuya, M., *Bunseki Kagaku* **11,** 1247 (1962).
(52) Galliford, D. J. B., Yardly, J. T., *Analyst* **88,** 653 (1963).
(53) Gilman, A. R., Isserow, S., *U. S. Atomic Energy Comm. Rept.* **NMI-1234,** 24 pp. (1960).
(54) Ginsberg, H., Pfundt, H., *Z. Metallk.* **53,** 695 (1962).
(55) Girardi, F., Pietra, R., ANAL. CHEM. **35,** 173 (1963).
(56) Gomiscek, S., *Rudarsko-Met. Zbornik,* **1961**, p. 403.
(57) Gordievskii, A. V., Ustyugov, G. P., *Izv. Vysshikh Uchebn. Zavedenii, Khim. i Khim. Tekhnol.* **4,** 366, (1962).
(58) Gornaya, R. I., *Zavodsk. Lab.* **29,** 1083 (1963).
(59) Gromoshinskaya, T. F., *Tr. Vses. Nauchn.-Issled. Alyumin.-Magnievyi Inst.* **49,** 200 (1962).
(60) Grossmann, O., Doege, H. G., *Kernenergie* **7,** 113 (1964).
(61) Guerreschi, L., Romita, R., *Ric. Sci., R. C., A,* **2,** 603 (1962).
(62) Gusarskii, V. V., Kuzovlev, I. A., *Zavodsk. Lab.* **25,** 1464 (1959).
(63) Gusarskii, V. V., Tarasevich, N. I., *Ibid.*, **28,** 183 (1962).
(64) Hasegawa, M., *Nippon Kinzoku Gakkaishi* **24,** 493 (1960).
(65) Hashimoto, S., Tanaka, R., *Bunseki Kagaku* **8,** 564 (1959).
(66) Hashitani, H., *Nippon Genshiryoku Gakkaishi* **4,** 287 (1962).
(67) Hayes, M. R., Metcalfe, J., Elwell, W. T., Wood, D. F., *Analyst* **88,** 471 (1963).
(68) Hibbits, J. O., Kallmann, S., *Talanta* **10,** 181 (1963).
(69) Hine, R. A., Bates, J. F., *Appl. Mater. Res.,* **2,** 215 (1963).
(70) Hine, R. A., Bates, J. F., *Metallurgia,* **65,** 101 (1962).
(71) Hirano, S., Mizuike, A., Iida, Y., *Kogyo Kagaku Zasshi* **62,** 1491 (1959).
(72) Ichiryu, A., Hashimoto, S., *Bunseki Kagaku* **10,** 1137 (1961).
(73) Ishibashi, M., Komaki, H., *Ibid.*, **11,** 43 (1962).
(74) *Ibid.*, p. 756.
(75) Ivanova, Z. I., Kovalenko, P. N., Tsyvenkova, T. V., *Elektrokhim. i Optich. Metody Analiza Sb.*, 191 (1963).
(76) Jackson, H., Phillips, D. S., *Analyst* **87,** 712 (1962).
(77) Kajzer, M., *Glasnik Hem. Drustva, Beograd,* **25/26,** 217 (1960–61).
(78) Kalinin, Yu. S., Kondrashova, G. P., Mironov, D. E., Yalymov, G. I., *Tr. po Khim. i Khim. Tekhnol.* **4,** 472 (1961).
(79) Kalinnikov, V. T., Shteinberg, A. N., *Titan i Ego Splavy, Akad. Nauk SSSR., Inst. Met.* **8,** 260 (1962).
(80) Karalova, Z. K., Nemodruk, A. A., *Zh. Analit. Khim.* **17,** 985 (1962).
(81) Karev, V. N., Matyushenko, N. N., *Zavodsk. Lab.* **30,** 45 (1964).
(82) Kern, R., *Physik Kondensierten Materie* **1,** 105 (1963).
(83) Keys, J. H., Rowan, J. H., *U. S. At. Energy Comm. Rept.* **Y-1447,** 14 pp. (1963).
(84) Kharin, A. N., Soroka, N. N., *Zh. Prikl. Khim.* **37,** 672 (1964).
(85) Khasnobis, S. K., *Indian J. Appl. Chem.* **25,** 71 (1962).
(86) Kida, K., Abe, M., Nishigaki, S., Kobayashi, K., *Bunseki Kagaku* **9,** 1031 (1960).
(87) Kida, K., Abe, M., Nishigaki, S., Kusaka, T., *Ibid.*, **10,** 358 (1961).

(88) *Ibid.*, p. 1217.
(89) Kiesl, W., Bildstein, H., Hecht, F., *Radiochim. Acta* 1 (3), 123 (1963).
(90) Kiesl, W., Bildstein, H., Sorantin, H., *Mikrochim. Ichnoanal. Acta*, **1963**, 151.
(91) Kiesl, W., Sorantin, H., Pfeifer, V., *Mikrochim. Ichnoanal. Acta* **1963**, 996.
(92) Klug, Otto, Sajo, Istvan, *Kohasz. Lapok* **95**, 425 (1962).
(93) Klyachko, Yu. A., Kunin, L. L., Chistyakova, E. M., *Sb. Tr. Tsentr. Nauchn.-Issled. Inst. Chernoi Met.*, **24**, 42 (1962).
(94) Kobayashi, M., Nagatsuka, S., Sawai, T., *Radioisotopes* **13**, 26 (1964).
(95) Kolthoff, I. M., Elving, P. J., eds., "Treatise on Analytical Chemistry," Part II, Vol. **5**, p. 41, Interscience, New York, 1961.
(96) Korenman, Ya. I., *Tr. Khim. i Khim. Tekhnol.* (*Gor'kii*) 384 (1962).
(97) Kotsis, Tivadar, *Femip. Kut. Int. Kozlemen.* **5**, 333 (1961).
(98) Kovalenko, T. V., *Zavodsk. Lab.* **25**, 1519 (1959).
(99) Krejzova, E., Kruml, J., Plocek, L., *Sklar Keram.* **9**, 244 (1959).
(100) Kukula, F., Slunecko, J., Simkova, M., *Jaderna Energie* **9**, 166 (1963).
(101) Kuzovlev, I. A., Gusarski, V. V., *Zavodsk. Lab.* **28**, 1076 (1962).
(102) Landi, M. F., *Alluminio* **31**, 577 (1962).
(103) Laszlo, P., *Magyar Kem. Foly.*, **68**, 523 (1962).
(104) Laverlochere, J., *Bull. Soc. Chim. France*, **1963**, 457.
(105) Laverlochere, J., *Chim. Anal.* **44**, 388 (1962).
(106) Lemieux, P. E., *Appl. Spectry.* **17**, 153 (1963).
(107) Lewis, R. W., Earl, C. F., Potter, J. L., Fehler, R., *U. S. Bur. Mines, Rept. Invest.* 6108, 15 pp. (1962).
(108) Lihl, Franz, Fischhuber, Johann, *Z. Metallk.* **53**, 186 (1962).
(109) Lin, Robert, *U. S. At. Energy Comm. Rept.* **UCRL-6639**, 10 (1961).
(110) Linanov, V. A., Bukhanova, A. A., Kolachev, B. A., *Tr. Mosk. Aviats. Tekhnol. Inst.* **55**, 90 (1962).
(111) Linanov, V. A., Gorokhov, V. P., Golofaev, T. I., Malyavkina, V. P., *Izv. Akad. Nauk SSSR, Ser. Fiz.*, **26**, 914 (1962).
(112) McCrary, J. H., Morgan, I. L., Baggerly, L. L., *Proc. Intern. Conf., Modern Trends Activation Anal.*, College Station, Tex., p. 24 (1961).
(113) Malevannyi, V. A., *Lakokrasochnye Materialy i ikh Primenenie*, **1962**, 54.
(114) Matelli, G., Attini, E., *Alluminio* **29**, 227 (1960).
(115) Mendlina, N. G., *Zh. Analit. Khim.*, **18**, 408 (1963).
(116) Meyer, R. A., Austerman, S. B., Swarthout, D. G. Anal. Chem. **35**, 2144 (1963).
(117) Mikheeva, L. M., Wihitill, I., *Zh. Analit. Khim.* **17**, 84 (1962).
(118) Mills, E. C., Hermon, S. E., *Metal Ind.* (*London*), **101**, 492 (1962).
(119) Milos, Jozefa, *Chem. Anal.* (*Warsaw*) **7**, 373 (1962).
(120) Miyajima, T., *Bunseki Kagaku* **12**, 742 (1963).
(121) *Ibid.*, **13**, 257 (1964); cf. Ishiwatari, *et al.*, *Ibid.* **12**, 603 (1963).
(122) Monnier, D., Prod'hom, G., *Anal. Chim. Acta* **30**, 358 (1964).
(123) Morita, K., *Nagoya Kogyo Gijutsu Shikensho Hokoku* **12**, 296 (1963).
(124) *Ibid.*, p. 406.
(125) Morris, W. F., *U. S. At. Energy Comm. Rept.* **UCRL-6639**, 16 (1961).
(126) Mukai, K., Mochizuki, Y., *Bunseki Kagaku* **13**, 259 (1964).
(127) Munshi, K. N., Dey, A. K., *J. Prakt. Chem.*, **18**, 233 (1962).
(128) Musil, F. J., *U. S. At. Energy Comm. Rept.* **GAT-T-1053**, 27 pp. (1962).
(129) Navyazhskaya, E. A., Sporykhina, V. S., *Lakokrasochnye Materialy i ikh Primenenie*, No. 4, 52 (1962).
(130) Nazarenko, V. A., Shustova, M. B., Shitareva, G. G., Yagnyatinskaya, G. Ya., Ravitskaya, R. V., *Zavodsk. Lab.* **28**, 645 (1962).
(131) Neeb, K. H., *Z. Anal. Chem.*, **194**, 255 (1963).
(132) Nemodruk, A. A., Palei, P. N., Hun-I, He, *Zavodsk. Lab.* **28**, 406 (1958).
(133) Neverovsky, E., *Rev. Brasil. Quim.* (*Sao Paulo*) **56**, 191 (1963).
(134) Oda, N., Kubo, M., *Bunseki Kagaku* **10**, 1262 (1961).
(135) Oda, N., Norishima, K., Kubo, M., *Ibid.*, **11**, 526 (1962).
(136) Oda, N., Tsunoo, S., Hashimoto, T., *Trans. Japan Inst. Metals*, **1**, 130 (1960).
(137) Oida, K., Fujita, K., Takahashi, I., *Bunseki Kagaku* **11**, 1136 (1962).
(138) Onishi, K., *Ibid.*, **12**, 959 (1963).
(139) Orlova, N. M., Petrov, A. A., *Vestn. Leningr. Univ. Ser. Fiz. i Khim.* **1964**, 69.
(140) Orlovtsev, Yu. V., Krapukhin, V. V., Krestovnikov, A. I., *Izv. Vysshikh Uchebn. Zavedenii, Tsvetn. Met.* **5**, 132 (1962).
(141) Ottolini, A. C., *Develop. Appl. Spectry.* **1**, 91 (1962).
(142) Owens, E. B., *Appl. Spectry.* **16**, 86 (1962).
(143) Penner, E. M., Inman, W. R., *Can. Dept. Mines Tech. Surv., Mines Branch, Tech. Bull.* **TB-49**, 16 pp. (1963).
(144) Peter, L., *Magy. Kem. Folyoirat* **69**, 231 (1963).
(145) Peter, L., *Talanta* **10**, 513 (1963).
(146) Petit, J., Engelmann, Ch., *Prod. Use Short-Lived Radioisotopes Reactors, Proc. Seminar, Vienna*, **2**, 29 (1962) (Pub. 1963).
(147) Pilgrim, W. E., Ford, W. R., Anal. Chem. **35**, 1735 (1963).
(148) Pitwell, L. R., *Analyst* **87**, 684 (1962).
(149) Pocze, L., *Kohasz. Lapok* **96**, 179 (1963).
(150) Pohl, H., *Aluminum* **34**, 710 (1958).
(151) Pollock, E. N., Zopatti, L. P., *Anal. Chim. Acta* **28**, 68 (1963).
(152) Postma, F. W., Jr., McMurray, C. S., Jr., *U. S. At. Energy Comm. Rept.* **TID-7655**, 90 (1962) (Pub. 1963).
(153) Pribil, R., Vesely, V., *Chemist Analyst* **52**, 43 (1963).
(154) Romand, J., Balloffet, G., Vodar, B., *Colloq. Spectros. Intern., 8th, Lucerne, Switzerland*, 160, **1959** (Pub. 1960).
(155) Rosenberg, K., *U. S. At. Energy Comm. Rept.* **IA-672**, 11 pp. (1961).
(156) Ross, W. J., Anal. Chem. **36**, 1114 (1964).
(157) Sajo, I., Pozsgay, G., Klug, O., *Kohasz. Lapok* **96**, 412 (1963).
(158) Sawada, T., *Keikinzoku* **11**, 55 (1961).
(159) Sawada, T., Kato, S., *Bunseki Kagaku* **11**, 544 (1962).
(160) Sawada, T., Kato, S., *J. Japan Inst. Metals, Sendai*, **26**, 190 (1962).
(161) Schroeder, W. S., Strasheim, A., *Colloq. Spectros. Intern., 9th, Lyons*, **2**, 117 (1961) (Pub. 1962).
(162) Shimazaki, K., Nakayama, T., Nakamura, R., *Nippon Kinzoku Gakkaishi* **25**, 269 (1961).
(163) Shkaravskii, Yu. F., *Zh. Analit. Khim.* **19**, 514 (1964).
(164) Shustova, M. B., Nazarenko, V. A., *Khim. Prom., Nauk.-Tekhn. Zb.*, No. 4, 78 (1962).
(165) Sikes, J. H., Wade, M. A., Yamamura, S., CONF-157-5, (1959). ACS, Northwest Regional Meeting, Bellingham, Wash., June, 1963; *Nucl. Sci. Abstr.* **18**, 706 (1964).
(166) Smart, A., *Metallurgia* **69**, 245 (1964).
(167) Steckel, L. M., Hall, J. R., *U. S. At. Energy Comm. Rept.* **Y-1406**, 18 pp. (1962).
(168) Stross, W., Clark, J., *Metallurgia* **67**, 47 (1963).
(169) *Ibid.*, **68**, 191 (1963).
(170) Suzuki, T., *Bunseki Kagaku* **12**, 655 (1963).
(171) Tah, Fu-Hai, *Wu Li Hsueh Pao* **17**, 495 (1961).
(172) Tajima, N., Kurobe, M., *Bunseki Kagaku* **10**, 146 (1961).
(173) Tajima, N., Kurobe, M., Terada, H., *Ibid.*, p. 1340.
(174) Tanaki, R., *Ibid.*, p. 336.
(175) Teillac, J., *Microtecnic* **17**, 258 (1963).
(176) Thieme, O., *Hermsdorfer Tech. Mitt.* (*HTM*) **1**, 8 (1960).
(177) Tikhonov, V. N., *Izv. Vysshikh Uchebn. Zavedenii, Khim. i Khim. Tekhnol.* **5**, 31 (1962).
(178) *Ibid.*, p. 214.
(179) Tikhonov, V. N., *Zh. Analit. Khim.* **17**, 422 (1962).
(180) Tikhonov, V. N., Grankina, M. Ya., *Ind. Lab.* **29**, 696 (1963).
(181) Tikhonov, V. N., Nikitina, A. P., *Zavodsk. Lab.* **28**, 662 (1962).
(182) Todd, R., Cuthbert, G., Dickinson, R., *U. K. At. Energy Authority, Prod. Group PG Rept.* **337**, 35 pp. (1962).
(183) Torok, T., *Talanta* **10**, 527 (1963).
(184) Tsuji, N., Abe, K., Ando, M., *Bunseki Kagaku* **11**, 648 (1962).
(185) Tucholka-Szmeja, B., *Chem. Anal.* (*Warsaw*) **7**, 463 (1962).
(186) Tuma, H., Kabicky, V., *Talanta* **8**, 749 (1961).
(187) Tumanov, A. A., Sidorenko, A. N., *Tr. Khim. i Khim. Tekhnol.* (*Gor'kii*), 378 (1962).
(188) *U. K. At. Energy Authority Rept.* **PG-348**(S), 1962.
(189) *Ibid.*, **PG-409** (S), 1963.
(190) Vahldick, F. W., Lynch, D. T., Robinson, L. B., Anal. Chem. **34**, 1667 (1962).
(191) Valach, R., *Czech.* **105,355**, Oct. 15, 1962.
(192) Valnshtein, E. E., Korolev, V. V., Savinova, E. N., *Chem. Anal., Warsaw*, **7**, 187 (1962).
(193) Van Sandt, W. A., Santomassino, V. C., Rumble, R. P., Barlow, O. M., *Am. Ind. Hyg. Assoc. J.* **23**, 203 (1962).
(194) Vasil'eva, L. N., Vinogradova, E. N., *Zh. Analit. Khim.* **18**, 454 (1963).
(195) Vilnat, J., Voinovitch, I. A., *Publ. Group. Avan. Methodes Spectrog.* (10), 307 (1962).
(196) Vinogradova, E. N., Vasil'eva, L. N., *Zh. Analit. Khim.* **17**, 579 (1962).
(197) Volkova, A. I., Get'man, T. E., Emtsova, N. A., *Ukr. Khim. Zh.* **30**, 102 (1964).
(198) Volodarskaya, R. S., Derevyanko, G. N., *Zavodsk. Lab.* **29**, 148 (1963).
(199) Voronezhskaya, I. A., Mladentseva, O. I., Aksenova, A. V., Gradoboeva, R. A., *Ibid.*, **28**, 557 (1962).
(200) Vvedenskii, L. E., Shekhobalova, V. I., Novikova, A. S., *Izv. Akad. Nauk SSR, Ser. Fiz.*, **26**, 896 (1962).
(201) Wakamatsu, S., *Nippon Kinzoku Gakkaishi* **24**, 504 (1960).
(202) Walkden, J., Heathfield, K. E., *At. Energy Research Estab.* (*Gt. Brit.*) **AM 32**, 1 (1959).
(203) Wallace, F. J., *Analyst* **88**, 259 (1963).

(204) Wallace, F. J., *Foseco Developm.*, (8), 72 (1963).
(205) Wallace, F. J., *Hillger J.* **7**, 39 (1962).
(206) Wells, J. R., Carpenter, Ll., *U. S. Bur. Min. Rep. Invest.* **6105**, 23 pp. 1962.
(207) Will, Fritz, III, Moss, M. L., ANAL. CHEM. **35**, 96R (1963).
(208) Wilson, L., *Anal. Chim. Acta* **30**, 377 (1964).
(209) Wolna, Jozefa, Studencki, Jerzy, *Rudy Metale Niezelazne* **7**, 557 (1962).
(210) Wood, D. F., McKenna, R. H., *Analyst* **87**, 880 (1962).
(211) Wood, G. C., Marron, V. J. J., Lambert, B. W., *Nature* **199**, 239 (1963).
(212) Yakovlev, P. Ya., Malinina, R. D., *Sb. Tr. Tsentr. Nauchn.-Issled. Inst. Chernoi Met.* **24**, 136 (1962).
(213) Zinchenko, V. A., Barinova, O. D., *Titan i Ego Splavy, Akad. Nauk SSSR, Inst. Met.* **8**, 251 (1962).
(214) Zinchenko, V. A., Ershova, N. A., Gertseva, N. M., *Titan i Ego Splavy, Akad. Nauk SSSR, Inst. Met.* **8**, 242 (1962).

Nonferrous Metallurgy

II. Zirconium, Hafnium, Vanadium, Niobium, Tantalum, Chromium, Molybdenum, and Tungsten

Robert Z. Bachman and Charles V. Banks
Institute for Atomic Research and Department of Chemistry, Iowa State University, Ames, Iowa

THIS REVIEW, which is appearing for the first time, is concerned with methods for the determination of zirconium, hafnium, vanadium, niobium, tantalum, chromium, molybdenum, and tungsten which appeared in the literature between January 1962 and July 1964. A recent review (*21*) discusses the literature on the analytical chemistry of these elements along with titanium for the period from 1950 through 1960. A review of the literature appearing in 1961 was included in the Nonferrous Metallurgy Section of Analytical Reviews—1963. Because this is the first review in Analytical Reviews dealing exclusively with these elements, enough different methods and types of methods are included to try to present a representative cross-section of the material being published on the determination of these elements. To make this review more useful to those engaged in the determination of these metals, an effort was made to include a brief statement as to the content of each paper.

Those references which appeared in the Ferrous or Nonferrous Metallurgy Sections of Analytical Reviews—1963 were intentionally omitted from this review. Although titanium has been included in some reviews of the analytical chemistry of these elements, it was felt in this case that better continuity would be maintained by including titanium with the other "light-structural metals" in Nonferrous Metallurgy, Part I. The elements vanadium, chromium, molybdenum, and tungsten are discussed separately; however, it was found to be more satisfactory to discuss zirconium and hafnium together and niobium and tantalum together. Analytical methods are summarized in Tables I–XI.

ZIRCONIUM AND HAFNIUM

Zirconium and hafnium are found together in nature, are usually discussed together in the literature, and most wet chemical methods work equally well for both. Consequently, they will be considered together in this review to avoid repetition.

Elwell and Wood (*84*) have recently authored a book on the analysis of zirconium entitled "The Analysis of Titanium, Zirconium, and Their Alloys." Recent reviews of the analytical chemistry of zirconium and hafnium include a two-part review by Ito and Hoshino (*123, 124*) and one by Elinson (*80*). McKaveney (*165*) used round-robin analysis on three niobium-base alloys to evaluate several types of procedures for the determination of a number of elements including zirconium in niobium.

Activation analysis is a technique which has proved useful for the determination of zirconium in the presence of hafnium and of hafnium in the presence of zirconium. Oka, Kato, and Sasaki (*203*) added copper as an internal standard to mixtures of zirconium and hafnium and then measured the ratio of radioactivity of zirconium-89 to copper-62 to determine zirconium in hafnium. Zitnansky and Sebestian (*293*) irradiated pure zirconium along with the unknown sample to determine the zirconium correction for the determination of hafnium in zirconium. Kamemoto and Yamagishi (*132*) irradiated a standard along with the sample to determine 0.06 to 1.1% hafnium in zirconium. They used the γ-rays from hafnium-181 rather than hafnium-179. Hafnium along with a number of elements was determined by Fournet (*89*) using activation analysis in pure zirconium prepared by two different methods. Ehmann and Setser (*78*) applied activation analysis and radiochemical separations to the determination of both hafnium and zirconium in stone meteorites. Chinaglia *et al.* (55) included hafnium in a discussion of the use of short-lived radionuclides in activation analysis. Girardi and Pietra (*97*) used an activation method coupled with separations for the determination of hafnium in aluminum. Activation analysis techniques were used by Gruverman and Henninger (*101*) for the determination of zirconium in alloy steels and electroetch residues. Radiochemical methods have also been frequently used to determine the amount of zirconium-95 in various substances under different conditions. Seyb (*250*) proposed a mathematical treatment of the decay curves which allows the determination of both zirconium-95 and niobium-95 in mixtures. Overman (*210*) used a β–γ coincidence counting technique to resolve mixtures such as zirconium-95 and niobium-95. Maeck, Marsh, and Rein (*167*) utilized zirconium-97 to evaluate critical nuclear incidents in which large levels of fission products were present prior to the incident. Park, Kim, and Suo (*215*) determined the concentration of zirconium-95 and a number of other elements in air to find the level of fallout from weapons tests. MacDonald *et al.* (*163*) determined some γ-emitting nuclides including zirconium-95 in newborns, infants, and children, and Cofield (*60*) worked out techniques for the determination of zirconium-95 and other nuclides in human lungs. Techniques for the determination of important fission products including zirconium have been applied to rain water by Buchtela and Lesigang (*36*); to vegetables by Park, Kim, and Suo (*216*); to marine sediments by Osterberg, Kulm, and Byrne (*209*); to seaweed and seawater by Hampson (*108*); and to soil and river-bed samples by Sakagishi, Ueno, and Minami (*241*).

During the period covered by this review there were also a number of papers published which described gravimetric methods for the determination of zirconium. Several of these were merely procedures for finishing the determination after a separation had been achieved by some other means, while some involved the use of reagents which were claimed to separate zirconium from some other elements. Machlan and Hague (*164*) used an anion exchange method for the separation of zirconium and hafnium and then finished the determination by means of a cupferron precipitation of both elements. Rafiq, Rulfs, and Elving (*229*) studied the precipitation of zirconium by mandelic acid, *p*-bromomandelic acid, and *m*-nitromandelic acid and determined the optimum conditions for quantitative precipitation at the 0.1- and 1-mg. levels. These same authors (*230*) used *p*-bromomandelic acid for the determination of zirconium in refractory samples. Sheskol'skaya (*254*) used a cupferron precipitation for the separation and determination of zirconium in the presence of large amounts of molybdenum and tungsten. Maienthal and Taylor (*172*) also used cupferron for the determination of zirconium in zirconia–yttria mixtures by what is claimed to be a simple and accurate procedure. Takahashi (*263*) used a cupferron precipitation for the determination of zirconium in zirconium diboride. Thiosalicylic acid was used by Dema (*68*) for the precipitation separation of zirconium from many elements including uranium, aluminum, vanadium, molybdenum, tungsten, the rare earths, and the alkaline earths and for the determination of zirconium. Other organic compounds which have been used for the precipitation of zirconium are 2,3-dihydroxynaphthalene-6-sulfonic acid (*69*), *p*-iodoamygdalic acid (*253*), chelidonic acid (*173*), phenylacetylhydroxamic acid (*175*), *N*-benzoylphenylhydroxylamine (*10*), tartrazine (*224*), and three aromatic carboxylic acids: 1,3,5-benzenetricarboxylic acid; 1,2,4-benzenetricarboxylic acid; and 1,2,4,5-benzenetetracarboxylic acid (*194*). Adams and Holness (*2*) made a thermogravimetric study of the selenites of zirconium and hafnium and found them suitable for the determination of the two elements in mixtures if the proper conditions are carefully maintained.

The majority of the titrimetric methods for zirconium involve the use of EDTA and either use a new indicator for the detection of the end point or recommend a new separation to remove interfering ions prior to the titration. The amperometric titration methods will be included with the polarographic methods. Elbeih and Garba (*79*) described a paper chromatographic separation using an antipyrine–dioxane–nitric acid solvent system in which zirconium and hafnium do not move to separate the alkali metals, rare earths, copper, mercury, tin, thorium, chromium, nickel, and other elements before the zirconium and hafnium are titrated with EDTA. Volodarskaya and Derevyanko (*276*) titrated zirconium to the xylenol orange end point after removing the interference of iron(III) by reducing it with hydroxylammonium chloride. Tsuchiya (*270*) titrated zirconium and hafnium with EDTA to the xylenol orange end point in a boiling solution containing 0.9 ± 0.3*M* hydrochloric acid. Up to 1 gram of uranium(VI), iron(II), aluminum, zinc, tin, and magnesium do not interfere. Pribil and Vesely (*226*) also titrated zirconium with EDTA using xylenol orange as the indicator in a hot solution which was 0.3 to 0.6*M* in nitric acid. Hoshino (*118*) determined small amounts of hafnium in zirconium by extracting the hafnium into cyclohexanone from an aqueous solution containing ammonium thiocyanate, ammonium sulfate, and hydrochloric acid. After a number of scrubbing steps the hafnium was back-extracted into 2*M* sulfuric acid and titrated with EDTA. The quantitative oxidation of mandelic acid to benzoic acid by vanadium(V) was used by Panwar and Gaur (*213*) as a titrimetric method for zirconium. Matthes, Boethin, and Rybak (*182*) used a somewhat different titrimetric method. Zirconium hydroxide was treated with potassium fluoride in the presence of an excess of 0.1*M* nitric acid followed by the titration of the excess acid with 0.05*M* potassium hydroxide. Singh and Agarwala (*257*) used essentially the same method except they performed a direct titration of the liberated hydroxide which was formed from the reaction of potassium fluoride and zirconium hydroxide. A number of cations including aluminum, lanthanum, thorium, and the alkaline earths interfere. A 30-megacycle, high-frequency titrimeter was used by Kumar and Singh (*151*) to detect the end point of a precipitation titration of zirconium with potassium dihydrogen phosphate.

Table I. Spectrophotometric

Element	Reagent: Material (References)
Zirconium	Xylenol orange: Study of conditions and/or interferences (*37, 51, 52*); alkali metals, alkaline earths (*49*); Nb, Ta, W (*110*)
	Semixylenol orange: Study of complexes (*207*)
	Arsenazo III: Study of conditions and/or interferences (*243*); Zr-Th (*208*); Ti, Th, Fe, Ca, Mg (*269*); Zr, Hf, Pa, Sc, Th, U mixtures (*244, 245*)
	Sodium alizarin sulfonate: U (*119, 121*); Ti (*71, 288*); study of conditions and/or interferences (*214*)
	Purpurogallin: Study of conditions and/or interferences (*73*); Fe (*8*)
	Chlorophosphinazo III: Study of conditions and/or interferences (*85*)
	8-Quinolinol: U (*193*)
	Flavinol-2′-sulfonic acid: Study of conditions and/or interferences (*204*)
	Galangin: Study of conditions and/or interferences (*135*)
	N,*N*′-di-(2-hydroxy-5-sulfophenyl)-*c*-cyanoformazon: Study of conditions and/or interferences (*273*)
	Molybdosulfatozirconate: Study of conditions and/or interferences (*67*)
	Methylthymol blue: Study of conditions and/or interferences (*52*)
Hafnium	Arsenazo III: Zr, Hf, Pa, Sc, Th, U mixtures (*244, 245*)
	Mandelic acid, chloranilic acid, quercetin, xylenol orange: Zr (*127*)
Vanadium	Xylenol orange: U (*37*)
	Vanadium(V) ion: Study of conditions and/or interferences (*242*)
	Vanadium(II), (III), (IV) ions: Study of conditions and/or interferences (*180*)
	Vanadate and vanadyl ions: Study of conditions and/or interferences (*90*)
	Phosphotungstovanadic acid: Cr, W, V (*286*); Al (*31*)
	8-Quinolinol: Study of conditions and/or interferences (*171*); U (*17*)
	Catechol: Complex alloys (*110*)
	3,3′-Diaminobenzidine: Study of conditions and/or interferences (*53*)
	Tetraphenyl phosphonium chloride: Zn, Al, Cu, Co, Ti (*159*)
	Thiocyanate-pyridine: Study of conditions and/or interferences (*18*)
	Thiophene-2-hydroxamic acid: Study of conditions and/or interferences (*187*)
	2,6-Pyridinedicarboxylic acid: Study of conditions and/or interferences (*218*)
	Solochrome Fast Red: Study of conditions and/or interferences (*145*)
	Solochrome Fast Grey: Fe, Cu (*125*)
Niobium	Xylenol orange: Study of conditions and/or interferences (*20, 54, 82, 122*)
	4-(2-Pyridylazo)-resorcinol: Study of conditions and/or interferences (*23, 24*)
	1-(2-Pyridylazo)-resorcinol: Ta, Ti, Zr (*7*); Ti, Zr (*81*)
	Thiocyanate: Study of conditions and/or interferences (*186, 221, 222*); U (*162*); Ti (*179*); complex alloys (*110*)
	Strychnine-thiocyanate: Study of conditions and/or interferences (*169*); Ti, Nb, Ta (*181*)
	Pyrogallol: Mo, W, Cr (*46*); minerals and ores (*279*)
	Hydroquinone: Be, BeO (*115*)

The papers describing spectrophotometric methods for the determination of zirconium are for the most part applications of, or additional research on, previously published methods. Cheng (*52*) studied the factors affecting color formation and the properties of the complexes formed by zirconium and hafnium with xylenol orange and methylthymol blue. It was observed that zirconium formed

Methods

Element	Reagent: Material (References)
	Tribromopyrogallol: Study of reagents (*1*)
	Acid Chrome Violet K: Ores (*268*)
	Lumogallion: Ta, Zr (*6*)
	Derivatives of 2,3,7-trihydroxy-6-fluorone: Study of reagents (*201*)
	Compounds containing *O,O′*-dihydroxyazo: Study of reagents (*9*)
	Phosphomolybdic acid: Study of conditions and/or reagents (*256*)
	Peroxide: Pyrochlore soils (*22*)
Tantalum	Pyrogallol: Ti, Nb (*181*); Ti (*179*); Be, BeO (*115*); V (*47*); minerals and ores (*279*); complex alloys (*110*)
	Methyl violet: Study of reagent (*221, 222*)
	1-(2-Pyridylazo)-resorcinol: Nb, Ti, Zr (*7*)
	Pyrocatechol Violet: Study of reagents (*19*)
	12-Molybdotantalic acid: Nb, Ti (*102*)
Chromium	*s*-Diphenylcarbazide: Study of reactions (*178, 294*); Be (*223*); U, U oxides (*119, 237*); W, Mo, V (*286*); Nb, Ta, W (*110*); sapphire and ruby (*56*); diphenyl (*190*)
	EDTA, NTA, HEDTA, DCTA, DTPA: Study of absorption spectra (*33*)
	EDTA: Ores (*25*)
	1,2-Diaminocyclohexanetetraacetic acid: Ores, refractories (*248*)
	Tribenzylamine: Study of conditions and/or interferences (*86*)
	Thioglycolic acid: Study of reactions (*143*)
Molybdenum	Thiocyanate: Study of interferences (*116*); W (*219*); U (*119*); Cr, W, V (*286*); steel (*92*); sea water (*281*)
	Diantipyrylmethane-thiocyanate: Steel (*264*)
	8-Quinolinol: Study of conditions and/or interferences (*171*); U (*192*); nuclear reactor materials (*75*)
	Xylenol orange: U (*38*)
	Thioglycolic acid: Study of conditions and/or interferences (*143*)
	8-Quinolinol-5-sulfonic acid: Alkaline earths, Ni, Cd, Al, Th, Mn, Cr (*41*)
	8-Mercaptoquinoline: Study of conditions and/or interferences (*170*)
	1,10-Phenanthroline: Study of conditions and/or interferences (*109*)
	2-Amino-4-chlorobenzenethiolhydrochloride: Steel and cast iron (*139*)
	Chloroanilic acid: Study of conditions and/or interferences (*158*)
	Diethyldithiocarbamic acid: Study of conditions and/or interferences (*103*)
	1-Mercaptopropionic acid: Study of reagent (*42*)
	Solochrome Fast Red: Rare earths (*145*)
	Toluene-3,4-dithiol: Nb (*117*); Nb, Ta, W (*110*)
	Molybdenum(V): Study of conditions (*183*)
Tungsten	Thiocyanate: Fe, steel (*65, 92, 161, 219*); Cr, V, Mo (*286*)
	8-Quinolinol: V, Mo (*171*); steel, nuclear materials (*74*)
	Toluene-3,4-dithiol: Nb (*117*); Nb, Ta (*110*)
	Tetraphenylarsonium-thiocyanate: Steel and heat-resistant alloys (*4*)
	Thioglycolic acid: Study of reactions (*143*)

more stable complexes than hafnium but that the zirconium complexes are completely masked by hydrogen peroxide. Mandelic acid, chloranilic acid, quercetin, and xylenol orange were investigated by Johnson (*127*) as possible reagents for the differential spectrophotometric determination of hafnium in zirconium. Xylenol orange was found to be the most satisfactory. Budesinsky (*37*) investigated the reactions of a number of cations including zirconium with xylenol orange and calculated the formation and stability constants. Chen and Chu (*51*) also described the determination of zirconium with xylenol orange. Cerrai and Testa (*49*) determined zirconium after extraction with tri-*n*-octylamine by using xylenol orange. Headridge and Dixon (*110*) first separated zirconium from niobium, tantalum, and tungsten by means of ion exchange and then determined it with xylenol orange. Olson and Margerum (*207*) studied the zirconium complexes with a related compound, semixylenol orange, and described a method for the determination of zirconium using this reagent. Savvin (*243*) proposed the use of arsenazo (III) for the determination of zirconium. Savvin (*244, 245*) also described a method for the determination of zirconium, hafnium, protactinium, scandium, thorium, and uranium in mixtures using arsenazo (III) without separation. Onishi (*208*) also worked out the conditions for the determination of thorium and zirconium without separation by using arsenazo III. Tserkovnitskaya and Borovaya (*269*) used arsenazo (III) for the determination of zirconium following separation by extraction of the mandelate into isobutanol. Fadeeva and Alimarin (*85*) studied the conditions necessary for the determination of zirconium and other elements with chlorophosphinazo (III). Dehne and Mellon (*67*) used the reduced form of a complex molybdosulfatozirconate for the spectrophotometric determination of zirconium. Other reagents used for the spectrophotometric determination of zirconium are sodium alizarin sulfonate (*71, 119, 121, 214, 288*), 8-quinolinol (*193*), purpurogallin (*8, 73*), flavonol-2′-sulfonic acid (*204*), *N,N′*-di-(2-hydroxy-5-sulfophenyl)-*c*-cyanoformazon (*273*), and galangin (*135*). Hercules (*114*) used the fluorescence of the zirconium–quercetin complex for the determination of zirconium following separation from iron, vanadium, and titanium by an extraction with thenoyltrifluoroacetone.

A few polarographic and amperometric titration methods have been proposed for zirconium and hafnium even though these elements are not well suited to this type of determination and therefore reducible reagents or back-titrants are required. Goldstein, Manning, and Zittel (*98*) used the vanadyl ion as a back-titrant for the excess EDTA in an amperometric method for zirconium with a standard deviation of about 1%. Gallai, Alimarin, and Sheina (*94*) used *N*-benzoylphenylhydroxylamine for the titration of zirconium and titanium in the presence of nickel, manganese, cobalt, aluminum, chromium, and iron. Cupferron was used as the titrant by Kubota and Surak (*149*) in an amperometric titration of zirconium in highly radioactive solutions. The amperometric titration of zirconyl chloride using potassium ferrocyanide as the titrant was carried out by Gaur (*95*). Sharma and Gaur (*252*) used potassium tellurite as the titrant in amperometric, conductometric, and potentiometric titrations of zirconium. Hu, Wang, and Chang (*120*) precipitated zirconium with *m*-nitrobenzoic acid and then dissolved the precipitate and determined the *m*-nitrobenzoic acid polarographically. Pan, Sun, and Wong (*212*) performed a polarographic determination of zirconium in a cadmium–EDTA solution at pH 9 to 10.

The determination of hafnium in zirconium which is very difficult by ordinary wet chemical methods can be accomplished by spectrographic techniques. Naude and Zeeman (*200*) used the tape method whereby the sample is carried by a moving adhesive-coated tape into the spark gap so each spark contacts a new portion of sample for the spectrographic determination of from 2 to 99% hafnium in zirconium. Moroshkina and Smirnova (*191*) used a spectrographic method for the simultaneous determination of zirconium, hafnium, thorium, and titanium in natural samples with an accuracy of ±10%. Other applications of spectrochemical techniques include the determination of zirconium in vanadium by Muzgin, Zolotavin, and Gavrilov (*196*); in high-purity chromium by Heffelfinger *et al.* (*113*); of zirconium and hafnium in europium oxide by Rozsa and Stone (*240*); of zirconium and other cations in alkali metals by Vinogradov, Dronova, and Korovin (*274*); of hafnium in ores and minerals by Smirnova and Moroshkina (*258*); of zirconium in rocks and minerals by Joensuu and Suhr (*126*); of zirconium and hafnium in ores and rocks by Tsykhanskii and Krinberg (*271*); of zirconium in graphite by Goleb, Faris, and Meng (*99*), and of a number of elements including zirconium in industrial solutions and silicate rocks by Vorobev and Rusanov (*277*). D'Silva, Kniseley, and Fassel (*72*) introduced an improved burner for a premixed oxyacetylene flame which enabled them to detect, for the first time in a simple flame, analytically useful lines for several elements including zirconium. Additional information on this technique was reported by Fassel, Myers, and Kniseley (*88*).

Another technique which has been used for the determination of zirconium is based on x-ray fluorescence but frequently this method must include a prior separation. Karttunen (*134*) used x-ray fluorescence for the determination of zirconium in uranium-base alloys following a separation. Kriege and Rudolph (*148*) separated zirconium by precipitation with *p*-bromomandelic acid from iron, cobalt, and nickel alloys and finished the determination by x-ray fluorescence. The heterogeneity of distribution of various elements including zirconium in ores was determined by Hahn-Weinheimer and Ackermann (*105*) by means of x-ray fluorescence. Mamaev (*176*) determined zirconium, niobium, and yttrium by x-ray fluorescence and Bykov and Sorokin (*43*) determined zirconium, niobium, and germanium. Tomkins, Borun, and Fahlbusch (*266*) separated zirconium from tantalum, tungsten, and niobium by cupferron precipitations prior to determination of the zirconium by x-ray fluorescence.

A different type of analytical technique was used by Yatsimirskii and Raizman (*289, 290*) for the determination of hafnium and zirconium in which they utilized the catalytic effect which zirconium and hafnium have on the oxidation of iodide by hydrogen peroxide. They found that the rate of oxidation was dependent upon the concentration of iodide, hafnium, and zirconium but not peroxide and that the maximum catalytic effect of hafnium and zirconium occurs at different pH values.

Table II. Other Optical Methods

Element	Method-reagent: Material (References)
Zirconium	Flame photometric: Study of conditions (*72, 88*) Fluorometric–quercetin: Fe, V, Ti (*114*)
Vanadium	Flame photometric: Study of conditions (*72, 88*) Atomic absorption spectrophotometric: Study of conditions (*87*)
Niobium	Fluorometric–lumogallion: Ta (*140*) Fluorometric–NaF: Study of conditions (*272*) Flame photometric: Study of conditions (*72, 87, 88*) Infrared spectrophotometry–8-quinolinol: Steel (*168*)
Tantalum	Infrared spectrophotometry–8-quinolinol: Steel (*168*)
Chromium	Atomic absorption spectrophotometric: Study of conditions (*128*); steel (*137*)
Molybdenum	Flame photometric: Study of method (*88*)
Tungsten	Fluorometric–flavanol: Ni-W (*35*) Flame photometric: Study of method (*72, 88*)

Table III. Titrimetric Methods

Element	Reagent: Material (References)
Zirconium	EDTA: Alkali metals, rare earths, Cu, Hg, Th (*79*); Fe (*276*); U, Fe, Al, Zn, Sn, Mg (*270*); glass (*226*) Mandelic acid–vanadium(V): Study of conditions (*213*) KF-HNO_3-KOH: Hf-Zr mixtures (*182*) KF-HNO_3: Cr, Ni, Co, Zn, Mn (*257*) KH_2PO_4: Study of conditions (*151*)
Hafnium	EDTA: Zr (*118*) KF-HNO_3-KOH: Hf-Zr mixtures (*182*)
Vanadium	Ce(IV): Study of reactions (*70, 232*) Fe(II): Ce, Fe, Cr, W, Mo, U, Co, Ni (*233,-235*) $AgNO_3$: Study of reactions (*246*) EDTA: Study of separations (*91*) $NaVO_3$: Study of reactions (*195*)
Niobium	Nitrilotriacetic acid: Study of reactions (*156*) 8-Quinolinol–$KMnO_4$: Study of reactions (*177*) Cd reduction–$K_2Cr_2O_7$: Study of conditions (*147*)
Chromium	1,2-Diaminocyclohexanetetraacetic acid: Cr(III) in Cr(VI) (*227*) Fe(II): Study of reactions (*233*); fluoride salts (*14*) K_2CO_3, Na_2O_2, $NaHCO_3$: Molten KNO_3 (*251*)
Molybdenum	Fe(II): Study of reactions (*236*); V, W, Mn (*234*) $Pb(C_2H_3O_2)_4$: Study of reactions (*28*) $K_2Cr_2O_7$: Fe, W (*111*) Citric acid–diphenyl carbazone: Carbides, borides, silicides, nitrides (*150*)
Tungsten	Cerium(IV): Steel (*285*) $K_2Cr_2O_7$: Fe, Mo (*111*)

VANADIUM

During the last two years the majority of the papers dealing with methods for the determination of vanadium have been concerned with applications of activation analysis, spectrochemical, and spectrophotometric techniques.

Aoki and Okada (*12*) determined the most effective sample-source position relationship for the irradiation of vanadium-containing samples with a radium–beryllium neutron source in a paraffin moderator. These same authors (*13*) determined vanadium in petroleum without chemical separation by irradiating the sample in a reactor for 3 minutes at a flux of about 4×10^{10} *n*/sq. cm./second. The range of vanadium content to which the method could be applied was 10^{-1} to 10^3 p.p.m. Okada (*206*) has also applied the activation analysis method to the determination of vanadium in graphite. Kamemoto and Yamagishi (*133*) described a study of the activation analysis method for vanadium in conjunction with an extraction of vanadium 8-quinolinolate into benzene. Colombo *et al.* (*61*) determined vanadium in crude oils, distillation fractions, asphalts, and related substances without prior separation. Kaiser and Meinke (*129*) determined submicrogram amounts of vanadium in tissue by an activation analysis technique and reported a lower limit of 3×10^{-9} gram for vanadium. Soltys and Morrison (*259*) used an activation analysis method for the determination of the stoichiometry of sodium-vanadium bronze single crystals. Activation analysis methods have also been applied to the determination of vanadium in plants (*220*), stainless steel (*104*), zirconium (*89*), marine organisms (*93*), and milk powder (*189*). Chinaglia *et al.* (*55*) have studied vanadium along with other radionuclides and reported the sensitivity and characteristic γ-ray spectra.

Several spectrochemical methods have also been described in the literature during the past 2 years. Berthelot (*29*) studied the line intensity ratio as a function of d-c arc temperature for vanadium and other elements. Oda and Idohara (*202*) and Laib (*154*) used d-c arc methods for the determination of vanadium and other impurities in tantalum. Adell, Ruiz, and Gonzalez (*3*) used a quantometer for the determination of vanadium in nuclear grade graphite. The best results for vanadium were obtained when the sample was diluted with 25% cupric fluoride. Goleb, Faris, and Meng (*99*) determined 60 elements in graphite using either a d-c arc or copper spark method. Spectrographic methods were used for the determination of vanadium in high-purity chromium by Heffelfinger *et al.* (*113*); in aluminum oxide and tin oxide by Owens (*211*); in rocks, minerals, and related materials by Joensuu and Suhr (*126*); and in soils and plants by Chamberlain (*50*). Birks, Weldrick, and Thomas (*32*) employed the carrier distillation technique with an internal standard in the determination of a number of elements including vanadium in uranium dioxide. The copper spark method was used by Hainski and Rossi (*106*) for the determination of vanadium in polyphenyls.

Table IV. Gravimetric Methods

Element	Reagent: Material (References)
Zirconium and Hafnium	Cupferron: ZrB_2 (*263*); ZrO_2–Y_2O_3 (*172*); Mo, W (*254*); Zr-Hf mixtures (*164*) Mandelic acid and derivatives: Study of conditions (*229*); refractory materials (*230*) Thiosalicylic acid: U, V, Mo, W, rare earths (*68*) 2,3-Dihydroxynaphthalene-6-sulfonic acid: U, Ti (*69*) *p*-Iodoamygdalic acid: Zr alloys and ores (*253*) Chelidonic acid: Ti (*173*) Phenylacetylhydroxamic acid: Nb (*175*) *N*-benzoyl-*N*-phenylhydroxylamine: Fe, Al, Cr, Ti, Nb, Ta (*10*) Tartrazine: Zr concentrates (*224*) 1,3,5- and 1,2,4-Benzenetricarboxylic acid and 1,2,4,5-benzenetetracarboxylic acid: Study of reactions (*194*) Selenious acid: Zr-Hf mixtures (*2*)
Vanadium	None
Niobium	*N*-benzoyl-*N*-phenylhydroxylamine: Nb-Ta (*174*); Zr (*175*) Cupferron: Mo (*255*); minerals, ores, concentrates (*131*) Tannin: Nb-Ta (*100*) 8-Quinolinol: Study of conditions and/or interferences (*146*) 7,8-Dihydroxy-4-methylcoumarin: Nb, Ta, Ti (*217*)
Tantalum	*N*-Benzoyl-*N*-phenylhydroxylamine: Nb-Ta (*174*) Tannin: Nb-Ta (*100*) Cupferron: Minerals, ores, concentrates (*131*) Phenylarsonic acid: Study of conditions and/or interferences (*198*) 7,8-dihydroxy-4-methylcoumarin: Nb, Ta, Ti (*217*)
Chromium	None
Molybdenum	8-Quinolinol: Steel (*92*) Thioacetamide: Study of reactions (*40*)
Tungsten	Hydrolysis from acidic peroxide solution: Alkali tungsten bronze (*228*); steel (*64*) Hydrogen reduction: Alkali tungsten bronze (*228*) Tris-(tri-*n*-butylammonium)-12-tungstophosphate: Iron (*184*)

Table V. Electrometric Methods

Element	Method; Reagent: Material (References)
Zirconium	Amperometric; EDTA–V(V): Fluoride materials (*98*) Amperometric; *N*-benzoyl-*N*-phenylhydroxylamine: Ni, Mn, Co, Al, Cr, Fe (*94*) Amperometric; cupferron: Radioactive solutions (*149*) Amperometric; potassium ferrocyanide: Zr solutions (*95*) Amperometric; potassium tellurite: Study of conditions (*252*) Polarographic; *M*-nitrobenzoic acid: Quartz (*120*) Polarographic; Cd–EDTA: Study of conditions (*212*)
Vanadium	Amperometric; Fe(II)–$K_2Cr_2O_7$: Pure V (*155*) Controlled potential coulometric: Fused LiCl-KCl (*48*) Oscillopolarographic: Vegetable material (*199*) Voltammetric: Study of conditions (*185*)
Niobium	Polarographic: Ta (*138*); W (*152*)
Chromium	Controlled potential coulometric: Fused LiCl-KCl (*48*) Voltammetric: Study of conditions (*185*)
Molybdenum	Polarographic: Study of conditions (*292*); study of species (*142*); U (*15*); vegetable material (*199*) Square-wave polarographic: Zr (*287*)
Tungsten	Polarographic: Study of reactions (*152*) Square-wave polarographic: Zr (*287*)

A variety of methods for the spectrophotometric determination of vanadium was published. Budesinsky (*37*) studied the reaction between vanadium and xylenol orange, found the optimum conditions for the formation of the complex, and calculated the formation and stability constants. Sarma (*242*) used the color of the vanadium(V) ion in acid solution as the basis of a method for the determination of vanadium. The absorption spectra for vanadium(II), (III), and (IV) in hydrochloric, sulfuric, and perchloric acids were measured by Martin and Bentley (*180*). Headridge and Dixon (*110*) determined the distribution coefficients of several elements including vanadium between hydrochloric acid solutions and De-Acidite FF and Zeocarb 225 ion exchange resins and determined the vanadium spectrophotometrically with catechol at pH 8 to 9. Frederickson and Hausen (*90*) correlated the structure of several vanadium–oxygen compounds with the infrared absorption bands characteristic of these compounds. A spectrophotometric method for the determination of vanadium based on the oxidation of 3,3′-diaminobenzidine with vanadate was developed by Cheng (*53*). Liteanu, Lukacs, and Strusievici (*159*) determined vanadium spectrophotometrically by measuring the absorbance of the vanadium–tetraphenylphosphonium chloride complex in ethylene chloride. Minczewski and Skorko-Trybula (*187*) used thiophene-2-hydroxamic acid as a reagent for the spectrophotometric determination of vanadium. Ayres and Scroggie (*18*) determined vanadium by extracting the thiocyanate into a pyridine–chloroform mixture and measuring the resulting absorbance. Witwit, Magee, and Wilson (*286*) used phosphotungstovanadic acid as the colored species for the determination of vanadium following separation from chromium, tungsten, and molybdenum by paper partition chromatography. Biechler, Jordan, and Leslie (*31*) measured the absorbance of vanadium phosphotungstate after extraction into *n*-hexanol. Janauer and Korkisch (*125*) used Solochrome Fast Grey for the spectrophotometric determination of vanadium after an ion exchange separation using Dowex-1 and alcoholic hydrochloric acid solutions. Korkisch, Hazan, and Arrhenius (*145*) used Solochrome Fast Red as the chromogenic reagent after separation of vanadium using Dowex-1 and a nitric acid–alcohol mixture. 8-Quinolinol was used for the spectrophotometric determination of vanadium by Awasthi and Khasgiwale (*17*) who measured the absorbance in a chloroform solution at 550 mμ and by Magee and Witwit (*171*) who measured the absorbance in the infrared region at 10.50 microns. Pearse (*218*) used 2,6-pyridinedicarboxylic acid for the spectrophotometric determination of vanadium.

Rao and coworkers have published a number of papers describing titrimetric methods for the determination of vanadium, including the titration of vana-

Table VI. Spectrochemical Methods

Element	Material (References)
Zirconium	Cr (*113*); V (*196*); Eu_2O_3 (*240*); alkali metals (*274*); graphite (*99*); ores, rocks, minerals (*126, 271, 277*); natural materials (*191*)
Hafnium	Zr (*200*); Eu_2O_3 (*240*); ores, rocks, minerals (*258, 271*); natural materials (*191*)
Vanadium	Study of conditions and/or interferences (*29*); Cr (*113*); Ta (*154, 202*); Al_2O_3 or SnO_2 (*211*); UO_2 (*32*); graphite (*3, 99*); rocks and minerals (*126*); soils and plants (*50*); polyphenyls (*106*)
Niobium	Mo (*249*); Ta (*202*); Nb-Ta (*197*); alkali metals (*274*); rocks and ores (*231, 271*)
Tantalum	Mo (*249*); Pu (*141*); Nb-Ta (*197*); alkali metals (*274*); rocks and ores (*231, 271*)
Chromium	Study of conditions and/or interferences (*265*); Bi (*144*); Ta (*154, 202*); Nb (*153*); Np (*283*); V (*196*); Mo (*66*); Eu_2O_3 (*240*); UO_2 (*32*); Al_2O_3 and SnO_2 (*211*); graphite (*99*); ruby and sapphire (*56*); soils and plants (*50*); rocks and minerals (*126*); polyphenyls (*106*)
Molybdenum	Cr (*113*); Ta (*202*); alkali metals (*274*); UO_2 (*32*); Eu_2O_3 (*240*); graphite (*99*); rocks (*76*); plant tissue (*50, 62*); polyphenyls (*106*)
Tungsten	Study of conditions and/or interferences (*265*); Ta (*202*); alkali metals (*274*); UO_2 (*32*); graphite (*99*)

dium with sodium metavanadate (*195*), with cerium(IV) sulfate (*70, 232*), and with iron(II) in strong phosphoric dica (*233–235*). Saxena and Sharma (*246*) performed a potentiometric titration of vanadium with silver nitrate using a silver indicator electrode. Fritz and Abbink (*91*) separated vanadium from several other cations on a Dowex-50 column with a dilute hydrochloric acid solution containing a small amount of hydrogen peroxide and then titrated the vanadium with EDTA. A controlled-potential coulometric titration was utilized by Caton and Freund (*48*) for the determination of vanadium in the fused lithium chloride–potassium chloride eutectic.

Other methods reported to be useful for the determination of vanadium include a flame photometric method using a fuel-rich, oxyacetylene flame with which D'Silva, Kniseley, and Fassel (*72*) reported observing vanadium lines and Fassel, Myers, and Kniseley (*88*) detected vanadium in the 1- to 10-p.p.m. range. Fassel and Mossotti (*87*) used the fuel-rich, oxyacetylene flame in an atomic absorption method for the determination of vanadium. Ginsberg and Pfundt (*96*) used x-ray fluorescence for the determination of vanadium and other elements in aluminum at the 0.1- to 0.001-wt. % level. Townsend (*267*) determined vanadium in silica- and alumina-base materials by an x-ray fluorescence technique. Carter (*45*) worked out the optimum conditions for the x-ray fluorescence determination of several elements including vanadium.

An amperometric titration was used by Lannoye (*155*) for the determination of vanadium in pure vanadium metal. Nangniot (*199*) used an oscillopolarographic method for the determination of vanadium and other trace metals in vegetable material. Miller and Zittel (*185*) used a pyrolytic graphite electrode for the voltammetric determination of vanadium in sulfuric acid or sulfuric and phosphoric acids. A different approach to the determination of vanadium was used by Bontschev (*34*), who described a method based on the ability of vanadium to catalyze the oxidation of aryl amines.

NIOBIUM AND TANTALUM

Although not so similar as zirconium and hafnium, niobium and tantalum usually occur together in ores and minerals and the analytical methods for their determination will be discussed together in this review.

Porter (*225*) described and evaluated the methods for the separation and determination of niobium and tantalum and described methods for the determination of niobium in tantalum and tantalum in niobium. Cockbill (*59*) and Elwell and Wood (*83*) reviewed the analytical chemistry of niobium and tantalum. Alimarin and Bilimovech (*5*) also reviewed the literature on the analytical chemistry of niobium and tantalum and included 130 references. A review with 57 references was prepared by Yu (*291*).

Among the papers reporting work on the spectrophotometric determination of niobium are some using new or uncommon reagents and some in which well known reagents such as thiocyanate are used. Belcher, Ramakrishna, and West (*23, 24*) in two papers described the use of 4-(2-pyridylazo)-resorcinol as a reagent for the spectrophotometric determination of niobium. Of some 40 ions examined only vanadium(V), uranium(VI), and phosphate interfere seriously when the recommended procedure is followed. The slight tantalum interference is negligible if the tantalum-to-niobium ratio is not too high. The use of 1-(2-pyridylazo)-resorcinol as a chromogenic reagent for the spectrophotometric determination of niobium was reported by Alimarin and Han (*7*) and also by Elinson and Pobedina (*81*). These authors worked out the proper conditions for the determination of as little as 5 μg. of niobium in 50 ml. of solution in the presence of titanium or zirconium. A relatively new reagent which has been used for the determination of niobium is xylenol orange which was studied by Cheng and Goydish (*54*) who reported a molar absorptivity of 16,000 at 535 mμ. Methods for the determination of niobium with this reagent were described by Babko and Shtokalo (*20*) who stated that in the presence of tartaric acid the xylenol orange reacts with niobium in a ratio of 2:1 and by Elinson and Pobedina (*82*) who stated that in the presence of oxalate the xylenol orange reacts with niobium in the ratio of 3:2. Ishiwatari and Onishi (*122*) studied the xylenol orange–niobium complex by both the continuous variation method and the slope-ratio method and found evidence for a mixture of both the 2:1 and 1:1 complexes. McCown and Kudera (*162*) used the thiocyanate method for the determination of niobium in uranium-fission element alloys. Minczewski and Bozycki (*186*) studied the effect of various organic solvents for the extraction of the niobium–thiocyanate complex. Magee and Martin (*169*) studied the effect of various organic bases on the niobium–thiocyanate complex and proposed a method in which a niobium–strychnine–thiocyanate complex is used. They also developed a method based on the measurement of the absorbance of the reduced species of niobium formed by reaction with zinc dust in sulfuric acid. Martin and Magee (*181*) used the strychnine–thiocyanate method for niobium and the pyrogallol method for tantalum after separating niobium, tantalum, and titanium by partition chromatography using a solvent composed of hydrofluoric acid, nitric acid, and diethyl ketone. Mari (*179*) worked out extraction methods for separating niobium and tantalum prior to the determination of the niobium as the thiocyanate and the tantalum with pyrogallol. Webb, Ashworth, and Hills (*279*) determined both niobium and tantalum with pyrogallol following a cupferron separation. Headridge and Dixon (*110*) used anion exchange resins and hydrochloric acid solutions to separate the constituents of complex alloys before determining the niobium spectrophotometrically with thiocyanate and the tantalum with pyrogallol. Catoggio and Rogers (*46*) increased the absorbance of the niobium–pyrogallol complex by the addition of a long-chain quaternary ammonium compound, Ethoquad 18/25 (polyethoxy-15-stearyl-methyl-ammonium chloride) to the solution and eliminated the interference from molybdenum, tungsten, and chromium by extraction of the niobium complex into ethyl acetate. These same authors (*47*) eliminated the fluoride interference in the pyrogallol method for tantalum by the addition of boric acid and eliminated the vanadium interference by extraction of the tantalum–pyrogallol complex into ethyl acetate in the presence of Ethoquad 18/25. Hibbits *et al.* (*115*) determined tantalum with pyrogallol and niobium with hydroquinone in beryllium and beryllium oxide after first precipitating them with cupferron and then separating them on a strongly basic ion exchange resin. After a systematic study, Ackermann and Koch (*1*) recommended tribromopyrogallol as a selective and sensitive reagent for niobium. Pilipenko and Eremenko (*221, 222*) studied various reagents and recommended thiocyanate as a reagent for niobium and methyl violet for tantalum. Acid chrome violet K was used by Tramm and Pevzner (*268*) for the determination of niobium in ores. Nazarenko and Yagnyatinskaya (*201*) studied 16 derivatives of 2,3,7-trihydroxy-6-fluorone and found that some were suitable for the determination of niobium. Alimarin, Savvin, and Dedkov (*9*) studied various compounds containing the *o,o′*-dihydroxyazo group as possible reagents for niobium. The niobium complex with phosphomolybdic acid was used by Shkaravskii (*256*) for the determination of niobium. Guyon (*102*) determined tantalum spectrophotometrically by measuring the absorbance of 12-molybdotantalic acid at 820 mμ. Niobium was determined in pyrochlore soils by Bakes, Gregory, and Jeffery (*22*) by means of the niobium–peroxide complex in mixed sulfuric and phosphoric acids after separation of the niobium by precipitation with tannin. Babko and Shtokalo (*19*) studied the complexes formed by 16 indicators with tantalum and found that the most effective for the determination of tantalum were pyrocatechol violet and hematoxylin. Alimarin and Han (*6*) also investigated lumogallion as a reagent for niobium.

Very little has been published recently on titrimetric methods for the determination of niobium and tantalum. Lassner (*156*) used the complex formed between nitrilotriacetic acid and the niobium–peroxide complex for the titrimetric determination of niobium by adding an excess of the reagent and back-titrating the excess with copper using a fluorescent indicator, methylcalcein. Kotlyar and Nazarchuk (*147*) made a study of various reductants for niobium and developed a method for an oxidimetric determination of niobium using a cadmium metal reductor which reduces niobium to an average oxidation state of 3.2. The results compare favorably with those obtained by the gravimetric cupferron procedure. Mambetov and Rzaeva (*177*) precipitated niobium with 8-quinolinol then dissolved the precipitate and titrated the 8-quinolinol with permanganate.

Table VII. X-Ray Fluorescence Analysis

Element	Material (References)
Zirconium	Fe, Co, Ni alloys (*148*); U alloys (*134*); Nb, Ta, W (*266*); Y_2O_3 or Nb_2O_5 (*176*); ores (*105*); study of conditions and/or interferences (*43*)
Hafnium	None
Vanadium	Al (*96*); Fe alloys (*45*); SiO_2 or Al_2O_3 (*267*)
Niobium	Sn (*30*); ZrO_2 or Y_2O_3 (*176*); Fe alloys (*160*); ores, minerals, or slags (*43, 44, 239, 278*); high-temperature alloys (*266*)
Tantalum	Ores, minerals, or slags (*239, 278*); Fe alloys (*160*); high-temperature alloys (*266*)
Chromium	SiO_2 or Al_2O_3 materials (*267*); Fe, Cr, or Mn base materials (*188*); Fe alloys (*45*); mixed oxides of Cr and Ag (*30*)
Molybdenum	U (*134*); Fe alloys (*160*); SiO_2 or Al_2O_3 materials (*267*)
Tungsten	W-Re alloys (*30*); high-temperature alloys (*160, 266*)

Majumdar and Pal (*175*) separated zirconium from niobium by precipitating the zirconium with phenylacetylhydroxamic acid and then determined the niobium by precipitation with *N*-benzoyl-*N*-phenylhydroxylamine. These same authors (*174*) also separated and determined tantalum and niobium using the same reagents. Goroshchenko *et al.* (*100*) separated niobium and tantalum from each other and from other materials by extraction with cyclohexanone and then precipitated the elements individually with tannin. Sheskol'skaya (*255*) separated niobium from molybdenum and determined the niobium by precipitation with cupferron. Patrovsky (*217*) precipitated niobium and tantalum with 7,8-dihydroxy-4-methylcoumarin. Nakata, Kusaka, and Kikkawa (*198*) included tantalum in a study of the precipitation of 21 cations with phenylarsonic acid and its derivatives. Kosta and Dular (*146*) successfully precipitated niobium 8-quinolinolate of a definite composition by precipitation from homogeneous solution and determined the formula, the solubility in various organic solvents, and the ultraviolet and infrared absorption spectra. Kallmann, Oberthin, and Liu (*131*) determined niobium and tantalum by cupferron precipitations after first separating them from each other and from other interfering elements by ion exchange with hydrochloric acid and hydrofluoric acid solutions.

Bertin (*30*) used a technique for the x-ray fluorescence determination of niobium in a niobium–tin alloy in which the intensity ratio of the fluorescence from the two elements was compared with intensity ratio versus concentration ratio plots prepared from standards. Luke (*160*) determined niobium and tantalum in ferrous alloys and steels by the borax disk x-ray fluorescence method after chemical separations. Bykov and Sorokin (*43, 44*) described an x-ray fluorescence technique for the determination of niobium and applied it to the determination of niobium in minerals. Rothmann *et al.* (*239*) used an x-ray fluorescence method for the determination of niobium and tantalum in ores, slags, and minerals after separation. Tantalum and niobium were determined in minerals and oxides by de Wappner (*278*) using an x-ray fluorescence method. Mamaev (*176*) also used an x-ray fluorescence method for the determination of niobium. Tomkins, Borun, and Fahlbusch (*266*) separated niobium and tantalum along with zirconium and tungsten from the other constituents of high-temperature alloys by a cupferron precipitation and then determined them by x-ray fluorescence.

Spectrographic methods have been used for the determination of niobium and tantalum in a variety of materials. Nakano (*197*) studied the effects of various buffers in the spectrographic determination of niobium in tantalum and tantalum in niobium. Semenenko and Tarasevich (*249*) studied the effect of phosphomolybdate on the spectrographic determination of small amounts of niobium and tantalum. Niobium was determined along with a number of other elements in tantalum in impurity amounts by Oda and Idohara (*202*). Tantalum and niobium were determined in the alkali metals by Vinogradov, Dronova, and Korovin (*274*). Tsykhanskii and Krinberg (*271*) used a spectrographic technique for the determination of niobium and tantalum in rocks following a separation. A scintillation spectrographic method in which a ground sample of a mineral is fed into the arc and the flashes of the mineral lines are recorded by a scintillation counter was described by Raikhbaum, Malhkh, and Luzhnova (*231*). Ko (*141*) used a spectrographic method for the determination of tantalum in plutonium.

Neutron activation analysis is another technique which has been successfully

Table VIII. Neutron Activation Analysis

Element	Material (References)
Zirconium	Steel and electro-etch residues (*101*); Zr-Hf mixtures (*203*); meteorites (*78*)
Hafnium	Zr (*89, 132, 293*); Al (*97*); meteorites (*78*); study of conditions and/or interferences (*55*)
Vanadium	Study of conditions and/or interferences (*55, 133*); V (*12*); Zr (*89*); steel (*104*); Na-V bronze (*259*); petroleum (*13, 61*); graphite (*206*); rat liver (*129*); plant tissue (*220*); marine organisms (*93*); milk powder (*189*)
Niobium	Nb-Ta (*136*)
Tantalum	W (*93*); Be (*157*); Zr (*89*); meteorites (*77*); sea water (*107*)
Chromium	Study of conditions and/or interferences (*86, 261*); Al (*97*); Be (*157*); ruber maser and laser crystals (*280*); steel (*26, 101*); silicon, silicon carbide, molybdenum sulfide (*247*); petroleum (*61*); polyphenyls (*57*); terphenyls (*16*); nucleic acids (*262*)
Molybdenum	Ge (*260*); SiO_2 (*130*); steel (*26, 101*); polyphenyls (*57*); marine organisms (*93*)
Tungsten	SiO_2 (*130*); meteorites (*11, 77*); steel (*205*); bone (*27*); marine organisms (*93*)

applied to the determination of tantalum and, to some extent, of niobium. Corth (*63*) used activation analysis for the determination of tantalum in tungsten with no separation. Kim and Meinke (*136*) used an activation analysis method in conjunction with radiochemical separations for the simultaneous determination of niobium and tantalum. Tantalum and other impurities were determined in beryllium by Laverlochere and May (*157*) using an activation analysis method. Additional applications of activation analysis to the determination of tantalum along with other elements were made by Fournet (*89*) who determined tantalum in zirconium, Ehmann (*77*) who determined tantalum in meteorites, and Hamaguchi *et al.* (*107*) who determined tantalum in sea water.

A number of other techniques were also described in the literature as being useful for the determination of niobium or tantalum. A polarographic method in phosphoric acid solutions was used by Kurbatov and Skorynina (*152*) for the determination of niobium in the presence of tungsten. Kirby and Freiser (*138*) developed a polarographic method for niobium based upon the behavior of the niobium–EDTA complex. Electron paramagnetic resonance was used by Vinokurov *et al.* (*275*) for the determination of niobium in zircon. Fluorescence methods were used by Klimov and Didkovskaya (*140*) and Ushakova (*272*). The infrared spectra of niobium and tantalum 8-quinolinolates were used by Magee and Martin (*168*) for the determination of these elements. D'Silva, Kniseley, and Fassel (*72*) reported the existence of analytically useful lines for niobium using the fuel-rich, oxyacetylene flame. Fassel, Myers, and Kniseley (*88*) described the flame spectrum of niobium in a fuel-rich, oxyacetylene flame. Fassel and Mossotti (*87*) found an analytically useful atomic absorption spectrum for niobium in the highly fuel-rich, oxyacetylene flame.

Table IX. Radiochemical Analysis

Element	Material (References)
Zirconium	Zr-Nb (*210*); Zr-Hf (*250*); newborns, infants, and children (*163*); human lungs (*60*); rain water (*36*); vegetables (*216*); marine sediments (*209*); seaweed and sea water (*108*); soil and river bed (*241*); to evaluate critical nuclear incidents (*167*); to determine fallout level (*215*)
Hafnium	Zr-Hf (*250*)
Vanadium	None
Niobium	Zr-Nb (*210*); Zr-Hf (*250*); newborns, infants, and children (*163*); human lungs (*60*); rain water (*36*); vegetables (*216*); marine sediments (*209*); seaweed and sea water (*108*); soil and river bed (*241*); to evaluate critical nuclear incidents (*167*); to determine fallout level (*215*)
Tantalum	None
Chromium	Marine sediments (*209*); fission products and corrosion products (*166*); biological materials (*58, 282*)
Molybdenum	Biological materials (*282*); rain water (*36*); fission product mixtures (*284*); bone (*112*)
Tungsten	Biological materials (*282*)

Radiochemical methods can also be used for niobium; however, the niobium procedures are identical to those of zirconium, and because they are mentioned in the discussion of zirconium they are omitted from this part.

CHROMIUM

Neutron activation analysis has been applied to the determination of chromium in a number of different materials. Steele and Meinke (*261*) examined specific activities of 7 elements including chromium in an investigation of fast neutron activation analysis to determine the applicability to routine analytical operation and reported the conditions used for the determination of these elements. Gruverman and Henninger (*101*) utilized activation analysis to determine chromium in the analysis of a high-alloy steel and residues from electro-etching procedures. Benson and Gleit (*26*) determined the chromium content of stainless steel by means of activation analysis. Chromium was determined in beryllium by Laverlochere and May (*157*) by an activation analysis method following an ion exchange separation of the other impurities present. Neutron activation analysis was also applied to the determination of 13 trace impurities including chromium in aluminum by Girardi and Pietra (*97*). Activation analysis methods have been applied to the determination of chromium in ruby maser and laser crystals by Weiner, O'Connor, and Rubin (*280*); in petroleum by Colombo *et al.* (*61*); in polyphenyls by Ciuffolotti, Fasolo, and Malvano (*57*); in terphenyls by Aubouin (*16*); in nucleic acids by Stehlik and Altmann (*262*). Fasolo, Malvano, and Massaglia (*86*) first separated chromium by extracting it into a chloroform solution of tribenzylamine and then determined it by an activation analysis method. Schmied and Bergamin (*247*) gave a series of examples showing how impurity amounts of a number of elements including chromium could be determined in many different materials including silicon, silicon carbide, and molybdenum sulfide.

A number of spectrochemical methods for the determination of chromium were also reported in the literature. Tappe and van Calker (*265*) used various high-frequency plasma flames for the determination of chromium. Konovalov *et al.* (*144*) reported the use of either the d-c arc or spark excitation for the determination of 18 impurities including chromium in bismuth by a single spectrogram. Twenty-nine impurities including chromium were determined in uranium dioxide by the carrier distillation method with an internal standard by Birks, Weldrick, and Thomas (*32*). Oda and Idohara (*202*) reported a method for the spectrographic determination of 14 elements including chromium in high-purity tantalum. Fifteen elements including chromium were determined by Kuznetsova and Krauz (153) in niobium following the removal of the niobium by solvent extraction. In this way the interference caused by the complex niobium spectrum was removed and the sensitivity of all the elements was increased. Chromium and other impurities were separated from neptunium by ion exchange preceding spectrographic determination in a method reported by Wheat (283). Rozsa and Stone (240) applied a quantitative spectrographic procedure to the determination of chromium in europium oxide. Chromium in highly pure vanadium was determined by a spectrographic technique after removal of the vanadium by distillation as vanadium (IV) chloride and vanadium(V) oxychloride in a method described by Muzgin, Zolotavin, and Gavrilov (196). Concentration of the impurities by the evaporation of them onto a graphite electrode was also used prior to the spectrographic determination of chromium in molybdenum by Degtyreva and Ostrovskaya (66). The spark method using silver electrodes and palladium as the internal standard was used by Hainski and Rossi (106) in the determination of chromium and other metallic impurities in polyphenyls. Spectrographic methods were used for the determination of chromium in rocks, minerals, and related materials by Joensuu and Suhr (126); in fused aluminum oxide and tin oxide crystals by Owens (211); in tantalum and tantalum oxide by Laib (154); in graphite by Goleb, Faris, and Meng (99); in sapphire and ruby maser crystals by Chirnside *et al.* (56) and in soils and plants by Chamberlain (50).

Table X. Miscellaneous Methods

Element	Method: Material (References)
Zirconium	Catalytic effect on the oxidation of iodide by hydrogen peroxide: Hf (289, 290)
Hafnium	Catalytic effect on the oxidation of iodide by hydrogen peroxide: Zr (289, 290)
Vanadium	Catalytic effect on the oxidation of *p*-phenetidine by potassium chlorate: Study of interferences (34)
Niobium	Paramagnetic resonance spectra: Zircon (275)
Tantalum	None
Chromium	Gas chromatographic hexafluoroacetylacetonate: Study of conditions (238)
Molybdenum	Catalytic effect on the oxidation of iodide by hydrogen peroxide: LiF, CdS (39)
Tungsten	Catalytic effect on the oxidation of iodide by hydrogen peroxide: LiF, CdS (39)

Although it appears that spectrophotometric methods for the determination of chromium are somewhat less popular than they were in the past, some additional papers report the application of the *s*-diphenylcarbazide method for the determination of chromium in beryllium (223), uranium and uranium oxides (119, 237), diphenyl (190), and sapphire and ruby maser crystals (56). *s*-Diphenylcarbazide was also used by Witwit, Magee, and Wilson (286) for the determination of chromium following separation from tungsten, molybdenum, and vanadium by partition chromatography, and by Headridge and Dixon (110) following ion exchange separations from alloys containing niobium, tantalum, and tungsten. Zittel (294) found that exposure to γ-ray radiation introduces very little error into the *s*-diphenylcarbazide method for chromium. Marchart (178) found that chromium (III) reacts with diphenylcarbazone to form the same complex as is formed by the reaction of chromium(VI) with *s*-diphenylcarbazide. den Boef and Poeder (33) reported wavelengths of maximum absorbance and molar absorptivities for the chromium complexes with EDTA, NTA, HEDTA, DCTA, and DTPA. Bennett and Marshall (25) used the chromium–EDTA complex for the spectrophotometric determination of chromium following separation from ores by means of liquid ion exchange resins. Selmer-Olsen (248) used 1,2-diaminocyclohexanetetraacetic acid as a reagent for the spectrophotometric determination of chromium. The absorbance of the chromium–tribenzylamine complex in chloroform was used by Fasolo, Malvano, and Massaglia (86) for the spectrophotometric determination of chromium. Kono and Ozawa (143) used thioglycolic acid as the chromogenic agent for the spectrophotometric determination of chromium. This reagent reacts with chromium(VI) at pH 6 to 9, and the maximum absorbance occurs at 475 mμ.

Several other types of methods have been described in the literature as useful for the determination of chromium. One of these is an atomic absorption method described by Kahn and Slavin (128) in which they determined 0.1 μg. of chromium per ml. of solution with a coefficient of variation of less than 10%. Kinson, Hodges, and Belcher (137) used an atomic absorption spectrophotometric method for the determination of chromium in low-alloy irons and steels. Carter (45) reported on the best conditions for the determination of a number of elements including chromium by the x-ray fluorescence technique. Townsend (267) used x-ray fluorescence for the determination of chromium in silica- and alumina-base catalyst materials. Mitchell and O'Hear (188) determined chromium in iron-, chromium- and/or manganese-bearing material by an x-ray fluorescence method. Bertin (30) used an x-ray fluorescence spectrometric method in which he measured the intensity ratio *vs.* concentration ratio for the determination of chromium and silver in mixed oxides of the two elements. A titration with 1,2-diaminocyclohexanetetraacetic acid was used by Pribil and Vesely (227) for the determination of chromium(III) in the presence of chromium(VI). Rao and Dikshitulu (233) titrated chromium(VI) with iron(II) in $10.5M$ phosphoric acid. An amperometric titration with iron(II) sulfate was used by Apple and Zittel (14) for the determination of chromium in mixtures containing lithium fluoride, beryllium fluoride, zirconium fluoride, and uranium fluoride. In the range of 1 to 50 μg. of chromium the relative standard deviation was less than 2%. The controlled-potential coulometric method for the determination of chromium in fused lithium chloride–potassium chloride eutectic was studied by Caton and Freund (48). The chromium was determined by the reduction of chromium(III) to chromium(II) with a relative error of 0.38%. Shams El Din and Gerges (251) titrated potassium dichromate as an acid with potassium carbonate, sodium peroxide, and sodium bicarbonate in molten potassium nitrate. Miller and Zittel (185) used the pyrolytic graphite electrode for the polarographic determination of chromium in either sulfuric acid or mixed sulfuric–phosphoric acid solution. A quantitative gas chromatographic method for the determination of chromium using chromium(III) hexafluoroacetylacetonate was reported by Ross and Wheeler (238).

Chromium has also been determined simply by measuring the γ-ray emission of chromium-51. This method has been used for the determination of chromium in marine sediments (209), in fission and corrosion products (166), and in biological materials (58, 282).

MOLYBDENUM

The determination of molybdenum has not received as much attention as has zirconium and niobium; however, the recent literature does include a wide variety of methods for the determination of molybdenum in a number of different materials.

A number of analytical techniques for the determination of alloying amounts of molybdenum and other elements in niobium were compared by McKaveney (*165*) by means of round-robin analysis to ascertain which ones need further development. The results showed good agreement between the spectrographic spark solution technique and thiocyanate spectrophotometric technique in his laboratory and fair agreement between laboratories.

Motojima *et al.* (*192*) separated molybdenum from uranium by extracting the molybdenum 8-quinolinolate into chloroform prior to the spectrophotometric measurement. Hibbits and Williams (*116*) extracted the molybdenum thiocyanate complex into methyl isobutyl ketone and measured the absorbance of the organic phase at 500 mμ. They found interference only from rhenium, platinum, palladium, rhodium, selenium, and tellurium. Peng and Sandell (*219*) separated molybdenum together with tungsten from other elements by extracting them into a solution of α-benzoinoxime in chloroform and then, after destroying the α-benzoinoxime with acid, determined them by measuring the absorbance of their thiocyanate complexes in isopropyl ether at 2 wavelengths. Weiss and Lai (*281*) found that they could concentrate molybdenum from sea water by cocrystallization with α-benzoinoxime. They then determined the molybdenum spectrophotometrically as the thiocyanate. Fritz and Hedrick (*92*) determined molybdenum as the thiocyanate after separating it from the other constituents of stainless steel by retaining it on a Kel-F column impregnated with methyl isobutyl ketone and then eluting it with hydrochloric acid. Hostein and Sifferlen (*119*) included a discussion of the molybdenum–thiocyanate method in a report on the sensitivity and precision of methods for analyzing technically-pure uranium. Witwit, Magee, and Wilson (*286*) determined molybdenum using thiocyanate after separating the molybdenum, chromium, tungsten, and vanadium by partition paper chromatography. Tananaiko and Blucke (*264*) found that the reduced molybdenum–diantipyrylmethane–thiocyanate complex was stable when extracted into chloroform or dichloroethane, had a sensitivity of 10 μg. in 10 ml. of the extract, and could be used for the determination of molybdenum in steel without interference from nickel and chromium. Budesinsky (*38*) determined 0.1 to 1.2 mg. of molybdenum in uranium without prior separation by measuring the absorbance at 490 mμ of the molybdenum–xylenol orange complex at pH 3.6 in a solution containing sodium acetate. Kono and Ozawa (*143*) reported a sensitivity of 0.017 μg. per sq. cm. at 368 mμ for the yellow molybdenum–thioglycolic acid complex in aqueous solution at pH 3 to 5. Molybdenum was determined in nuclear reactor materials by Eberle and Lerner (*75*) by extracting the molybdenum–8-quinolinol complex into chloroform from a sulfate solution in the absence of halide and measuring the absorbance directly in the organic phase. Magee and Witwit (*171*) determined molybdenum by measuring the absorbance of the 8-quinolinol complex in the infrared region at 10.80 microns. It was also found by Magee and Witwit (*170*) that 8-mercaptoquinoline could be used as a reagent for the determination of molybdenum. The complex was extracted into chloroform and the absorbance was measured in the organic phase at 425 mμ. Busev and Fan (*41*) used 8-quinolinol-5-sulfonic acid for the spectrophotometric determination of molybdenum. They found 540 mμ to be the wavelength of maximum absorbance and the molar absorptivity to be 5180. Havermans, Verbeek, and Hoste (*109*) determined 2 to 9 μg. of molybdenum(V) per ml. with 1,10-phenanthroline being used as the reagent. The molar absorptivity is 6290 at 508 mμ which is the wavelength of maximum absorbance. Kirkbright and Yoe (*139*) investigated 2-amino-4-chloro-benzenethiol hydrochloride as a reagent for the spectrophotometric determination of molybdenum. The precipitate formed by the reagent with molybdenum is extracted into chloroform where the maximum absorbance occurs at 720 mμ with a sensitivity of 0.0054 μg. of molybdenum per sq. cm. Lee, Shastri, and Amis (*158*) studied the molybdenum–chloranilic acid complex in acid solution and found that the pH had to be controlled to ±0.05 and that the dissociation constants change linearly with pH. Haas and Schwarz (*103*) used the ammonium salt of diethanoldithiocarbamic acid for the spectrophotometric determination of molybdenum. In a study of the reaction of 1-mercaptopropionic acid and some of its derivatives with molybdenum Busev, Nacu, and Rudzit (*42*) developed an extraction–spectrophotometric method for the determination of molybdenum. Korkisch, Hazan, and Arrhenius (*145*) developed a method for the determination of molybdenum with Solochrome Fast Red following an ion exchange separation from the interfering elements. Hobart and Hurley (*117*) extracted the molybdenum complex with toluene-3,4-dithiol into carbon tetrachloride and measured the absorbance at 680 mμ. An ion exchange separation employing De-Acidite FF or Zeocarb 225 and up to 10*M* hydrochloric acid was used by Headridge and Dixon (*110*) to separate the constituents of complex alloys before determining the molybdenum with toluene-3,4-dithiol. Mikhail and Paddelford (*183*) studied the effect of temperature on the absorbance of molybdenum (V) in aqueous solution.

Molybdenum has been included in work on the development of procedures for the spectrographic determination of impurities in metals and other materials. Oda and Idohara (*202*) worked out the conditions for the spectrographic determination of molybdenum along with other cations in tantalum and tantalum compounds. Vinogradov, Dronova, and Korovin (*274*) used a spectrographic method for the determination of molybdenum in alkali metals after separation by solvent extraction. Twenty elements including molybdenum were determined in europium oxide in the range of 1 to 10,000 p.p.m. by Rozsa and Stone (*240*). Birks, Weldrick, and Thomas (*32*) used the carrier-distillation method for the determination of molybdenum and other impurities in uranium dioxide. Heffelfinger *et al.* (*113*) determined molybdenum and other impurities in high-purity chromium by first volatilizing the chromium as chromyl chloride and then examining the remaining solution spectrographically. Goleb, Faris, and Meng (*99*) determined a number of elements including molybdenum in graphite by the copper spark method after first ashing the sample. Molybdenum was spectrographically determined by Edge, Dunn, and Ahrens (*76*) in granitic and related rocks after first concentrating it along with iron by extracting them into tri-*n*-butylphosphate. Connor and Bass (*62*) developed a method for the spectrographic determination of molybdenum in plant tissue in which they reduced the matrix problems of biological systems by mixing the ashed sample with a buffer powder consisting of potassium sulfate and graphite. Chamberlain (*50*) also used a spectrographic method for the determination of molybdenum and other trace elements in soils and plants. The copper spark method with palladium as an internal standard was used by Hainski and Rossi (*106*) for the determination of molybdenum in polyphenyls after destruction of the organic material by dry ashing. Fassel, Myers, and Kniseley (*88*) described the use of a fuel-rich, oxyacetylene flame for the flame photometric determination of molybdenum.

X-ray fluorescence methods have also been found useful for the determination of molybdenum. Luke (*160*) employed a borax disk x-ray fluorescence method for the determination of molybdenum in ferrous alloys and high-alloy steels. Karttunen (*134*) used an ion exchange procedure for the separation of molybdenum from uranium-base fission product alloys prior to determination of the molybdenum by an x-ray fluorescence method. The measurement of the x-ray fluorescence was used by Townsend (*267*) for the determination of molybdenum in silica- and alumina-base catalysts.

Titrimetric methods are also to be found among the proposed procedures for the determination of molybdenum. Rao and Sagi (*236*) used iron(II) in $12M$ phosphoric acid for the titration of molybdenum(VI) to molybdenum(V). The end point was detected potentiometrically using a bright platinum rod and saturated calomel electrode. Rao and Dikshitulu (*234*) used this method for the simultaneous determination of vanadium and molybdenum without interference from tungsten and manganese. Berka *et al.* (*28*) titrated molybdenum (III) to molybdenum(VI) in 5 to $7M$ hydrochloric acid or to molybdenum(V) in 8 to $10M$ hydrochloric acid using lead tetraacetate as the oxidizing agent with a potentiometric end point. Headridge; and Taylor (*111*) titrated molybdenum, tungsten, and iron in mixtures of the 3 elements by using both a Jones and a silver reductor, various concentrations of hydrochloric and hydrofluoric acids, and various temperatures. Molybdenum is quantitatively reduced to molybdenum(III) by a Jones reductor in $2M$ hydrochloric acid–$0.5M$ hydrofluoric acid solution. Kugai and Nazarchuk (*150*) proposed a titrimetric determination of molybdenum using citric acid as the titrant in the presence of diphenylcarbazone.

Athavale, Kalyanaraman, and Khasgiwale (*15*) described a polarographic method for the determination of 0.01 to 1.0% molybdenum in uranium with a relative accuracy of 3%. Zahnow and Robinson (*292*) determined molybdenum polarographically without a calibration curve by complexing the reduced species and thereby preventing the catalytic current due to the reduction of perchlorate by molybdenum(IV) or nitrate by molybdenum(III). Kolthoff and Hodara (*142*) described the different polarograms obtained from solutions of varying molybdenum and sulfuric acid concentration which are attributed to different species of molybdenum. Wood and Clark (*287*) used a square-wave polarographic technique for the determination of molybdenum and other impurities or alloying constituents in zirconium. Nangniot (*199*) determined molybdenum and other trace metals in vegetable material by oscillopolarography. Bulgakova and Zalubovskaya (*39*) followed amperometrically the rate of iodide oxidation with hydrogen peroxide as a method for determining molybdenum in cadmium sulfide and lithium fluoride crystals.

Neutron activation analysis methods have also been worked out for the determination of molybdenum in a variety of materials. Gruverman and Henninger (*101*) used a neutron activation analysis method for the determination of 16 elements including molybdenum in stainless steel and electro-etch residues. Stary, Ruzicka, and Zeman (*260*) used an activation analysis method after separation of the molybdenum by precipitation with substoichiometric amounts of 8-quinolinol. The molybdenum content of stainless steel microspheres was determined by Benson and Gleit (*26*) using an activation analysis method. Molybdenum and several other cations were determined in silicon dioxide by Kalinin, Kuznetsov, and Moiseev (*130*) using neutron activation analysis along with ion exchange separations. Activation analysis methods were also used by Ciuffolotti, Fasolo, and Malvano (*57*) for the determination of molybdenum in polyphenyls; by Stehlik and Altmann (*262*) for the determination of molybdenum in nucleic acids; by Colombo *et al.* (*61*) for the determination of molybdenum in petroleum; and by Fukai and Meinke (*93*) for the determination of molybdenum in marine organisms.

Wester, Brune, and Samsahl (*282*) coupled an ion exchange separation with γ-ray spectrometry for the determination of 23 elements including molybdenum in biological material. Buchtela and Lesigang (*36*) used radiochemical techniques for the determination of molybdenum and other fission products in rain water. Wish (*284*) separated molybdenum-99 from fission product mixtures by extraction into a solution of α-benzoinoxime in chloroform from $1.0M$ hydrochloric acid and used a radiochemical determination. Healy and McCabe (*112*) extracted molybdenum-99 into a solution of cupferron in chloroform in the radiochemical determination of molybdenum in bone samples.

Molybdenum was determined gravimetrically by precipitation with 8-quinolinol by Fritz and Hedrick (*92*) following separation from the constituents of steels by means of a reversed-phase partition chromatographic technique using methyl isobutyl ketone on Kel-F as the stationary phase. Burriel-Marti and Vidan (*40*) precipitated molybdenum quantitatively as the sulfide from homogeneous solution with thioacetamide without the use of pressure flasks.

TUNGSTEN

The papers describing methods for the determination of tungsten are much fewer in number than those of the other elements included in this review and many of them merely include tungsten in a study of many elements. McKaveney (*165*) considered tungsten along with other elements in his discussion of chemical and spectrographic methods for the determination of alloying amounts of these elements in niobium.

Table XI. Compilation of Methods

Element	Review
Zirconium	Review (*80, 84, 123, 124*); evaluation of methods (*165*)
Vanadium	None
Niobium and tantalum	Review and evaluation of methods (*225*); review (*5, 59, 83, 291*)
Molybdenum	Evaluation of methods (*165*)
Tungsten	Evaluation of methods (*165*)

A spectrophotometric determination of tungsten in nuclear materials and steels was described by Eberle (*74*) in which he measured the absorbance of the tungsten–8-quinolinol complex in chloroform following extraction. Luke (*161*) determined tungsten in iron and steel spectrophotometrically by extracting the thiocyanate into methyl isobutyl ketone and measuring the absorbance at 410 mμ after first separating the iron by extraction into methyl isobutyl ketone and other interfering elements by extraction into a solution of cupferron in chloroform. Peng and Sandell (*219*) separated tungsten from iron by extracting it into a solution of α-benzoinoxime in chloroform and, after destroying the α-benzoinoxime with acid, determined the tungsten by measuring the absorbance of the tungsten–thiocyanate complex in isopropyl ether. A tri-*n*-butyl phosphate extraction with a water strip was used by De and Rahaman (*65*) to separate tungsten from the other constituents of steel prior to the determination of tungsten as the thiocyanate. Fritz and Hedrick (*92*) found that tungsten could be separated along with molybdenum from the other constituents of steel and also separated from the molybdenum by means of reversed-phase partition chromatography using methyl isobutyl ketone on Kel-F as the stationary phase. After separation the tungsten was determined as the thiocyanate. Witwit, Magee, and Wilson (*286*) separated tungsten from chromium, vanadium, and molybdenum by paper partition chromatography and then determined the tungsten as the thiocyanate. Magee and Witwit (*171*) determined tungsten spectrophotometrically by measuring the absorbance of the 8-quinolinol complex in the infrared region at either 10.61 or 10.90 microns. Headridge and Dixon (*110*) separated the constituents of complex alloys by ion exchange and then determined the tungsten by measuring the absorbance of the toluene-3,4-dithiol complex in carbon tetrachloride after extraction. Hobart and Hurley (*117*) determined tungsten in niobium by extracting the tungsten–toluene–3,4-dithiol complex into carbon tetrachloride and measuring the absorbance at 640 mμ. Affsprung and Murphy (*4*) determined tungsten in steels and heat-resisting alloys by forming a tungsten–tetraphenylarsonium-thiocyanate which was extracted into chloroform and the absorbance measured at 406 mμ. The method is said to be selective for tungsten when niobium is masked with fluoride. Kono and Ozawa (*143*) determined tungsten with thioglycolic acid which forms a water-soluble complex with tungsten at pH 3 to 5. The complex has an absorption maximum at 306 mμ and a sensitivity of 0.034 μg. of tungsten per sq. cm.

Bottei and Trusk (*35*) used flavanol to produce a fluorescence with tungsten in the pH range 2.5 to 5.5 which can be used to determine tungsten between the limits 6 and 42 μg. per 100 ml. of solution. Some nickel, cobalt, manganese, and copper can be tolerated.

Tappe and van Calker (*265*) studied the use of high-frequency plasma flames in quantitative spectrochemical determinations of tungsten and other materials. A total of 45 impurity elements including tungsten were determined in uranium dioxide by Birks, Weldrick, and Thomas (*32*) using an emission spectrographic method. Vinogradov, Dronova, and Korovin (*274*) used solvent extraction separations followed by spectrographic determinations for the determination of tungsten in the alkali metals. Oda and Idohara (*202*) carried out experiments to determine optimum conditions for the spectrographic determination of impurities including tungsten in tantalum metal and tantalum compounds. Goleb, Faris, and Meng (*99*) used the copper spark method for the spectrographic determination of tungsten in graphite after ashing the sample. D'Silva, Kniseley, and Fassel (*72*) described an improved burner using premixed oxyacetylene. Under fuel-rich conditions they were able to observe flame emission spectra from tungsten. Fassel, Myers, and Kniseley (*88*), using this premixed, fuel-rich, oxyacetylene flame, were able to detect 90 p.p.m. tungsten.

Bertin (*30*) proposed a method of preparing calibration curves for the x-ray fluorescence determination of both the constituents of binary alloys in which he prepared a log–log plot of intensity ratio versus concentration ratio instead of the usual intensity *vs.* concentration. This method was applied to the analysis of tungsten-rhenium alloys. A borax disk technique was used by Luke (*160*) for the x-ray fluorescence determination of tungsten in heat-resisting and corrosion-resisting alloys and high-alloy steel. Tomkins, Borun, and Fahlbusch (*266*) determined tungsten by x-ray fluorescence after the tungsten along with zirconium, niobium, and tantalum was separated from the other constituents of high-temperature alloys by cupferron precipitation.

The polarographic behavior of tungsten in pyrophosphoric acid solutions was studied by Kurbatov and Skorynina (*152*) who found that a clearly defined wave is obtained in 18*M* pyrophosphoric acid. Wood and Clark (*287*) used a square-wave polarograph for the direct polarographic determination of tungsten in zirconium. Bulgakova and Zalubovskaya (*39*) used an amperometric technique to follow the rate of oxidation of the iodide ion with hydrogen peroxide which is proportional to the amount of tungsten present and therefore can be used as a method of determination of tungsten.

Gravimetric methods were used by Raby and Banks (*228*) for the determination of tungsten in alkali metal tungsten bronzes. In bronzes, except for lithium bronzes, the tungsten was reduced to the metal with hydrogen after the sample was decomposed with bromine trifluoride. In the case of lithium compounds the tungsten was precipitated from a nitric acid–hydrogen peroxide solution after decomposing the sample by a basic oxidizing fusion. Dams and Hoste (*64*) also precipitated tungsten from homogeneous solution by the thermal decomposition of peroxytungstate from nitric acid–hydrogen peroxide solution. Miller and Thow (*184*) separated tungsten from iron by adsorption of tungstophosphoric acid on a cellulose column from 6*M* hydrochloric acid and then determined the tungsten by precipitation of tris-(tri-*n*-butylammonium)-12-tungstophosphate.

Tungsten was determined titrimetrically by Witwit and Magee (*285*) who reduced tungsten(VI) to tungsten(V) with bismuth amalgam in concentrated hydrochloric acid and titrated it with cerium(IV) sulfate using diphenylamine as the indicator. Headridge and Taylor (*111*), by using Jones and silver reductors and various concentrations of mixed hydrochloric acid and hydrofluoric acid along with various temperatures, were able to determine tungsten, molybdenum, and iron in mixtures of the three elements. Tungsten was quantitatively reduced to tungsten(III) with a Jones reductor in 2*M* hydrochloric acid–0.5*M* hydrofluoric acid solution.

Several papers which report the use of activation analysis techniques for the determination of tungsten were published during this period. In general, these papers describe the determination of tungsten or a group of elements in some specific matrix. Bergman and Soeremark (*27*) used an activation analysis method for the determination of a number of elements including tungsten in bone. Okada (*205*) proposed a neutron activation analysis method for the rapid, nondestructive determination of tungsten in steel. Tungsten abundances in meteoritic and terrestrial materials were determined by Amiruddin and Ehmann (*11*) using an activation analysis method. Ehmann (*77*) also reported on the determination of tungsten and other elements in meteorites by activation analysis. Neutron activation analysis was also used by Kalinin, Kuznetsov, and Moiseev (*130*) for the determination of tungsten in silicon dioxide and by Fukai and Meinke (*93*) for the determination of tungsten in marine organisms.

Wester, Brune, and Samsahl (*282*) used γ-ray spectrometry and ion exchange separations for the determination of some 23 elements including tungsten in biological materials.

LITERATURE CITED

(1) Ackermann, G., Koch, S., *Talanta* **9**, 1015 (1962).
(2) Adams, R. W., Holness, H., *Analyst* **89**, 31 (1964).
(3) Adell, M. R., Ruiz, E. B., Gonzalez, F. A., *Anales Real Soc. Espan. Fis. Quim.* (*Madrid*), *Ser. B.* **59**, 335 (1963).
(4) Affsprung, H. E., Murphy, J. W., *Anal. Chim. Acta* **30**, 501 (1964).
(5) Alimarin, I. P., Bilimovech, G. N., "Metody Opredeleniya i Analiza Redkikh Elementov," p. 487, Publishing House of the Academy of Sciences, Moscow, 1961.
(6) Alimarin, I. P., Han, H., *Zh. Analit. Khim.* **18**, 82 (1963).
(7) *Ibid.*, p. 182.
(8) Alimarin, I. P., Puzdrenkova, I. V., Dolnikova, S. Ya., *Ibid.*, **17**, 700 (1962).
(9) Alimarin, I. P., Savvin, S. B., Dedkov, Yu. M., *Ibid.*, **19**, 328 (1964).
(10) Alimarin, I. P., Yung-Schaing, T., *Talanta* **9**, 9 (1962).
(11) Amiruddin, A., Ehmann, W. D., *Geochim. Cosmochim. Acta* **26**, 1011 (1962).
(12) Aoki, F., Okada, M., *Tokyo Kogyo Shikensho Hokoku* **54**, 121 (1959).
(13) *Ibid.*, **58**, 49 (1963).
(14) Apple, R. F., Zittel, H. E., Anal. Chem. **36**, 983 (1964).
(15) Athavale, V. T., Kalyanaraman, R., Khasgiwale, K. A., *Anal. Chim. Acta* **29**, 280 (1963).
(16) Aubouin, G., *Radiochim. Acta* **1**, 117 (1963).
(17) Awasthi, S. P., Khasgiwale, K. A., *Indian J. Chem.* **2**, 102 (1964).
(18) Ayres, G. H., Scroggie, L. E., *Anal. Chim. Acta* **26**, 470 (1962).
(19) Babko, A. K., Shtokalo, M. I., *Ukr. Khim. Zh.* **29**, 963 (1963).
(20) Babko, A. K., Shtokalo, M. I., *Zh. Analit. Khim.* **17**, 1068 (1962).
(21) Bachman, R. Z., Banks, C. V., "Progress in Nuclear Energy," Series IX, Vol. **3**, part 4, p. 95, Pergamon Press, Oxford, England, 1963.
(22) Bakes, J. M., Gregory, G. R. E. C., Jeffery, P. G., *Anal. Chim. Acta* **27**, 540 (1962).
(23) Belcher, R., Ramakrishna, T. V., West, T. S., *Talanta* **9**, 943 (1962).
(24) *Ibid.*, **10**, 1013 (1963).
(25) Bennett, H., Marshall, K., *Analyst* **88**, 877 (1963).
(26) Benson, P. A., Gleit, C. E., Anal. Chem. **35**, 1029 (1963).
(27) Bergman, B., Soeremark, R., *Nucl. Med.* **3**, 175 (1963).

(28) Berka, A., Dolezal, J., Nemec, I., Zyka, J., *J. Electroanal. Chem.* **3,** 278 (1962).

(29) Berthelot, C., *Spectrochim. Acta* **19,** 1323 (1963).

(30) Bertin, E. P., ANAL. CHEM. **36,** 826 (1964).

(31) Biechler, D. G., Jordan, D. E., Leslie, W. D., *Ibid.*, **35,** 1685 (1963).

(32) Birks, F. T., Weldrick, G. J., Thomas, A. M., *Analyst* **89,** 36 (1964).

(33) Boef, G. den, Poeder, B. C., *Anal. Chim. Acta* **30,** 261 (1964).

(34) Bontschev, P. R., *Mikrochim. Acta* **1962,** p. 577.

(35) Bottei, R. S., Trusk, B. A., ANAL. CHEM. **35,** 1910 (1963).

(36) Buchtela, K., Lesigang, M., *Radiochim. Acta* **1,** 208 (1963).

(37) Budesinsky, B., *Collection Czech. Chem. Commun.* **28,** 1858 (1963).

(38) Budesinsky, B., *Zh. Analit. Khim.* **18,** 1071 (1963).

(39) Bulgakova, A. M., Zalubovskaya, N. P., *Ibid.*, **18,** 1475 (1963).

(40) Burriel-Marti, F., Vidan, A. M., *Anal. Chim. Acta* **26,** 163 (1962).

(41) Busev, A. I., Fan, C., *Talanta* **9,** 107 (1963).

(42) Busev, A. I., Nacu, A., Rudzit, G. P., *Zh. Analit. Khim.* **19,** 337 (1964).

(43) Bykov, V. P., Sorokin, I. V., *Zavodsk. Lab.* **29,** 1065 (1963).

(44) *Ibid.*, p. 1074.

(45) Carter, G. F., *Appl. Spectry.* **16,** 159 (1962).

(46) Catoggio, J. A., Rogers, L. B., *Talanta* **9,** 377 (1962).

(47) *Ibid.*, p. 387.

(48) Caton, R. D., Jr., Freund, H., ANAL. CHEM. **36,** 15 (1964).

(49) Cerrai, E., Testa, C., *Anal. Chim. Acta* **26,** 204 (1962).

(50) Chamberlain, G. T., *Appl. Spectry.* **16,** 1 (1962).

(51) Chen, Y., Chu, C., *Hua Hsueh Hsueh Pao* **27,** 181 (1961).

(52) Cheng, K. L., *Anal. Chim. Acta* **28,** 41 (1963).

(53) Cheng, K. L., *Talanta* **8,** 658 (1961).

(54) Cheng, K. L., Goydish, B. L., *Ibid.*, **9,** 987 (1962).

(55) Chinaglia, B., Ciuffolotti, L., Fasolo, G. B., Malvano, R., *Energia Nucl.* (*Milan*) **9,** 503 (1962).

(56) Chirnside, R. C., Cluley, H. J., Powell, R. J., Proffitt, P. M. C., *Analyst* **88,** 851 (1963).

(57) Ciuffolotti, L., Fasolo, G. B., Malvano, R., *Energia Nucl.* (*Milan*) **10,** 381 (1963).

(58) Clapham, W. F., Hayter, C. J., *Phys. Med. Biol.* **7,** 313 (1962).

(59) Cockbill, M. H., *Analyst* **87,** 611 (1962).

(60) Cofield, R. E., *Health Phys.* **9,** 283 (1963).

(61) Colombo, U. P., Sironi, G., Fasolo, G. B., Malvano, R., ANAL. CHEM. **36,** 802 (1964).

(62) Connor, J., Bass, S. T., *Appl. Spectry.* **16,** 150 (1962).

(63) Corth, R., ANAL. CHEM. **34,** 1607 (1962).

(64) Dams, R., Hoste, J., *Talanta* **8,** 664 (1961).

(65) De, A. K., Rahaman, M. S., *Ibid.*, **11,** 601 (1964).

(66) Degtyreva, O. F., Ostrovskaya, M. F., *Zavodsk. Lab.* **30,** 174 (1964).

(67) Dehne, G. C., Mellon, M. G., ANAL. CHEM. **35,** 1382 (1963).

(68) Dema, I., *Rev. Chim.* (*Bucharest*) **12,** 231 (1961).

(69) Dev, B., Jain, B. D., *Current Sci.* (*India*) **32,** 306 (1963).

(70) Dikshitulu, L. S. A., Rao, G. G., *Talanta* **9,** 857 (1962).

(71) Dragulescu, C., Simonescu, T., Policec, S., *Ibid.*, **11,** 747 (1964).

(72) D'Silva, A. P., Kniseley, R. N., Fassel, V. A., ANAL. CHEM. **36,** 1287 (1964).

(73) Dutt, Y., Singh, R. P., *Indian J. Chem.* **1,** 471 (1963).

(74) Eberle, A. R., ANAL. CHEM. **35,** 669 (1963).

(75) Eberle, A. R., Lerner, M. W., *Ibid.*, **34,** 627 (1962).

(76) Edge, R. A., Dunn, J. D., Ahrens, L. H., *Anal. Chim. Acta* **27,** 551 (1962).

(77) Ehmann, W. D., "Proc. Intern. Conf. Mod. Trends Activation Anal.," p. 41, A & M College of Texas, College Station, Texas, 1961.

(78) Ehmann, W. D., Setser, J. L., *Science* **139,** 594 (1963).

(79) Elbeih, I. I. M., Garba, G. G., *Chemist-Analyst* **52,** 13 (1963).

(80) Elinson, S. V., "Methody Opredeleniya i Analiza Redkikh Elementov," p. 303, Publishing House of the Academy of Sciences, Moscow, 1961.

(81) Elinson, S. V., Pobedina, L. I., *Zh. Analit. Khim.* **18,** 189 (1963).

(82) *Ibid.*, p. 734.

(83) Elwell, W. T., Wood, D. F., *Anal. Chim. Acta* **26,** 1 (1962).

(84) Elwell, W. T., Wood, D. F., "The Analysis of Titanium, Zirconium, and Their Alloys," p. 198, Wiley, London, 1961.

(85) Fadeeva, V. I., Alimarin, I. P., *Zh. Analit. Khim.* **17,** 1020 (1962).

(86) Fasolo, G. B., Malvano, R., Massaglia, A., *Anal. Chim. Acta* **29,** 569 (1963).

(87) Fassel, V. A., Mossotti, V. G., ANAL. CHEM. **35,** 252 (1963).

(88) Fassel, V. A., Myers, R. B., Kniseley, R. N., *Spectrochim. Acta* **19,** 1187 (1963).

(89) Fournet, L., *Ann. Chim.* (*Paris*) **7,** 763 (1962).

(90) Frederickson, L. D., Jr., Hausen, D. M., ANAL. CHEM. **35,** 818 (1963).

(91) Fritz, J. S., Abbink, J. E., *Ibid.*, **34,** 1080 (1962).

(92) Fritz, J. S., Hedrick, C. E., *Ibid.*, **36,** 1324 (1964).

(93) Fukai, R., Meinke, W. W., *Limnol. Oceanog.* **7,** 186 (1962).

(94) Gallai, Z. A., Alimarin, I. P., Sheina, N. M., *Zh. Analit. Khim.* **18,** 1442 (1963).

(95) Gaur, J. N., *Z. Anal. Chem.* **194, 47** (1963).

(96) Ginsberg, H., Pfundt, H., *Z. Metallk.* **53,** 695 (1962).

(97) Girardi, F., Pietra, R., ANAL. CHEM. **35,** 173 (1963).

(98) Goldstein, G., Manning, D. L., Zittel, H. E., *Ibid.*, **35,** 17 (1963).

(99) Goleb, J. A., Faris, J. P., Meng, B. H., *Appl. Spectry.* **16,** 9 (1962).

(100) Goroshchenko, Ya. G., Volkova, M. I., Babkin, A. G., Pyryaev, N. K., *Zh. Analit. Khim.* **18,** 739 (1963).

(101) Gruverman, I. J., Henninger, W. A., ANAL. CHEM. **34,** 1680 (1962).

(102) Guyon, J. C., *Anal. Chim. Acta* **30,** 395 (1964).

(103) Haas, W., Schwarz, T., *Microchim. Ichnoanal. Acta* **2,** 253 (1963).

(104) Haerdi, W., Martin, E., Monnier, D., *Helv. Chim. Acta* **46,** 1572 (1963).

(105) Hahn-Weinheimer, P., Ackermann, H., *Z. Anal. Chem.* **194,** 81 (1963).

(106) Hainski, Z., Rossi, G., *Met. Ital.* **55,** 391 (1963).

(107) Hamaguchi, H., Kuroda, R., Hosohara, K., Shimizu, T., *Nippon Genshiryoku Gakkaishi* **5,** 662 (1963).

(108) Hampson, B. L., *Analyst* **88,** 529 (1963).

(109) Havermans, E., Verbeek, F., Hoste, J., *Anal. Chim. Acta* **26,** 326 (1962).

(110) Headridge, J. B., Dixon, E. J., *Analyst* **87,** 32 (1962).

(111) Headridge, J. B., Taylor, M. S., *Ibid.*, **88,** 590 (1963).

(112) Healy, W. B., McCabe, W. J. ANAL. CHEM. **35,** 2117 (1963).

(113) Heffelfinger, R. E., Blosser, E. R., Perkins, O. E., Henry, W. M., *Ibid.*, **34,** 621 (1962).

(114) Hercules, D. M., *Talanta* **8,** 485 (1961).

(115) Hibbits, J. O., Oberthin, H., Liu, R., Kallmann, S., *Ibid.*, **8,** 209 (1961).

(116) Hibbits, J. O., Williams, R. T., *Anal. Chim. Acta* **26,** 363 (1962).

(117) Hobart, E. W., Hurley, E. P., *Ibid.*, **27,** 144 (1962).

(118) Hoshino, Y., *Bunseki Kagaku* **11,** 1032 (1962).

(119) Hostein, C., Sifferlen, R., *Chim. Anal.* (*Paris*) **45,** 634 (1963).

(120) Hu, C., Wang, S., Chang, S., *Kexue Tongbao* **2,** 34 (1962).

(121) Ishii, D., Takeuchi, T., *Bunseki Kagaku* **11,** 267 (1962).

(122) Ishiwatari, N., Onishi, H., *Ibid.*, **11,** 576 (1962).

(123) Ito, T., Hoshino, Y., *Ibid.*, **12,** 587 (1963).

(124) *Ibid.*, p. 695.

(125) Janauer, G. E., Korkisch, J., *Talanta* **8,** 569 (1961).

(126) Joensuu, O. I., Suhr, N. H., *Appl. Spectry.* **16,** 101 (1962).

(127) Johnson, A. R., Jr., Ph.D. thesis, Oregon State University, 1962.

(128) Kahn, H. L., Slavin, W., *Appl. Opt.* **2,** 931 (1963).

(129) Kaiser, D. G., Meinke, W. W., *Anal. Chim. Acta* **29,** 211 (1963).

(130) Kalinin, A. I., Kuznetsov, R. A., Moiseev, V. V., *Radiokhimiya* **4,** 575 (1962).

(131) Kallmann, S., Oberthin, H., Liu, R., ANAL. CHEM. **34,** 609 (1963).

(132) Kamemoto, Y., Yamagishi, S., *Nippon Kagaku Zasshi* **84,** 270 (1963).

(133) Kamemoto, Y., Yamagishi, S., *Talanta* **11,** 27 (1964).

(134) Karttunen, J. O., ANAL. CHEM. **35,** 1044 (1963).

(135) Katyal, M., Singh, R. P., *Proc. Indian Acad. Sci. Sect. A* **57,** 259 (1963).

(136) Kim, C. K., Meinke, W. W., ANAL. CHEM. **35,** 2135 (1963).

(137) Kinson, K., Hodges, R. J., Belcher, C. P., *Anal. Chim. Acta* **29,** 134 (1963).

(138) Kirby, R., Freiser, H., ANAL. CHEM. **35,** 122 (1963).

(139) Kirkbright, G. F., Yoe, J. H., *Talanta* **11,** 415 (1964).

(140) Klimov, V. V., Didkovskaya, O. S., *Zavodsk. Lab.* **29,** 147 (1963).

(141) Ko, R., ANAL. CHEM. **36,** 1290 (1964).

(142) Kolthoff, I. M., Hodara, I., *J. Electroanal. Chem.* **4,** 369 (1962).

(143) Kono, N., Ozawa, T., *Tokyo-to Ritsu Kogyo Shoreikan Hokoku* **15,** 59 (1963).

(144) Konovalov, E. E., Peizulaev, Sh. I., Pinchuk, G. P., Larionova, I. E., Kondrat'eva, L. I., *Zh. Analit. Khim.* **18,** 624 (1963).

(145) Korkisch, J., Hazan, I., Arrhenius, G., *Talanta* **10,** 865 (1963).

(146) Kosta, L., Dular, M., *Ibid.*, **8,** 265 (1961).

(147) Kotlyar, E. E., Nazarchuk, T. N., *Zh. Analit. Khim.* **18,** 474 (1963).

(148) Kriege, O. H., Rudolph, J. S., *Talanta* **10,** 215 (1963).

(149) Kubota, H., Surak, J. G., ANAL. CHEM. **35,** 1715 (1963).

(150) Kugai, L. N., Nazarchuk, T. N., *Zh. Analit. Khim.* **17,** 1082 (1962).

(151) Kumar, A. N., Singh, R. P., *Indian J. Chem.* **1,** 514 (1963).

(152) Kurbatov, D. I., Skorynina, I. S., *Zh. Analit. Khim.* **17,** 711 (1962).

(153) Kuznetsova, N. N., Krauz, L. S., *Ibid.*, **18,** 1090 (1963).

(154) Laib, R. D., *Appl. Spectry.* **17**, 160 (1963).
(155) Lannoye, R. A., ANAL. CHEM. **35**, 558 (1963).
(156) Lassner, E., *Talanta* **10**, 1229 (1963).
(157) Laverlochere, J., May, S., *Bull. Soc. Chim. France* **3**, 457 (1963).
(158) Lee, W. F., Shastri, N. K., Amis, E. S., *Talanta* **11**, 685 (1964).
(159) Liteanu, C., Lukacs, I., Strusievici, C., *Anal. Chim. Acta* **29**, 574 (1963).
(160) Luke, C. L., ANAL. CHEM. **35**, 56 (1963).
(161) *Ibid.*, **36** 1327 (1964).
(162) McCown, J. J., Kudera, D. E., *Ibid.*, **35**, 1545 (1963).
(163) MacDonald, N. S., Hutchinson, D. L., Moyer, D. L., Chez, R. A., *Science* **141**, 1033 (1963).
(164) Machlan, L. A., Hague, J. L., *J. Res. Natl. Bur. Std.*, *A* **66**, 517 (1962).
(165) McKaveney, J. P., ANAL. CHEM. **35**, 2139 (1963).
(166) Maeck, W. J., Kussy, M. E., Rein, J. E., *Ibid.*, **34**, 1602 (1962).
(167) Maeck, W. J., Marsh, S. F., Rein, J. E., *Ibid.*, **35**, 292 (1963).
(168) Magee, R. J., Martin, I., *Anal. Chim. Acta* **28**, 366 (1963).
(169) Magee, R. J., Martin, I., "Analytical Chemistry 1962," p. 193, Elsevier, New York, 1963.
(170) Magee, R. J., Witwit, A. S., *Anal. Chim. Acta* **29**, 27 (1963).
(171) *Ibid.*, p. 517.
(172) Maienthal, E. J., Taylor, J. K., ANAL. CHEM. **36**, 1286 (1964).
(173) Majumdar, A. K., Bag, S. P., *Anal. Chim. Acta* **28**, 293 (1963).
(174) Majumdar, A. K., Pal, B. K., *Ibid.*, **27**, 356 (1962).
(175) *Ibid.*, **29**, 168 (1963).
(176) Mamaev, V. E., *Met. i Khım. Prom. Kazakhstana, Nauchn.-Tekhn. Sb.* **4**, 76 (1961).
(177) Mambetov, A. A., Rzaeva, N. A., *Tr. Azerb. Sel'skokhoz. Inst.* **11**, 75 (1960).
(178) Marchart, H., *Anal. Chim. Acta* **30**, 11 (1964).
(179) Mari, E. A., *Ibid.*, **29**, 312 (1963).
(180) Martin, E. L., Bentley, K. E., ANAL. CHEM. **34**, 354 (1962).
(181) Martin, I., Magee, R. J., *Talanta* **10**, 1119 (1963).
(182) Matthes, F., Boethin, T., Rybak, R., *Chem. Tech.* (*Berlin*) **14**, 550 (1962).
(183) Mikhail, S. Z., Paddelford, D. F., ANAL. CHEM. **35**, 24 (1963).
(184) Miller, C. C., Thow, D. H., *Talanta* **8**, 43 (1961).
(185) Miller, F. J., Zittel, H. E., *J. Electroanal. Chem.* **7**, 116 (1964).
(186) Minczewski, J., Bozycki, C., *Chem. Anal.* (*Warsaw*) **8**, 977 (1963).
(187) Minczewski, J., Skorko-Trybula, Z., *Talanta* **10**, 1063 (1963).
(188) Mitchell, B. J., O'Hear, H. J., ANAL. CHEM. **34**, 1620 (1962).
(189) Molinski, V. J., Wahl, W. H., Strain, W. H., *Trans. Am. Nucl. Soc.* **6**, 398 (1963).
(190) Monnier, D., Haerdi, W., Martin, E., *Helv. Chim. Acta* **46**, 1042 (1963).
(191) Moroshkina, T. M., Smirnova, M. N., *Zh. Analit. Khim.* **19**, 325 (1964).
(192) Motojima, K., Hashitani, H., Kimie, I., Yoshida, H., *Bunseki Kagaku* **11**, 47 (1962).
(193) Motojima, K., Hashitani, H., Yoshida, H., *Ibid.*, **11**, 659 (1962).
(194) Mukherji, A. K., ANAL. CHEM. **36**, 1064 (1964).
(195) Murty, V. S. R., Rao, G. G., *Talanta* **8**, 426 (1961).
(196) Muzgin, V. N., Zolotavin, V. L., Gavrilov, F. F., *Zh. Analit. Khim.* **19**, 111 (1964).
(197) Nakano, M., *Appl. Spectry.* **16**, 165 (1962).
(198) Nakata, H., Kusaka, Y., Kikkawa, S., *Bull. Chem. Soc. Japan* **35**, 1611 (1962).
(199) Nangniot, P., *J. Electroanal. Chem.* **7**, 50 (1964).
(200) Naude, W. J., Zeeman, P. B., *Spectrochim. Acta* **19**, 2075 (1963).
(201) Nazarenko, V. A., Yagnyatinskaya, G. Ya., *Dopovidi Akad. Nauk Ukr. RSR* **9**, 1218 (1963).
(202) Oda, N., Idohara, M., *Nippon Kinzoku Gakkaishi* **28**, 39 (1964).
(203) Oka, Y., Kato, T., Sasaki, M., *Nippon Kagaku Zasshi* **84**, 588 (1963).
(204) Oka, Y., Yanai, M., *Bunseki Kagaku* **13**, 207 (1964).
(205) Okada, M., *Nature* **196**, 1088 (1962).
(206) Okada, M., *Tokyo Kogyo Shikensho Hokoku* **58**, 7 (1963).
(207) Olson, D. C., Margerum, D. W., ANAL. CHEM. **34**, 1299 (1962).
(208) Onishi, H., *Bunseki Kagaku* **12**, 1153 (1963).
(209) Osterberg, C., Kulm, L. D., Byrne, J. V., *Science* **139**, 916 (1963).
(210) Overman, R. F., *Nucl. Sci. Eng.* **14**, 249 (1962).
(211) Owens, E. B., *Appl. Spectry.* **16**, 86 (1962).
(212) Pan, K., Sun, P., Wong, W., *J. Chinese Chem. Soc.* (*Taiwan*) **8**, 298 (1961).
(213) Panwar, K. S., Gaur, J. N., *Indian J. Chem.* **1**, 361 (1963).
(214) Parissakis, G., Kontoyannakos, J., *Anal. Chim. Acta* **29**, 220 (1963).
(215) Park, T., Kim, T., Suo, K., *Kwayon Hwibo* **5**, 189 (1960).
(216) *Ibid.*, p. 190.
(217) Patrovsky, V., *Collection Czech. Chem. Commun.* **27**, 1824 (1962).
(218) Pearse, G. A., Jr., ANAL. CHEM. **34**, 536 (1962).
(219) Peng, P. Y., Sandell, E. B., *Anal. Chim. Acta* **29**, 325 (1963).
(220) Pijck, J., Kim, J. I., *J. Pharm. Belg.* 19 (1–2), 3 (1964).
(221) Pilipenko, A. T., Eremenko, O. M., *Ukr. Khim. Zh.* **29**, 532 (1963).
(222) *Ibid.*, p. 538.
(223) Pollock, E. N., Zopatti, L. P., *Anal. Chim. Acta* **28**, 68 (1963).
(224) Popa, G., Popea, F., Cluceru, D., Baiulescu, G., *Ibid.*, **26**, 434 (1962).
(225) Porter, G., "Columbium and Tantalum," p. 535, Wiley, New York, 1963.
(226) Pribil, R., Vesely, V., *Chemist-Analyst* **53**, 43 (1964).
(227) Pribil, R., Vesely, V., *Talanta* **10**, 1287 (1963).
(228) Raby, B. A., Banks, C. V., ANAL. CHEM. **36**, 1107 (1964).
(229) Rafiq, M., Rulfs, C. L., Elving, P. J., *Talanta* **10**, 696 (1963).
(230) *Ibid.*, p. 827.
(231) Raikhbaum, Ya. D., Malhkh, V. D., Luzhnova, M. A., *Zavodsk. Lab.* **29**, 677 (1963).
(232) Rao, G. G., Dikshitulu, L. S. A., *Talanta* **9**, 289 (1962).
(233) *Ibid.*, **10**, 295 (1963).
(234) *Ibid.*, p. 1023.
(235) Rao, G. G., Rao, P. K., *Ibid.*, **11**, 703 (1964).
(236) Rao, G. G., Sagi, S. R., *Ibid.*, **10**, 169 (1963).
(237) Roellig, H. E., Trommer, E., Minenko, A., *Acta Chim. Acad. Sci. Hung.* **32**, 159 (1962).
(238) Ross, W. D., Wheeler, G., Jr., ANAL. CHEM. **36**, 266 (1964).
(239) Rothmann, H., Schneider, H., Niebuhr, J., Pothmann, C., *Arch. Eisenhuettenw.* **33**, 17 (1962).
(240) Rozsa, J. T., Stone, J., "Developments in Applied Spectroscopy," Vol. **1**, p. 187, Plenum Press, New York, 1962.
(241) Sakagishi, S., Ueno, K., Minami, K., *Radioisotopes* (*Tokyo*) **12**, 11 (1963).
(242) Sarma, P. L., ANAL. CHEM. **36**, 1076 (1964).
(243) Savvin, S. B., *Talanta* **8**, 673 (1961).
(244) *Ibid.*, **11**, 1 (1964).
(245) Savvin, S. B., *Zavodsk. Lab.* **29**, 131 (1963).
(246) Saxena, R. S., Sharma, O. P., *Talanta* **11**, 863 (1964).
(247) Schmied, H., Bergamin, U., *Neue Tech.* **5**, 732 (1963).
(248) Selmer-Olsen, A. R., *Anal. Chim. Acta* **26**, 482 (1962).
(249) Semenenko, K. A., Tarasevich, N. I., *Zh. Analit. Khim.* **18**, 88 (1963).
(250) Seyb, K. E., *Radiochim. Acta* **1**, 167 (1963).
(251) Shams El Din, A. M., Gerges, A. A. A., *J. Electroanal. Chem.* **4**, 309 (1962).
(252) Sharma, V. K., Gaur, J. N., *Ibid.*, **5**, 375 (1963).
(253) Shen, H., *Hua Hsueh Hsueh Pao* **25**, 231 (1962).
(254) Sheskol'skaya, A. Ya., *Zh. Analit. Khim.* **17**, 949 (1962).
(255) *Ibid.*, **18**, 782 (1963)
(256) Shkaravskii, Yu. F., *Ibid.*, **18**, 196 (1963).
(257) Singh, D., Agarwala, V. S., *Z. Anorg. Allgem. Chem.* **321**, 81 (1963).
(258) Smirnova, M. N., Moroshkina, T. M., *Vestn. Leningr. Univ.*, *No.* **22**, *Ser. Fiz. i Khim.*, *No. 4*, 172 (1963).
(259) Soltys, M. N., Morrison, G. H., ANAL. CHEM. **36**, 293 (1964).
(260) Stary, J., Ruzicka, J., Zeman, A., *Anal. Chim. Acta* **29**, 103 (1963).
(261) Steele, E. L., Meinke, W. W., "Proceedings, 1961 International Conference on Modern Trends in Activation Analysis," p. 161, College Station, Texas, 1961.
(262) Stehlik, G., Altmann, H., *Monatsh. Chem.* **94**, 1163 (1963).
(263) Takahashi, Y., *Bunseki Kagaku* **13**, 193 (1964).
(264) Tananaiko, M. M., Blucke, L. A., *Ukr. Khim. Zh.* **29**, 974 (1963).
(265) Tappe, W., van Calker, J., *Z. Anal. Chem.* **198**, 13 (1963).
(266) Tomkins, M. L., Borun, G. A., Fahlbusch, W. A., ANAL. CHEM. **34**, 1260 (1962).
(267) Townsend, J. E., *Appl. Spectry.* **17**, 37 (1963).
(268) Tramm, R. S., Pevzner, K. S., *Zavodsk. Lab.* **30**, 20 (1964).
(269) Tserkovnitskaya, I. A., Borovaya, N. S., *Vestn. Leningr. Univ.* **17**, No. 16, *Ser. Fız. ı Khım.*, *No. 3*, 148 (1962).
(270) Tsuchiya, Y., *Bunsekı Kagaku* **11**, 1176 (1962).
(271) Tsykhanskii, V. D., Krinberg, I. A., *Izv. Sibirsk. Otd. Akad. Nauk SSSR* **11**, 125 (1963).
(272) Ushakova, A. M., *Zh. Analit. Khim.* **18**, 79 (1963).
(273) Vasil'eva, N. L., Ermakova, M. I., *Ibid.*, **18**, 545 (1963).
(274) Vinogradov, A. V., Dronova, M. I., Korovin, Yu. I., *Ibid.*, **18**, 29 (1963).
(275) Vinokurov, V. M., Zoripov, M. M., Polskii, Yu. E., Stepanov, V. G., Chirkin, G. K., Shekun, L. Ya., *Geokhimiya* **11**, 1002 (1963).
(276) Volodarskaya, R. S., Derevyanko, G. N., *Zavodsk. Lab.* **29**, 28 (1963).
(277) Vorobev, V. S., Rusanov, A. K., *Zh. Analit. Khim.* **19**, 286 (1964).
(278) Wappner, B. G. de, *Anales Asoc. Quim. Arg.* **49**, 285 (1961).
(279) Webb, H. W., Ashworth, V., Hills, J. M., *Analyst* **88**, 142 (1963).

(280) Weiner, J. R., O'Connor, J. J., Rubin, B., *J. Electrochem. Soc.* **110**, 1160 (1963).
(281) Weiss, H. V., Lai, M. G., *Talanta* **8**, 72 (1961).
(282) Wester, P. O., Brune, D., Samsahl, K., *Intern. J. Appl. Radiation Isotopes* **15**, 59 (1964).
(283) Wheat, J. A., *Appl. Spectry.* **16**, 108 (1962).
(284) Wish, L., ANAL. CHEM. **34**, 625 (1962).
(285) Witwit, A. S., Magee, R. J., *Anal. Chim. Acta* **27**, 366 (1962).
(286) Witwit, A. S., Magee, R. J., Wilson, C. L., *Talanta* **9**, 495 (1962).
(287) Wood, D. F., Clark, R. T., *Analyst* **87**, 342 (1962).
(288) Wood, D. F., McKenna, R. H., *Ibid.*, **87**, 880 (1962).
(289) Yatsimirskii, K. B., Raizman, L. P., *Zh. Analit. Khim.* **18**, 829 (1963).
(290) Yatsimirskii, K. B., Raizman, L. P., *Zh. Neorgan. Khim.* **7**, 1819 (1962).
(291) Yu, J., *Hua Hsueh Tung Pao* **6**, 343 (1962).
(292) Zahnow, E. W., Robinson, R. J., *J. Electroanal. Chem.* **3**, 263 (1962).
(293) Zitnansky, B., Sebestian, I., *Chem. Listy* **57**, 518 (1963).
(294) Zittel, H. E., ANAL. CHEM. **35**, 329 (1963).

Work was performed in the Ames Laboratory of the U. S. Atomic Energy Commission.

Vacuum Fusion and Vacuum Extraction Analysis for Nitrogen in Metals

Basic Principles and Current Status

G. W. Goward, Advanced Materials Research and Development Laboratory, Pratt and Whitney Aircraft, North Haven, Conn.

VACUUM FUSION is the most widely used method for the determination of gaseous constituents in metals. In a typical analysis, a specimen of metal is introduced into molten iron or platinum in a graphite crucible and the gases oxygen, nitrogen, and hydrogen are released as carbon monoxide, elemental nitrogen, and hydrogen, respectively. In the past, the greatest emphasis has been placed on the determination of oxygen. Values for nitrogen, obtained with little or no extra expenditure of time and energy, are usually considered to be less accurate than those from wet chemical methods. There are exceptions to these generalizations; extensive studies have been made on the determination of nitrogen in steels by vacuum fusion (*5, 12, 29*) and a few studies have been made on other metals (*4, 28*). Nitrogen values obtained by vacuum fusion are still, however, regarded with suspicion. This is particularly true for the reactive metals (Ti, Zr, and Hf) and to a lesser extent, the refractory metals (V, Nb, Ta and Cr, Mo, W).

In general terms, the vacuum fusion method is potentially as sensitive as, or more sensitive than, any other method currently in use for the determination of nitrogen. It is not unreasonable to assume that it could be used to determine as little as a few tenths of a part per million of nitrogen were it not for the uncertainty over the quantitative release of nitrogen from the reaction medium. In view of these considerations, it has been deemed worthwhile to re-examine the method, principally in terms of reaction mechanisms in the vacuum fusion process and comparison of results obtained by vacuum fusion with those from other methods. For practical purposes, this review is limited to the reactive and refractory metals and steels. Since hot vacuum extraction (without fusion) has been shown to be useful for the determination of nitrogen in a few cases (*16, 28*), some consideration is also given to this method in this review. A limited review is also given on the state and solubility of nitrogen in the reactive and refractory metals and steels.

NATURE AND SOLUBILITY OF NITROGEN IN METALS

When nitrogen dissolves in a metal it is usually found that the relation between pressure and composition at constant temperature follows Sieverts' law, which states that the amount of nitrogen in solution is proportional to the square root of the equilibrium nitrogen pressure:

$$P_{N_2}^{1/2} = K\ (\%\text{N}) \qquad (1)$$

Symbolizing nitrogen in solution as [N], the implication to be drawn from this relation is that the reaction involved during dissolution is

$${}^1/_2\ \text{N}_{2_g} \rightarrow [\text{N}] \qquad (2)$$

or that nitrogen goes into solution as single atoms. At any given temperature, the upper limit of adherence to Sieverts' law is governed by the stability of the nitride of the metal in question—i.e., by the reaction

$$\text{M} + {}^1/_2\ \text{N}_2 \rightarrow \text{MN} \qquad (3)$$

At a pressure corresponding to the equilibrium pressure of nitrogen over the nitride at the stated temperature, Sieverts' law no longer holds because the pressure is invariant (provided that the composition of the nitride is invariant). If the nitride is extremely stable the limits of temperature and pressure over which Sieverts' law can be expected to apply may be so restrictive as to prohibit direct experimental study of the relation. For example, the equilibrium nitrogen pressure over TiN is about 10^{-3} mm. at 2330° C. and 1 mm. at 2910° C; thus, Sieverts' law could be expected to be demonstrable only below 10^{-3} mm. at 2330° and only below 1 mm. at 2910° C. These temperatures are both well above the melting point of the metal (1812° C.) and so there is little hope of direct study of solution equilibria for the solid state. Approximately the same limitations can be expected to hold for other reactive metals such as Zr, Hf, and Th, which also form very stable nitrides. As a consequence of these limitations, the solubility of nitrogen in these metals can only be measured in equilibrium with the precipitated nitride. Reasonably reliable values for these terminal solubilities are available in the literature for many metals (*18, 24*).

However, if the terminal solubility of the nitride and the equilibrium pressure of nitrogen over the nitride are known at a given temperature the upper limiting point of Sieverts' law at this temperature, and thus the Sieverts' law constant, *K*, can be calculated. The rationale upon which this calculation is based is that the equilibrium pressure over the nitride and the solid solution are necessarily identical at this point.

For those elements which form nitrides of somewhat lower stability (V, Nb, Ta; Cr, Mo, W; Fe, in order of decreasing stability) direct studies of the Sieverts' law relation are possible. Such studies have been performed for Nb (*2, 7, 33*), Ta (*1, 33*), Cr(*40*), Mo, W (*32, 41*), and Fe and numerous iron alloys (*34*). In all but one of these cases it has been observed that Sieverts' law is obeyed in the ranges studied. The only exception is the high temperature (2300–3000° C.) behavior of the tantalum–nitrogen system recently reported by Pemsler (*33*). The experimental difficulties involved at such high temperatures may be the cause of this apparent deviation.

It is of some significance to the analytical determination of gases in the reactive and refractory metals that two groups of these metals (Ti, Zr, Hf and V, Nb, Ta) are exothermic occluders of nitrogen (heat is evolved during the reaction with nitrogen) and the other group (Cr, Mo, W) apparently consists of endothermic occluders (heat is absorbed during the reaction with nitrogen). Actually two sets of data [Norton and Marshall (*32*) *vs.* Sieverts and Bruning (*41*)] are in conflict for molybdenum; the measurement is difficult for tungsten; and the data are rather inconclusive for chromium (*40*). The significance of a negative heat of reaction is that the solubility of nitrogen in the metal in equilibrium with the gas decreases with increasing temperature, the reverse being true for a positive heat of reaction. The iron–nitrogen system is interesting in this respect in that in the αFe region (up to 900° C.), solubility increases with temperature; in the γFe region (900–1400° C.), solubility decreases with increasing temperature; and in both the δFe region (1400–1535° C.) and the liquid state, solubility again increases with increasing temperature.

A very brief summary of solubilities and temperature coefficients of solubility is presented in Table I. For further details the reader is referred to the very extensive literature on this subject. The most significant point to be deduced from the data in Table I is that the analyst must almost invariably deal with a solid solution of nitrogen in the metal for the elements Ti, Zr, Hf, V, Nb, Ta (and perhaps Cr) rather than a nitride. The significance of this will be enlarged upon in later discussions in this report on the mechanism of vacuum fusion reactions. Suffice it to state at this point that most, if not all, discussions on the mechanism of release of nitrogen during the vacuum fusion reaction have been based on the free energy of formation of nitrides, which nitrides are rarely found to be present in the above metals. It will be shown that it is more pertinent to consider these mechanisms in terms of the Sieverts' law constants, or partial molar free energies of solution of nitrogen in the metal, which free energies are quite different than those of the nitrides. Even in those cases where nitrides might be present, their decomposition is still only a first consideration; the removal of nitrogen from solid or liquid solution is still the thermodynamic limiting factor in the analysis.

The analysis of steels presents a somewhat different condition in that a large part of the nitrogen present may be in the form of precipitated nitrides such as AlN, BN, NbN, Si_3N_4, TiN, VN, and ZrN. The analyst is then to be concerned with mechanisms for decomposing the nitrides. The high stabilities of some of these compounds have apparently proved to be the limiting factor in quantitative release of nitrogen from some steels (*5, 21, 29*) although this question is still the subject of some controversy.

HOT VACUUM EXTRACTION ANALYSIS FOR NITROGEN IN METALS

Theoretical Considerations. THERMODYNAMICS. The mechanism for the hot vacuum extraction process may be postulated as follows (assuming the initial presence of nitride),

$$MN \rightarrow M + {}^1/_2N_2 \qquad (4)$$

$$[N] \rightarrow {}^1/_2N_2 \qquad (5)$$

where [N] denotes nitrogen in solid solution. If nitrogen is initially present only in solid solution, Equation 4 does not apply. If it is present as nitride, both equations still apply because the nitride will be in equilibrium with the saturated solid solution initially, and at all stages of the decomposition. It should be obvious at this point that the nitrogen is more difficult to remove from the solid solution than from the nitride. The feasibility of quantitatively decomposing the nitride can be evaluated from data on the free energy of formation of the nitrides, or more directly from published equilibrium nitrogen pressures at various temperatures. Data for TiN, ZrN, VN (*45*), Nb_2N (*7*), TaN (*45*), Cr_2N (*24*), Mo_2N (*45*), and W_2N (*40*) are presented in Table II. It appears that V_2N and Ta_2N are the phases actually in equilibrium with V and Ta metals, respectively, but no reasonable thermodynamic data exist for these nitrides.

The data indicate that the nitrides of Cr, Mo, W, V, N, Ta (and Al, Si, B) should be decomposable at temperatures readily attainable in a standard vacuum fusion apparatus. The nitrides of Ti, Zr, and Th decompose only at much higher temperatures. It should be noted at this point that the above considerations are purely thermodynamic; no account can be taken of kinetic factors which can only be evaluated experimentally.

Following thermal decomposition of the nitride, the remaining metal will still contain some amount of nitrogen in solution. If the initial sample was pure nitride, the amount of nitrogen remaining in the residual metal will be inconsequential in comparison to the total amount of nitrogen initially present and therefore the extraction of the nitrogen from the solution will not be important. If, however, the initial sample was principally metallic with only a small amount of precipitated nitride present, the large proportion of nitrogen remaining in solid solution must be extracted to obtain a quantitative analysis. The same considerations apply to a sample containing only nitrogen in solid solution with no precipitated nitride present.

To estimate the feasibility of extracting nitrogen from solid solution, use has been made of the Sieverts' law in the form given by Equation 1. If the nitrogen concentration is known at a given equilibrium pressure, *K* may be calculated directly. It is then assumed that the expression holds down to low (1 p.p.m.) concentrations and pressures. Table III presents calculated equilibrium pressures of nitrogen, for various temperatures, for 1 p.p.m. of nitrogen, it being assumed that to perform a

Table I. Solubility of Nitrogen in Various Metals

Element	Temp., °C.	p_{N_2} mm.	Solubility	Temp. coeff. of solubility
Cr	1100	1.5	400 p.p.m.	pos.
Cr liq.	1600	760	4.1%	neg.
Mo	1200	760	10.5 p.p.m.	pos.
W	1200	760	0.013 p.p.m.	pos.
V	900	a	<1%	. . .
Nb	1100	b	0.05%	pos.[a]
Ta	1000	c	0.32%	pos.[a]
αTi	1000	d	7.5%	pos.[a]
αZr	1000	e	4.5%	pos.[a]
Th	1000	f	0.11%	. . .
γFe	1000	760	250 p.p.m.	neg.
δFe	1500	760	123 p.p.m.	pos.
liq. Fe	1600	760	450 p.p.m.	pos.

a In equilibrium with VN
b In equilibrium with Nb_2N
c In equilibrium with Ta_2N
d In equilibrium with TiN
e In equilibrium with ZrN
f In equilibrium with ThN

[a] Negative when in equilibrium with N_2 gas.

sensible quantitative analysis, the residual concentration of nitrogen must be reduced to some value less than 1 p.p.m. (if a residual nitrogen content of 10 p.p.m. is acceptable, the corresponding equilibrium nitrogen pressures will be greater by a factor of 10^2). It is seen from the data in Table III that the quantitative extraction of nitrogen from δFe, liquid Fe, Mo, and W is thermodynamically feasible at reasonable temperatures. Although the data in the literature for Cr are somewhat uncertain, it seems that the temperature coefficient of solubility of nitrogen is positive and that therefore, quantitative extraction will be somewhat difficult for this element, even in the liquid state. Equilibrium pressures for 1 p.p.m. nitrogen in Nb and Ta are quite low at all temperatures up to the melting point and so quantitative extraction from these metals will be quite difficult. A rough calculation for zirconium from terminal solubility and free energy data for ZrN and extrapolation of Sieverts' law yields an equilibrium pressure of 2.8×10^{-15} mm. at 1600° C. for 1 p.p.m. of nitrogen in this metal. Obviously, this is an impossible case. Similar results can be expected for Ti.

Table II. Dissociation Pressures of Various Nitrides

Nitride	Temp. (°C.) at which nitrogen pressure is equal to 1 mm.	10^{-2} mm.	10^{-3} mm.
TiN	2912	2489	2331
ZrN	2603	2205	2046
VN	1242	1024	933
(NbN)	(1946)	(1620)	(1491)
Nb_2N	2050	1747	1614
TaN	1921	1599	1460
"Cr_2N"($Cr_2N_{0.76}$)	1050	782	675
Mo_2N	329	243	209
W_2N	344	256	221
AlN	1487	1275	1187
Si_3N_4	1421	1164	1060
BN	1340	1075	927
Th_3N_4	2401	2037	1890

KINETICS. If it is assumed that diffusion of nitrogen through the metal to the surface is the limiting rate process in hot extraction—i.e., no interference by surface films—then, by the use of diffusion theory, an evaluation can be made of the length of time involved in removal of nitrogen from the various metals. A convenient form of the diffusion law for this type of problem has been presented by Fast and Verrijp (*17*) who used the following equation to determine the diffusion coefficient of nitrogen in αFe by measurement of degassing times of cylindrical wires:

$$\log\left(\frac{\bar{C} - C_e}{C_a - C_e}\right) \cong -0.160 - 0.4343\,\frac{t}{\tau} \qquad (6)$$

where C_a = initial concentration of nitrogen;
$\bar{C}$ = mean concentration at time t (seconds);
C_e = final equilibrium concentration;
and $\tau = \dfrac{R^2}{5.784D}$
where R = radius of the cylinder in cm.;
D = diffusion coefficient in cm.^{2}/second

The equation has been shown to be a valid approximation for $t > \tau/3$ or

$$\log\left(\frac{\bar{C} - C_e}{C_a - C_e}\right) < -0.3$$

For outgassing conditions C_e approaches zero and the equation reduces to

$$\log\left(\frac{\bar{C}}{C_a}\right) \cong -0.160 - \frac{2.51\,tD}{R^2} \qquad (7)$$

Since the release of nitrogen by hot extraction can be represented by the first order equation

$$[N] \rightarrow {}^1/_2 N_2$$

(note that Equation 7 is a first order rate equation), it is convenient to calculate extraction times in terms of $t_{1/2}$, the time required to reduce the nitrogen content to one half of its initial concentration. When $t = t_{1/2}$, $\bar{C} = {}^1/_2\, C_a$ and $\log \dfrac{\bar{C}}{C_a} = 0.301$. Using published values of diffusion coefficients, $t_{1/2}$ values have been calculated and are given in Table III. Although literature values of diffusion coefficients for many of these metals are quite varied, estimates of $t_{1/2}$ should be good to within a factor of two. It is observed from the listed $t_{1/2}$ values that hot extraction should be kinetically feasible for all of the elements listed in Table III. No value for the diffusion coefficient of nitrogen in chromium is available but it should be of the same order of magnitude as those for molybdenum and iron.

Practical Applications. The application of hot vacuum extraction to the determination of nitrogen in metals has received very little attention. Fagel, Smith, and Witbeck (*16*) reported "by product" nitrogen values obtained by hot extraction (2000° C.) of Mo, W, Ta and 0.5% Ti–Mo alloy. Comparisons with values obtained by vacuum fusion analysis (iron–tin bath) were favorable for molybdenum and tungsten (some scatter is observed) in agreement with the above theoretical calculations. Recovery from an 0.5% Ti–Mo alloy was also reasonable. Thermodynamic quantities are not currently available for a theoretical evaluation of such a system; a linear interpolation between individual Sieverts' law constants for Ti and Mo to describe the Ti–Mo system is not warranted. Recovery from Ta was poor (10 *vs.* 84 p.p.m. for extraction *vs.* fusion, respectively) in agreement with the above calculations. Mallett and Griffith (*28*) reported the successful determination of nitrogen in molybdenum by vacuum extraction at 1800° C. (apparently in the 20- to 900-p.p.m. range). The method was validated by comparison with values obtained by the Kjeldahl method. Time of extraction was 30 to 50 minutes for 4-mm. thick samples. Thus both aspects, thermodynamic and kinetic, of the above theoretical calculations are confirmed.

The calculations presented in Table III indicate that vacuum extraction of nitrogen from pure iron should be relatively easy. It is unlikely that there is any practical application of this method since analyses of pure iron are rarely needed. The more complicated analysis of steels containing precipitated nitrides would have to be evaluated for each particular case with regard to the types of nitrides present.

Thermodynamic and particularly, Sieverts' law data describing interaction of nitrogen with chromium and vanadium are nonexistent or not very ac-

Table III. Calculated Equilibrium Pressures for 1 p.p.m. Nitrogen for Various Metals

Element	Temp., °C.	Equilibrium N_2 pressure (mm.)	$t_{1/2}$[a] (min.)
δFe	1500	4.8×10^{-2}	1.6
liq. Fe	1600[b]	4.8×10^{-3}	1.4
Cr	1300	9.9×10^{-6}	...
liq. Cr	1600	4.6×10^{-7}	
Mo	2000	6.9×10^{-2}	7
W	2000	6.3×10^{-2}	31
Nb	2000	1.2×10^{-9}	1.9
	2420	3.8×10^{-9}	
Ta	2000	2.7×10^{-9}	2.8
	2960	1.4×10^{-6}	...

[a] 0.4-cm. diameter cylinder.

[b] Molten Fe, 1.6-cm. crucible diameter. For comparison, the equilibrium pressure of 1 p.p.m. of hydrogen in Nb is 1.5×10^{-2} mm. at 1000° C. and 4×10^{-1} mm. at 2000° C.

curate. Either new thermodynamic data should be obtained or more directly, a test of the hot extraction procedure should be performed to evaluate the method. Very rough calculations, based on presently available data, indicate that the method has some chance of success.

In all of the practical applications above, the samples were contained in a graphite crucible at about 2000° C. during the extraction. The question arises as to the degree and effects of interaction of the samples with carbon. Fagel *et al.* (*16*) report "no significant carbon pickup by molybdenum after several hours in the crucible at 2000° C." Contrary to this statement, Ehrlich and Friedrich (*10*) report that molybdenum and tungsten form lamellated surface layers of Mo_2C and W_2C, respectively, during the extraction and that tantalum forms concentric layers of TaC and Ta_2C. In regard to the influence of carbon, three possible effects should be considered: a carbide layer on the surface could prevent diffusion of nitrogen through the surface at practical rates; the equilibrium nitrogen pressure could be decreased by the formation of MC_xN_{1-x} type compounds; and carbon in solid solution could increase the equilibrium nitrogen pressure in analogy to the Fe–C–N system.

For the specific determination of nitrogen by hot vacuum extraction, it is possible to heat the specimens in other types of containers, or in no container at all by induction or, less favorably, by resistance heating techniques. These aspects of the hot vacuum extraction process warrant further study.

VACUUM FUSION ANALYSIS FOR NITROGEN IN METALS

Theoretical Considerations. THERMODYNAMICS. The first attempt at a definitive study of the thermodynamics and mechanisms of the vacuum fusion process was made by Sloman, Harvey, and Kubaschewski (*42*) in 1951. In so far as nitrogen analyses were concerned, the temperature to decompose quantitatively a model compound, TiN, in an iron–carbon bath, was first determined experimentally to be 1800° C. From thermodynamic data, the equilibrium pressure of the system was then calculated for each of the following mechanisms at 1800° C:

(a) thermal dissociation of the nitride

$$MN_{solid} \rightarrow M_{liq.} + {}^1/_2N_2$$

(b) thermal dissociation of the nitride and solution of the metal in liquid iron

$$MN_{solid} \rightarrow [M]_{Fe} + {}^1/_2N_2$$

and (c) reaction of the nitride with carbon to form nitrogen and the metal carbide

$$MN_{solid} + C_{solid} \rightarrow MC_{solid} + {}^1/_2N_2$$

It was then assumed that to decompose quantitatively other nitrides (Mo_2N, Si_3N_4, VN, AlN, VN, ZrN, Th_3N_4), nitrogen pressures equal to those calculated for TiN would be necessary and the required temperatures to yield such pressures were calculated for each of three mechanisms for all of the above nitrides. Comparison of these temperatures with experimentally measured temperatures required for quantitative decomposition of the nitrides was then used to deduce the mechanism involved for each nitride. It was concluded that the principal mechanism was (b) above and that carbide formation played little or no part in the decomposition of the nitrides under consideration.

All subsequent calculations appearing in the literature (*4*, *23*, *45*) have been but slight variations of the above method. All have assumed that the mechanism must be based on some type of decomposition of a nitride. Turovtseva and Kunin (*45*) criticize the method of calculation used by Sloman but offer no real alternative. Turovtseva and Kunin did present some data leading to the following treatment but did not carry the subject to its logical conclusion.

In view of the fact that many of the metals of concern to the vacuum fusion analyst can dissolve relatively large amounts of nitrogen in comparison to the levels normally subjected to analysis, it seems unrealistic to base the mechanism of release of nitrogen on the decomposition of a nitride. This viewpoint is best illustrated by considering the case of analysis of a niobium specimen containing 100 p.p.m. of nitrogen. From information on the solubility of nitrogen in niobium (*2*, *7*), this amount of nitrogen would be present totally in solution; no nitride would be present. If 1 gram of this specimen were dissolved in a 10-gram iron-carbon bath, the total concentration of nitrogen in the 11-gram mixture would be about 10 p.p.m. At 1600° C., this amount of nitrogen would still exist only as a solute in the molten Fe–Nb–C mass [Pehlke and Elliott (*34*) found no nitride precipitation in an Fe–Nb–N solution at 1600° C. containing up to about 2000 p.p.m. of nitrogen and about 9% niobium]. Neglecting for the moment the kinetics of dissolution of the niobium and release of nitrogen from the bath, the problem is reduced to estimating feasibility of quantitatively extacting this dissolved nitrogen from the molten Fe–Nb–C–N system. No consideration need be given to nitride formation or decomposition.

Based on these considerations, the mechanism of the vacuum fusion process for nitrogen in niobium can be formulated as follows:

$$[N]_{Nb_s} + (Fe - C)_l \rightarrow [N]_{(Fe-Nb-C)_l} \quad (8)$$

$$[N]_{(FeNbC)_l} \rightarrow {}^1/_2N_2 \quad (9)$$

where $[N]_{Nb_s}$ denotes nitrogen in solid solution in Nb;
$(Fe–C)_l$ denotes the liquid Fe–C bath;
$[N]_{(Fe-Nb-C)_l}$ denotes solution of nitrogen in the liquid Fe–Nb–C bath;
and N_2 denotes gaseous nitrogen released from the bath.

Thermodynamic data recently made available by metallurgists interested in nitrogen in alloy steels, notably Chipman (*6*) and Pehlke and Elliott (*34*), allow a reasonably rigorous evaluation of the extent of Reaction 9 under vacuum fusion conditions and, therefore, of the feasibility of performing vacuum fusion analysis for nitrogen in Nb and many other metals by the iron bath technique.

Possible secondary reactions or interactions such as

$$Nb + C \rightarrow NbC_s \quad (10)$$

where both Nb and C can be in the solid or solution state and

$$[Nb] + [C] = [Nb_xC_y] \quad (11)$$

where $[Nb_xC_y]$ denotes complex formation or association in the solution, are more difficult to evaluate but will be discussed in a qualitative manner in turn.

The treatment which follows has been adapted from the theories developed principally by Chipman (*6*) and Wagner (*46*) and is presented here in terms originally formulated by Chipman in 1955 (*6*).

When a solvent metal 1, in this case iron, contains a number of solute metals (or nonmetals) 2, 3, 4, etc., the activity coefficient of one of these solutes, f_2 for example, can be expressed as a product of factors which represent the effect of each of the other components on component 2. Thus in a solution of mole fractions X_2, X_3, X_4, etc.

$$f_2 = f_2' \times f_2^{(3)} \times f_2^{(4)} \;.\;.\;.$$

where f_2' is the activity coefficient of component 2 in the binary 1–2 system of mole fraction X_2 and $f_2^{(3)}$ is the activity coefficient of component 2 in the ternary system 1–2–3 at mole fractions X_1, X_2, X_3, and so on. Wagner (*46*) has derived an equivalent expression

$$\ln f_2(X_2, X_3, X_4 \;.\;.\;.) = \ln f_2^{\circ} + X_2 \left(\frac{\partial \ln f_2}{\partial X_2}\right)_{T,P} +$$

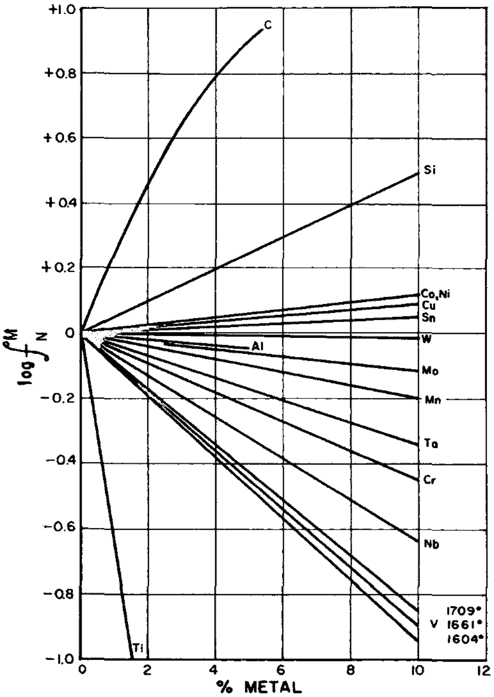


Figure 1. Log activity coefficient of nitrogen vs. per cent metal in iron at 1600° C.

$$X_3\left(\frac{\partial \ln f_3}{\partial X_3}\right)_{T,P} + X_4\left(\frac{\partial \ln f_4}{\partial X_4}\right)_{T,P} + \; . \; . \; . \; . \; .$$

where higher order terms of a Taylor series are neglected. Both expressions are rigorously applicable only at infinite dilution but have proved to be useful first approximations at "low" finite concentrations, possibly up to 10% in many cases.

The partial derivatives are represented by the symbols $\epsilon_2^{(3)}$, etc., defined as

$$\epsilon_2^{(2)} = \left(\frac{\partial \ln f_2}{\partial X_2}\right)_{T,P}; \quad \epsilon_2^{(3)} = \left(\frac{\partial \ln f_2}{\partial X_3}\right)_{T,P} \text{ etc.}$$

and so the equation becomes

$$\ln f_2\,(X_2, X_3, X_4 \; . \; . \; . \; . \; .) = X_{2}\epsilon_2^{(2)} + X_{3}\epsilon_2^{(3)} + X_{4}\epsilon_2^{(4)} + \; . \; . \; .$$

where the term $\ln f_2^\circ$ is made zero by taking $f_2^\circ = 1$ at infinite dilution. Using common logarithms, and expressing concentrations in weight per cent,

$$\log f_2(\%2, \%3, \%4 \; . \; .) = e_2^{(2)}(\%2) + e_2^{(3)}(\%3) + e_2^{(4)}(\%4) + \; . \; . \; . = \log f_2' + \log f_2^{(3)} + \log f_2^{(4)} + \; . \; . \; . \qquad (12)$$

$$\text{where } e_2^{(3)} = \left(\frac{\partial \log f_2}{\partial\,(\%3)}\right)_{T,P} \text{ etc.}$$

Wagner has also shown that

$$e_i^{(j)} = \left(\frac{0.2425}{\mathrm{M}_j}\right) \epsilon_i^{(j)}$$

where M_j is the atomic weight of component j.

The equilibrium between nitrogen in the gaseous state and in a solution of pure molten iron can be written as

$$[\mathrm{N}]_{\mathrm{Fe}} \rightleftarrows {}^1/_2\mathrm{N}_2 \qquad (13)$$

and the equilibrium constant for this reaction is

$$K = \frac{P_{\mathrm{N}_2}{}^{1/2}}{(\%\mathrm{N})\,f_{\mathrm{N}}'} \qquad (14)$$

where f_N' is the activity coefficient of nitrogen in the melt. If, as above, the reference state is taken as the infinitely dilute solution of nitrogen in pure iron, then in this state $f_N' = 1$. Further, if K remains constant over all concentrations and pressures of interest—i.e., if the system obeys Sieverts' law, then f_N' must be unity over the concentration range of interest. Experimentally, it has been found that this system does obey Sieverts' law, and so

$$K = \frac{P_{\mathrm{N}_2}{}^{1/2}}{(\%\mathrm{N})} \qquad (15)$$

If additional components are introduced into the system, the equilibrium

$$[\mathrm{N}]_{\mathrm{Fe,M_1,M_2}} \; . \; . \; . \; . \rightleftarrows {}^1/_2\mathrm{N}_2$$

applies where $[\mathrm{N}]_{\mathrm{Fe,M_1,M_2}}$ . . denotes nitrogen in solution in the multicomponent melt. This is the basic Equation 9 previously proposed for the vacuum fusion process. The activity coefficient of nitrogen in this multicomponent melt may be greater or less than unity, reflecting the effect of the additional components on the activity and the resulting partial pressure of nitrogen. Therefore,

$$K = \frac{P_{\mathrm{N}_2}{}^{1/2}}{(\%\mathrm{N})\,f_{\mathrm{N}}} \text{ or } K' = Kf_{\mathrm{N}} = \frac{P_{\mathrm{N}_2}{}^{1/2}}{\%\,\mathrm{N}} \qquad (16)$$

where f_N is the activity coefficient of nitrogen in the multicomponent melt of specified composition. Again it is assumed that the multicomponent system follows Sieverts' law—this has also been found to be experimentally valid for all systems studied to date. If f_N can be calculated for the multicomponent system (in the case of vacuum fusion conditions, f_N for the system Fe–N–$\mathrm{M_1}$–$\mathrm{M_2}$, etc., where $\mathrm{M_1}$ is the metal to be analyzed and $\mathrm{M_2}$ is usually carbon) then K' can be calculated and, therefore, the concentration of nitrogen in the molten bath at any given equilibrium nitrogen pressure can be evaluated.

The activity coefficient, f_N is calculated by Equation 12 in the form

$$\log f_{\mathrm{N}} = e_{\mathrm{N}}^{\mathrm{M_1}}\,(\%\mathrm{M_1}) + e_{\mathrm{N}}^{\mathrm{C}}\,(\%\mathrm{C}) = \log f_{\mathrm{N}}^{\mathrm{M_1}} + \log f_{\mathrm{N}}^{\mathrm{C}}$$

where $\%\mathrm{M_1}$ is the concentration of metal in the iron bath and %C is the concentration of carbon in the bath, taken as 5.3%, the saturation value at 1600° C. (*18*).

The most comprehensive collection of e_N^M values available is that presented by Pehlke and Elliott (*34*) for various Fe–N–M systems at 1600° C. The data were obtained by measurement of Sieverts' law behavior for various concentrations of metals (and carbon, oxygen, sulfur) in molten iron. A graphical summary of log f_N *vs.* % metal in iron at 1600° C. is presented in Figure 1. The data for vanadium are from a report by El Tayeb and Parlee (*11*). Although the curves are not always linear, they have been idealized into linear form for the purposes of this report. The error involved in this adjustment is insignificant for the present purposes. Temperature coefficients of solubility of nitrogen in the various Fe–N–M systems, and thus temperature coefficients of f_N^M, have also been measured by Pehlke and Elliott (*34*) over the range of 1550° to 1775° C. and so K' may be evaluated over this temperature range.

It should be noted at this point, that the above theory (Equation 12) has been validated for the Fe–Ni–Cr–C–N system by Langenberg (*26*) by calculation of equilibrium concentrations of nitrogen in various melts and confirmation of the calculations by measurement of the nitrogen contents of the rapidly solidified alloys. Kashyap and Parlee (*22*) also obtained reasonable agreement between experimental and calculated nitrogen contents for a series of Fe–Mo–V alloys. No validation for systems containing the very high carbon and low nitrogen contents treated in the present report has been reported.

Using Equations 12, 15, and 16 and the data in Figure 1, equilibrium nitrogen concentrations in various molten baths have been calculated for various equilibrium nitrogen pressures. The results are presented in Table IV. For convenience, all values have been calculated for 1600° C. unless otherwise indicated.

The assessment of a realistic equilibrium pressure of nitrogen under vacuum fusion conditions is rather difficult. In this laboratory, it is usually found that nitrogen constitutes about 10% of the blank value for both empty crucibles and crucibles containing iron and the metal analyzed. If the total pressure in the furnace is 10^{-5} mm., then an equilibrium partial pressure of nitrogen of 10^{-6} mm. is not an unreasonable assumption. For reasons to be discussed in a following section, the actual effective pressure near the gas–metal interface may be much larger than that indicated by gross pressure measurement. For purposes of complete development of the foregoing arguments, it is desirable to assume equilibrium pressures of the order of 10^{-5} to 10^{-6} mm. and reserve further discussion of this point to the section dealing with practical applications.

Table IV. Equilibrium Nitrogen Contents in Molten Fe and Molten Fe–M Solutions at Various Nitrogen Pressures

(All at 1600° C. unless otherwise indicated)

System	Metal in bath, %	C in bath, %	Equil. N_2 content in bath (p.p.m.) at indicated pressure 10^{-4} mm.	10^{-5} mm.	10^{-6} mm.
Fe	. . .	0	0.152	0.0479	0.0152
		5.3	0.0170	0.00537	0.00170
Fe–Ni	5	0	0.13	0.043	0.013
	5	5.3	0.015	0.0046	0.0015
	10	0	0.12	0.036	0.012
	10	5.3	0.013	0.0041	0.0013
Fe–Al	5	0	0.17	0.054	0.017
	5	5.3	0.019	0.0060	0.0019
	10	0	0.19	0.061	0.019
	10	5.3	0.021	0.0068	0.0021
Fe–Cr	5	0	0.26	0.083	0.026
	5	5.3	0.029[a]	0.0091[a]	0.0029[a]
	5	5.3	0.027[b]	0.0087[b]	0.0027[b]
	10	0	0.44	0.14	0.044
	10	5.3	0.045[a]	0.014[a]	0.0045[a]
	10	5.3	0.044[b]	0.014[b]	0.0044[b]
Fe–Mo	5	0	0.18	0.055	0.018
	5	5.3	0.019	0.0061	0.0019
	10	0	0.20	0.062	0.020
	10	5.3	0.022	0.0069	0.0022
Fe–W	5	0	0.15	0.049	0.015
	5	5.3	0.017	0.0055	0.0017
	10	0	0.16	0.050	0.016
	10	5.3	0.018	0.0056	0.0018
Fe–V	5	0	0.48	0.15	0.048
	5	5.3	0.050	0.016	0.0050
	10	0	1.32	0.42	0.132
	10	5.3	0.15	0.048	0.015
	1700° C. { 10	0	1.07	0.34	0.107
	10	5.3	0.12	0.038	0.012
Fe–Nb	5	0	0.33	0.10	0.033
	5	5.3	0.035	0.011	0.0035
	10	0	0.70	0.22	0.070
	10	5.3	0.073	0.023	0.0073
Fe–Ta	5	0	0.23	0.074	0.023
	5	5.3	0.023	0.0073	0.0023
	10	0	0.34	0.11	0.034
	10	5.3	0.037	0.012	0.0037
Fe–Ti	2	0	2.8%	0.87%	0.28%
	2	5.3	0.32	0.10	0.032
	5	5.3	23	7.4	2.3

[a] Uncorrected for chromium–carbon interaction.

[b] Corrected for chromium–carbon interaction.

Note: $e_C^{Mo} = -0.002$, e_C^{Ni}, e_C^{W} are unknown.
e_C^{V}, e_C^{Nb}, e_C^{Ta}, e_C^{Ti} are unknown but are probably large.

A specific example, the analysis of nitrogen in tungsten, follows to illustrate the use of the data in Table IV. If a 1-gram sample is dissolved in a 10-gram iron–carbon bath, the equilibrium nitrogen concentration remaining in the bath, after all of the excess nitrogen has been pumped into the analytical system, should be (at 10^{-6} mm. of N_2) 0.0018 p.p.m. or 0.02 μg. Since this amount is negligible compared to the initial nitrogen content of the sample, for all practical purposes the yield of nitrogen from the sample is 100%. Even at an equilibrium pressure of 10^{-4} mm., only 0.2 μg. of nitrogen should remain in the bath, which value is still satisfactory for most purposes. Note, however, the case for titanium, where for only 5% Ti in the bath, the equilibrium nitrogen content should be 230 μg. at 10^{-4} mm. and 23 μg. at 10^{-6} mm. The effect of carbide formation will probably modify this value in a favorable direction—this will be discussed in a following section.

The following points can be deduced from the data in Table IV and Figure 1:

(a) The final equilibrium pressure of nitrogen over the bath has an important effect on nitrogen recovery. For every 100-fold decrease in pressure, the equilibrium nitrogen content of the bath is reduced by a factor of 10. For each particular system, there is a unique equilibrium pressure required to obtain a practical quantitative recovery of nitrogen. This pressure is higher for high nitrogen contents in the sample than for low contents. In general, the more reactive metals (Ti, Nb, Ta, etc.) require lower pressures than do the less reactive metals (Cr, Mo, W, Ni, etc.).

(b) In general, the difficulty of removal of nitrogen increases with increasing affinity of the solute metal for nitrogen. This relationship is modified, however, by the affinity of the solute metal for iron. Note in Figure 1, that chromium lies between tantalum and niobium whereas the partial molar free energy of solution of nitrogen in chromium (*13*) is much less than that for nitrogen in tantalum (*11*). Niobium and tantalum have a high affinity for iron and therefore, their affinities for nitrogen in the iron solution are decreased, whereas chromium interacts very little, if at all, with iron, and its affinity for nitrogen in the iron solution is not largely different from what it would be in pure chromium. Differences in affinities for iron are reflected by the activity coefficients of these elements in molten iron; 0.2 for Nb and Ta and 1 for Cr (*28*). Note also in Figure 1, that $\log f_N^{Si}$ is positive; since the affinity of silicon for nitrogen is rather high ($\Delta F_{Si_3N_4} = -29$ kcal./mole at 1600° C.), the positive value of $\log f_N^{Si}$ indicates a strong interaction between iron and silicon. This conclusion is in agreement with the measured activity coefficient of 0.0072 for silicon in iron (*28*). In general, then, there is some agreement with the conclusions of Sloman *et al.* (*21*), that dissolution of the metal in the iron bath is important in the release of nitrogen but this conclusion is reached in an entirely different manner and is only one of the factors to be considered.

(c) The effect of carbon on nitrogen solubility in molten iron is quite large. For most of the elements considered in Table IV, only this direct effect of carbon is considered—i.e., $\log f_N^C$ and $\log f_N^M$ are added linearly and any secondary interaction effects are ignored. For some of the elements considered, the interaction coefficients between carbon and the solute metal are known ($e_C^{Mo} = -0.002$, $e_C^{Cr} = -0.002$, $e_C^{Mn} = -0.002$, $e_c^{Si} = +0.008$) and can be used to calculate the activity of the metal in the presence of carbon. This has been done for Cr in Table IV; the effect is negligible. The effect has been justifiably ignored for Mo because e_C^{Mo} is quite small. The effect has also been ignored for W and Ni, since the carbon interaction coefficients for these elements, while unknown, are probably also very small. For those elements which have a high affinity for carbon (V, Nb, Ta, Ti in order of increasing affinity), the effect of carbon must be very large. Approximate thermodynamic calculations indicate that all of these elements will form some solid MC carbides in the molten bath under vacuum fusion conditions. The solubility of the carbide, and thus the effective concentration of metal in the bath at equilibrium, will be much less than the nominal concentration of the metal and will depend on the free energies of formation of the carbides and the free energies of solution of the metals in the molten iron bath. Available thermodynamic data do not allow a very exact calculation of the solubilities of these carbides but it is indicated that the concentration of Nb in equilibrium with the solid carbide in molten iron–carbon at 1600° C. should be between 0.1 and 1%. (This has been roughly confirmed in this laboratory by equilibrating melts of 10% Nb in Fe saturated with C for 15 minutes—the usual time of a vacuum fusion analysis. The amount of Nb left in solution in the matrix, after rapid quenching of the melt, was determined by electron microprobe analysis. Values of 0.8 to 1% Nb were found in the matrix.) For Ti, the formation of carbide should be essentially 100% complete and therefore the calculations for Nb and Ti in Table IV should be modified to reflect the true equilibrium concentrations of metal in the bath. Similar considerations apply to V, Ta, Zr, etc. In the case of Ti where the effective concentration of the metal in the bath is essentially zero, the equilibrium nitrogen pressures should be virtually the same as for a pure Fe–C–N system (note that in a graphite crucible, the supply of carbon is essentially inexhaustible and so saturation is always achieved at equilibrium). Since practical experience has indicated that the release of nitrogen from the Fe–N–Ti–C system at 1600° C. is rarely, if ever, complete, the above conclusions are seriously at odds with practice.

It has been proposed (*45*) that the formation of MC_xN_{1-x} (*38*) compounds either slows or prevents the release of nitrogen from this system. Portnoi and Levinskii (*36*, *37*) have recently measured equilibrium constants of reactions of the type

$$MN + C \rightleftarrows MC + {}^1/_2\,N_2$$

for titanium, zirconium, and hafnium carbonitrides. In each case, the nitride and carbide are isomorphous and form complete series of solid solutions. From their data for the hafnium–carbon–nitrogen system at 1700° C., the equilibrium nitrogen content of Hf(C,N) is calculated to be $5 \times 10^{-4}\%$ at an equilibrium pressure of 10^{-5} mm. of nitrogen. This is equivalent to about 5 μg. of nitrogen in the carbide which would be formed from 1 gram of hafnium dissolved in a typical vacuum fusion bath. The corresponding value at 1930° C., at the same nitrogen pressure, would be about 3 μg. of nitrogen; increasing temperature promotes the release of nitrogen from the compound. Thus, the equilibrium nitrogen contents of such compounds range from barely significant to negligible, depending on the nitrogen contents of the samples to be analyzed.

To summarize, carbon does play an important role in the release of nitrogen from all of these systems. For those elements which do not form carbides in the melt, the presence of carbon assists in the process by reducing the solubility of nitrogen in the melt. For those elements which form carbides the effect is twofold: (a) reduction of solubility of nitrogen in the melt and (b) reduction of concentration of the reactive metal in the melt which further lowers the solubility and equilibrium concentration of nitrogen. It is interesting to note that for the elements Ni (and probably Co, Mn, etc.) the presence of carbon is not at all necessary to obtain complete release of nitrogen even at equilibrium pressures as high as 10^{-4} mm. If the equilibrium pressure can truly be reduced to 10^{-6} mm. (10^{-5} mm. for Al) then the presence of carbon should not be necessary for the successful analysis of Cr, V, Nb, Ta, Mo, W, and Al. Dilution with ron seems to be the only requisite for essentially complete release of nitrogen. It is planned to attempt to verify this conclusion experimentally; the results should be extremely interesting.

Finally, the results of this treatment should be compared with those of the treatment of Sloman (*42*). In agreement with Sloman, the presence of carbon and, therefore, the occurrence of carbide formation is probably not important for Mo and V. In disagreement with Sloman, the presence of carbon and the formation of carbides must be essential for successful analysis of Ti (and probably also Zr and Th). Again, these conclusions are based on a different mechanism from that proposed by Sloman. While the present treatment as yet has had no experimental verification, it is believed that it does more realistically represent the actual processes occurring than that based on decomposition of nitrides which are not present at any point in the process. For those special cases when nitrides are initially present the mechanism of their decomposition must first be considered, according to Sloman's treatment. The final release of nitrogen from the Fe–C–N–M system should then be treated according to the above presentation.

All of the above calculations have been performed for vacuum fusion analysis using an iron–carbon bath but no consideration has been given to the use of a platinum–carbon bath, which medium has become increasingly popular during the last decade. Those experimental metallurgists who are interested in interactions in molten iron are motivated by the very practical need to understand variables in steel making processes. The measurement of similar interaction coefficients for nitrogen in Pt–N–M–C systems is of no industrial significance and so similar calculations for vacuum fusion analysis using the platinum bath are not feasible at this time. (f_N^M values for molten platinum, or any other system, could be calculated if the following individual thermodynamic values were available: $\overline{\Delta F}$ for solution of M in Pt, C in Pt, N_2 in M, and C in M. A few data exist for the last two parameters, but data for the first three are virtually nonexistent.) Certain qualitative speculations, based on meager information in the literature can be made. As far as can be ascertained, the solubility of nitrogen in molten platinum is vanishingly small in comparison to that for iron. The solubility of carbon in molten platinum is of the order of 1.2 to 1.4% *(18)*. Based on observations of vacuum fusion analysts, the dissolution of metals such as Ti and Zr in Pt is highly exothermic indicating strong interactions between these metals and platinum. Two of these effects, low solubility of nitrogen and strong interaction of the metal with platinum, would tend to promote release of nitrogen from the platinum bath. Beyond this, no reasonable speculations can be made. Sieverts'-law studies of platinum–metal–carbon–nitrogen systems would be very useful in understanding the advantages and disadvantages of the platinum bath for nitrogen analysis.

KINETICS. The foregoing considerations are thermodynamic in nature; the feasibility of vacuum fusion analysis for nitrogen has been evaluated only in terms of final equilibrium conditions existing in molten F–M–C–N systems. Since the rate of approach to equilibrium is also of considerable importance, it is in order to consider the critical rate processes involved. They are as follows:

(a) dissolution of the metal specimen in the bath to form a completely homogeneous solution;

(b) formation of the carbide, for those metals where carbide formation is of importance; the reaction could be represented by any or all of the following:

$$[\mathrm{M}]_{\mathrm{Fe}} + [\mathrm{C}]_{\mathrm{Fe}} \rightarrow \mathrm{MC}$$

$$\text{or}\quad \mathrm{M}_{\mathrm{solid}} + [\mathrm{C}]_{\mathrm{Fe}} \rightarrow \mathrm{MC}$$

$$\text{or}\quad \mathrm{M}_{\mathrm{solid}} + \mathrm{C}_{\mathrm{solid}} \rightarrow \mathrm{MC}$$

(c) possible formation of carbonitrides

$$\mathrm{MC} + [\mathrm{N}]_{\mathrm{FeC}} \rightarrow \mathrm{MC}_x\mathrm{N}_{1-x}$$

(d) dissolution of carbon from the crucible to replace that consumed by carbide formation

$$\mathrm{C}_{\mathrm{solid}} \rightarrow [\mathrm{C}]_{\mathrm{Fe}}$$

(e) release of nitrogen from the solution in which equilibrium with respect to solution and carbide formation has been achieved

$$[\mathrm{N}]_{\mathrm{Fe,M,C}} \rightarrow {}^1/_2\,\mathrm{N}_2$$

For those metals which form carbides in the melt (Ti, Zr, Nb, Ta, etc.), processes (a) and (b) above will occur simultaneously and the limiting factor may be (d), dissolution and diffusion of carbon to the reaction site. Complete equilibrium will only be achieved when the precipitated carbide is in equilibrium with the saturated solution (saturated with respect to carbon and carbide). As long as carbon content remains below the saturation value, the calculations in Table IV are invalid since $f_N{}^C$ has been calculated for 5.3% C, the saturation value.

For those metals which do not form carbides in the melt, only processes (a) and (b) need be considered. It must be emphasized that in order for the conditions in Table IV to be realized, complete homogeneity in the melt must be achieved. Local volumes high in concentration of a metal such as chromium will not release nitrogen to the extent calculated for the equilibrium conditions in Table IV.

The complexity of any theroetical quantitative evaluation of the rates of processes (a), (b), (c), and (d) is immediately apparent. It is possible, however, to make some quantitative evaluation of process (e) and to decide qualitatively which of the above five processes are most likely to be rate limiting.

Pehlke and Elliott *(35)* have measured rates of absorption and evolution of nitrogen from a number of molten Fe–M systems. It is observed experimentally that the absorption (and evolution) process is a first-order reaction, with the rate being proportional to the concentration of nitrogen in the bath at any time t. Thus,

$$\frac{d\,[\log C_e - C]}{\mathrm{dt}} = -k'A$$

where C_e = final equilibrium concentration
C = concentration at time t
k' = apparent rate constant
A = surface area of the melt

Integrating between the limits C_0 at time t_0 and C at time t

$$\log \frac{C_e - C_0}{C_e - C} = k'A\ (t - t_0)$$

Using Pehlke and Elliott's value of 1.8×10^{-3} cm.$^{-2}$ sec.$^{-1}$ at 1600° C. for k' for pure iron, 2 cm.^{2} for A (crucible diameter 1.6 cm.), a calculated equilibrium nitrogen content of 0.0054 p.p.m. (C_e from Table IV at 10^{-5} mm. equilibrium pressure) the equation reduces to

$$\log \frac{C_0}{0.5\,C_0} = 3.6 \times 10^{-3} t_{1/2} \qquad (13)$$

where $t_0 = 0$ and $t_{1/2}$ is the time to reduce the nitrogen content to one half its initial value.

Solving for $t_{1/2}$ gives a half time of 84 seconds. For an initial nitrogen content of 50 p.p.m. in the bath (1-gram sample, 500 p.p.m., dissolved in 10-gram bath), 12.5 minutes would be required to reduce the nitrogen content to 0.09 p.p.m. or 0.2% of its original value. Note that since C_e is always very small in comparison to C_0, $t_{1/2}$ is essentially independent of final equilibrium pressure in the range of 10^{-4} to 10^{-6} mm. The effects of third components (O, S, Al, Cr, Nb, Ni, Si, and W) on the rate constant have also been measured by Pehlke and Elliott. The effects of the metallic elements are rather small; for 10% Nb in the bath, $t_{1/2}$ under the same conditions listed above is 168 seconds and the time to degas from 50 to 0.09 p.p.m. is about 25 minutes. The effects of the other metals studied are all less than that for niobium. Relatively small amounts of both oxygen and sulfur decrease the rate of outgassing quite markedly; 400 p.p.m. of oxygen decreases k' by about a factor of three. This would be of little significance under vacuum fusion conditions, however, because equilibrium oxygen concentrations would be vanishingly small. The influence of carbon was not determined in this study and so the effect of this very important element on outgassing under vacuum fusion conditions cannot be theoretically assessed. In those few cases where nitrogen analysis by vacuum fusion has been shown to be successful, the degassing times are of the same order (10 to 30 minutes) as those calculated above and so it can be assumed that the effect of carbon is no greater than that of niobium.

It is pertinent to note that stirring effects from induction heating (frequency not stated) used by Pehlke and Elliott were observed to influence the rate constant, but the effects are believed to be rather small; a factor of 5 decrease in current density in the melt, achieved by placing a molybdenum susceptor around the crucible, reduced k' by only 30%. This effect may only be related to the change in surface area of the melt due to stirring.

One further point of importance is that all rate measurements were done under conditions such that bubble formation in the melt was prevented. Therefore, all mass transfer was by diffusion to, and through the interface between the metal and the gas phase, and so was slower than would be the case were bubbles allowed to form in the melt. Since bubble formation under practical conditions of vacuum fusion analysis is highly probable, degassing times should be shorter than the above calculated values.

Since the criterion of a successful vacuum fusion analysis is that the nitrogen be quantitatively released in less than 20 ± 10 minutes, the transfer of nitrogen from the melt to the gas phase could be the rate limiting process in a successful analysis. When much longer times are required to recover all of the nitrogen (or if it is not recoverable at all) it would appear that any or all of processes (a), (b), (c), and (d) are rate limiting.

It follows from these considerations that efforts to promote stirring in the melt by the use of low frequency (30 kc. *vs.* the normally used 500 kc.) induction heating are fruitless insofar as increasing the rate of gas evolution is concerned. If it is desirable to speed up the mass transfer of nitrogen from the melt to the gas phase, investigations of large diameter crucibles to expose a greater surface area of the melt to the vacuum system would be much more to the point.

Since nitrogen transfer does not appear to be seriously rate limiting, it is logical that research efforts should be directed to determining which of the remaining four processes, sample dissolution, carbide formation, formation of $\mathrm{MC}_x\mathrm{N}_{1-x}$ compounds, or carbon saturation, are limiting. Conditions should then be altered to accelerate or eliminate the rate limiting process or processes.

Two methods of accelerating complete dissolution are stirring by the use of low frequency induction heating and maintaining the bath in a fluid rather than a pasty or solid state. McDonald, Fagel, and Balis (*30*) attempted to assess the effects of stirring at 30 kc. *vs.* 500 kc. for an iron bath used for the analysis of zirconium for oxygen content. The experiment was complicated by solidification of the bath due to graphite precipitation, and no clear cut conclusion was obtained. It was, however, demonstrated that solidification of the melt due to prolonged heating occurred frequently and that this was the main deterrent to successful analysis in this case. Solidification was prevented by minimizing the time that the melt was held at operating temperature and it was suggested that the occasional addition of more iron to the melt to reduce carbon saturation and promote fluidity could also be helpful. Further work on the effect of stirring on the rate of solution is still necessary.

For those metals which form carbides in the melt (Ti, Zr, V, Nb, Ta, etc.), solution of the metal and carbide precipitation probably occur simultaneously. The attainment of thermodynamic equilibrium may be retarded or even prevented by complete coverage of the surface of the sample by a protective carbide layer which could effectively prevent further dissolution and/or carbide formation at a sensible rate and/or slow solution of carbon from the crucible walls and diffusion of this carbon to the reaction area to replenish that consumed by carbide formation.

The first situation could be alleviated by reacting the sample in a medium containing a lower initial concentration of carbon. This could be achieved either by diluting the melt with iron at the same time the sample is added (iron flux or iron capsule technique) or by the use of a melt such as platinum, which dissolves much less carbon than does iron. The second process, equilibrium solution of carbon, could be accelerated by low frequency induction stirring.

In regard to the possible retention of nitrogen in the bath due to the formation of carbonitrides, thermodynamic calculations given in a preceding section indicate that the amounts of nitrogen retained by such compounds at equilibrium should be quite small. If the compounds were formed during initial carbide formation, however, subsequent release of nitrogen by solid state diffusion through the carbide would be a very slow process and final equilibrium would probably not be attained in practical periods of time. Carbonitride formation could, therefore, be a major deterrent to successful analysis of the strong carbide formers (Ti, Zr, Hf, V, Nb, and Ta) for nitrogen by conventional vacuum fusion.

GETTERING EFFECTS. During initial outgassing of the crucible assembly and metal bath, and subsequent analysis of the metal in question, varying amounts of carbon, bath metal, and sample metal are evaporated from the crucible and condensed in the cooler parts of the furnace tube. Significant amounts of nitrogen (and other gases), following approximately the same path as these evaporating elements, may be irreversibly absorbed by several possible gettering reactions, causing the method to give low analytical results.

The following factors are of importance in considering the causes, effects, and means of minimization of gettering

(a) vapor pressure and rates of vaporization of carbon and metals at operating temperatures
(b) reduction of vapor pressure of metals by interaction with carbon, by the use of a diluent metal, or by the increase of ambient pressure in the system
(c) nature and rate of gettering reactions and the effect of pumping speed
(d) deactivation of gettering surfaces.

Not all of the above factors are amenable to exact theoretical evaluation but qualitative deductions based on physical principles can be of considerable value to the practicing analyst. Brief discussions of each of the above factors follow.

(a) Vapor Pressures and Rates of vaporization of Carbon and Metals. Estimates of vapor pressures and rates of vaporization of various metals of interest to this report are presented in Table V. The values have been derived from data presented by Dushman (*9*). To reflect practical vacuum fusion conditions the data have been calculated at temperatures of 1600, 1900, and 2400° C. The calculations show that appreciable amounts of many of the metals listed can volatilize in the periods of time used for vacuum fusion analyses. Considerable evaporation of carbon, about 1.3 grams/20 minutes from the inner surfaces of an average crucible at 2400° C., can also occur under conditions used for initial outgassing of the furnace assembly. Fortunately, several conditions commonly used in the vacuum

Table V. Vapor Pressures and Rates of Vaporization of Carbon and Metals at Various Temperatures (9)

Metal	M. p., °C.	Vapor pressure (microns)			Rate of vaporization (gram/2 cm.²/15 min.)[a]		
		1600° C.	1900° C.	2400° C.	1600° C.	1900° C.	2400° C.
C	...	...	2×10^{-3}	8.5	...	1.8×10^{-5}	5.4×10^{-2}
Al	659	1.7×10^{3}	2.0×10^{4}	...	25	144	...
Ti	1812	2.4	1.5×10^{2}	...	1.8×10^{-2}	1.1	...
Zr	1852	2.1×10^{-4}	2.8×10^{-2}	...	1.9×10^{-6}	3.1×10^{-4}	...
Th	1827	2.2×10^{-3}	2.7×10^{-1}	...	2.1×10^{-5}	4.1×10^{-2}	...
V	1857	2.3×10^{-1}	2.1×10^{1}	...	2.2×10^{-3}	1.8×10^{-1}	24
Nb	2450	4.0×10^{-8}	5.6×10^{-5}	1.7×10^{-1}	...	9.0×10^{-7}	1.9×10^{-3}
Ta	2997	...	3.5×10^{-6}	1.0×10^{-2}	...	6.3×10^{-7}	1.4×10^{-4}
Cr	1903	2.0×10^{2}	5.4×10^{3}	...	1.7	43	...
Mo	2577	1.3×10^{-5}	2.8×10^{-3}	1.2	1.5×10^{-7}	3.3×10^{-5}	1.3×10^{-2}
W	3380	...	1.3×10^{-7}	1.8×10^{-3}	...	1.3×10^{-8}	2.5×10^{-5}
Fe	1539	6.6×10^{1}	1.7×10^{3}	6.4×10^{4}	6.3×10^{-1}	14	...
Ni	1452	4.2×10^{1}	1.3×10^{3}	6.0×10^{4}	3.1×10^{-1}	13	...
Pt	1770	1.0×10^{-2}	1.0	2×10^{2}	1.7×10^{-4}	1.1×10^{-2}	9.0×10^{-1}

[a] Exposed surface area of melt in an average vacuum fusion crucible is about 2 cm.²

Table VI. Vapor Pressure and Vaporization Rate Lowering by Solution in Iron Bath and Carbon–Metal Interactions

(All values at 1600° C. unless otherwise indicated)

Metal	p_M (microns)	Activity coeff. of metal in Fe (*8*)	p_M (microns) for 10% metal in Fe[a]	Approximate vaporization rate (gram/2 cm.^{2}/15 min.)[a]	Nature and effect of carbon–metal interaction
Cr	2.0×10^2	1.0	19	3.4×10^{-1}	Cr–C complex formation. $p_{Cr} = 2.7$ microns
Mo	1.3×10^{-5}	1.0	2.1×10^{-6}	4.9×10^{-8}	Mo–C complex formation. $p_{Mo} = 1.4 \times 10^{-6}$
W	1.3×10^{-7} (1900° C.)	1.0	3.5×10^{-8} (1900° C.)	2.2×10^{-9}	W–C complex formation, similar to Mo
Ti	2.4	0.05	1.0×10^{-2}	...	TiC formation. p_{Ti} becomes very small
Zr	2.1×10^{-4}	0.0076	2.4×10^{-7}	...	ZrC formation. p_{Zr} becomes very small
V	2.3×10^{-1}	0.12	2.5×10^{-3}	...	VC formation probable
Nb	4.0×10^{-8}	0.2	1.2×10^{-9}	...	NbC formation. p_{Nb} becomes very small
Ta	Activity coefficient not known.		Behavior will be same as for Nb.		

[a] Assuming no interaction with carbon.

fusion process serve to lower the vapor pressures of the metals being analyzed. These are discussed in the following section.

(b) Reduction of Vapor Pressure. The two most important methods of reduction of vapor pressure are interaction of the subject metal with carbon and dilution with another metal. Direct interaction with carbon is important only for those metals, such as titanium, which form stable carbides and for those metals, such as iron, which dissolve sufficient amounts of carbon in the molten state to cause an appreciable reduction in metal vapor pressure. Direct reaction with carbon in the absence of any other reaction medium is rarely used in modern vacuum fusion practice; the one important exception is the Walter method for the analysis of oxygen in titanium (*47*).

The degree of vapor pressure and consequent vaporization rate lowering which can be achieved by dilution with a second metal are indicated by the data given in Table VI. The data were obtained by application of Raoult's law and activity coefficients for various metals in molten iron obtained from the literature (*8*). The effects of carbon in solution in the iron with respect to further reduction of the activity coefficient by metal–carbon interaction or complex formation, or essentially complete removal of the metal from solution by solid carbide formation, cause a further lowering of vapor pressure over and above that based on simple Raoult's law calculations. The nature of such carbon–metal interactions and their significance are indicated in Table VI for those elements for which such information is available. For those metals exhibiting only weak interaction with carbon, approximate values of vaporization rates are also presented. Of the metals of interest to this report, only chromium should have vapor pressures and vaporization rates sufficiently high to form significant amounts of surface active films under practical vacuum fusion conditions. Since iron will still have an appreciable vapor pressure (of the order of 40 microns) under practical conditions, the formation and effects of films of this bath metal, along with carbon films formed during crucible outgassing, may also be significant.

A major criticism of the foregoing calculations is that the instantaneous achievement of homogeneous solution, carbon–metal interaction, and carbide formation is assumed. That is, the calculations are for final equilibrium conditions only. Practical considerations indicate that when a metal, such as titanium, is dropped into a molten iron bath, the pure metal will exist as such for a finite length of time at temperatures up to 1600° C. Solution of the metal, over a finite length of time will then occur. From the time that the metal contacts the surface of the bath until final equilibrium with respect to homogeneous solution and carbide formation is achieved, vapor pressures will be greater than those indicated in Table VI. Quantitative evaluation of such effects is virtually impossible. It is important to note, however, that dissolution can be hastened, and these nonequilibrium effects minimized, by the use of the so-called flux technique, wherein the sample is wrapped in iron (in platinum for platinum bath) prior to introducing it into the molten bath (*14*).

A third method of decreasing the rate of vaporization of the graphite components and metals in question is to increase the ambient pressure in the furnace tube by the introduction of an inert gas such as argon or helium. Calculation of rates of vaporization in the presence of an inert gas is a complicated problem which apparently has not been completely solved. Seybolt and Burke (*39*) present a short treatment of the problem and cite the decrease in vaporization rate of magnesium in argon as an example; the rate of vaporization of magnesium in vaccum at 650° C. is 11 mg./cm.^{2}/sec. but with only 1 mm. of argon present, the rate is reduced to 0.07 mg./cm.^{2}/sec. The amount of decrease is a complex function of temperature, inert gas pressure, and the ratios of the densities and atomic weights of the metal and the inert gas. Argon is preferable to helium because of the greater atomic weight and density of the former gas.

Vaporization rate suppression at inert gas pressures of a few millimeters has not yet been applied in practical vacuum fusion analysis. Suppression of carbon vaporization by the use of a full atmosphere of argon during initial outgassing of graphite crucibles is practiced in some laboratories (*15*). The use of a full atmosphere of argon during fusion is the basis of the well known inert gas fusion method. Higher temperatures than those for vacuum fusion are commonly used in this method and adverse effects due to gettering appear to be rare, but no definitive study has yet been made to validate these apparent advantages.

The use of controlled overpressures of inert gas would complicate the analysis of the evolved gases in the conventional vacuum fusion method, but application of specific methods of gas analysis such as gas chromatography or mass spectrometry would overcome this complication. Further study of this method of minimizing gettering effects is warranted.

(c) Nature of Gettering Reactions. Under the conditions of vacuum fusion analysis, two major types of gettering and combinations thereof are possible; bulk gettering or reaction of gases with previously deposited active metallic films, and flash gettering wherein gases are chemically or physically occluded during condensation of the metal vapor. Both processes may be enhanced by the presence of an electrodeless or glow discharge, a condition which while normally not present in the furnace tube, can occur during a pressure rise accompanying rapid evolution of gases from the bath. A further pertinent property of both processes is that under conditions practically attainable in the furnace tube, they are both irreversible—once occluded, the gases cannot be released by any practical means.

It is currently impossible to assess which of the two gettering reactions preponderates; it is certain that both operate in the presence of significant amounts of metal evaporation. Certain physical conditions will minimize bulk gettering but given the condition that metal vaporization can occur, within the limitations of a practical, conventional, vacuum fusion apparatus, very little can be done to minimize flash gettering. The remainder of this section is, therefore, devoted to a short discussion of bulk gettering with the full realization that minimization or elimination of this type of reaction may only alleviate a part of the problem.

Beach and Guldner (*3*) have shown that no significant losses of nitrogen occur in the presence of either carbon or iron–carbon films. This is to be expected since neither of these materials has a high chemical affinity for nitrogen. The equilibrium solubility of nitrogen in solid iron at the final background pressure in the furnace would be vanishingly small (of the order of 0.001 to 0.01 p.p.m.) at all of the temperatures encountered in the furnace. Conversely, loss of nitrogen to films of reactive metals at the indicated temperatures is highly probable. The degree of loss by such gettering will depend on:

(i) the surface area of the active metal film
(ii) the temperature of the film
(iii) the rate of reaction of nitrogen with the metal
(iv) the time of contact of the nitrogen with the film

A complete evaluation of these four factors is beyond the scope of this review. For a comprehensive discussion of bulk gettering rates and mechanisms, the reader is referred to Chapter 9 of Dushman (*9*). The following qualitative conclusions can, however, be drawn:

(i) The geometric surface area, or the area of the furnace tube covered by the film, can and should be minimized by cooling. If a very sharp temperature drop from the top of the crucible to the immediate surrounding area of the furnace tube is maintained, most of the active deposit will be confined to a small area, thus minimizing the time of contact of the evolving gas with the active film. Unfortunately, the vacuum conditions in the furnace tube (10^{-4} to 10^{-6} mm.) are highly conducive to the production of films of high catalytic surface area; i.e., the films are very rough on a micro scale. No practical means of mitigating this factor are available.

(ii) Contrary to popular opinion, maintaining the films at high temperature will not decrease sorption of the evolved gases; since the process is undoubtedly irreversible even at quite high temperatures, higher temperatures will increase the rate of gettering. Every effort should be made to cool the furnace tube to as low a temperature as possible.

(iii) Many studies of rates of reaction of metals with nitrogen have been made and summaries of data are available (*25*). Qualitative predictions of possible losses of nitrogen by gettering can be made on the basis of these studies. For example, serious loss of nitrogen could be expected due to gettering by films of titanium, zirconium, niobium, and tantalum. Losses to films of chromium, molybdenum, tungsten, iron, nickel platinum, etc., are expected to be minimal.

(iv) Gettering will be minimized by the rapid removal of the evolved gases from the furnace tube. Large-diameter tubing from the furnace coupled to a high speed diffusion pump is highly desirable. Pumping speeds up to 60 liters per second at a few microns pressure are feasible and appropriate.

(d) Deactivation of Gettering Surfaces. The addition of a volatile, inert metal, such as tin to the vacuum fusion bath during analysis of metal samples is widely practiced. The reasons for this use of tin are obscure; one of its proposed functions is to cover the surface of active films with a layer of this inert metal and thus prevent gettering by the underlying reactive metal. McDonald, Fagel, and Balis (*30*) have demonstrated that a 15% loss of carbon monoxide passed through a furnace containing an iron–zirconium melt is eliminated by the addition of tin to the bath. The conclusion in this case is equivocal because the actual presence of a zirconium film in the furnace was not demonstrated. Everett and Thompson (*14*) have shown that tin apparently causes desorption of carbon monoxide from active films of beryllium and may also inhibit the initial gettering reaction.

An alternate function of tin may be simply to increase the ambient pressure over the immediate surface of the melt (the vapor pressure of tin at 1600° C. is 1 mm.) and thereby decrease the vaporization rate of the bath and the metal being analyzed. It is also possible that the high concentration of tin in the vapor phase may decrease flash gettering by simple dilution of the more active metal—e.g., zirconium by the less active metal, tin. Considerable experimental work is still needed to demonstrate the true nature of the function of tin in aiding in complete recovery of gas from the vacuum fusion furnace.

Further mention should be made of the systematic study of gettering under vacuum fusion conditions made by Beach and Guldner (*3*). Known amounts of gases (N_2, CO, H_2) were passed over films evaporated from carbon, molten iron, platinum, platinum–titanium, germanium, nickel, aluminum, and manganese at operating temperatures. Within acceptable error, no significant losses of nitrogen were observed for any of these metals. Standard deviations of recovery of nitrogen were, however, significantly greater in the presence of films of carbon, nickel, aluminum, and manganese than those for the remaining materials. Pure titanium, as an example of a highly reactive metal was not investigated and no evidence was presented that the platinum–titanium mixture actually formed a titanium containing film. Thus, while this study does present a valid method of studying gettering and some useful information for a few metals, much experimental work remains to be done to provide more complete information on this important vacuum fusion parameter.

Current Status: Comparison of Vacuum Fusion Nitrogen Results with Those of Other Methods. Several methods are available for assessment of the accuracy of vacuum fusion methods for nitrogen analysis. The usual procedure is to compare vacuum fusion results with those obtained by another independent method, commonly the wet chemical or Kjeldahl method and less often, isotope dilution or neutron or charged particle activation. Alternatively, samples containing known additions of nitrogen, prepared by gas absorption or nitride addition methods, are used to measure the accuracy of the vacuum fusion method. A representative cross section of such data, gathered and evaluated to assess the current status of the method follows.

Reactive Metals—Ti, Zr, Hf(Th). It is generally accepted that vacuum fusion analyses for nitrogen in these elements are not reliable. This conclusion is based largely on verbal communication among analysts active in the field for there are few, if any, published results to support or deny the validity of the conclusion. Under the conditions of the Walter (*47*) method for oxygen in Ti (graphite crucible plus tin, 1900° C.) no nitrogen is evolved. Booth, Bryant, and Parker (*4*) reported that vacuum fusion analyses for nitrogen in Ti, Zr, and Th were low and erratic in comparison to values obtained by the Kjeldahl method. The approximate conditions were—Pt to metal ratio, 100 to 1 or better. Thorium nitride (Th_3N_4), apparently of questionable purity, was reported to release 80% of its nitrogen under these conditions. Very limited experiments indicated 100% recovery of nitrogen from zirconium doped with nitrogen at 1200° C. but less than 100% recovery when the nitrogen was added at 1500° to 1600° C.

Sloman (*42*) reported successful nitrogen analysis by vacuum fusion for Ti, Zr, and Th (iron bath, 1750° C., <30% metal in bath for Zr; 1800° C., <20% metal in bath for Ti; 1900° C., <20%

metal in bath for Th). Presumably, quantitative analysis of TiN, ZrN, and Th_3N_4 was first demonstrated and then various samples of the metals were analyzed for nitrogen. No data are given for analysis of the thorium by other methods. Two samples of titanium containing known amounts of nitrogen added by arc melting were analyzed but the results are not particularly convincing (0.18% and 0.26% recovered from 0.23% and 0.33% thought to be present). No comparative results are given for analysis of zirconium by other methods.

A very limited amount of data obtained in the author's laboratory on NBS #360 Zircaloy 2 indicates that satisfactory values can be obtained for nitrogen by vacuum fusion, at least on this chip sample. Conditions were as follows: Pt bath–Pt flux, 1900° C., <10% Zr in bath.

Holt and Goodspeed (*19*) recently reported nitrogen values for two zirconium samples obtained by a platinum flux–inert gas fusion method, which were in good agreement with Kjeldahl results (48 *vs.* 45 and 50 *vs.* 54 for IGF and Kjeldahl methods, respectively).

Everett (*13*) reported successful comparisons between vacuum fusion (Pt bath, 1900° C.) and the Kjeldahl method for Zr, but no supporting data have been published in the open literature.

Refractory Metals. (a) V, Nb, Ta. Sloman (*42*) established suitable iron bath conditions for analysis of vanadium for gases (1560° C., <20% V in bath), but no comparative results for nitrogen by other methods were obtained by these authors.

The vacuum fusion method for nitrogen in niobium has been validated by comparison with Kjeldahl values in the laboratories of the Du Pont Experimental Station. Vacuum fusion conditions were as follows: Pt flux, 1900° C., 15-minute extraction. Results on an ASTM round-robin sample (*31*) were as follows: vacuum fusion, average of 10 analyses 106, relative standard deviation 7.5%; Kjeldahl, average of 5 analyses 104, relative standard deviation 3.6%. Turovtseva and Kunin (*45*) reported successful analyses for nitrogen in niobium using an iron bath at 1650° C., <20% Nb in bath, extraction time 15 minutes. Samples with known additions of nitrogen (method of addition not stated) in the range of 29 to 120 p.p.m. were satisfactorily analyzed.

No comparisons of analyses of tantalum for nitrogen by vacuum fusion and other methods have been published.

(b) Cr, Mo, W (U). Mallett (*27*) found that recovery of nitrogen from chromium by vacuum fusion is about 80% complete. Supporting data and conditions were not given.

Mallett and Griffith (*28*) obtained satisfactory vacuum fusion results on nitrogen-doped samples of molybdenum (iron bath) only when the nitrogen was present in the surface layer. However, when the nitrogen was dispersed (nitride or solid solution) low results were obtained by vacuum fusion. Satisfactory results were obtained on similar dispersed samples (analyzed by Kjeldahl method) by hot extraction (carbon crucible, 1800° C., extraction time 30–50 minutes). Samples covering a range of 20 to 1000 p.p.m. were analyzed. Turovtseva and Kunin (*45*) reported satisfactory analysis of doped samples of molybdenum in an iron bath (1650° C., 15 minutes, <30% Mo in bath) in the range of 400 to 600 p.p.m. of nitrogen. Doping was carried out by physical addition of a nitrided Mo powder (5% nitrogen) to a cavity in a high purity Mo cube. Consideration of the results of Mallett and Griffith quoted above indicates that this method of doping may yield fortuitous results. Sloman (*42*) reported complete recovery of nitrogen from a mixture of Mo_2N and nitrogen saturated metal (iron bath, 1560° C., <40% Mo in bath, extraction time 2–3 minutes). Again, this validation may not be legitimate in view of the results of Mallett and Griffith quoted above.

Table VII. Analysis of Steels for Nitrogen Content by Vacuum Fusion, Isotope Dilution, and Kjeldahl Methods

(All values in p.p.m. nitrogen)

		Vacuum Fusion			
Sample	Sample comp.	Masson & Pearce[a]	Fassel[b]	Kjeldahl[g]	Isotope dilution
JM 2	Pure Fe	210–228[c]		224 (n = 3)	229 (n = 6)
NBS 1040[e]	0.3 Mn	29.5 (n = 2)	37	26 (n = 5)	20 (n = 8)
NBS 1042[f]	0.7 Mn	139 (n = 3)	160	151 (n = 4)	159 (n = 4)
APM 80	Pure Fe	21 (n = 3)		30 (n = 3)	32 (n = 4)
BPL 21	Al killed	85 (n = 3)		99 (n = 3)	106 (n = 2)
E	Pure Fe	32 (n = 2)		52[d]	39 (n = 2)
1296-1	3% Si	43, 60 (1650° C.) 76, 78 (2200° C.)		40 (n = 3) 42[d]	80 (n = 2)
RH 836	0.5% Mn 0.25% Si 0.28% Mo	33, 38, 29		40 (n = 3)	27–41 (n = 4) Mean 31

[a] Masson and Pearce vacuum fusion values at 1650° C. unless otherwise indicated.

[b] Vacuum fusion values by Evens and Fassel (*12*) Pt bath, Pt flux, 1850° C., <20% sample in bath.

[c] 2100° C.

[d] Kjeldahl analysis by Staley and Svec (*44*).

[e] NBS value 30 p.p.m.

[f] NBS value 140 p.p.m.

[g] British Standards Institution method.

No data on validation of vacuum fusion nitrogen analysis in tungsten are available.

Booth, Bryant, and Parker (*4*) reported that generally low and scattered nitrogen values are obtained for uranium by vacuum fusion in comparison to the Kjeldahl method. Conditions were as follows; Pt bath, 1820° C., probably <1% U in bath. Sloman (*42*) established that UN is quantitatively decomposed in an iron bath at 1550° C. but no validating data for the analysis of metal were given.

Steels and Related Alloys. Numerous studies have been made on vacuum fusion analyses for nitrogen in a wide variety of steels. No attempt will be made to list and evaluate all of these studies in this report. Rather, a few recent definitive publications will be discussed to present a general idea of the present status of the method and indicate areas worthy of further development.

One of the most comprehensive studies on comparative nitrogen values obtained by the Kjeldahl and vacuum fusion methods for steel is that carried out under the auspices of the British Iron and Steel Research Association (*5*). Approximately seven laboratories performed Kjeldahl analysis on a variety of steels, and vacuum fusion analyses were obtained on the same steels by six different laboratories. Steels containing low and high carbon, aluminum, boron, chromium, cobalt, copper, manganese, molybdenum, nickel, niobium, silicon, titanium, tungsten, vanadium, and zirconium were analyzed. It is not immediately obvious from the report, but apparently all vacuum fusion analyses were performed by simply fusing the samples in the graphite crucible; no bath materials were used. The major conclusion reached was that the results of the two methods were completely equivalent (the chemical method was unsatisfactory for certain silicon- and boron-containing steels). Steels containing TiN could not be satisfactorily analyzed on vacuum fusion instruments employing resistance heating because of temperature limitations. These general conclusions may be somewhat clouded by the following considerations:

(a) chemical methods were not always adequate for 4% Si steels and some boron steels—thus there was not always an adequate method available with which to compare the vacuum fusion values;

(b) no legitimate comparisons were obtainable on zirconium steels because of sample segregation effects;

(c) while statistical analysis of the data indicated agreement between methods, for low nitrogen contents (30 to 100 p.p.m.) spreads of 10 p.p.m. were common. Inspection of individual results indicates that further checking would be desirable at these levels.

Karp, Lewis, and Melnik (*21*) recently reported on cooperative analysis of 3% Si steel and concluded that for certain heat treatment conditions, neither the Kjeldahl nor vacuum fusion methods was satisfactory. Higher, and more concordant, results were obtained by a caustic fusion method. Only one laboratory out of seven reporting vacuum fusion values used a platinum bath technique. Significantly better agreement with the caustic fusion results was obtained by this technique.

A definitive comparative study of the vacuum fusion, isotope dilution and Kjeldahl methods as applied to high purity iron, low carbon manganese, aluminum killed, and high silicon steels has recently been published by Masson and Pearce (*29*). Vacuum fusion was usually done on a conventional apparatus, with no bath and at various temperatures from 1600° C. to 2400° C. Some crucible modifications were made and a very high speed diffusion pump was used for some samples in attempts to raise apparently low values. The results of this study are reproduced in Table VII. Vacuum fusion results on two of the samples, obtained by a platinum bath-flux method recently described by Evens and Fassel (*12*) are also included. Masson and Pearce conclude that the vacuum fusion method tends to yield slightly lower (10 to 15 p.p.m.) values than the isotope dilution method, although the vacuum fusion results do tend to increase as the temperature is raised. This effect is observed both for high silicon steels and high purity iron. Ihida (*20*) has also rigorously confirmed that nitrogen is incompletely extracted from iron at 1800° C. The conditions used were approximately the same as those used by Masson and Pearce—no bath metal was used. Such results are in serious disagreement with the conclusions drawn in preceding sections of this paper. Reasons for this disagreement will be proposed and discussed in the following Discussion and Conclusions section.

It is significant that Fassel obtains higher results on both NBS 1040 and 1042, by using a platinum bath at 1850° C. It is also reported that this method is satisfactory for all steels, including the high silicon steels. For example, the method gives 46 *vs.* 51 and 90 *vs.* 98 p.p.m. in comparison to the caustic fusion method of Karp, Lewis, and Melnick (*21*) for 3% silicon steels. This appears to be the first instance where a concerted study of the platinum bath method has been made for steels; all previous studies usually involved the use of no bath, and occasionally an iron bath. In view of the success attained with the platinum bath for the reactive and refractory metals, it is surprising that it has not been applied to any great extent in the steel industry up to this time.

DISCUSSION AND CONCLUSIONS

Thermodynamic calculations based on experimentally determined interaction coefficients predict that iron bath vacuum fusion nitrogen analysis should be feasible for all metals, including iron, discussed in this paper. Consideration of the kinetics of nitrogen extraction indicates that, with a few possible exceptions, the release of the gas from the molten iron bath should not limit the success of the method. Conversely, a large body of practical experimental evidence, admittedly somewhat beclouded for the refractory and reactive metals, but very clear for pure iron, indicates that the method as currently applied is largely unsuccessful. Since the greatest discrepancy between theory and experiment exists for the analysis of pure iron, it is instructive to examine the experimental data for this system in terms of the proposed theory in some detail. The work of Ihida (*20*), supported by data presented by Masson and Pearce (*29*) and Evens and Fassel (*12*) is particularly pertinent to this examination. Ihida analyzed pure iron–carbon residues remaining after vacuum fusion analysis at 1850° C. and found that some 30 to 40 p.p.m. of nitrogen remained in the bath. Addition of these residual nitrogen values to the amounts extracted by the vacuum fusion process gives results which agree with those obtained by the Kjeldahl method. Ihida calculated that for pure iron, this residual nitrogen corresponds to an equilibrium nitrogen pressure of 7.6 mm. at 1600° C., decreasing to 6.3 mm. at 1900° C. It was also noted that the effect of carbon would be to increase this equilibrium pressure but no quantitative calculation was presented. Using data previously presented in this paper, it is calculated that if the bath were saturated with carbon, the equilibrium nitrogen pressure for 40 p.p.m. of nitrogen would be of the order of one atmosphere. Although no direct analyses of residual nitrogen contents in bath materials were performed by Masson and Pearce (*29*) or Evens and Fassel (*12*), low nitrogen recoveries from pure iron obtained by these investigations indicate the same situation reported by Ihida.

It must be concluded that the main deterrent to success of the method is an inability to achieve thermodynamic equilibrium. The failure of the method for pure iron precludes its successful application to any other metals or alloys, not for the usually accepted reason of inability to decompose refractory nitrides, but because elemental nitrogen cannot be extracted to its equilibrium limits of concentration.

Speculative thought indicates that incomplete extraction of nitrogen from the bath may be due to inhomogeneity of composition of the iron carbon system. Smith (*43*) has shown that graphite flakes can precipitate near the surface of the bath, apparently because of surface cooling due to iron evaporation, to form a semisolid network of graphite and carbon-saturated iron. In addition to the graphite flakes observed by Smith, a discontinuous surface film of what is apparently carbon is observed on the top of solidified iron–carbon baths. Both of these conditions could prevent practical rates of diffusion of nitrogen to and through the bath–gas interface. These conditions, if present before a sample was dropped into the bath, would be disrupted by the sample entering the surface of the bath and the initial violent release of gas bubbles. Subsequent reformation of the semisolid layer would then prevent the release of residual gas via the normally expected diffusion process. The fact that some, but not all, of the nitrogen is normally released lends some evidence to this explanation. Mechanical or electrical stirring by low frequency induction heating should break up this impervious layer and allow complete release of the entrapped nitrogen.

Evens and Fassel have shown that the use of platinum in place of iron for a bath material leads to a method which appears to be quantitative for all types of steels. It is believed that the platinum bath method will eventually prove to be successful for nitrogen analysis in most metals and alloys, including the reactive and refractory metals. However, for theoretical and economic reasons it is desirable to continue research which may lead to solutions of the problems inherent in the iron bath method. To this end a research program, based on the propositions presented in this paper, is being carried out in this laboratory as a part of a larger program on investigation of gases in metals analysis. It is hoped that this paper will help to stimulate similar research efforts in other laboratories concerned with this important problem.

LITERATURE CITED

(1) Andrews, M. R., *J. Am. Chem. Soc.* **54**, 1845 (1932).
(2) Ang, C. Y., Wert, C., *Trans. AIME* **197**, 1032 (1953).

(3) Beach, A. L., Guldner, W. G., ANAL. CHEM. **31**, 1722 (1959).
(4) Booth, E., Bryant, F. S., Parker, A., *Analyst* **82**, 50 (1957).
(5) British Iron and Steel Research Association, "Determination of Nitrogen in Steel," Special Rept. No. **62**, Iron and Steel Institute, London, 1962.
(6) Chipman, J., *J. Iron Steel Inst.* **180**, 97 (1955).
(7) Cost, J. R., Wert, C., *Acta Met.* **2**, 231 (1963).
(8) Dealy, J. M., Pehlke, R. D., *Trans. AIME* **227**, 1030 (1963).
(9) Dushman, S., "Scientific Foundations of High Vacuum Technique," Wiley, New York, 1962.
(10) Ehrlich, G., Friedrich, K., *Neue Huette* **8**, 27 (1963).
(11) El Tayeb, N. M., Parlee, N., *Trans. AIME* **227**, 929 (1963).
(12) Evens, F. M., Fassel, V. A., ANAL. CHEM. **36**, 2115 (1964).
(13) Everett M. R., *Analyst* **83**, 321 (1958).
(14) Everett, M. R., Thompson, G. E., *Ibid.*, **87**, 515 (1962).
(15) Fagel, J. E., General Electric Co., private communication, Cleveland, Ohio, 1963.
(16) Fagel, J. E., Smith, H. A., Witbeck, R. F., ANAL. CHEM. **31**, 1115 (1959).
(17) Fast, J. D., Verrijp, M. F., *J. Iron Steel Inst.* **176**, 24 (1954).
(18) Hansen, M., "Constitution of Binary Alloys," McGraw-Hill, New York, 1958.
(19) Holt, B. D., Goodspeed, H. T., ANAL. CHEM. **35**, 1510 (1963).
(20) Ihida, M., *Bunseki Kagaku* **8**, 786 (1959).
(21) Karp, H. S., Lewis, L. L., Melnick, L. M., *J. Iron Steel Inst.* **200**, 1032 (1962).
(22) Kashyap, V. C., Parlee, N., *Trans. AIME* **212**, 1 (1958).
(23) Klyachko, Yu. A., Chistyakova, E. M., *Zavod. Lab.* **25**, 1519 (1960).
(24) Kubaschewski, O., Catterall, J. A., "Thermochemical Data of Alloys," Pergamon Press, London, 1956.
(25) Kubaschewski, O., Hopkins, B. E., "Oxidation of Metals and Alloys," Academic Press, New York, 1952.
(26) Langenberg, F. C., *Trans. AIME* **206**, 1099 (1956).
(27) Mallett, M. W., *Talanta* **9**, 133 (1962)
(28) Mallett, M. W., Griffith, C. B., *Trans. ASM* **46**, 375 (1954).
(29) Masson, C. R., Pearce, M. L., *Trans. AIME* **224**, 1134 (1962).
(30) McDonald, R. S., Fagel, J. E., Balis, E. W., ANAL. CHEM. **27**, 1632 (1955).
(31) McKinley, T. D., E. I. du Pont de Nemours & Co., Wilmington, Del., private communication,
(32) Norton, F. J., Marshall, A. L., *Trans. AIME* **156**, 351 (1944).
(33) Pemsler, J. P., Wright Air Development Division, *WADD-TR* **60-655**, 1961.
(34) Pehlke, R. D., Elliott, J. F., *Trans. AIME* **218**, 1088 (1960).
(35) Pehlke, R. D., Elliott, J. F., *Ibid.*, **227**, 844 (1963).
(36) Portnoi, K. I., Levinskii, Yu. V., *Zhur. Fiz. Kim.* **37**, 2627 (1963), *Russ. J. Phys. Chem.* **37**, 1424 (1963).
(37) Portnoi, K. I., Levinskii, Yu. V., *Zhur. Fiz. Khim.* **37**, 2467 (1963), *Russ. J. Phys. Chem.* **37**, 1333 (1963).
(38) Schwarzkopf, P., Kieffer, R., "Refactory Hard Metals," Macmillan, New York, 1955.
(39) Seybolt, A. U., Burke, J. E., "Experimental Metallurgy," Wiley, New York, 1953.
(40) Seybolt, A. U., Oriani, R. A., *Trans. AIME* **206**, 556 (1956).
(41) Sieverts, A., Bruning, K., *Arch. Eisenhuttenw.* **7**, 641 (1934).
(42) Sloman, H. A., Harvey, C. A., Kubaschewski, O., *J. Inst. Met.* **80**, 391 (1951–52).
(43) Smith, W. H., ANAL. CHEM. **27**, 1636 (1955).
(44) Staley, H. G., Svec, H. J., *Anal. Chim. Acta* **21**, 289 (1959).
(45) Turovtseva, Z. M., Kunin, L. L., "Analysis of Gases in Metals," Consultants Bureau, New York, 1961.
(46) Wagner, C., "Thermodynamics of Alloys," Addison-Wesley Press, Inc., Cambridge, 1952.
(47) Walter, D., ANAL. CHEM. **22**, 297 (1957).

Pesticide Residues

J. William Cook and Sidney Williams

Division of Food Chemistry, Food and Drug Administration, U. S. Department of Health, Education, and Welfare, Washington, D. C.

METHODOLOGY for residue analysis has advanced rapidly during the current review period, from November 1962 through October 1964. Notable progress has been made in the development and refinement of methods of analysis by which any or all of a large number of pesticide residue chemicals can be detected and measured in one general operation. This is of particular significance because great interest has developed—outside the scientific community as well as within—in the possible presence of pesticide chemicals in all parts of our environment, including man himself. Only by the use of improved methodology will it be possible to accomplish the task of detecting, identifying, and measuring the many possible residual pesticide chemicals. It is only after the presence or absence of these chemicals in any part of our environment has been proved unequivocally that the medical man, the lawyer, the lawmaker, the administrators in government and in industry, and other interested groups can assess the significance of such residues.

There are 300 to 400 chemicals registered for use on food products alone, and a few hundred more are registered for other uses whereby they may become part of our environment. The chemist cannot know which of the hundreds of possible pesticide chemical residues to look for in samples of air, water, soil, plants, human and animal tissues, prepared foods, etc. There is an urgent need for general procedures that can identify and measure a large number of chemicals at one time. They must be highly sensitive and accurate, since it is essential that all monitoring of our environment be at a level considerably below any "tolerance" or otherwise critical level, so that trends can be more readily recognized and assessed for significance. Upward or downward trends in any portion of our environment will be recognizable only when the methodology becomes sufficiently sensitive and accurate so that analyses in the fraction-of-a-part-per-million or even part-per-billion range become routinely dependable (Fischbach, H. Pub. 1082, National Research Council, p. 55, Nov. 29, 1962).

The multiple detection procedure of Mills (*145*) for chlorinated pesticides is still being extended and modified. The determinative steps most useful for these general procedures are still the forms of chromatography—gas liquid, thin layer, and paper. There is growing recognition that, for these determinative steps to be useful and dependable, great care must be exercised to avoid interferences, false responses, and incorrect interpretations of chromatograms.

Lykken (*134*) emphasizes the point that if a sample is not representative of the lot of material from which it is obtained, results of analysis will not be valid or useful. He presents valuable information for anyone involved in residue work and discusses factors which must be considered in designing experiments, such as proper sampling, compositing, quartering, storage, and shipment of samples.

Greater importance has been assigned to efficient extraction of the pesticide residue from the sample. The use of mixtures of solvents such as hexane and isopropanol to achieve a single continuous phase with the aqueous medium of food products has been studied (*14, 101, 141, 147, 176, 192*). Some investigators dehydrated the tissues by using anhydrous sodium sulfate, which improved the extraction of the residue with an organic solvent (*10, 50, 141, 185*).

The use of solvents which dissolve the pesticidal chemicals while maintaining miscibility with the aqueous medium of the tissues, such as acetone (*93*, *127*, *185*) and acetonitrile (*14*, *54*, *157*), is reported. Such solvents give generally higher values than those obtained by the use of solvents that are immiscible with tissue media, such as hexane alone. Care must be exercised not to pass judgment on the efficiency of extraction merely on the basis of high recoveries of the chemical added during the analytical procedure. Good recoveries of the chemical added may be achieved when the degree of extraction of the actual weathered residue is poor.

Gas chromatography columns currently available are sensitive to small amounts of certain impurities. A number of papers emphasize the necessity of good cleanup before the extracts can be chromatographed to yield unambiguous results (*22*, *27*, *44*, *45*, *63*, *130*, *133*). These columns must be carefully and thoroughly "conditioned" (*44*, *45*, *63*, *177*) before they can be used routinely to obtain good qualitative and quantitative results.

Barry and Hundley (*17*) have edited a "Pesticide Analytical Manual" comprising a compilation of methods and other information useful to a residue analyst. Although developed as a guide for chemists in the laboratories of the Food and Drug Administration, it has been distributed to many others as well. It presents information on sampling, extraction, cleanup, and determinative procedures; techniques for preparing and conditioning columns; and lists of relative retention times and detectable quantities of many pesticides for different chromatographic procedures and detectors.

Although gas chromatography with various detectors is now the most popular technique in residue analysis, other procedures have not been neglected. Thin layer chromatography, with its greater speed of development and increased resolution and sensitivity, has to some extent replaced paper chromatography. Colorimetric, ultraviolet, and infrared procedures also are used.

GAS CHROMATOGRAPHY—GENERAL

The greatest advances during this period have been made in gas chromatography. Because of the extreme sensitivity of the electron capture detector, it has been used by many workers. Dimick and Hartmann (*64*) have published a general description of electron capture gas chromatography as used in pesticide analysis. They discuss the principle of electron capture and the geometry and operating parameters of the detector. Although only one specific instrument is described and some of the steps in the outlined procedure have since been improved, this report does provide a good introduction to those unfamiliar with the subject.

With the search for ever-increasing sensitivity and speed of analysis has come the realization that these desirable goals encourage the production of methods in which unrecognized side effects, minor interferences, slight amounts of contaminants, and any lack of care in the use of equipment or interpretation of responses can produce greatly misleading and inaccurate results.

Lovelock (*133*), in a general discussion of electron absorption detectors, points out that with complex mixtures (such as are usually present in residue analysis) these detectors may give "... not only inaccurate but even totally false results." Causes of various false responses, both positive and negative, are discussed and a pulse-sampling technique which minimizes the errors is described. Barney, Stanley, and Cook (*16*), working with Systox, have shown that in a poorly designed detector, electron capture and ionization may take place at the same time and that pulse mode of detector operation will not eliminate interferences from ionization.

Burke and Giuffrida (*44*) point out the need for adequate cleanup before a sample extract is injected into the electron capture gas chromatograph. They show that injection of poorly cleaned extracts may contaminate a column and result in weak or spurious responses. Since solvents used must be "pure," redistillation is frequently required. The use of plastic containers for solvents is discouraged, since extractables in the plastics may cause response of the electron capture detector. In some cases these spurious effects are so strong that responses due to pesticide residues may be completely masked. The need for proper preconditioning of the gas chromatography column is also thoroughly discussed. Unless properly conditioned, the column may cause degradation of some pesticides. Not only may the degradation result in loss of the pesticide but also the degradation products may cause responses at the retention times of some other common pesticides for which they may be mistaken. Equipment and operating parameters described permit detection of chlorinated pesticides, such as heptachlor epoxide, at levels of 0.01 to 0.001 p.p.m. Relative retention times are listed for 65 pesticides.

Bonelli, Hartmann, and Dimick (*35*) describe two columns used with electron capture gas chromatography. Pesticides which cannot be resolved on one column may be resolved on the other. Operating parameters, sensitivity data, and retention times for a number of pesticides, including chlorinated, organophosphorus, and organosulfurs, are given.

Burke and Holswade (*45*) present similar data for microcoulometric gas chromatography. Retention times relative to aldrin are listed for 87 chlorinated compounds, and those relative to sulfone for 26 thio compounds. A table which lists the amount of each pesticide required to give a half-full scale recorder deflection may be found very useful. Recovery data indicate that responses are linear when the pesticide is present above a definite minimum quantity. The need for proper cleanup of sample extract before injection is emphasized, and conditions and precautions for most effective use are described. The general level of sensitivity obtainable is given as 0.01 p.p.m.

Shuman and Collie (*177*) describe the preparation of a gas chromatography column; they also emphasize the need for proper conditioning. They recommend a 6-foot, 6-mm. i.d. column packed with 10% Dow Corning 200 (12,500 centistokes) silicon fluid on Anakrom ABS. Other workers (*44*, *45*) have also found this type of column superior for pesticide work.

De Faubert-Maunder, Egan, and Roburn (*63*) give details for preparing a column. Good columns, columns which decompose pesticides, construction, linearity of responses and cleaning of detectors, effects of rate of injection, peak measurements, and chromatogram interpretation are discussed. The use of a glass injection liner is recommended.

Beckman and Bevenue (*24*) studied the effect of column tubing composition on recovery of chlorinated hydrocarbons. Working with 6-foot by $^1/_4$-inch columns and a microcoulometric detector, they checked columns made of copper, stainless steel, aluminum, and quartz. Copper tubing gave the poorest recoveries, quartz the best. Aluminum and stainless steel were satisfactory.

It has been apparent for some time that complete reliance on the retention time for identification of a compound may yield erroneous results. Robinson and Richardson (*170*) emphasized the need for caution in interpreting the results of gas chromatography of plant and animal extracts, both as to identity and quantity, when only one column is used. They described four different columns and tabulated the resolution of pairs of pesticides on these various columns.

Goulden, Goodwin, and Davies (*88*, *89*) were also concerned about improving the certainty of identification. They found that a column packed with a 2.5% silicon oil and 0.25% Epikote 1001 on Celite gave good resolution. With column temperature of 163° and gas flow of 100 ml. nitrogen per minute, they obtained complete separation of at least 11 pesticides in 30 minutes. They also proposed the use of simultaneous chromatography using five parallel columns leading to one electron capture detector. The stationary phases of the columns differ so that 3 to 5 peaks may be obtained for each pesticide. They called this a "spectrochromatogram" and stated that the pattern is characteristic of the specific pesticide. They also described the use of a halogen-sensitive cell of the type used in detecting refrigerator leaks. The response to individual chlorinated pesticides of this cell differs from that of the electron capture cell. By connecting this detector in series with the electron capture detector and recording responses from both detectors, identification of individual compounds is made more reliable.

Programmed temperature gas chromatography is also coming into use as a means of improving resolution and separation, speeding up runs, and chromatographing mixtures containing both very fast and very slowly eluting compounds. Burke (*43*) used programmed temperature with a microcoulometric detector; he tabulated relative retention times for 22 compounds. Other workers have also used this technique (*20, 36, 118*).

The technique of preparation of a derivative of a pesticide before injection into the gas chromatograph has been continued. For some time, it has been the practice to convert 2,4-D and other chlorinated phenoxy acid herbicides to their methyl esters because the free acids will not pass through the common gas chromatographic columns. Derivatives are now being used for other reasons. Klein and Watts (*120*) found that Perthane, *o,p'*-DDT, and *p,p'*-DDD have similar retention times and are difficult to resolve on many gas chromatographic columns; however, olefins of these three compounds prepared by refluxing cleaned-up sample extracts with 2% NaOH in ethanol were separated on a 3-foot gas chromatographic column of Celite 545 with a 2.5% coating of SF-96 and 2,2 - diethyl - 1,3 - propandioliso-phthalate polyester (1:1). Klein and Watts obtained recoveries ranging from 84 to 105% from samples of leafy vegetables containing residues added at levels of 1 to 10 p.p.m. One striking benefit from the use of the olefins is that the Perthane olefin gives an electron capture response about ten times greater than Perthane.

Beckman and Berkenhotter (*20*) used derivatives to increase the reliability of identification of pesticide residues. They separated the individual compounds by gas chromatography with a thermal conductivity detector and then dechlorinated the individual fractions with sodium and liquid ammonia. After that they chromatographed the dechlorinated portions again and obtained chromatograms. The results obtained from the two chromatograms can be used to characterize the pesticide.

Gutenmann and Lisk (*97, 99*) prepared brominated derivatives which had strong electron capturing ability and chromatographed these as a means of obtaining increased sensitivity. They worked with diphenyl, Guthion, MCP, and MCPB as pure compounds, and also used this technique to determine residues of CIPC, monuron, diuron, and linuron in fruits and vegetables. Recoveries from crops of 76 to 116% were obtained at levels of 0.05 to 1.2 p.p.m. when only one pesticide was present at a time.

A word of caution may be in order about using this technique in any procedure which does not include extensive cleanup of residue before gas chromatography. Valuable information may be obtained when only one pesticide is added and when the untreated crop is available so that chromatograms of sample and control can be compared. However, if the technique is used on crops of unknown spray history with no control crop available for comparison, chromatograms would probably contain so many unidentified and unidentifiable peaks that accurate interpretation would be impossible.

Bache, Lisk, and Loos (*13*) prepared nitro derivatives of MCP, MCPB, and NAA in order to increase the response of these herbicides on the electron capture gas chromatograph. They used this technique to determine MCP and MCPB in timothy and peas and NAA in apples, and reported finding residues of MCP on snap bean plants treated with MCPB.

The above discussion has been concerned primarily with gas chromatograph detectors for halogenated compounds. One of the most exciting and promising developments of the past year was the appearance of two dissimilar detector systems, each of which is reported to be highly specific for phosphorus - containing compounds. Giuffrida (*86*) modified a conventional flame ionization detector by fusing a sodium salt onto the electrode. The result was a detector 600 times as responsive to a compound containing 10 carbons and 1 phosphorus atom as was the conventional flame detector. Response to compounds containing six chlorine atoms was twenty times as great, while the response to compounds containing neither Cl nor P was the same as that of the conventional flame ionization detector. When the extraction procedure of Mills, Onley, and Gaither (*146*) was used, diazinon, ronnel, parathion, ethion, and Trithion, when added to broccoli at levels of 0.05 and 0.1 p.p.m., were easily detected. There was no interference from crop materials even when the equivalent of 5 grams of original sample was injected. Construction and operating conditions of the detector are described, and retention time lists for 23 organophosphate compounds are given.

Burchfield, Rhoades, and Wheeler (*42*) report the development of a microcoulometric detection system which is specific for phosphorus. The effluent from the usual gas chromatographic column is passed through a quartz tube heated to 950° C., with hydrogen as the carrier gas. Organic compounds are reduced to hydrocarbons, water, PH_3, H_2S, and HCl. The latter three compounds precipitate silver ion and so register on a microcoulometric titration cell. Insertion of a short silica gel column removes HCl; substitution of Al_2O_3 for silica gel removes both HCl and H_2S and permits measurement of PH_3 with absolute specificity. Response of the cell to PH_3, H_2S, and HCl is in the ratio of 2:2:1. When a model C-100 microcoulometer at maximum sensitivity is used, 0.1 μg. of P gives a peak area of 5 square inches. Cleaned-up extracts from crops examined do not interfere with the reduction or detection steps.

CHLORINATED PESTICIDES—GENERAL PROCEDURES

More attention has been given to the development of multiple detection procedures for the chlorinated pesticides than for any other class of pesticides. This is only natural, since these compounds are widely used and many are so persistent that traces of some compounds, such as DDT, are being found almost everywhere. Moreover, these compounds have been found to be more amenable to this type of analytical method.

Mills, Onley, and Gaither (*146*) have combined and modified previously reported methods to provide a rapid, simple procedure for extracting and cleaning up residues from nonfatty foods. Used with gas chromatography, thin layer chromatography, or paper chromatography, the procedure will determine 21 chlorinated pesticides. Good recoveries were obtained of 5 pesticides added to 11 products at levels from 0.02 to 0.2 p.p.m.

Taylor, Rea, and Kirby (*185*) extracted chlorinated pesticide residues from animal tissue by blending the tissue with acetone and anhydrous Na_2SO_4. The pesticides were transferred to hexane and injected into a gas chromatograph. Recoveries for lindane, endrin, dieldrin, *p,p'*-DDE, and heptachlor epoxide ranged from 75 to 99% at 2.5 to 10 p.p.m. levels.

Several procedures have been reported for extracting chlorinated pesticide residues from water. Kahn and Wayman (*113*) describe a continuous extractor using refluxing petroleum ether.

The water sample was passed through the extractor at a rate of 0.5 to 1.0 liter per hour. Nonpolar compounds were extracted by the petroleum ether, concentrated, and determined by electron capture gas chromatography. Some of the intermediates in the manufacture of aldrin and endrin were determined at levels as low as 0.3 p.p.b. by using a 135-liter sample. Infrared spectra can be run on extracts after cleanup on alumina columns.

Breidenbach *et al.* (*38*) describe equipment and procedures for collecting large volume samples of water by carbon adsorption as well as analysis of discrete bottled samples of water. The procedure used by the Public Health Service–Water Pollution Surveillance System for analysis of carbon-chloroform extract by thin layer chromatography, electron capture, and microcoulometric gas chromatography, and infrared is reported, but no data on the efficiency of such a system are presented.

Schwartz *et al.* (*175*) have used electron capture gas chromatography for determining "Polystream," a mixture of chlorinated benzenes, in clams and oysters.

Minyard and Jackson (*147*) analyzed 101 samples of commercial animal feeds, using electron capture gas chromatography. They extracted the samples with an isopropanol–Skellysolve B mixture (1 + 3) and used a Florisil column cleanup. They state that they were able to detect less than 1 p.p.b. of most of the chlorinated pesticides.

Baetz (*14*) reported using Norit-A for cleaning up sample extracts. The sample was extracted by blending with acetonitrile or mixed solvents and the residues were partitioned into petroleum ether. An aliquot was evaporated to dryness, taken up in benzene, shaken with Norit-A, and filtered. The filtrate was reported to be suitable for injection into the microcoulometric gas chromatograph but it could not be used for electron capture gas chromatography because of excessive interference. The method is reported to have given satisfactory recoveries of 11 chlorinated pesticides from carrots, collards, okra, and peas at 0.1 to 1.0 p.p.m. levels. Recoveries of lindane and BHC, however, were as low as 53%.

Moats (*152*) reported a one-step cleanup procedure using a column of Nuchar C 190–Celite 545 (1 + 2). With paper chromatography as the determinative step, sensitivity was believed to be about 0.03 to 0.1 p.p.m.

McKinley, Coffin, and McCully (*137*) have reviewed cleanup procedures for both chlorinated and organophosphate pesticide residues. They point out the advantages and limitations of the various methods, and list 41 references. Gutenmann and Lisk (*93*) have used electron capture gas chromatography as the determinative step in which the samples were extracted with acetone and the residues partitioned into Skellysolve B, a portion of which was injected into the gas chromatograph without additional cleanup. They used technical grade solvents without purification and reported recoveries ranging from 60 to 112% for 11 pesticides, with sensitivities from 0.04 to 0.001 p.p.m. (The writers believe that it is well to caution readers once more against using such abbreviated procedures unless the spray history of the crop is known and untreated samples of the same product are available for comparison determination.)

The determination of chlorinated pesticide residues in fatty foods has presented a special problem, since the pesticides are fat- and oil-soluble and separation is difficult. The analysis of milk presents an added challenge in that, for many procedures, the fat must first be separated from the milk.

Onley (*161*) has reported a rapid method for milk which combines the usual two steps into one. Instead of first separating the fat from the milk and then extracting the pesticide from the fat, the milk is blended with a mixture of acetonitrile, ethyl ether, dioxane, and acetone (3:1:1:1) and anhydrous sodium sulfate. After filtering, water is added and the residues are transferred to petroleum ether. From this point, a modification of the Mills procedure is followed. By electron capture gas chromatography, satisfactory recoveries were obtained for 19 pesticides at levels ranging from 0.005 to 0.1 p.p.m.

Henderson (*106*) reported a collaborative study involving two samples of milk and 22 laboratories. Advantages and disadvantages of various methods were discussed, and results by paper chromatography and microcoulometric and electron capture gas chromatography were compared.

Several methods have been reported for separating fat from dairy products prior to pesticide analysis. Langlois, Stemp, and Liska (*131*) used the conventional Babcock test procedure and reported that although endrin was apparently destroyed, DDT, DDE, lindane, heptachlor, heptachlor epoxide, and dieldrin were recovered satisfactorily. Lampert (*128*) used a detergent solution and a Babcock cream test bottle for separating the fat from milk.

A number of different ways have been suggested for separation or cleanup of pesticide residues from fats and oils. Eidelman (*67*) used dimethylsulfoxide to extract the residues from acetone and petroleum ether solutions of fat. After water was added and the residues were partitioned into petroleum ether, they were further cleaned up by the Mills Florisil column procedure and then determined by microcoulometric gas chromatography. Fish oil samples required additional treatment of the 15% eluate from the Florisil column to eliminate interferences. Saponification and MgO-Celite column cleanup were used prior to injection into the gas chromatograph.

McCully and McKinley (*135*) used a freezing technique to separate chlorinated pesticide residues from fats and oils. The fat or oil was dissolved in a benzene-acetone mixture (1 + 19) and the fat precipitated by cooling to −70° C. The solution was filtered through a charcoal-wood cellulose column and concentrated for injection into the electron capture gas chromatograph. A special apparatus for use in this procedure was described in a second publication (*136*). Working with organophosphate compounds, Crosby and Laws (*57*) reported that freezing out of waxlike substances from acetone solution removed some impurities but also removed pesticides.

Ott and Gunther (*163*) used forced volatilization to separate pesticide residues from butter fat by use of a newly designed device. The fat was heated to about 190° C. and volatiles were carried to a cooled trap by a stream of nitrogen. Determination was made by microcoulometric gas chromatography; the entire analysis required about one hour. Sensitivity was reported at about 0.5 p.p.m. for some of the more common chlorinated pesticides. However, DDT broke down to form some DDE and DDD.

De Faubert-Maunder, Egan, and Roburn (*62*) compared dimethylformamide and dimethylsulfoxide for extracting residues from hexane solutions of fats, and reported that dimethylformamide gave better recoveries. They described procedures for analyzing samples of fat, milk, butter, and eggs. Moats (*151*) used a column containing 100 grams of standardized Florisil to clean up as much as 2 grams of fat. Pesticides were eluted from the column with 20% methylene chloride in petroleum ether in a form adequate for spotting the entire sample for paper chromatography.

Langlois, Stemp, and Liska (*130*) extracted pesticide residues from dairy products by grinding the samples with Florisil that was partially deactivated by the addition of 5% water to the adsorbent. The mixture was added to the top of a Florisil column and the pesticides were eluted with 20% methylene chloride in petroleum ether. The eluate was evaporated and the residue taken up in hexane for injection into the electron capture gas chromatograph. The procedure took 30 to 90 minutes and recoveries were reported as being consistently better than 90%. The sensitivity was stated to be 0.05 p.p.m.

for DDT and endrin and 0.1 p.p.m. for some of the other chlorinated pesticides. The same procedure was also adapted for analyzing egg yolk and poultry tissue (*181*, *182*). The fact that in this procedure different volumes of eluting solvent are used for different pesticides presents a difficulty. For example, to remove endrin, 650 ml. of eluting solvent and 90 minutes of eluting time are required, and in screening for all pesticides this most time-consuming version of the procedure would have to be used routinely.

Onley and Mills (*162*) modified the conventional Mills procedure for use on eggs. To eliminate interferences, they passed an acetone solution of the extracted oil through a filter paper pulp column. They obtained recoveries of 73 to 110% for seven pesticides at levels down to 0.02 p.p.m.

Thin layer chromatography has become increasingly important in pesticide residue analysis. Kovacs (*124*) studied the chromatography of 16 pesticides on alumina and silica gel plates. He found that for the silica gel plates, prewashing was desirable and the ultraviolet exposure time was critical. R_f's relative to DDD are listed and results by thin-layer chromatography and microcoulometric gas chromatography are compared, using the extraction and cleanup procedure of Mills, Onley, and Gaither (*146*). Use of good cleanup before thin layer chromatography, allowed determination of many pesticides in the p.p.b. range.

Walker and Beroza (*196*) have made an extensive study of thin layer chromatography. They list the R_f's in 19 solvent systems for each of 62 pesticides, including chlorinated compounds, organophosphates, and carbamates, and they discuss chromogenic sprays, choice of solvent system, and the use of thin layer chromatography as a cleanup procedure.

Kawashiro and Hosogai (*114*) have reported a new spray reagent for detecting chlorinated pesticides on silica gel thin layer chromatography plates. The plates are sprayed with 0.5% *o*-tolidine or *o*-dianisidine and then irradiated with ultraviolet at 2536 A. The pesticides appear as green spots against a white background. Amounts of 0.5 to 1 μg. are detectable for many of the pesticides. This reagent does not appear to be as sensitive as the conventional $AgNO_3$.

Morley and Chiba (*153*) have used thin layer chromatography as a cleanup procedure for gas chromatography. Samples were spotted and developed on each half of a plate. One half of the plate then was covered with aluminum foil and the other half sprayed and exposed to ultraviolet light to locate the spots. Similar areas on the covered half then were scraped off and extracted for gas chromatography. They also report the use of thin layer chromatography on uncleaned plant extracts as a rapid screening method for DDT and DDE in plants.

Paper chromatography continues to receive attention. Mitchell (*148*) reports the minimum detectable quantities of 22 chlorinated pesticides, using the $AgNO_2$-phenoxyethanol chromogenic agent. Data for an aqueous system and a nonaqueous system are given along with R_f values. Krzeminski and Landmann (*126*) describe a spray reagent for paper chromatography which gives no curtain effect and in which impurities in the paper do not interfere. The reagent, an alcoholic water solution of methyl yellow, was tested on 14 chlorinated pesticides and showed detection limits of 2 to 8 μg.

Heinisch and Neubert (*104*) report the use of wedge-shaped strips for the ascending paper chromatography of chlorinated pesticide residues. They state that this procedure gives better separation than other methods.

A matter of concern to all residue chemists is the possible presence of unsuspected degradation or metabolic products of pesticides. Roburn (*171*) studied the effect of sun and ultraviolet light on several chlorinated pesticides. Using gas chromatography, he found that grass treated with dieldrin and exposed to sunlight for several months gave an unknown second peak with response approaching that of dieldrin in magnitude. Fifty 100-μg. quantities of pesticides were then deposited as films on glass and exposed to a germicidal ultraviolet lamp for 2 to 3 hours. With gas chromatography as the examining medium, dieldrin so exposed showed one derivative; endrin showed one main product and several minor ones; aldrin showed dieldrin and a small amount of another derivative; *p,p'*-DDE, three main products and several minor ones; *p,p'*-TDE, a small amount of a dehydrochlorinated product; and *p,p'*-DDT, a small amount of DDE. The α, β, γ, and δ isomers of BHC did not show any reaction products.

CHLORINATED PESTICIDES—SPECIFIC PROCEDURES

Friestad (*77*) reports a spectrophotometric method for aldrin which requires prior cleanup of the sample. The aldrin is reacted with nitrosyl chloride, then heated in acid to form dihydrochlorketo aldrin. This compound reacts in alcoholic solution with *m*-dinitrobenzene and potassium hydroxide to give a red-violet color, which is extracted with chloroform and its absorbance read at 525 mμ. Dieldrin and endrin interfere with the assay. This reaction is sensitive to about 10 μg. aldrin.

Bache (*11*) used thin layer chromatography to determine amiben in tomatoes. The sample was treated with sodium hydroxide; the hydrolyzed amiben was extracted and, after transfer to acetone, spotted on silica gel plates. Bache, Gutenmann, and Lisk (*12*) used electron capture gas chromatography for the same determination, methylating the hydrolyzed amiben prior to injection into the gas chromatograph. They report recoveries of 70 to 123% at levels of 0.05 to 1.25 p.p.m.

Klayder (*119*) has modified the A.O.A.C. method for captan for use on green vegetables. A collaborative study involving eight laboratories showed average recovery of 95% at levels of 50 to 105 p.p.m. He also reports that captan was largely destroyed by a canning process in which the food was heated at 14-pound pressure for 35 minutes to one hour.

Beckman and Bevenue (*22*) determined chlorobenzilate in grapes and cottonseed, using microcoulometric gas chromatography. They report a sensitivity of 0.05 p.p.m. and state that rigorous cleanup is essential; otherwise the gas chromatographic column quickly becomes contaminated.

Schafer, Busch, and Campbell (*173*) have reported a rapid screening method for DDT in milk, using electron capture gas chromatography. The milk was treated with alcoholic potassium hydroxide to saponify the fat and convert DDT to DDE. A hexane extract of the milk was then injected into the gas chromatograph. Recoveries of 95% are reported at levels of 0.04 to 0.12 p.p.m. on a whole milk basis.

Blinn and Gunther (*31*) present the results of a collaborative study of two versions of a colorimetric procedure for DDT in milk and butter fat. They prefer the version which includes an oxidation step in the cleanup.

Klein, Watts, and Damico (*121*) used the conversion of DDT to DDE as confirmation of identity in the analysis of butter and oils for DDT. They used the Mills procedure for extraction and cleanup and determined DDT by electron capture gas chromatography. A second aliquot was converted to DDE by treatment with sodium hydroxide, and again chromatographed.

Hardin and Sarten (*101*) compared five procedures for extracting DDT from field-treated collards. They found that blending first with isopropyl alcohol and then with hexane gave better recoveries than did tumbling or grinding with hexane.

Espadas and Loaeza (*70*) used aniline in place of alcoholic sodium methylate or alcoholic potassium hydroxide in the color development after nitration of

DDT. They state that a more stable color was obtained and the method was simplified.

Beckman and Bevenue (*23*) determined Dilan residues in pears by using microcoulometric gas chromatography after cleanup with Nuchar. DDT, if present, was removed by elution from a Florisil column.

Albert (*6*) has suggested a modification which saves about two hours in the determination of endrin by the Mills procedure. He reports that the 15% eluate can be cleaned up and fat eliminated by passing the eluate through a potassium hydroxide–Celite column. This replaces the lengthy saponification step. By electron capture gas chromatography, the recoveries from carrots, onions, collards, and broccoli at the 0.05 p.p.m. level ranged from 100 to 106%. EDITOR'S NOTE. This procedure may work equally well for dieldrin.

Gordon, Haines, and Martin (*87*) determined Kelthane in milk colorimetrically with a sensitivity of 0.01 p.p.m. based on whole milk or about 0.25 p.p.m. on the fat basis. They extracted the fat from the milk and hydrolyzed the Kelthane with tetramethylammonium hydroxide. The released chloroform was determined by the Fujiwara reaction. The procedure eliminated the need for a special steam system.

Ackermann, Carbone, and Kuchar (*4*) modified an earlier spectrophotometric method for the determination of pentachloronitrobenzene in soil and crops. They used a direct extraction from food products with ethanol and partition into petroleum ether to eliminate pigments. The method was said to be sensitive to about 3 μg.

The A.O.A.C. method for sulfone was studied by Shuman (*176*). He found that hexane as the stripping solvent gave better recovery from field-sprayed peaches, whereas benzene worked better for apples. He checked the remainder of the fruit by blending with an isopropanol-benzene mixture and found that the stripping had removed over 90% of the residues.

Two gas chromatographic columns are described by Carey (*48*) for use in the determination of 2,3,5,6-tetrachloronitroanisole in vegetables and grains by electron capture gas chromatography. By using a cleanup column of activated magnesia and diatomaceous earth, as little as 0.02 p.p.m. can be detected without interference from crops.

Beckman and Bevenue (*21*) describe a method for the determination of tetrachlorothiophene and 1,2-dibromo-3-chloropropane in brussels sprouts and walnut meats, using electron capture gas chromatography. The sample is blended with petroleum ether, and the extract is passed through a Florisil column and injected into the gas chromatograph. The sensitivity of the method is reported as 0.01 p.p.m. Recoveries of 90 to 100% were obtained at levels of 0.05 to 1.0 p.p.m.

Burke and Mills (*46*) used a modification of the Mills procedure to determine Thiodan and Tedion residues. DDT was removed by elution from the Florisil columns with 6% ethyl ether in petroleum ether. Thiodan and Tedion then were eluted with 30% ethyl ether in petroleum ether. Determination was made by microcoulometric gas chromatography. Extracts of broccoli required additional cleanup on a column of sodium sulfate, attapulgus clay, Celite 545, and Nuchar 190 N. Dieldrin and endrin, if present, gave overlapping peaks with the column used in the work.

A colorimetric method for the determination of Thiodan in vegetables and beef fat is described by Maitlen, Walker, and Westlake (*141*). Vegetables were extracted by tumbling with an *n*-hexane–isopropanol mixture (2 + 1). Sugar beet extracts required cleanup by shaking with a charcoal–magnesium oxide mixture. Beef fat was ground with anhydrous sodium sulfate and extracted with *n*-pentane. Cleanup included acetonitrile partitioning and the use of a Florisil column. The actual determination was carried out in a single test tube; an aliquot was evaporated, methanolic sodium hydroxide–pyridine reagent was added, and the developed color was read at 520 mμ. Of 45 pesticides tested, only captan, chlordane, heptachlor, and ovex caused any interference. A somewhat similar procedure had been used earlier by Butler, Maitlen, and Fahey (*47*) to determine Thiodan in strawberries and alfalfa.

A modification of the colorimetric method for toxaphene has been reported by Nikolov and Donev (*160*). They state that the sensitivity of the determination was increased about tenfold by preliminary treatment with nitric acid before development of the color.

HERBICIDES

A number of methods have appeared for the determination of the chlorophenoxy acids and their esters in food products. The free acids are generally converted to their methyl esters for determination by gas chromatography. This is necessary because the free acids will not pass through the common silicon oil gas chromatographic column.

Bevenue, Zweig, and Nash (*27*) describe a method for determining 2,4-D and its esters in potatoes, using microcoulometric gas chromatography. Cleanup on a Florisil column was found necessary to remove unidentified interferences, and diazomethane was used to esterify any free 2,4-D. The method was sensitive to 0.01 p.p.m.

Both paper and thin layer chromatography were used by Abbott *et al.* (*1*) to determine MCPA, MCPB, 2,4-D, 2,4-DB, and 2,4,5-T in soil and water. The thin layer procedure separated dinoseb and DNOC from the above herbicides and detected their presence. Other chromatographic procedures for determining some of these herbicides in various crops are described by Bevenue, Zweig, and Nash (*26*) for 2,4-D in dry crops and walnuts; by Yip (*198*) for 2,4-D in wheat; by Gutenmann and Lisk (*94*) for 2,4-D and 2,4-DB in forage; by (*100*) for MCP in soil; and by (*98*) for silvex in water.

Daoud and Luh (*59*) describe a colorimetric method for determining 2,4,5-T in canned apricots at a level of about 1 p.p.m. After cleanup on activated basic aluminum oxide, the extracted residue was reacted with chromotropic acid and sulfuric acid. The resulting color was read at 565 mμ. The authors state that about 40% of the 2,4,5-T was converted to a combined form not extractable with ether.

Coakley, Campbell, and McFarren (*52*) used a somewhat similar color reaction to determine 2,4-D and its butoxyethanol ester in fish and shellfish at a sensitivity reported to be 0.1 p.p.m. The blended samples were treated with sodium hydroxide to hydrolyze the ester to 2,4-D which, after acidification, was extracted with benzene. A Florisil column was used for cleanup, after which the color was developed with chromotropic acid and read at 570 mμ. Gas chromatography was used to verify the identity of the 2,4-D.

Kirkland and Pease (*118*) used temperature-programmed microcoulometric gas chromatography for the determination of the herbicides Trysben (2,3,6-trichlorobenzoic acid) and Zobar (polychlorinated benzoic acid). The herbicides were extracted from samples of sorghum, wheat, barley, pineapple, and sugar cane by blending with methyl ethyl ketone. The extracted residues were cleaned up and converted to the methyl esters before they were chromatographed. Satisfactory analyses were carried out at 0.04 p.p.m.

Getzendaner (*84*) reports a method for the determination of dalapon in cranberries, bananas, and corn cobs. The determination was made by electron capture gas chromatography using a column consisting of 3.85% diethylene glycol adipate polyester and phosphoric acid on Gas Chrom S or Chromosorb W.A.W. These columns permitted the successful chromatographing of dalapon as the free acid so long as the acidity of the column (H_3PO_4) was maintained. Recoveries at levels of 0.25 to 5 p.p.m. ranged from 80 to 100%.

ORGANOPHOSPHATES—GENERAL PROCEDURES

The development of multiple detection schemes for the organophosphate pesticides has proved more difficult than for the chlorinated. Extraction and cleanup procedures, to be useful, must be capable of handling compounds of widely differing polarities, since the parent pesticides and their metabolites range from oil-soluble to water-soluble.

Coffin and Savary (*54*) report a procedure which includes acetonitrile extraction, elution from polyethylene-coated alumina with slightly acidified 40% acetonitrile, partitioning into chloroform, and final elution of the organophosphates from Magnesol with successive portions of chloroform, acetone, and methanol. The pesticides were separated by paper chromatography and the spots were located by one of several means. The portions of the paper chromatogram containing the spots were cut out and phosphorus was determined after digestion in a Schöniger flask. Recoveries of 80 to 107% were obtained for 41 organophosphate standards and for 25 organophosphates added to lettuce at levels of 0.4 to 1.5 p.p.m. Eight compounds tested were not determined by this procedure.

As mentioned earlier, a big step forward in the methodology for organophosphate residues has been the development of gas chromatographic detectors highly specific and sensitive for phosphorus-containing compounds (*42*, *86*). It is anticipated that as they become more readily available these detectors will find widespread use in the development of complete procedures that will be adequate for the determination of both the parent pesticide chemical and the significant metabolic products. Meanwhile earlier detectors are being used. Nelson (*157*) made use of microcoulometric gas chromatography with the sulfur cell for determining 10 thiophosphate pesticides in fruits and vegetables at levels ranging from 0.15 to 1.5 p.p.m. Samples were extracted by blending with acetonitrile, and the residues were partitioned into petroleum ether. No additional cleanup was used. It was pointed out that the study was made only on parent compounds. Many of these formed toxic metabolites which contained no sulfur or were water-soluble and so would not be detected by this procedure.

Egan, Hammond, and Thomson (*66*), using electron capture gas chromatography, obtained recoveries of 73 to 91% for a number of the parent organophosphates from lettuce, onions, apples, etc. The samples were blended with an ethyl methyl ketone–hexane mixture (3 + 2) and the extract was washed with sodium sulfate solution, passed through anhydrous sodium sulfate and then through an alumina or magnesia column, concentrated, and injected into the gas chromatograph. Two columns are described, and relative retention times and sensitivities for 19 organophosphate compounds are reported.

Anyone using conventional electron capture gas chromatography for determining organophosphate residues should remember that the electron capture detector is much more responsive to halogenated compounds. Chlorinated pesticide residues, if present even in trace amounts, can be mistaken for significant amounts of organophosphates with similar retention times.

Gutenmann and Lisk (*96*) took advantage of this high electron capture response to halogenated compounds in a method they used to determine ethion and malathion in solutions. They reported that organophosphates which contained methoxy or ethoxy groups reacted with HI (Zeisel alkoxyl reaction) to form methyl and ethyl iodides. These were injected into the gas chromatograph. It is pointed out that alcohols, ethers, and esters must be removed, since they also undergo the Zeisel reaction. This procedure, of course, does not identify the compound being measured other than as one containing a methoxy or ethoxy group.

Crosby and Laws (*57*) used gas chromatography as an additional cleanup step in preparing extracts for infrared determination. The entire effluent from the gas chromatograph was caught by passing it through methylene chloride. After evaporation of the methylene chloride, the residue was dissolved in carbon disulfide and the infrared spectrum was determined over the 5- to 15-micron range, using a cavity cell with beam condenser and scale expander. Good spectra were obtained with as little as 1 μg. residue. Recoveries of 50 to 80% were obtained from fruit and vegetables at 0.2 to 4.0 p.p.m. levels, and 40 to 50% at the lower limit of 0.1 p.p.m. Data are reported for 15 organophosphate compounds.

Frehse (*74*) has written a most extensive review on the use of infrared in pesticide residue analysis. Although it covers all classes of pesticides, the greatest emphasis is on organophosphates. Among the subjects discussed are extraction and cleanup procedures (where it is pointed out that thorough cleanup is indispensable), cells, solvents, analysis of solid substances, special equipment, and the infrared characteristics of organophosphate pesticides.

MacRae and McKinley (*139*) used a Solka-Floc and activated charcoal column to clean up residues prior to paper chromatography. Two systems are described which can be used to identify 13 parent organophosphates. However, when added to crop extracts, many of the compounds were not recovered.

Zadrozinska (*200*) determined parathion, methyl parathion, malathion, and diazinon in strawberries, cabbage, spinach, etc., at levels of 0.5 to 2 p.p.m. by a paper chromatographic procedure. After bromination, fluorescein was used to detect the spots on the chromatograms.

Three color reagents (metanil yellow, yellow RFS, and methyl orange) for detecting thiophosphates on paper chromatograms are reported by Dutt and Seow (*65*). Metanil yellow was found to be the best of the three when tested on parathion, malathion, diazinon, and dimethoate. The limit of detection was 1 to 2 μg.

Getz and Friedman (*83*) studied cholinesterase inhibition methods of detecting organophosphates on paper chromatograms. They developed two procedures. In one, a direct method, the developed chromatogram itself was sprayed first with enzyme-indicator solution and then with the substrate. In the other, or indirect, procedure, after a second sheet of paper had been sprayed with the enzyme indicator solution, it was placed in firm contact with the developed chromatogram and incubated for 15 minutes. Then the second sheet was treated with substrate and the spots were developed on it.

McKinley and Johal (*138*) described the use of liver esterase inhibition for detection of organophosphate spots on paper chromatograms. The substrate was 1-naphthyl acetate and the color reagent was azoene fast blue RR. About 30 organophosphate pesticides and metabolites, as well as carbaryl, were studied. Most of the compounds were detectable at levels between 0.01 and 0.50 μg.; some required as much as 3 μg.

Thin layer chromatography has also been used in the determination of organophosphates. Uchiyama and Okui (*190*) list R_f values for 14 compounds chromatographed on silica gel plates using a hexane-acetone (4 + 1) mixture as developing solvent. Bunyan (*41*) adapted both the bromophenol blue–silver nitrate reagent for thiophosphates and the cholinesterase inhibition method for use on thin layer chromatography. The bromophenol blue–silver nitrate reagent was found to be more sensitive on silica gel plates (<0.1 to 0.6 μg.) than on alumina (about 0.5 μg.). The cholinesterase inhibition technique would not work directly on the thin layer plates and required the use of sprayed paper placed in contact with the plate, similar to the method described above (*83*). Again, silica gel plates worked better than alumina.

Several procedures based upon the molybdenum blue method for determination of phosphorus after extraction, cleanup, and oxidation to inorganic phosphate have been reported. Blinn (*28*) used a Schöniger combustion flask to determine dimethoate in a number of agricultural products, with a sensitivity of 0.1 p.p.m. Brewerton (*39*) used perchloric acid digestion. Isaeva and Enoshevskaya (*109*) used a mixture of nitric and sulfuric acids and potassium permanganate as the digesting and oxidizing agent in determining a number of the organophosphates.

These last three procedures, of course, do not identify the pesticide but simply measure the total phosphorus. However, when combined with paper or thin layer chromatography, the method becomes more specific. The cleaned-up sample extracts may be chromatographed on paper or thin layer and the identity of the residue determined by the R_f of the spot. The spots then may be cut out from the paper chromatogram or scraped off the thin layer plate and total phosphorus determined to obtain a quantitative value. Ruelene in milk (*132*) and phosphamidon in vegetables and fruits (*8*) have been determined in such a manner by paper chromatography. Thin layer chromatography has been used similarly in the determination of dimethoate (*180*).

A rather novel approach for semiquantitative determination of organophosphates was taken by Bruaux, Dormal, and Thomas (*40*). It is based upon the fact that esterases from various bovine organs separate into five to seven zones when extracts of the organs are submitted to agar-gel electrophoresis on microscope slides. Total or partial disappearance of one or more of the zones occurred when organophosphates were added to the extracts prior to electrophoresis. Inhibition patterns for 14 compounds and procedures for the analysis of samples of unknown history are described. With kidney extracts, the sensitivity is reported as 0.05 p.p.m. for some of the pesticides.

ORGANOPHOSPHATES—SPECIFIC PROCEDURES

Van Middelem, Waites, and Wilson (*191*) used both electron capture gas chromatography and the dinitrochlorobenzene colorimetric method to determine dimethoate in snap beans and found results by the two procedures in good agreement. Only the parent compound was actually measured, since the oxygen analog was not recovered by the procedures.

A modification of the colorimetric method for diazinon was used by Enos and Frear (*69*) to determine dimethoate in fruits and forage. The sample was extracted with a solvent (varying with nature of sample) and, after cleanup by solvent extraction, the dimethoate was extracted from hexane with hydrobromic acid. Acid hydrolysis produced hydrogen sulfide, which was swept into a receiver containing zinc acetate and reacted with *N,N*-dimethyl-*p*-phenylenediamine hydrochloride to form methylene blue, the absorbance of which was read at 670 mμ.

Giang and Schechter (*85*) determined dimethoate in milk and various crops by a colorimetric procedure, which measured both the parent compound and its oxygen analog. The compounds were hydrolyzed with alkali to thioglycolic acid, which was reacted with sodium phospho-18-tungstate, and the absorbance was measured at 720 mμ.

Enos and Frear (*68*) used paper chromatography to determine dimethoate in milk. The dimethoate was extracted from milk with an ethyl ether–hexane mixture. After transfer to hexane, the extract was cleaned up on a Florisil column and then spotted for paper chromatography. After development the paper was sprayed with 2,6-dibromo-*N*-chloro-*p*-quinoneimine. Dimethoate showed up as a red spot. Diazinon, Guthion, Systox, Trithion, and malathion did not react.

Cerna (*49*) reports a colorimetric method, based on the Fujiwara reaction, for the determination of Dipterex (trichlorfon) in foods.

Mitsui *et al.* (*149*) describe a colorimetric method for DDVP based on an orange-red complex that is formed between DDVP and acetone in the presence of alcoholic potassium hydroxide. Absorption at 370 mμ follows the Beer-Lambert law. They report that the method is also applicable to Dipterex and Dibrom, and the procedure for Dibrom is described (*150*).

Sun and Johnson (*183*) have developed a fly bioassay procedure which can determine as little as 0.1 p.p.m. DDVP in the presence of many other insecticides.

Archer *et al.* (*9*) report a nonspecific cholinesterase inhibition procedure for the determination of ethion in olives. They used peracetic acid to oxidize the ethion because the olefinic compounds present in olives interfered with the usual bromine treatment. The method, with modified cleanups, worked well on a number of fruits and vegetables.

Graham and Orwoll (*90*) describe a procedure in which the ethion is hydrolyzed with ethanolic sodium hydroxide and the diethyl phosphorodithioic acid formed is determined spectrophotometrically as its complex copper salt absorbing at 418 mμ. To make the procedure specific for ethion, Delnav is eliminated by a mercuric chloride treatment and other phosphate pesticides are eliminated by a dilute sodium hydroxide wash. The method is reported as applicable to a number of fruits and vegetables.

Dawson, Donegan, and Thain (*61*) used electron capture gas chromatography to determine Fenitrothion [dimethyl(3-methyl-4-nitrophenyl phosphorothionate)], parathion, Chlorthion, and paraoxon in cocoa beans.

Cox (*55, 56*) studied the colorimetric procedure for Guthion in which the pesticide is hydrolyzed to anthranilic acid, diazotized, and coupled with *N*-(1-naphthyl)ethylenediamine dihydrochloride. He reports a collaborative study in which Guthion was added to various fruits and vegetables at levels of 0.3 to 1.56 p.p.m. Recoveries ranged from 53 to 137%.

Miles (*144*) describes a new and rapid colorimetric method for Guthion, Ethyl Guthion, and their oxygen analogs. It is based on the direct coupling of the pesticide with *N*-(1-naphthyl)ethylenediamine dihydrochloride in the presence of acetic and hydrochloric acids to produce a purple solution with absorption maximum at 556 mμ. The samples were extracted by blending or tumbling with chloroform and were cleaned up with an Attaclay-Celite mixture. The oxygen analogs were separated from the parent compounds on a Florisil column. Recoveries from fruits and vegetables ranged from 78 to 97%.

Frehse, Niessen, and Tietz (*75, 76*) report an infrared method for fenthion (Lebaycid) in beet leaves, lettuce, cabbage, apples, and cherries as well as olives and olive oil. After extraction and cleanup, the residue was oxidized with potassium permanganate. The sulfone band at 7.55 microns was used for quantitation and the spectrum from 7 to 11 microns for identification. A micro phosphorus determination may also be run on the cleaned-up residue.

Bates and Rowlands (*18, 19*) have studied the conventional colorimetric method for malathion. This procedure involves the alkaline decomposition of malathion to sodium dimethyldithiophosphate, which is extracted and complexed with copper. They found that many stored food products, such as citrus pulp, coconut meal, copra, flour, etc., gave troublesome emulsions unless a preliminary chromatographic cleanup on alumina or silica gel column was included. They also found that recoveries from some rice brans were low (*18*). This they believe to be due to the formation of free fatty acid in the bran during storage. Rowlands (*172*) eliminated interferences in the determination of malathion in pimento by using polyethylene-coated alumina and acid-washed alumina cleanup columns.

Fischer and Uhlich (*72*) report an infrared method for the determination of malathion in kohlrabi, lettuce, and cauliflower. The cleaned-up extracts were dissolved in carbon disulfide and the absorption band at 9.82 microns was used to determine the malathion.

Considerable work has been done on the determination of parathion and related compounds. Van Middelem, Waites, and Wilson (*192*) studied various extraction and cleanup procedures for parathion in leafy vegetables. They found blending with a mixture of isopropyl alcohol and benzene to be the preferred method of extraction. Straight tumbling with benzene gave very low recoveries. They also describe a chromatographic cleanup column which is superior to shaking the raw extract with a decolorizing mixture.

George (*82*) reports a micro method for the determination of parathion and such similar compounds as methyl parathion, binapacryl, EPN, and Guthion. This method is based on the Averell-Norris colorimetric procedure but is said to be ten times more sensitive. In the determination, Guthion was first hydrolyzed to break the nitrogen ring. Karathane and Chlorthion interfere in the method but unhydrolyzed Guthion gives only a slight color.

Coffin and McKinley (*53*) report both a colorimetric and a paper chromatographic method for parathion, methyl parathion, EPN, and their oxons. To determine the total *p*-nitrophenol, the cleaned-up extract is treated with hydrogen peroxide and potassium hydroxide and the *p*-nitrophenate is measured colorimetrically at 400 mμ. To determine individual compounds, the cleaned-up extracts are chromatographed on paper, the developed chromatogram is treated with bromine and potassium hydroxide, and the individual spots of *p*-nitrophenate are eluted and read at 400 mμ. Recoveries of 84 to 101% are reported from lettuce, strawberries, and apples at levels from 0.4 to 1.3 p.p.m. As little as 1 μg. of each compound was readily detected on the paper chromatogram, and aromatic amines did not interfere.

Kubistova (*127*) described a method for parathion and *p*-nitrophenol in animal tissue. The sample was blended with acetone, the residue transferred to chloroform, and *p*-nitrophenol extracted with a sodium carbonate solution. In the determination of total parathion and *p*-nitrophenol, a second sample was extracted and parathion was hydrolyzed to *p*-nitrophenol. The *p*-nitrophenol was determined as an indophenol blue after reduction with titanium trichloride and reaction with *o*-cresol.

Gajan (*79*) developed a polarographic method for the determination of parathion. It was tested on green beans, apples, tomatoes, broccoli, spinach, and brussels sprouts and was able to detect as little as 0.1 p.p.m. parathion. Methyl anthranilate and *p*-nitrophenol did not interfere.

Several methods have been proposed for the determination of phorate. Waldron *et al.* (*195*) used an improved colorimetric method in which phorate is hydrolyzed to release formaldehyde which is then reacted with chromotropic acid. Reagent and crop blanks are required, and the authors point out possible interference from formaldehyde in the air or from phosgene in the chloroform.

To determine phorate, Blinn (*29*) used thin layer chromatography with infrared or the colorimetric chromotropic acid method. The residue was oxidized by peracetic acid to the oxygen analog sulfone. By using thin layer chromatography the residue was separated from potential interfering pesticides and positively identified. A palladium chloride chromogenic agent did not interfere with the colorimetric or infrared determination of the eluted spots. Excellent infrared spectra were obtained with as little as 7 μg. by using ultramicropotassium bromide pellets and beam condenser. Blinn (*30*) later compared the ability of 12 oxidants to convert phorate to its oxygen analog sulfone. He reports that *m*-chloroperbenzoic acid worked best. He also suggests the use of silica gel thin layer plates buffered at pH 6 to prevent hydrolytic decomposition of the organophosphate esters.

Although Archer *et al.* (*10*) also used peracetic acid to oxidize phorate, the phorate was determined after oxidation by cholinesterase inhibition. Potatoes were anlyzed by extracting with chloroform and anhydrous sodium sulfate without further cleanup. Sugar beet leaf extracts were cleaned up on a sodium carbonate–Celite 545–charcoal column, and cottonseed extracts on a Florisil column.

Cholinesterase inhibition was used by Blumen (*33*) to determine Phosdrin in fruits and vegetables. A modification of the procedure in which unhydrolyzed acetylcholine is converted to hydroxamic acid and reacted with ferric chloride to form a red complex was studied collaboratively. Recoveries from apples, cabbage, and tomatoes ranged from 70 to 117% at levels of 0.164 and 0.328 p.p.m.

Claborn and Ivey (*51*) report a colorimetric method for determining Nemacide (VC-13) and ronnel in animal tissue. After extraction and cleanup, the pesticides are hydrolyzed and the resulting chlorophenols are steam-distilled and reacted with 4-aminoantipyrine. The resulting color is extracted into a nitromethane-pyridine mixture and read at 490 mμ. Sensitivity is estimated to be 0.05 p.p.m. Teasley (*186*) used a different version of the colorimetric procedure to determine Nemacide [*O*,*O*-diethyl O-(2,4-dichlorophenyl)-phosphorothioate] in fruits and vegetables. The method was subjected to collaborative study, and although three collaborators obtained fair results, two others were unable to do so. Magat (*140*) describes a modified method for ronnel in meat in which the ronnel is also hydrolyzed and the trichlorophenol steam-distilled, but, in place of using a colorimetric determination, the trichlorophenol is determined directly by ultraviolet spectroscopy at 315 mμ. This method, however, is not applicable to samples containing less than 1 p.p.m.

Adams, Anderson, and McDougall (*5*) report a paper chromatographic method for determining Systox (demeton) and its toxic metabolites. An ethanol solution of the extracts is cleaned up on a column of acid-washed alumina and then is chromatographed on silicone-treated paper. The paper is sprayed with potassium permanganate and treated with potassium hydroxide. Systox and its most important metabolites form *O*,*O*-diethylphosphorothioic acid which is then detected by spraying with 2,6-dibromo-*N*-chloro-*p*-quinoneimine. This procedure is said to have a sensitivity of 0.3 p.p.m. and to distinguish residues of Systox and its metabolites in the presence of other organophosphate pesticides.

Trotsenko (*189*) describes a method for detecting Systox in air. The method is based on the ability of the thiol isomer to extinguish the fluorescence of eosin. Geldmacher-Mallinckrodt and Weigel (*81*) studied the reaction of the hydrolysis products of Systox and Meta-Systox with heavy metals. They suggest the use of copper and cobalt solutions as spray reagents after separation of the compounds by thin layer chromatography.

A spectrophotofluorometric method for Zinophos and its oxygen analog is described by Kiigemagi and Terriere (*115*). After extraction and cleanup, the residue is hydrolyzed and washed with strong alkali. It is then activated at 315 mμ and the fluorescence measured at 375 mμ. The method was tried on a number of fruits and vegetables and is said to have a sensitivity of 0.05 p.p.m.

CARBAMATES

A general infrared method for the determination of *N*-methyl carbamates in plants has been described by Niessen and Frehse (*158*). Samples were extracted by blending with acetone. Interfering plant material was precipitated with an ammonium chloride–phosphoric acid coagulating solution and, after additional cleanup on alumina, the infrared spectrum from 2.95 to 2.83 microns was recorded. The absorption at 2.88 microns due to the N—H stretching vibration was used for quantitation. As little as 0.2 p.p.m. of the pesticides could be determined.

After the infrared determination, the carbon disulfide solution was used for thin layer chromatography on alumina

G, which served to determine the identity of the pesticide. R_f values are listed for seven compounds.

An infrared method was used by Ferguson *et al.* (*71*) to determine CIPC in white potatoes. After extraction and cleanup the residue was dissolved in carbon disulfide and the infrared spectra were obtained of the solution in 0.5-mm. cells. Peaks at 1110 and 1210 cm.$^{-1}$ were used for calculation. Monuron and diuron did not interfere and the three compounds could be distinguished by their infrared spectra. The method was used on samples which contained between 2 and 15 p.p.m. CIPC. To determine CIPC in milk and urine, Gard and Ferguson (*80*) used modifications of other methods. The CIPC was hydrolyzed; the 3-chloroaniline was distilled, diazotized, and coupled with *N*-(1-naphthyl)-ethylene diamine dihydrochloride. In order to obtain consistent low blanks, it was necessary to add formalin to the urine and to age it for 48 hours prior to analysis.

Hardon, Brunink, and Van der Pol (*102*) made use of similar diazotization and coupling to determine dichloran (2,6-dichloro-4-nitroaniline), a fungicide. Although not a carbamate, it is listed here since it can, if present, interfere in the determination of some of the carbamates.

Johnson (*110*) conducted a collaborative study of the colorimetric method for carbaryl. After minor modifications were made to improve the method, an additional collaborative study was run (*111*) on samples of apples and lettuce. Recoveries averaged 87.8%.

Chiba and Morley (*50*) introduced a rapid thin layer chromatographic screening procedure for carbaryl without any prior cleanup. The sample was extracted by blending with methylene chloride and anhydrous sodium sulfate, evaporated, dissolved in petroleum ether, and spotted on silica gel plates. After development, the plates were sprayed with methanolic sodium hydroxide and the hydrolyzed 1-naphthol was coupled by spraying with a solution of *p*-nitrobenzene diazonium fluoborate. The authors note that with suitable cleanup much lower amounts of carbaryl can be detected.

Bracha (*37*) used a different diazonium salt in the determination of *O* - isopropoxyphenyl - *N* - methylcarbamate. For the determination of residues on various surfaces, he coupled the hydrolyzed insecticide with diazotized 3-nitroaniline-4-sulfonic acid and measured the absorbance at 490 mμ. The developed color was very stable in water. This method has been adapted for the determination of carbaryl, Isolan, Pyrolan, Dimetilan, and Hercules AC-5727 (*m*-isopropylphenyl-*N*-methylcarbamate).

Marquardt and Luce (*142*) report the use of a new color reagent in determining Zectran in peaches and cottonseed. The Zectran was extracted from the sample and hydrolyzed to yield 4-dimethylamino-3,5-xylenol, which then was reacted with luteoarsenotungstic acid. Absorbance was measured at 700 mμ. The luteoarsenotungstic acid is said to be highly specific for 4-dimethylamino-3,5-xylenol.

Cullen (*58*) modified the standard procedure for dithiocarbamates. The residue was decomposed directly on the crop and the evolved carbon disulfide was collected and reacted with a solution of cupric acetate and diethanolamine in ethanol. Absorbance was measured at 435 mμ. Cullen points out that the dithiocarbamates decompose very quickly when in a slurry of a crop or in contact with slightly polar solvents. Samples should either be analyzed immediately after harvest or frozen for storage. The method was tested on ferbam, ziram, maneb, zineb, thiram, and metiram.

DINITRO COMPOUNDS

From a study of methods for determining the dinitro compounds, Boggs (*34*) concludes that the paper chromatographic procedure is still the best general method. He lists R_f values for six compounds for both the aqueous and nonaqueous systems.

Potter (*168*) determined Dinoseb in potatoes by measuring its absorbance in ethyl methyl ketone at 379 mμ after extraction and cleanup.

Abbott and Thomson (*2*, *3*) used a wedge-layer type of plate chromatography as cleanup in the determination of Dinoseb in a number of fruits and vegetables. The plates were coated with a layer of silica gel-kieselguhr, which varied in thickness from 2 mm. at one edge to 0.1 mm. on the opposite edge. The sample extract was applied as a streak near the thick edge and the plate was developed. The yellow Dinoseb band was then scraped off; the pesticide was eluted with a solvent and determined by infrared or gas chromatography or colorimetrically by the method of Potter (*168*) described above.

Kilgore and Cheng (*116*) note that Karathane dissolved in *N*,*N*-dimethylformamide gives a strong yellow color without the addition of alkali. They used this phenomenon as a basis for the determination of Karathane in fruit. A hexane extract of the sample was cleaned up, either on a Florisil column or by washing with concentrated sulfuric acid, and evaporated. The residue was dissolved in *N*,*N*-dimethylformamide and absorbance read at 444 mμ. A sensitivity of about 0.05 p.p.m. was attained.

Heinisch and Panser (*105*) report a method for dinitro-*o*-cresol in plants used for fodder. A dilute sodium hydroxide extract of the sample was acidified and extracted with petroleum ether. The solvent was evaporated and the residue was dissolved in 5 ml. of ethanol and treated with 0.5 ml. of propanol and 2 drops of 10% aqueous potassium cyanide to produce an orange color, which was measured. The method is said to be good for residues as low as 0.1 p.p.m.

FUNGICIDES

Gunther, Blinn, and Barkley (*92*) describe a procedure for determining biphenyl and *o*-phenylphenol in citrus fruit. The sample was blended with water and the residues were isolated by steam distillation into cyclohexane. After separation, the *o*-phenylphenol was coupled with *p*-nitrobenzenediazonium fluoborate and determined colorimetrically at 540 mμ. Biphenyl was determined directly by measurement of its absorbance at 248 mμ.

Souci and Maier-Haarlaender (*179*) used a similar procedure for biphenyl but modified the steam distillation apparatus. Rajzman (*169*) reports a method for biphenyl in citrus fruit based upon the blue color given by biphenyl with sulfuric acid and traces of formaldehyde and ferric iron. The absorbance was measured at 610 mμ. There is no interference from *o*-phenylphenol; it does give a pink color but this disappears during treatment with sulfuric acid.

Vogel and Deshusses (*194*) used steam distillation to separate *o*-phenylphenol from citrus fruit. The *o*-phenylphenol was then reacted with 2,6-dibromoquinone-chloroimide and absorbance was measured at 619 mμ.

To determine diphenylamine in apples, Gutenmann and Lisk (*95*) used electron capture gas chromatography. The residue was extracted and brominated to form what was believed to be a hexabromo derivative of diphenylamine, which was then injected into the gas chromatograph. Solvents were redistilled and contact with rubber, which might contain diphenylamine, was avoided.

Anderson and Adams (*7*) report a colorimetric method for the determination of Dexon (*p*-dimethylaminobenzenediazo sodium sulfonate) in corn, cottonseed, and several other crops. The sample was blended with 1% sodium sulfite and the Dexon was isolated by dialysis. The Dexon was then reacted with resorcinol and sodium hydroxide and irradiated with light from two projection spotlights to produce a yellow color read at 450 mμ.

Pasarela (*164*) conducted a collaborative study of the colorimetric method [Steller *et al.*, *J. Agr. Food Chem.* **8**, 460 (1960)] for dodine in fruit at levels of 0.1 to 10.4 p.p.m. Recoveries ranged from 64 to 119%, with most values falling between 80 and 110%.

Kleinman (*122*) conducted a collaborative study of the colorimetric method for glyodin in pears and peaches. Recoveries averaged about 88% for pears and 85% for peaches. Two collaborators, however, reported difficulties with peaches.

Niessen, Frehse, and Tietz (*159*) developed a quantitative procedure for Fungilon (Bayer 32394) residues on apples, using a microtitration in a two-phase chloroform–water system. Apples were stripped with chloroform, waxes were removed by precipitation from cold methanol solution, and the extract was cleaned up on an alumina column. The Fungilon then was titrated with Aerosol OT (dioctylsodium-sulfosuccinate), with methylene blue as indicator. At the end point, color intensity was equal in the two layers. Glyodin and dodine are reported to interfere.

MISCELLANEOUS PESTICIDES

Analytical methods have been reported for a number of herbicides and other growth regulators in addition to those discussed above.

HEH (β-hydroxyethylhydrazine), also known as "Omaflora," is used to induce flowering in pineapples. Thomas and Ackermann (*187*) have developed a colorimetric method for its determination. After extraction with water and removal of interfering color pigments with ion exchange resins, the HEH is reacted with cinnamaldehyde to produce a yellow color which is read at 420 mμ.

Fletcher and Zalik (*73*) developed a method for 3-indoleacetic acid in which a methanolic extract of the plant material was chromatographed on paper and part of the chromatogram was sprayed with a chromogenic reagent to locate the indoleacetic acid. The corresponding R_f region from the unsprayed area was eluted with methanol and the ultraviolet spectrum was determined. Absorbance at 280 mμ was used for quantitative determination.

Lane (*129*) conducted a collaborative study of the colorimetric method for maleic hydrazide in potatoes. Recoveries were satisfactory.

Zweig *et al.* (*201*) developed a method for the determination of naphthaleneacetic acid in olives, using gas chromatography as part of the cleanup procedure. The olives were blended with chloroform and hydrochloric acid and the extract was passed through alumina and silica gel columns. The residue then was methylated with diazomethane and injected into the gas chromatograph. Fractions were collected. The eluate was nitrated and naphthaleneacetic acid was determined from the absorbance at 360 mμ.

Young, Shimabukuro, and Aono (*199*) determined naphthaleneacetic acid in pineapples by its ultraviolet absorbance after eliminating interferences by oxidation with potassium permanganate.

Petunova and Martinson (*165*) based their method for simazine in plant tissue on the ultraviolet absorbance of hydroxysimazine. After extraction and cleanup, the simazine was treated with sulfuric acid and hydrolyzed to hydroxysimazine. The absorbance then was measured at 225, 240, and 255 mμ.

Benfield and Chilwell (*25*) have proposed a method for determining the *s*-triazines in soil and in crops by gas chromatography of the cleaned-up extract. They used a 4-foot column packed with 0.1% ethylene glycol adipate polyester on glass beads. An unusual feature of their method was the addition of a second related triazine to the sample as an internal standard before extraction. Final determination involved only the ratio between the amounts of the two components present.

Blinn and Gunther (*32*) developed a procedure for distinguishing between residues of Aramite and OW-9 in foodstuffs. The two acaricides have similar structures, and OW-9 responds to the usual colorimetric procedure for Aramite. To distinguish between them, Blinn and Gunther used gas and thin layer chromatography as well as gas chromatography of their parent carbinols after hydrolysis.

Tietz *et al.* (*188*) made use of a red nickel chelate complex formed with ammonia to determine Eradex (2,3-quinoxalinedithiol cyclic trithiocarbonate) in fruit. Absorbance was measured at 530 mμ. Havens, Adams, and Anderson (*103*) used a similar reaction to determine Morestan (6-methyl-2,3-quinoxalinedithiol cyclic carbonate) in apples and pears. They read the absorbance at 540 mμ.

Sinclair, Lindgren, and Forbes (*178*) determined ethylene chlorobromide, using the procedure of Sinclair *et al.* for ethylene dibromide [*J. Econ. Entmol.* **55,** 236 (1962)]. This procedure consisted of steam distillation, alkaline hydrolysis, and determination of the bromide. It was pointed out that since ethylene chlorobromide undergoes degradation in products, inorganic bromides should also be determined.

Kimura and Miller (*117*) modified the colorimetric method to determine metaldehyde in plant material. Emulsification problems were resolved by passing the extract through a Florex column. Objectionable interfering colors were eliminated by evaporating the chloroform extract to dryness.

After minor modifications, the ultraviolet method for nicotine in fruits and vegetables was studied collaboratively by Martin and Schwartzman (*143*). Recoveries from apples, cabbage, spinach, and mustard greens at 1.4 to 2.4 p.p.m. levels ranged from 84 to 120%.

Munday (*155*) conducted a collaborative study in which the A.O.A.C. method for piperonyl butoxide was tested on a number of processed grain products. It was found that plant extractives gave an abnormal brown color and interfered in the determination.

Hoffman and Gordon (*107*, *108*) studied the A.O.A.C. colorimetric methods for arsenic and found that the arsine–molybdenum blue method gave slightly better reproducibility than did the silver diethyldithiocarbamate procedure, although both were suitable for determining arsenic in foods. They report that antimony does not interfere with the arsine–molybdenum blue method and that its interference with the silver dithiocarbamate method can be prevented by adding more stannous chloride to the generating mixture.

Methods for cyanide have been reviewed by Bark and Higson (*15*) who compare and evaluate the various procedures. Jones and Schwartzman (*112*) report a rapid method for determining mercury in wheat containing treated seed kernels. The treated kernels were picked out visually under ultraviolet light and burned in a Schöniger combustion flask. Mercury was determined with dithizone. Good agreement with the official A.O.A.C. method is claimed. An analysis can be completed in about two hours.

Pickard and Martin (*167*) describe a method for determining mercury in soil. The sample is digested with sulfuric and nitric acids and selenium, and the mercury is distilled from boiling sulfuric acid with hydrogen chloride gas. After treatment with EDTA and sodium thiosulfate, the mercury is determined with dithizone.

Phillips, Bowman, and Schultheiz (*166*) have developed a screening procedure, the main purpose of which is to single out samples that may contain overtolerance residues. Bioassay, organic chlorine, and acetylcholinesterase inhibition determinations were run on the same extract. Comparison of various ratios provided the basis for characterization and estimation of most insecticides that inhibited cholinesterase or that contained chlorine.

Polarography has come into wider use in residue analysis work. Davidek and Janicek (*60*) and Kosmatyi and Shlyapak (*123*) used polarography to determine DDT, and Gajan (*79*) made use of the technique to determine parathion. Nangniot and Dardene (*156*) describe three polarographic methods for determining captan and folpet (Phaltan) on plants.

Veksler and Tsukervanik (*193*) list a number of defoliants which can be determined quantitatively by polarography. They state that there is a relationship between the polarographic behavior of the compounds and their activities as defoliants.

Morris and Haenni (*154*) determined the infrared spectra (2 to 35 microns) of 24 pesticides, using potassium bromide disks. They discuss the relation of absorption band to structure and note the maxima of analytical significance.

Guillemin (*91*) separated the isomers of BHC by gas chromatography, using a 3 m. × 6 mm. stainless steel column packed with 40- to 60-mesh glass beads coated with 0.25% polypropylene glycol Niax 1025.

In spite of advances in instrumentation, bioassay methods continue to have their uses. Sum *et al.* (*184*) discuss factors that may affect results and suggest precautions to be taken in bioassays. Weinmann (*197*) describes methods of purifying the extracts and evaluating results. Funderburk and Lawrence (*78*) determined Diquat and Paraquat by measuring their bleaching effect on duckweed (*Lemna* minor L.). as little as 0.0005 p.p.m. Diquat or 0.00075 p.p.m. Paraquat can be detected.

Two methods make use of newer techniques to accomplish the familiar determination of organic chlorides in fat. Krzeminski and Landmann (*125*) used sodium in liquid ammonia to release the chloride from pesticide residues and then determined the chloride potentiometrically. Schmitt and Zweig (*174*) used neutron activation to determine total organic chloride in butter fat. The sensitivity is reported to be 10 p.p.b. total organic chloride and the time of analysis to be less than 1 hour per sample, which can be reduced considerably if many specimens are processed simultaneously.

BIBLIOGRAPHY

(1) Abbott, D. C., Egan, H., Hammond, E. W., Thomson, J., *Analyst* **89**, 480 (1964).
(2) Abbott, D. C., Thomson, J., *Ibid.*, p. 613.
(3) Abbott, D. C., Thomson, J., *Chem. Ind.* (*London*) **12**, 481 (1964).
(4) Ackermann, H. J., Carbone, L. J., Kuchar, E. J., *J. Agr. Food Chem.* **11**, 297 (1963).
(5) Adams, J. M., Anderson, C. A., McDougall, D., *Ibid.*, p. 178.
(6) Albert, R. A., *J. Assoc. Offic. Agr. Chemists* **47**, 659 (1964).
(7) Anderson, C. A., Adams, J. M., *J. Agr. Food Chem.* **11**, 474 (1963).
(8) Anliker, R., Menzer, R. E., *Ibid.*, p. 291.
(9) Archer, T. E., Winterlin, W. L., Zweig, G., Beckman, H. F., *Ibid.*, p. 471.
(10) Archer, T. E., Zweig, G., Winterlin, W. L., Francis, E. K., *Ibid.*, p. 58.
(11) Bache, C. A., *J. Assoc. Offic. Agr. Chemists* **47**, 355 (1964).
(12) Bache, C. A., Gutenmann, W. H., Lisk, D. J., *J. Agr. Food Chem.* **12**, 185 (1964).
(13) Bache, C. A., Lisk, D. J., Loos, M. A., *J. Assoc. Offic. Agr. Chemists* **47**, 348 (1964).
(14) Baetz, R. A., *Ibid.*, p. 322.
(15) Bark, L. S., Higson, H. G., *Analyst* **88**, 751 (1963).
(16) Barney, J. E., II, Stanley, C. W., Cook, C. E., ANAL. CHEM. **35**, 2206 (1963).
(17) Barry, H. C., Hundley, J. G., Pesticide Analytical Manual (U. S. Dept. of Health, Education, and Welfare, Food and Drug Admin.), Vol. 1 (1963).
(18) Bates, A. N., Rowlands, D. G., *Analyst* **89**, 286 (1964).
(19) *Ibid.*, p. 288.
(20) Beckman, H. F., Berkenhotter, P., ANAL. CHEM. **35**, 242 (1963).
(21) Beckman, H., Bevenue, A., *J. Agr. Food Chem.* **11**, 479 (1963).
(22) *Ibid.*, **12**, 183 (1964).
(23) *Ibid.*, p. 245.
(24) Beckman, H., Bevenue, A., *J. Chromatog.* **10**, 233 (1963).
(25) Benfield, C. A., Chilwell, E. D., *Analyst* **89**, 475 (1964).
(26) Bevenue, A., Zweig, G., Nash, N. L., *J. Assoc. Offic. Agr. Chemists* **45**, 990 (1962).
(27) *Ibid.*, **46**, 881 (1963).
(28) Blinn, R. C., *J. Agr. Food Chem.* **12**, 337 (1964).
(29) Blinn, R. C., *J. Assoc. Offic. Agr. Chemists* **46**, 952 (1963).
(30) *Ibid.*, **47**, 641 (1964).
(31) Blinn, R. C., Gunther, F. A., *Ibid.*, p. 191.
(32) *Ibid.*, p. 204.
(33) Blumen, N., *Ibid.*, **47**, 272 (1964).
(34) Boggs, H. M., *Ibid.*, p. 346.
(35) Bonelli, E. J., Hartmann, H., Dimick, K. P., *J. Agr. Food Chem.* **12**, 333 (1964).
(36) Bosin, W. A., ANAL. CHEM. **35**, 833 (1963).
(37) Bracha, P., *J. Agr. Food Chem.* **12**, 461 (1964).
(38) Breidenbach, A. W., Lichtenberg, J. J., Henke, C. F., Smith, D. J., Eichelberger, Jr., J. W., Stierli, H., *U. S. Dept. of Health, Education, and Welfare, Public Health Service Pub.* No. 1241 (1964).
(39) Brewerton, H. V., *N. Z. J. Sci.* **6**, 418 (1963).
(40) Bruaux, P., Dormal, S., Thomas, G., *Ann. Biol. Clin.* (*Paris*) **22**, 375 (1964); *C.A.* **61**, 6303a (1964).
(41) Bunyan, P. J., *Analyst* **89**, 615 (1964).
(42) Burchfield, H. P., Rhoades, J. W., Wheeler, R. J., Division of Agricultural and Food Chemistry of American Chemical Society, 148th Meeting, ACS, Chicago, Ill., September 1964.
(43) Burke, J., *J. Assoc. Offic. Agr. Chemists* **46**, 198(1963).
(44) Burke, J., Giuffrida, L., *Ibid.*, **47**, 326 (1964).
(45) Burke, J., Holswade, W., *Ibid.*, p. 845.
(46) Burke, J., Mills, P. A., *Ibid.*, **46**, 177 (1963).
(47) Butler, L. I., Maitlen, J. C., Fahey, J. E., *J. Agr. Food Chem.* **10**, 479 (1962).
(48) Carey, W. F., *J. Assoc. Offic. Agr. Chemists* **46**, 876 (1963).
(49) Cerna, V., *Nahrung* **7** (1) 60–6 (1963); *C.A.* **59**, 6895c (1963).
(50) Chiba, M., Morley, H. V., *J. Assoc. Offic. Agr. Chemists* **47**, 667 (1964).
(51) Claborn, H. V., Ivey, M. C., *Ibid.*, p. 871.
(52) Coakley, J. E., Campbell, J. E., McFarren, E. F., *J. Agr. Food Chem.* **12**, 262 (1964).
(53) Coffin, D. E., McKinley, W. P., *J. Assoc. Offic. Agr. Chemists* **46**, 223 (1963).
(54) Coffin, D. E., Savary, G., *Ibid.*, **47**, 875 (1964).
(55) Cox, W. S., *Ibid.*, **46**, 229 (1963).
(56) *Ibid.*, **47**, 280 (1964).
(57) Crosby, N. T., Laws, E. Q., *Analyst*, **89**, 319 (1964).
(58) Cullen, T. E., ANAL. CHEM. **36**, 221 (1964).
(59) Daoud, N. H., Luh, B. S., *Fruchtsaft-Ind.* **7**, 33 (1962); *C.A.* **60**, 16412a (1964).
(60) Davidek, J., Janicek, G., *Z. Anal. Chem.* **194**, 431 (1963); *C.A.* **59**, 4494 (1963).
(61) Dawson, J. A., Donegan, L., Thain, E. M., *Analyst* **89**, 495 (1964).
(62) De Faubert–Maunder, M. J., Egan, H., Godly, E. W., Hammond, E. W., Roburn, J., Thomson, J., *Ibid.*, p. 168.
(63) De Faubert–Maunder, M J., Egan, H., Roburn, J., *Ibid.*, p. 157.
(64) Dimick, K. P., Hartmann, H., *Residue Rev.*, Vol. 4, Academic Press, Inc., New York, 1963.
(65) Dutt, M. C., Seow, P. H., *J. Agr. Food Chem.* **11**, 467 (1963).
(66) Egan, H., Hammond, E. W., Thomson, J., *Analyst* **89**, 175 (1964).
(67) Eidelman, M., *J. Assoc. Offic. Agr. Chemists* **46**, 182 (1963).
(68) Enos, H. F., Jr., Frear, D. E. H., *J. Agr. Food Chem.* **10**, 447 (1962).
(69) Enos, H. F., Jr., Frear, D. E. H., *Ibid.*, **12**, 175 (1964).
(70) Espadas, R. S., Loaeza, J. O., (Comision Nacl. Erradicacion Paludismo), *CNEP Bol.* **4**, No. 2, 86 (1960); *C.A.* **58**, 10673f (1963).
(71) Ferguson, C. E., Jr., Gard, L. N., Shupe, R. H., Dress, K. W., *J. Agr. Food Chem.* **11**, 428 (1963).
(72) Fischer, W., Uhlich, U., *Nachrbl. Deut. Pflanzenschutzdienst* (*Brunswick*) **13**, 28 (1961); *C.A.* **59**, 8042e (1963).
(73) Fletcher, R. A., Zalik, S., *Nature* **199**, 903 (1963).
(74) Frehse, H., *Pflanzenschutz-Nachricten "Bayer"* **16**, 182 (1963–64).
(75) Frehse, H., Niessen, H., Tietz, H., *Ibid.*, **15**, 148 (1962–63).
(76) *Ibid.*, p. 160.
(77) Friestad, H. O., ANAL. CHEM. **35**, 1012 (1963).
(78) Funderburk, Jr., H. H., Lawrence, J. M., *Nature* **199**, 1011 (1963).
(79) Gajan, R. J., *J. Assoc. Offic. Agr. Chemists* **46**, 216 (1963).
(80) Gard, L. W., Ferguson, Jr., C. E., *J. Agr. Food Chem.* **11**, 234 (1963).
(81) Geldmacher-Mallinchrodt, M., Weigel, U., *Arch. Toxikol.* **20**, 114 (1963); *C.A.* **59**, 10708b (1963).
(82) George, D. A., *J. Assoc. Offic. Agr. Chemists* **46**, 960 (1963).
(83) Getz, M. E., Friedman, S. J., *Ibid.* p. 707.
(84) Getzendaner, M. E., *Ibid.*, p. 269.
(85) Giang, P. A., Schechter, M. S., *J. Agr. Food Chem.* **11**, 63 (1963).
(86) Giuffrida, L., *J. Assoc. Offic. Agr. Chemists* **47**, 293 (1964).
(87) Gordon, C. F., Haines, L. D., Martin, J. J., *J. Agr. Food Chem.* **11**, 84 (1963).
(88) Goulden, R., Goodwin, E. S., Davies, L., *Analyst* **88**, 941 (1963).
(89) *Ibid.*, p. 951.
(90) Graham, J. R., Orwoll, E. F., *J. Agr. Food Chem.* **11**, 67 (1963).
(91) Guillemin, C. L., *Anal. Chim. Acta* **27**, 213 (1962); *C.A.* **58**, 889a (1963).
(92) Gunther, F. A., Blinn, R. C., Barkley, J. H., *Analyst* **88**, 36 (1963).
(93) Gutenmann, W. H., Lisk, D. J., *J. Agr. Food Chem.* **11**, 301 (1963).
(94) *Ibid.*, p. 304.
(95) *Ibid.*, p. 468.
(96) *Ibid.*, p. 470.
(97) *Ibid.*, **12**, 46 (1964).

(98) Gutenmann, W. H., Lisk, D. J., *J. Am. Water Works Assoc.* **56,** 189 (1964).

(99) Gutenmann, W. H., Lisk, D. J., *J. Assoc. Offic. Agr. Chemists* **46,** 859 (1963).

(100) *Ibid.,* **47,** 353 (1964).

(101) Hardin, L. J., Sarten, C. T., *Ibid.,* **45,** 988 (1962).

(102) Hardon, H. J., Brunink, H., Van der Pol, E. W., *Deut. Lebensm.-Rundschau* **60,** 67 (1964); *C.A.* **61,** 7601c (1964).

(103) Havens, R., Adams, J. M., Anderson, C. A., *J. Agr. Food Chem.* **12,** 247 (1964).

(104) Heinisch, E., Neubert, P., *J. Prakt. Chem.* **22,** 267 (1963); *C.A.* **60,** 7385f (1964).

(105) Heinisch, E., Panser, G., *Nachrbl. Deut. Pflanzenschutzdienst (Berlin)* **17,** 85 (1963); *C.A.* **59,** 15851d (1963).

(106) Henderson, J. L., *J. Assoc. Offic. Agr. Chemists* **46,** 209 (1963).

(107) Hoffman, I., Gordon, A. D., *Ibid.,* p. 245.

(108) *Ibid.,* **47,** 629 (1964).

(109) Isaeva, G. Ya., Enoshevskaya, K. K., *Vopr. Pitaniya* **22** (3) 88, (1963); *C.A.* **59,** 12080b (1963).

(110) Johnson, D. P., *J. Assoc. Offic. Agr. Chemists* **46,** 234 (1963).

(111) *Ibid.,* **47,** 283 (1964).

(112) Jones, L., Schwartzman, G., *Ibid.,* **46,** 879 (1963).

(113) Kahn, L., Wayman, C. H., ANAL. CHEM. **36,** 1340 (1964).

(114) Kawashiro, I., Hosogai, Y., *Shokuhin Eiseigaku Zasshi* **5,** 54 (1964); *C.A.* **61,** 6262c (1964).

(115) Kiigemagi, U., Terriere, L. C., *J. Agr. Food Chem.* **11,** 293 (1963).

(116) Kilgore, W. W., Cheng, K. W., *Ibid.,* p. 477.

(117) Kimura, Y., Miller, V. L., *Ibid.,* **12,** 249 (1964).

(118) Kirkland, J. J., Pease, H. L., *Ibid.,* p. 468.

(119) Klayder, T. J., *J. Assoc. Offic. Agr. Chemists* **46,** 241 (1963).

(120) Klein, A. K., Watts, J. O., *Ibid.,* **47,** 311 (1964).

(121) Klein, A. K., Watts, J. O., Damico, J. N., *Ibid.,* **46,** 165 (1963).

(122) Kleinman, A., *Ibid.,* p. 238.

(123) Kosmatyi, E. S., Shlyapak, S. I., *Vopr. Pitaniya* **22,** (3) 83 (1963); *C.A.* **60,** 1035g (1964).

(124) Kovacs, Jr., M. F., *J. Assoc. Offic. Agr. Chemists* **46,** 884 (1963).

(125) Krzeminski, L. F., Landmann, W. A., *J. Agr. Food Chem.* **11,** 81 (1963).

(126) Krzeminski, L. F., Landmann, W. A., *J. Chromatog.* **10,** 515 (1963).

(127) Kubistova, J., *Pracovni Lekar* **15,** 108 (1963); *C.A.* **59,** 10682e (1963).

(128) Lampert, L. M., *J. Dairy Sci.* **47,** 1013 (1964).

(129) Lane, J. R., *J. Assoc. Offic. Agr. Chemists* **46,** 261 (1963).

(130) Langlois, B. E., Stemp, A. R., Liska, B. J., *J. Agr. Food Chem.* **12,** 243 (1964).

(131) Langlois, B. E., Stemp, A. R., Liska, B. J., *J. Dairy Sci.* **46,** 854 (1963).

(132) Leahy, J. S., Taylor, T., *Analyst* **88,** 882 (1963).

(133) Lovelock, J. E., ANAL. CHEM. **35,** 474 (1963).

(134) Lykken, L., *Residue Rev.* **3,** 19, Springer-Verlag, Berlin-Gottingen-Heidelberg (1963).

(135) McCully, K. A., McKinley, W. P., *J. Assoc. Offic. Agr. Chemists* **47,** 652 (1964).

(136) *Ibid.,* p. 859.

(137) McKinley, W. P., Coffin, D. E., McCully, K. A., *Ibid.,* p. 863.

(138) McKinley, W. P., Johal, P. S., *Ibid.,* **46,** 840 (1963).

(139) MacRae, H. F., McKinley, W. P., *J. Agr. Food Chem.* **11,** 174 (1963).

(140) Magat, A., *Bull. Assoc. Franc. Ingrs., Chimistes Techniciens Ind. Cuir Doc. Inform. Centre Tech. Cuir* **25,** 291 (1963); *C.A.* **59,** 9238g (1963).

(141) Maitlen, J. C., Walker, K. C., Westlake, W. E., *J. Agr. Food Chem.* **11,** 416 (1963).

(142) Marquardt, R. P., Luce, E. N., *Ibid.,* p. 418.

(143) Martin, R., Schwartzman, G., *J. Assoc. Offic. Agr. Chemists* **47,** 303 (1964).

(144) Miles, J. R. W., *Ibid.,* **47,** p. 882.

(145) Mills, P. A., *Ibid.,* **44,** 171 (1961).

(146) Mills, P. A., Onley, J. H., Gaither, R. A., *Ibid.,* **46,** 186 (1963).

(147) Minyard, J. P., Jackson, E. R., *Ibid.,* p. 843.

(148) Mitchell, L. C., *Ibid.,* p. 988.

(149) Mitsui, T., Ozaki, H., Kumano, H., Sano, H., *Chem. Pharm. Bull. (Tokyo)* **11,** 619 (1963); *C.A.* **59,** 5711g (1963).

(150) Mitsui, T., Ozaki, H., Toda, E., Sano, H., *Chem. Pharm. Bull. (Tokyo)* **11,** 624 (1963); *C.A.* **59,** 5711g (1963).

(151) Moats, W. A., *J. Assoc. Offic. Agr. Chemists* **46,** 172 (1963).

(152) *Ibid.,* **47,** 587 (1964).

(153) Morley, H. V., Chiba, M., *Ibid.,* p. 306.

(154) Morris, Jr., W. W., Haenni, E. O., *Ibid.,* **46,** 964 (1963).

(155) Munday, W. H., *Ibid.,* p. 244.

(156) Nangniot, P., Dardene, G., *Bull. Inst. Agron. Stat. Rech. Gembloux* **31** (1), 110 (1963); *C.A.* **60,** 12603a (1964).

(157) Nelson, R. C., *J. Assoc. Offic. Agr. Chemists* **47,** 289 (1964).

(158) Niessen, H., Frehse, H., *Pflanzenschutz-Nachr. "Bayer,"* **16,** 205 (1963–64).

(159) Niessen, H., Frehse, H., Tietz, H., *Ibid.,* **15,** 172 (1962–63).

(160) Nikolov, N. I., Donev, L., *Zh. Analit. Khim.* **18,** 532 (1963); *C.A.* **59,** 4494g (1963).

(161) Onley, J. H., *J. Assoc. Offic. Agr. Chemists* **47,** 317 (1964).

(162) Onley, J. H., Mills, P. A., *Ibid.,* **45,** 983 (1962).

(163) Ott, D. E., Gunther, F. A., *J. Agr. Food Chem.* **12,** 239 (1964).

(164) Pasarela, N. R., *J. Assoc. Offic. Agr. Chemists* **47,** 300 (1964).

(165) Petunova, A. A., Martinson, E. E., *Fiziol. Rast.* **10,** 729 (1963); *C.A.* **60,** 11302g (1964).

(166) Phillips, W. F., Bowman, M. C., Schultheiz, R. J., *J. Agr. Food Chem.* **10,** 486 (1962).

(167) Pickard, J. A., Martin, J. T., *J. Sci. Food Agr.* **14,** 706 (1963).

(168) Potter, J. A., *Analyst* **88,** 651 (1963).

(169) Rajzman, A., *Ibid.,* p. 117.

(170) Robinson, J., Richardson, A., *Chem. Ind. (London)* **35,** 1460 (1963).

(171) Roburn, J., *Ibid.,* **38,** 1555 (1963).

(172) Rowlands, D. G., *Analyst* **89,** 498 (1964).

(173) Schafer, M. L., Busch, K. A., Campbell, J. E., *J. Dairy Sci.* **46,** 1025 (1963).

(174) Schmitt, R. A., Zweig, G., *J. Agr. Food Chem.* **10,** 481 (1962).

(175) Schwartz, N., Gaffney, H. E., Schmutzer, M. S., Stefano, F. D., *J. Assoc. Offic. Agr. Chemists* **46,** 893 (1963).

(176) Shuman, H., *Ibid.,* p. 195.

(177) Shuman, H., Collie, J. R., *Ibid.,* p. 992.

(178) Sinclair, W. B., Lindgren, D. L., Forbes, R., *J. Econ. Ent.* **57,** 346 (1964).

(179) Souci, S. W., Maier-Haarlaender, G., *Z. Lebensm.-Untersuch.-Forsch.* **119,** 217 (1963); *C.A.* **59,** 5688e (1963).

(180) Steller, W. A., Curry, A. N., *J. Assoc. Offic. Agr. Chemists* **47,** 645 (1964).

(181) Stemp, A. R., Liska, B. J., Langlois, B. E., Stadelman, W. J., *Poultry Sci.* **43,** 273 (1964).

(182) See ref. 130.

(183) Sun, Y. P., Johnson, E. R., *J. Assoc. Offic. Agr. Chemists* **46,** 524 (1963).

(184) Sun, Y. P., Johnson, E. R., Pankaskie, J. E., Earle, N. W., Sun, J. Y. T., *Ibid.,* p. 530.

(185) Taylor, A., Rea, R. E., Kirby, D. R., *Analyst* **89,** 497 (1964).

(186) Teasley, J. I., *J. Assoc. Offic. Agr. Chemists* **47,** 287 (1964).

(187) Thomas, M. P., Ackermann, H. J., *J. Agr. Food Chem.* **12,** 432 (1964).

(188) Tietz, H., Osman, M. F., Frehse, H., Niessen, H., *Pflanzenschutz-Nachr. "Bayer,"* **15,** 166 (1962–63).

(189) Trotsenko, M. A., *Novoe v Oblasti Sanit.-Khim. Analiza (Raboty po Prom.-Sanit. Khim.)* p. 175 (1962); *C.A.* **59,** 4493*g* (1963).

(190) Uchiyama, M., Okui, S., *Shokuhin Eiseigaku Zasshi* **3,** 277 (1962); *C.A.* **58,** 3839a (1963).

(191) Van Middelem, C. H., Waites, R. E., *J. Agr. Food Chem.* **12,** 178 (1964).

(192) Van Middelem, C. H., Waites, R. E., Wilson, J. W., *Ibid.,* **11,** 56 (1963).

(193) Veksler, E. A., Tsukervanik, I. P., *Uzbeksk. Khim. Zh.* **6,** No. 4, 55 (1962); *C.A.* **58,** 3834c (1963).

(194) Vogel, J., Deshusses, J., *Mitt. Gebiete Lebensm. Hyg.* **54,** 330 (1963); *C.A.* **60,** 11289f (1964).

(195) Waldron, A. C., Pasarela, N. R., Woolford, M. H., Ware, J. H., *J. Agr. Food Chem.* **11,** 241 (1963).

(196) Walker, K. C., Beroza, M., *J. Assoc. Offic. Agr. Chemists* **46,** 250 (1963).

(197) Weinmann, W., *Z. Lebensm.-Untersuch.-Forsch.* **119,** 334 (1963); *C.A.* **59,** 6896b (1963).

(198) Yip, G., *J. Assoc. Offic. Agr. Chemists* **47,** 343 (1964).

(199) Young, H. Y., Shimabukuro, S., Aono, L., *J. Agr. Food Chem.* **11,** 132 (1963).

(200) Zadrozinska, J., *Roczniki Panstwowego Zakladu Hig.* **15,** 5 (1964); *C.A.* **61,** 6262b (1964).

(201) Zweig, G., Gutnick, D. L., Gulli, R., Archer, T. E., Hartmann, H. T., *J. Agr. Food Chem.* **12,** 59 (1964).

Petroleum

C. M. Gambrill, Editor

Petroleum Chemicals Division, Ethyl Corp., Detroit, Mich.

Preface

This review covers selected papers and reports, of interest to analytical chemists in petroleum activities, which have been published in the literature since the previous review (*AA*) until early 1964. It has been prepared through the cooperation of a number of petroleum chemists as an industry effort, sponsored by the Division of Petroleum Chemistry of the American Chemical Society. The search of the literature, organizing the selected material into subject sections, and compiling the subject sections have been the efforts of experts in the field. Their suggestions and assistance aided greatly in coordinating the work of the project.

Introduction

Ralph O. Clark

Gulf Research & Development Co., Pittsburgh, Pa.

During the period covered by this review a number of meetings of interest to the petroleum analytical chemist have taken place. The annual meetings of the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy and of ASTM Committee E-14 on Mass Spectrometry continued to be of general interest. Several symposia devoted to gas chromatography were scheduled, of which the Fourth International Gas Chromatography Symposia at Michigan State University, sponsored by the Instrument Society of America, and an International Symposium on Advances in Gas Chromatography at Houston, Tex., were of particular significance. The Division of Refining of the American Petroleum Institute sponsored two technical sessions at its May 1964 meeting on instrumental techniques applied to the analysis of petroleum products, one on activation analysis and the other on nuclear magnetic resonance.

The number of textbooks on analytical chemistry continues to grow. Milner (*14A*) describes in a compact volume procedures for determining a number of trace elements in petroleum. A book by Reilley (*16A*) provides considerable information to the petroleum chemist in keeping abreast of developments in analytical chemistry and instrumentation. And one by Bobbith (*7A*) on thin-layer chromatography should be of value to those desiring to explore the potential of the technique to petroleum-derived materials. The American Society for Testing and Materials has issued three special technical publications relating to infrared (*2A*), mass (*3A*), and ultraviolet and visible (*5A*) spectra, another on standard samples for emission spectrochemical analysis (*6A*), and a manual on hydrocarbon analysis (*4A*). The proceedings of a conference on analytical methods organized by the British Non-Ferrous Metals Research Associations contains discussions on a number of techniques used by petroleum analytical laboratories (*8A*). A Russian compilation (*1A*) covers the application of spectral, electrical, chromatographic, and other approaches for the analysis of petroleum products, catalysts, and petrochemicals.

Hakala (*10A*) has reviewed some 12 techniques finding increasing application in petroleum research, and Prinzler published a review on problems associated with mineral oil analysis (*15A*). The applications of emission x-ray spectroscopy (*12A*), emission spectrographic analysis (*11A*), and thin-layer chromatography (*13A*) to the oil industry have been discussed. Szepesy and Simon (*18A*) have covered the use of gas chromatography within the petroleum industry, including process control. Bruderreck (*9A*) has pointed out the savings possible when capillary columns and flame ionization detectors are used for refinery control. And Stemberger (*17A*) has discussed the application of infrared spectroscopy for the characterization of additive types in fuels and lubricants.

Crude, Condensates, and Shale Oil

John L. Ellingboe

Marathon Oil Co., Littleton, Colo.

Kerenyi and coworkers (*24B*, *25B*) have continued their efforts to characterize the composition and properties of Hungarian crude oils. These characterizations are then correlated with optimum processing conditions. Moiseikov, Dragunskaya, and Tolstenev (*40B*) studied the composition of crude oil from the Kotur-Tepe region as to suitability for various end uses. Similar studies on crude oils from the Malgobek and Ali-Yurt Upper Cretaceous formations have been carried out by Levchenko and Bobkova (*31B*).

Makovetskiĭ and Serdyuk (*35B*) have shown that crude oils from the Kachanov formations of the northwestern zone of stepped faults of the Dnieper-Donets depression show similarities in physical and chemical properties and hydrocarbon composition within a single stratigraphic section. Analyses of crude oils from the Gulf Coast area of Louisiana and Texas have been reported by McKinney and Ferrero (*34B*). Included are analyses from 51 Louisiana fields and 116 Texas fields that produced at least 1000 barrels per day during at least one of the years 1957 through 1961.

The compositions of the tarry fractions of crude oils from the Bashkiria stratum and the coal-bearing suite in the Pokrovskoe deposit were found to be very similar by Nezhevenko and Smirnova (*45B*). All of the component parts of the tar fractions were found to be surface active. The asphaltenes had lower surface activity than any of the other tar components.

Ball, Haines, and Helm (*4B*) reported on the minor constituents of a Wilmington, Calif., crude oil. Nitrogen compounds were found to be mostly materials of high molecular weight and form the predominant group in the asphalt. As many as 19 metals were identified in the crude oil.

Stable isotopes of carbon, hydrogen, nitrogen, sulfur, and oxygen were examined by Adel'berg and Dvali (*1B*) for the problem of petroleum genesis. Sattarzade, Farzaliev, and Sattarzade (*57B*) studied the effect of temperature on the optical activity of crude oil in the presence of catalysts. The data obtained are of interest for evaluating the role of aluminosilicate rocks in oil genesis.

Relative dispersion instead of specific dispersion was used by Baxa (*5B*) to characterize various crude oils and crude oil fractions of different origin.

A sensitive test to detect naphthenic

acids in crude oil or crude oil fractions was developed by Pelcik and Zapotockeho (*47B*). Higher fatty acids interfere and must be removed.

An accelerated method for the determination of the degree of crude oil emulsification was described by Sadykhov, Frankfurt, and Abdullaev (*55B*). Kuntz and Remke (*28B*) have shown a linear relation between basic sediment and water content (b.s. + w.) and dielectric constant of crude over the range from 0 to 3% b.s. + w. The viscosities of specific crude oil were studied by Koten and Stoyanovskaya (*26B*) and Lagoutte and de Saint-Palais (*29B*).

Infrared spectroscopy continues to be used to identify types of compounds in crude oils and crude oil fractions. Serbanescu (*58B*) investigated the porphyrin, hemin, and chlorophyll content of several Romanian crude oils. The infrared spectra of tarry products from Karaton petroleum were examined by Yatsenko and Gutsalyuk (*65B*). Muradova, Sergienko and Korotkiĭ (*42B*) used both infrared and ultraviolet spectroscopy for the identification of high-molecular-weight hydrocarbons in Aligul petroleum. A comparative study of crude petroleum from Eastern Sahara was conducted by Thomas and Lagoutte (*63B*) using ultraviolet spectrophotometry. They found that crude petroleums can be identified and distinguished by the ultraviolet spectra of the 70° to 150° C. distillation fraction in a 2,2,4-trimethylpentane solution. The sensitivity of this method is good. Mukashev and Shmais (*41B*) used the fluorescence spectra of various crude oils as a means of their identification.

Silica and alumina chromatography techniques have been used by many investigators. The composition of the naphthenic and aromatic fractions of several crude oils and bitumens was studied by Rodionova, Korchagina, and Il'inskaya (*53B*) and by Rodionova, Il'inskaya, and Nadel'shtein (*51B*). Both silica and alumina separations were used. Rozmanek (*54B*) determined the per cent of aromatics present in silica and alumina fractions of medium-viscosity oils obtained from a high-sulfur crude oil. Boyd and Montgomery (*7B*) combined chromatography with activated carbon with elution chromatography on silica gel in a study of the oil component of Athabasca bitumen. The oil component represented 33% of the bitumen and on the average appeared to contain three rings per molecule.

The content of normal and branched-chain paraffins was determined in narrow boiling range fractions of Ukranium petroleums by Gutyrya *et al.* (*20B*). Molecular Sieves were used to determine the normal paraffin content in the presence of branched paraffins, naphthenes, and aromatics. Gordash *et al.* (*19B*) were also concerned with paraffin separations on narrow boiling range fractions of Mukhanov petroleum by the use of chromatography and urea complexes. Silica gel chromatography was used by Kalantar, Glazunov, and Mannafova (*22B*) to remove paraffins from the 300° to 400° C. distillation fraction of Tvimazy crude oil. Sergienko and Garbalinskiĭ (*59B*) modified the above techniques to determine the amount of cyclic hydrocarbon rings in paraffinic-cycloparaffinic hydrocarbon mixtures. Selective catalytic dehydrogenation was followed by silica gel separations. Nasirov and Ashumov (*44B*) reported on the determination of aromatic and hydroaromatic hydrocarbons in the gasoline fractions of Peschanyi and Karadag crude oils. Three distillation fractions were examined by chromatographic and spectrometric techniques.

A quantitative determination of five- and six-membered cyclanes in Satskhenis petroleum was reported by Areshidze and Khechinashvili (*2B*). The light hydrocarbon fractions, boiling ranges 60° to 200° and 200° to 250° C., were analyzed for their aromatic, hexahydroaromatic, cyclopentane, and paraffinic hydrocarbon contents.

Crystalline hydrocarbons were isolated by Sanin and Druzhinina (*56B*) from the 325° to 375° C. fraction of Romashkino crude oil in the form of urea complexes. These crystalline hydrocarbons contain about 12% cyclohexane derivatives and about 60% normal paraffins. Urea complexes were also used by Rodionova, Il'inskaya, and Nadel'shtein (*52B*), attempting to determine the nature of the saturated hydrocarbons from several crude oils and dispersed bitumens.

Sulfur compounds, hydrocarbons, and organometallic compounds were studied in regard to their distribution *vs.* boiling range by Dean and Whitehead (*9B*) on residues and distillates above kerosine from Middle East crudes and of residues from some South American crudes. Musayev *et al.* (*43B*) gave a comprehensive summary of the results of the 170° to 300° C. distillate of a representative sulfur-bearing crude oil of the USSR at the Sixth World Petroleum Congress. A similar study was made by Skidanova and Chernozhukov (*61B*) on the high-molecular-weight fractions of Kotur-Tep petroleum.

Pashaev (*46B*) found that the surface tension of crude oil fractions increases linearly with increasing boiling range. The dearomatized fractions have lower surface tensions than straight-run fractions.

Gas-liquid chromatography has become a very important technique for the analysis of petroleum fractions. The combination of gas-liquid chromatography with other methods of analysis has proved to be a powerful tool and many investigators are active in the field. Budec (*8B*) studied the chemical composition of gasoline fractions of crude oils by the use of silica gel chromatography coupled with gas-liquid chromatography. Four isoprenoid hydrocarbons were isolated from a petroleum distillate by preparative-scale gas chromatography by Bendoraitis, Brown, and Hepner (*6B*). These compounds were identified by a combination of mass, infrared, and proton magnetic resonance spectroscopy. They also offer additional insight into the origin of petroleum; their most probable precursors are plant alcohols such as chlorophyll.

Martin and Winters (*36B*) have developed a method for the determination of hydrocarbons in crude oil by capillary column gas chromatography. This method and supplementary techniques were used by Martin, Winters, and Williams (*37B*) to analyze 18 crude oils with varying geological histories. This very extensive analysis was presented at the Sixth World Petroleum Congress. An apparent correlation between composition and geological age was noted and suggestions as to the origin of petroleum were presented.

Le *et al.* (*30B*) used a combination of fractional distillation, gas-liquid chromatography, dehydrogenation, and spectrography to establish the composition of gas condensate fractions boiling below 200° C. A relatively high content of cyclohexane and aromatic hydrocarbons was noted. Naphthalene and alkylnaphthalenes in crude oils were determined by Garilli, Lombardo, and Zerbo (*16B*) using a Celite–polyethylene glycol column at 120° C.

An integrated gas chromatograph–mass spectrometer combination was used by Lindeman and LeTourneau (*33B*) for the identification of a number of individual hydrocarbons not previously reported in petroleum. The gas chromatograph–mass spectrometer combination was used to survey fractions for new components. Preparative scale gas chromatography was then used to isolate materials of interest for further study by infrared and nuclear magnetic resonance spectroscopy.

The C_{1-4} hydrocarbons of a crude oil sample were determined by gas liquid chromatography without prior distillation by Garilli, Lombardo, and Zerbo (*17B*). Mazitova, Virobyants, and Ermakova (*38B*) developed a technique for the determination of the C_2 to C_7 hydrocarbons from crude oils. The column used was 1-chloronaphthalene on diatomaceous earth with helium as the carrier gas. The samples were fractionated by distillation prior to analysis. Menez (*39B*) has also developed a technique for the quantitative analysis of the paraffins (up to

C_7) from crude oil samples taken under pressure.

Gas analyses have also been applied to the exploration for petroleum. Bailly (*3B*) analyzed gas samples from the drilling effluent of 17 California wells by gas-liquid chromatography.

The composition of shale oil has been studied extensively since the last review. Drabkin (*11B*) determined the naphthalene and ammonia content of oil shale gas. He did considerable method development work on the determination of naphthalene and methylnaphthalenes. Drakin and Babin (*12B*) developed an analytical method for the gasoline content of oil shale gas. Lille, Murd, and Kundel (*32B*) were also concerned with the analysis and composition of gas from oil shale. They developed a gas-liquid chromatographic method for the determination of hydrocarbons in shale gas.

The composition and characteristics of light fractions boiling below 65° C. from shale tars were studied by Rang, Arumeel, and Eisen (*49B*).

Infrared techniques were used by Goodspeed and Montgomery (*18B*) for the determination of methyl and methylene groups in the oil and resin fractions of Athabasca bitumen. While Polovoi (*48B*) used infrared to study polymerized shale oils, he found extensive evidence of branching. The chain-length distribution of normal alkanes in paraffins from lignite low-temperature tar and petroleum was studied by Hildebrand *et al.* (*21B*). Comparative investigations were made by means of high-temperature gas chromatography. In the normal alkanes from the lignite tar in the range C_{20} to C_{32}, the even-numbered members were present in higher concentration, while the opposite was found with the petroleum normal alkanes.

The chemical composition of the olefinic hydrocarbons in shale oil was studied by Eisen and Rang (*14B*). Fractionations and separations were done by silica gel chromatography, while physical properties, infrared, and gas-liquid chromatography were used to identify individual compounds. Twenty-six individual hydrocarbons ranging from nonane to methyl calins were identified by Eisen, Rang, and Arumeel (*15B*) in the paraffinic-naphthenic portion of the 150° to 215° C. fraction of a shale oil.

Kalde (*23B*) evaluated two methods for the determination of free formaldehyde in the products of phenol condensation of bituminous shale. The advantages and shortcomings of both methods are discussed.

A detailed review and experimental study were made by Sipovskiĭ and Kamp (*60B*) of published methods for the determination of neutral oils in the phenolate-phenol fraction of tars. They concluded that only gravimetric methods with benzene or toluene as the solvent extractors may be used for determining the amount of neutral oils needed to control and improve the dephenolization of oil shale tars with 10% aqueous sodium hydroxide. A new procedure is described for determining neutral oils in the oil shale tars.

Eisen (*13B*) separated the tar from Estonian shale into four fractions by silica gel chromatography. Further separations were then accomplished by alumina and urea. The physical and chemical characteristics of the fractions as well as the distribution of sulfur compounds were tabulated. Many individual compounds were identified.

An analytical method for the study of the thermal degradation of oil shale was developed by Smith (*62B*). A conventional assay method for determining oil yield was modified to provide for a precise determination of all products. The apparatus and procedure are described in detail and the reliability of the method was evaluated by a standard statistical treatment.

Kuiv and Gubergrits (*27B*) computed the effective heat conductivity of kukersite oil shale, using experimental data from three different methods.

The nitrogen adsorption and desorption isotherms of Green River oil shale indicate that it is a highly consolidated organic-inorganic system with no significant micropore structure, pore volume, or internal surface, as reported by Tisot (*64B*). The mineral constituents have an appreciable surface area apparently limited mainly to the external surface. Surface area data also suggest that only a small amount of the organic matter is bonded either physically or chemically to the mineral constituents.

The formation of 1-hexene, 1-heptane, 1-octene, and methyl ketones during the dry distillation of kerogen from Baltic oil shale was found by Dobryanskiĭ (*10B*). The presence of these compounds is explained by partial decomposition of hydroxy acids. Robinson *et al.* (*50B*) found that Colorado oil shale kerogen contains only small amounts of aromatic and straight-chain paraffin structures. This study showed that the kerogen is composed primarily of small, saturated ring systems connected by short aliphatic chains.

Gases (LPG)

R. S. Silas and D. E. Smith

Phillips Petroleum Co.,
Bartlesville, Okla.

THIS section covers the C_1 to C_5 hydrocarbons and mixtures of these materials which occur in petroleum or are produced in various refining operations. Paraffins, mono- and diolefins, acetylenes, and a limited number of cyclics are included, in addition to nonhydrocarbon gases such as helium, argon, oxygen, nitrogen, hydrogen, and carbon monoxide, which often occur in these hydrocarbon mixtures.

The analysis of natural gas by gas chromatography is the subject of a number of papers. Erickson (*10C*) discusses the general application. Miller (*26C*) describes the method approved by the Natural Gas Processors Association, which features the use of a synthetic blend for peak height calibration and of reverse flow for hexanes and heavier. Kniebes (*19C*) discusses the results from cooperative testing of methods for natural gas, reformed gas, and LPG. Torocheshnikov and Semenova (*38C*) describe a simple portable chromatograph for analysis of mixtures containing H_2, N_2, O_2, CO, CH_4, and CO_2, using a Janak integral detector system and a glass column filled with activated carbon. Mueller and Freund (*27C*) describe the use of gas chromatography in natural gas and petroleum prospecting and production. Other specific methods for natural gas by gas chromatography are given by Tung and Yu (*39C*), Yurovskiĭ (*42C*), and Aliev and Aminova (*1C*). The latter discuss also the analysis of cracked and other petroleum gases and employ both differential and integral (Janak-type) detector systems. Lenac-Lukacevic (*21C*) discusses gas chromatography in general with specific applications to the analysis of natural gas and catalytic cracking gases. Baranenko (*4C*) has summarized briefly the methods used in the USSR for analysis of natural gas.

A closely related analysis is that of fuel and refinery gases containing paraffins and the nonhydrocarbon gases. Colson and Brown (*7C*) describe a method for this purpose. Alumina is used for the hydrocarbons and a combination of silica, activated carbon, and 5A Molecular Sieve for the other components. Kipping and Jeffery (*18C*) use Molecular Sieve and charcoal columns to analyze fuel gas for methane and the nonhydrocarbon gases. Cieplinski, Averill, and Ettre (*6C*) describe a four-valve, two-column system for the same type of analysis. The columns are Molecular Sieve and silica gel or hexamethylphosphoramide. Two runs are needed, one for CO_2 alone. Schuster (*32C*) determines the hydrocarbons in fuel gas by infrared spectroscopy and finds that the results deviate from those obtained by Orsat analysis, which he attributes to the approximations used in the latter.

ASTM Committees D-2 and D-3 have approved a tentative method for the analysis of liquefied petroleum (LP) gases by gas chromatography (*2C*).

ASTM has also published for information only a proposed method for interconversion of the analyses of C_5 and lighter hydrocarbons to gas-volume, liquid-volume, or weight basis (*3C*).

The growing interest in helium is reflected in the number of methods published for the determination of helium in natural gas. Skarvada and Tesarik (*34C*) describe a direct gas chromatographic method using 5A Molecular Sieve at room temperature. Zielinski (*47C*) measures helium, hydrogen, and argon from a combination of analyses using Molecular Sieve and charcoal columns and nitrogen and hydrogen carrier gases. Baranenko and Burnykh (*5C*) preroast the gas sample in a quartz tube containing CuO to remove hydrogen. Graf and Toth (*12C*) adsorb natural gas on dry ice–cooled activated charcoal columns and elute the hydrogen and helium with CO_2. An additional uncooled column separated the hydrogen and helium for analysis. Demenkov (*8C*) describes a mass spectrometric method using a hot-cathode source, calibrated by the absolute sensitivity of helium. Penchev and Pencheva (*28C*) describe a manometric method. Zhukhovitskiĭ *et al.* (*44C*) report that 8 to 12% steer blood on Inzen brick will separate argon and oxygen.

The determination of the hydrocarbons present in refinery gases has been a challenge to many analysts. The large number of components from C_1 to C_5, which includes paraffins, olefins, diolefins, and acetylenes, presents a very complex mixture difficult to separate completely. Most methods employ two or more columns to make the complete analysis. Lulova *et al.* (*22C*) use silica gel wetted with dimethylacetamide, silver nitrate–ethylene glycol, and quinoline columns. Zizin and Sokolova (*48C*) use quinoline and silver nitrate–diethylene glycol for C_1 and C_5 hydrocarbons. Jarzynska and Mirecka (*16C*) use diethylene glycol and silver nitrate–polyoxyethylene glycol for C_2 to C_4 hydrocarbons. Sojak (*36C*) describes and analysis scheme which uses Janak's method of measurement and specific chemical tests for isobutene, acetylenes, and total olefins. Innes, Bambrick, and Andreatch (*15C*) employ parallel chemical absorbers combined with dual flame ionization detectors.

Koons and Walker (*20C*) use silver nitrate–benzyl cyanide for C_1 to C_5 hydrocarbons, with an alternative of dimethylsulfolane and diisopropyl phthalate columns in series. They employ small diameter (1/8-inch) columns and a flame ionization detector for faster analyses. Mazitova, Ermakova, and Virobyants (*25C*) report that eleven C_1 to C_4 hydrocarbons can be separated in one hour on 2 meters of 2% silicone oil on 20- to 50-mesh alumina. Perhaps the most promising development in this area is reported by Guiochon and Landault (*13C*), who found that a 0.4-mm. column, 7.5 meters long, packed with 0.1 to 0.12 mm. diameter activated alumina, will separate all the C_1 to C_4 olefins and paraffins plus acetylene in 6 minutes, and butadiene and the pentanes in a little longer time, with complete resolution.

The determination of specific impurities in processed refinery streams has become of special interest because of the effect of poisons on catalytic processes. Trace components in the parts per million range must be determined routinely for plant control. Heuschkel, Wolny, and Skoczowski (*14C*) determine traces of propyne and allene by separating first on a silicon column and then resolving the trapped peaks on diethylacetamide. Mamedova, Shnulin, and Portyanskiĭ (*23C*) determine allene in propane-propylene mixtures by infrared spectroscopy at 1960 cm^{-1}. A concentration range of 0.5 to 10% is linear. Pribyl and Soucek (*29C*) determine ethyne and propyne by photometric measurement of soluble acetylides. Genkin (*11C*) determines butylenes in butadiene using dimethylacetamide as the stationary phase and hydrogen saturated with the stationary phase as the carrier gas. Dementeva, Naumova, and Prokopenko (*9C*) determine the purity of isobutylene and C_2 to C_5 impurities using three columns: (1) ester of triethylene glycol and butyric acid, (2) silver nitrate and ethylene glycol, and (3) propylene carbonate. Hydrogen is used as the carrier gas. Markosov, Zaichenko, and Lityaeva (*24C*) measure trace concentrations of CO in ethylene using an activated charcoal column. Sarycheva and Pinchevakaya (*30C*) report results on the adaptation of commercial chromatographs in the USSR to the analysis of hydrocarbon gases, particularly to propane-propylene and butane-butylene fractions. Kienitz (*17C*) has reviewed macro, micro, and trace chemical and instrumental methods for about 100 inorganic and 100 organic substances boiling below 20° C.

Special techniques include the development of new detectors and special applications of gas chromatography. Seiyama *et al.* (*33C*) describe a detector which uses a semiconductive thin film of zinc oxide. The change in electrical conductivity of the film as components are adsorbed is the measured variable. The detector is about 100 times as sensitive as a conventional thermal conductivity cell. Van Luik and Rippere (*40C*) measure trace impurities in air by determining the opacity of condensation nuclei in a special cloud chamber. The components to be determined are cooled to liquid or solid submicroscopic particles and then the air is injected into the cloud chamber, which is supersaturated with moisture. The contaminants act as nuclei for droplets of water which grow to uniform size. The number of particles formed, as measured by the opacity, is a measure of the concentration. The detector will detect as few as 10,000 molecules.

Smith and Merritt (*35C*) describe a method based on the difference in mobility between electrons and negative ions. Electrons are produced by a strontium-90 source. An a.c. field is applied to outer parallel plates of a central grid chamber and the ion current is measured with an electrometer. Sensitivity for oxygen in nitrogen and hydrocarbons is 3 p.p.m. and for water in nitrogen is 1 p.p.m.

Zhdanov *et al.* (*43C*) treat borosilicate glass with 3*N* HCl to obtain a porous glass which performs better than alumina and silica gel as a column packing. A 10-meter length of 0.5-m. i.d. glass tubing treated in this manner gives satisfactory separation of C_1 to C_4 hydrocarbons.

Two relatively new techniques, stepped chromatography and vacanto chromatography, are discussed by Zhukhovitskiĭ *et al.* (*45C*, *46C*). Stepped chromatography consists of adding a large amount of sample, 100 cc. or more, and obtaining flat steps for each component. The advantage of this technique is that complete separation is not necessary and only a flat portion is required to measure the component peak quantitatively. Vacanto chromatography involves the use of the sample as reference gas and the addition of an inert gas to develop the chromatogram. This technique has many possibilities in on-stream analyzers, one of which is that the ratio of peak height to concentration is linear. The effects of varying the carrier gas can be easily studied. Calibration may be made by addition of one component to the carrier gas.

Spencer (*37C*) discusses the change in elution time of isobutane from Molecular Sieves with change in temperature of the sieves. The temperature range covered was 40° to 100°C. Apparently, the branched nature of isobutane causes it to be adsorbed only on the surface and it becomes much more sensitive to temperature changes than the other light hydrocarbons.

Schubert (*31C*) utilizes the fluorescent indicator adsorption method (FIA) to determine small concentrations of light oil in natural gas. He passes 1 to 2 liters of coal gas through a silica gel cartridge to concentrate the oil. This silica gel is then transferred to the top of the conventional FIA column and developed using Sudan red indicator.

Vigdergauz and Gol'bert (*41C*) discuss the theoretical basis and the application of reverse flow techniques for rapid chromatographic analysis.

Gaseous and Liquid Fuels

Fred. D. Tuemmler

Shell Development Co.,
Emeryville, Calif.

THIS section directs attention to the analysis of petroleum-derived engine and heating fuels and to the analysis of gas mixtures of interest to the petroleum industry. It is generally further limited to the analysis of finished commercial fuels; methods applicable to, but not specifically intended for commercial fuels, are included in the sections on hydrocarbon types, sulfur, nitrogen, oxygen, or other elements.

Advances in the field of analysis of gases have been limited in the past year or two, since the powerful tools of chromatography and spectroscopy have long since been exploited. Abel and Schmertzing (*1D*) were able to improve upon a micro parallel plate detector permitting the analysis of microliter quantities of mixtures of noncondensables and hydrocarbons boiling at the propane level and below. Gas chromatography has been exploited by Colson and Brown (*16D*) for the determination of odorants, gum formers, and other trace components in refinery tail gases entering fuel gas lines. Likhachev (*38D*) used activated carbon to separate carbon monoxide and nitrogen-oxygen peaks, in studies on the composition of combustion products of fuels.

Claytor and Jones (*15D*) have concerned themselves with the very accurate determination of hydrogen sulfide contaminant of chemical feed stack gases. Hoggan and Battles (*30D*) report on their improvements in the application of the Wickbold oxyhydrogen burner for the determination of sulfur in liquefied petroleum gases (LPG) and also in higher boiling petroleum fuels. Pom̆ykala (*52D*) describes a new type of high-pressure sampling apparatus which does not interfere with the accurate determination of sulfur.

Clarkson and Gooderham (*14D*) have made a mechanical improvement in the Haldane combustion apparatus that simplifies replacement of the easily fouled platinum wire. Boyadzhiev (*10D*), using a pneumatic continuously equilibrated bridge, operated an apparatus for the analysis of binary gas mixtures based on variation in viscosity and density of the mixture. Caffo (*12D*) reports on two very precise methods for measurement of relative viscosity of gases with respect to air.

For the now familiar fluorescent adsorption (FIA) method for the analysis of liquid fuels, a new fundamental quantity, the apparent selective adsorbent quantity (the enclosed liquid quantity per 1 gram of adsorbent) was studied and proposed by Funakubo, Taniguchi, and Uejima (*20D*); a general equation was developed which expresses the weight of adsorbent necessary for perfect separation. Morris, Shively, and Constantin (*43D*) describe improvements (ASTM D 1319) to be expected by adding a small amount of water to the silica gel adsorbent. In the manufacture of fuel gas from gas oil, carry-over light oil is to be avoided; to determine it, Schubert (*62D*) passed the fuel gas over silica gel for a given period and, using this gel as the sample, placed it in the FIA analytical column for determination of composition. Krumbholz and Jaechel (*35D*) use a high boiling residue from catalytically reformed residue to provide a good indicator for aromatics marker in the FIA method. By combining gas chromatography and molecular sieve techniques in a four-step procedure, Albert (*5D*) was able to determine in a commercial gasoline, aromatics, olefins, normal paraffins, and cyclo- and isoparaffins, thus extending the usual FIA method by one additional separation. Beggi (*6D*) showed that the product of specific gravity and refractive index provided a quickly measured value that could be used for identifying several lots of a given grade of aviation gasoline and to detect contamination during transport and storage.

The determination of lead antiknock compounds in fuels continues to interest investigators. Nagypataki and Tamási (*44D*) made a critical laboratory comparison of many methods and recommended flame-photometric and complexometric methods. ASTM Committee D-2 (*42D*) described interlaboratory tests carried out with that society's standard D 526. Using x-ray fluorescence (molybdenum anticathode at 50 kv. and 13 ma. with tellurium fluoride crystal) Campo (*13D*) was able to obtain lead analysis with an error less than 1% with an analysis speed 8 times greater than with the ASTM method. Loroue and Paul (*41D*), successfully used an oxidative extraction ($KClO_3$ in dilute nitric acid) to remove the lead from the gasoline, the lead nitrate being dissolved in water and a precipitate with sodium sulfide compared colorimetrically with standards; variability was ±0.001 ml. per liter in gasoline containing 200 ml. of TEL per liter. The same authors (*40D*) extended this procedure to detect 10 ± 1 p.p.m. of TEL in aircraft turbine fuel resulting from pipeline contamination. Bellomo and D'Amore (*7D*) also obtained the lead nitrate by extraction, measuring it by means of an automatic high frequency conductometric titration with a potassium dichromate solution. Dawson (*18D*) determined industrial tetraethyl-, tetramethyl-, and ethyl-trimethyllead mixtures in motor fuels by programmed-temperature gas chromatography using an electron-capture detector; standard deviations as low as 0.01 gram of lead per gallon were obtained. Pedinelli (*51D*) went further by determining the same three components and in addition diethyldimethyl- and triethylmethyllead (all resulting from metathetical reactions occurring on storage of fuels containing both TEL and TML); the lead compounds were converted to their bromo derivative, separated, and analyzed by paper chromatography techniques. Parker, Smith, and Hudson (*50D*) determined mixed lead alkyls in gasoline by using gas chromatography to separate the TEL and TML, following their elution spectrophotometically. From this experience, Parker and Hudson (*49D*) devised a simplified field version of the test using a column packed with Apiezon M on Chromosorb W; no detector elements were needed, since the calibration was based on a fixed relationship of the retention time for each lead alkyl to that of TEL.

Methods for analysis of lead antiknock fluid have also been studied. Pedinelli (*51D*) applied his method described above by first diluting the fluid with hexane. Gelius and Preussner (*23D*) described a procedure in which the lead in TEL was mineralized with chlorine dissolved in carbon tetrachloride; the lead salt was dissolved in water, and the solution was buffered with hexamine and titrated with EDTA using xylenol orange as indicator. In a second procedure this author dissolved the sample in benzene, and added an excess of standard iodine solution, titrating the excess iodine. Lapisova and Gol'dshtein (*36D*) quantitatively oxidized hexaethyldiplumbane to $Pb(C_2H_5)_6O$ with potassium permanganate dissolved in acetone. Gol'dshtein, Lapisova, and Shtifman (*25D*) diluted the TEL fluid with acetone and used this reaction in high-frequency conductometric titration with a relative error of about 5%. Tagliavini, Belluco, and Riccoboni (*66D*) make the same determinations with a precision of 1% by coulometric iodination at constant current with an amperometric end point.

Powell (*53D*) described means for determining anti-icing agents (of the type that lower the melting point of ice) in motor and aircraft engine fuels at the 1% level by an infrared difference-extraction procedure. With this background, Jenkins and Scruton (*31D*) applied infrared spectrometric analysis to water extracts of the fuel to determine isopropyl alcohol and/or hexylene glycol in motor fuels and Cellosolve in aviation turbine fuels at the 0.1% level; both identification and analysis were possible. Nottes (*46D*) developed a method using a carburetor for estimating in fuels the amount of polar, surface-active agents that presumably prevent icing by film formation on the throttle valve.

Fuel filter plugging has been studied by Gross *et al.* (*26D*) who describe a procedure for measuring the relative filterability (to isooctane) of motor fuels containing gel-forming alkyl sulfonates and sulfates from alkylate-containing fuels. Rybakov, Zhukov, and Isaeva (*60D*) used standard nitrocellulose membrane biological filters for measurement of mechanical impurities. Hoffmann, Chapin, and Kelly (*29D*) described a glass single-barrel carburetor throttle-body on which mechanical deposits could be collected during engine operation for rating by a photoelectric device.

Among the more exciting developments in fuel knock rating has been the development of computer-controlled engines. One such, described by Jones, Ludt, and Kellogg (*33D*), requires the operator only to load the fuel and push a button. A new method (distribution octane number) is being studied by the ASTM and the IP (*48D*) for predicting road antiknock quality of fuels for manual transmission cars; this method is to account for gross fractionation of light and heavy ends in the manifold. Adams and McCullough (*2D*) describe the effect of some controllable variables for the ASTM Research octane number method (D 908) applicable to fuels above 80 ON; incomplete compensation for barometric pressure changes was found dominant. A new electronic detonation meter has been described by Zabryanskiĭ *et al.* (*69D*). Equations and tables have been published for ready reference by Nelson (*45D*) relating performance numbers, octane numbers (above and below 100), and milliliters of lead. Jentzsch (*32D*) describes the relationships between the motor method for estimating octane response of motor fuels and the ignition value method.

Apparatus for the measurement of viscosity of *n*-pentane, *n*-heptane, and *n*-octane at room and very low temperatures and at pressures from 1 to 500 atm. has been described by Agaev and Golubev (*3D*, *4D*).

Sharapov and Fomina (*63D*) believe that the usual formula for calculating vapor pressure as determined in a closed system does not allow for volume of liquid water evaporated simultaneously with the sample; as a result, values for motor gasolines, gas turbine fuels, and diesel fuels may be as much as 7.5% low, allowing incorrect interpretation of the vapor lock tendencies of the fuels. Budanov (*11D*) claims that the previous authors make the same correction twice and find values on pure materials in good agreement with published results by more fundamental procedures because no allowance was made for air present in the fuel. Donlan (*19D*) was also concerned about volatility; he points out the causes for differences obtained when two standard ASTM tests (D86 and D1078) are applied to solvents and naphthas.

Studies on fuel storage (oxidation) stability carried out in Europe and methods for predicting such stability have been described. Ruf (*58D*) reports on several accelerated tests applicable to motor and aircraft gasolines, gas turbine fuels, and diesel fuel; results are compared to actual storage over periods of 2 to 14 years with accelerated predictions correlating well with actual storage except for aircraft turbine fuels. Rozhkov and Marunin (*57D*) describe an 8-hour air oxidation procedure for predicting storage stability of motor fuels and tetraethyllead fluid (dissolved in heptane) that is reminiscent of other familiar and standard bomb oxidation tests (ASTM D 525).

As might be expected, there has been a lively interest in the analysis of aircraft turbine fuels. Hawks and Edgington (*28D*) describe the need to have clean fuel delivered to the aircraft and methods of measuring fuel cleanliness levels. Harris and Coker (*27D*) explain the problems of quality control during production in the refinery, in pipeline transfers, and airport loading; they give practical fuel-cleanliness standards and methods for their evaluation. Rogers, Krynitsky, and Churchill (*56D*) describe works sponsored by a CRC committee dealing with research techniques for assessing water separation from fuels and fuel-additive combinations, for detecting trace amounts of surfactants, and for studies on microbial contamination. George (*24D*) describes quantitative methods for detection of microbial contaminants based on measurement of carbon dioxide produced by cellular respiration.

Loomer and Graham (*39D*) report on a novel quick method for monitoring rate of injection of anti-icing additive to aviation turbine fuels during delivery to storage tanks; the method is based on observing the freezing point of water extract of the fuel and comparing with a prepared correlation chart. The IP (*34D*) describe the experimental work in the establishment of a procedure for establishing the limiting temperature (cold flow test) at which gas turbine fuels will flow from an aircraft tank during flight. High temperature stability for high performance aircraft turbine fuels is important. Criqui (*17D*) describes some of the elements responsible for instability, and Stone (*65D*) gives a progress report on the development of the ASTM-CRC coker test (D 1660) and future work planned in this area.

Storage stability of diesel engine fuels and accelerated tests for the detection thereof in the presence of sulfur compounds have been extensively studied by Sablina *et al.* (*61D*). Reznikov (*55D*) reviews methods for the determination of phenylenediamine additive, and Shimonaev and Stepanova (*64D*) describe the determination of butyl nitrate and phenylisopropyl ethyl peroxide additives.

In view of the operation of multifuel diesel engines in some parts of the world, knowledge is needed of the cetane number of high octane motor gasoline used as igniter fuels. Wolf (*68D*) discusses the problem from the test engine point of view, and Futterer and Muenz (*21D*) describe a test engine designed specifically for measuring cetane numbers of gasolines below their 20 level. In common with similar methods, Lavrent'ev (*37D*) has established a correlation between cetane number and aniline point to avoid the necessity of running the engine method.

The detection and determination of kerosine contamination of gas oil have occupied the attention of Bentur, Babitz, and Rocker (*8D*), while the same authors with Skurnik (*9D*) have extended these studied to the detection of heavy fuel oil and cracked gas oil in straight-run gas oils. Ruf *et al.* (*59D*) were concerned with the characterization of gas oils by simple chromatographic techniques for the detection of sources of leakage.

The determination of carbon and hydrogen content is considered a convenient means of evaluating the fuel value of heavy fuels. Ogawa (*47D*) found a simplified relationship based on other normally made tests (specific gravity, viscosity, sulfur, water and ash content) that gave the same useful information with only a maximum 0.4% error. Taylor (*67D*) has described a number of gadgets to facilitate sample and test bulb handling in carrying out the Ramsbottom carbon residue test (ASTM D 524).

Lubricating Oil

D. O. Alford

Union Oil Co. of Calif.,
Brea, Calif.

Several papers on methods for the general evaluation of new and used lubricating oils have appeared during the period of this review. Among these was an article by Keil, Milowsky, and Saxe (*24E*) on a number of methods for determining contaminants, physical properties, hydrocarbon types, stability, and the efficiency of additives. Papock *et al.* (*37E*) reported studies on the performance characteristics of various oils by laboratory and engine tests. General evaluation of lubricating properties by means of fluorescence and phosphorescence spectroscopy was described by Khalupovskiĭ (*26E*). Methods for evaluating turbine oils were proposed by Hall (*20E*), Ellis (*11E*), Milz and Bunting (*35E*), and Manley and Henrikson (*33E*).

The use of light scattering, electron microscopy, and polarography in oil stability studies was described by Loveland *et al.* (*31E*). Various laboratory procedures for oxidizing lubricating oils and correlating the results were reported by Keil, Eckardt, and Milowsky (*23E*). Decomposition products in used transformer oils were determined by Rost (*38E*). Methods for testing the oxidative stability of transformer oils were given by Dichter and Ogradowska (*7E*). Engine test methods for lubricating oils were reported by Hall and Martin (*19E*), McLaughlin (*32E*), and ASTM Committee D-2 (*1E*).

Lopez de Azcona and Sanz de la Rosa (*30E*) described an emission spectrographic method for determining trace metals in oils, in which the sample is forced through a hole drilled in an aluminum or tin electrode. Another emission method for metals was reported by LaSell (*28E*), who used a rotating electrode, high-voltage spark, and a line emitted by the carbon in the sample as an internal standard. Lock and Mason (*29E*) also published an emission spectrograph procedure for trace metal determination. Cummins and Mason (*5E*) reported on factors effecting the spectrographic determination of barium, calcium, phosphorus, and zinc. The same elements were determined in a method by Mashireva and Zimina (*34E*).

Iron contamination of engine oil was measured by an x-ray fluorescence method developed by Florkowski and Gilewicz (*18E*). X-ray fluorescence was also used by Schreiber, Ottolini, and Johnson (*39E*) to determine zinc, barium, phosphorus, sulfur, and chlorine in thin films of additives on metal parts.

Tennyson (*44E*) utilized colorimetric methods for identification of metals and dark-field microscopy for evaluating the dispersion of particles in used oils. Zinc, barium, and calcium were measured in oils by titration with (ethylenedinitrilo)tetraacetic acid in methods given by Fisher (*17E*) and Palovcikova (*36E*). Hensley and Bergmann (*22E*) analyzed oils and additives for the same three metals by extraction with trifluoroacetic acid and complexometric titration with diethylenetriaminepentaacetic acid. Complexometric titration was also used by Faerman and Chigrin (*14E*) for determining barium in additives.

An ultraviolet method for measuring the phenol content of lubricating oil was given by Khudominskaya and Aksenova (*27E*). The simultaneous determination of water and fuel contamination in used oils by automatic vacuum distillation was reported by Siegfriedt and DeWoody (*42E*). Water in transformer oil was the subject of papers by Stannett (*43E*) and Engel and Moebes (*13E*). Engel (*12E*) also published on the determination of dissolved gases in transformer oil.

Bor (*2E*) used infrared spectroscopy to analyze polymethacrylates in additives. Dialkyldithiophosphate additives were determined in oils with an infrared method reported by Kendall and Rimmer (*25E*). Aromatic secondary amines were measured by infrared spectra of chromatographic extracts, according to Hasegawa, Kajikawa, and Kawaguchi (*21E*). Infrared spectroscopy was also applied to the determination of DBPC antioxidant in turbine oils by Serboli and Golgowski (*41E*) and in an anonymous article appearing in *Scientific Lubrication* (*40E*). The limitations of titrimetric and colorimetric procedures for DBPC in turbine and transformer oils were explored by Drescher and Teubel (*9E*). Delves (*6E*) employed paper chromatography to identify aromatic amine and phenothiazine antioxidants in turbine oils.

Finke and Heinze (*15E*, *16E*) and Bornemann, Finke, and Heinze (*3E*) reported on the use of elution chromatography to separate a lubricating oil into compound type fractions. Brewer (*4E*) also used elution chromatography to fractionate oils into their major components and to separate additives from compounded oils. Infrared spectroscopy was employed by Eckardt, Keil, and Rentrop (*10E*) to determine compound types in lubricating oils. The types of aromatic compounds in transformer oils were measured by ultraviolet and mass spectral analysis of chromatographic extracts in a method described by Dornenburg and Serboli (*8E*).

Greases

J. W. Johnson

The Atlantic Refining Co., Philadelphia, Pa.

Bird and Turian (*1F*) studied viscous heating effects in a plate and cone viscometer. Variational analysis indicates that these effects are sufficiently large to account for errors observed experimentally when the instrument is operated at high speed. Brunstrum (*2F*) developed a nomograph for determination of grease viscosity from flow constants. Cox (*4F*) observed radial flow in a cone-plate viscometer using a transparent acrylic plate. He concluded that the radial component may be an important factor in the viscometry of non-Newtonian fluids. Gabrysh, Eyring, *et al.* (*8F*) described an automatic rotational viscometer and high pressure apparatus for the study of the non-Newtonian behavior of materials. Herold (*9F*) described the determination of consistency and thixotropy of greases with an electrically driven tester modified to record the bending of the sieve plate by means of an oscillograph.

Ewbank *et al.* (*6F*) used permeability coefficients as a measure of the structure of lubricating grease. Flow of oil through a grease structure was shown to obey Darcy's law. Febles, Bresciano, and Maccoll Piriz (*7F*) used a phase-contrast microscope, polarized light, and an electron microscope to study structure of a calcium grease at various stages of manufacture. Rooney and Bird (*13F*) described the preparation of carbon replicas of grease fibers for study under an electron microscope. Kryn, Meshchaninov, and Zabelina (*11F*) used a polarizing microscope to study the crystal form, orientation, and dispersion of greases.

Jenkins (*10F*) indicated that infrared spectroscopy can be used to characterize the lubricant, soap, filler, and some additives. Use of ultraviolet was also described for identification of amine inhibitors. Smithson and Matta (*14F*) listed infrared spectra of oils, grease additives, and related materials used as a qualitative aid in lubricant identification.

Carter and Baumann (*3F*) used gas chromatography to identify fatty acids.

Criddle (*5F*) described an instrument for measurement of the color of greases.

Pope (*12F*) described the correlation of field data with useful grease tests for general plant lubrication.

Waxes

R. T. Edwards

Socony Mobil Oil Co., Inc., Paulsboro, N. J.

Interest in the hydrocarbon-type analysis of petroleum waxes and its relation to performance has continued. Ferris (*10G*) summarized methods for the characterization of these products. Phillips (*31G*) proposed characterization of waxes on the basis of performance of their blends with oil or other waxes. McMahon and Wood (*23G*) analyzed waxes by a combination of silica gel percolation and urea and thiourea adduction, followed by gas chromatography, and related results to wax performance. Brown, Lewis and Smith (*5G*) interpreted performance in terms of mass analyses. Guseva, Ashkinadze, and Leifman (*13G*) used infrared absorption to characterize wax fractions separated by chromatography and urea adduction. Negoro (*26G–29G*) published a series of studies on fractionation of microcrystalline waxes together with spectroscopic examination of the fractions. Gas chromatography has found increasing use in the analysis of wax. Levy and his coworkers (*18G, 19G*) have reported further on their work in this field. Dietz, Starnes, and Brown (*7G*) and Hildebrand, Peper, and Dahlke (*17G*) also published analytical methods of this type. Teubel, Roesner, and Leschner (*36G*) examined chlorinated paraffin waxes by elution chromatography.

Barmby, Bostwick, and Huston (*1G*) studied the solid-state properties of paraffin waxes by x-ray diffraction, differential thermal analysis, and dilatometry. Templin (*35G*) reported further on relations between composition and thermal expansion of waxes. Edwards (*8G*) estimated the crystallinity of waxes by means of x-ray diffraction. Moyer and Zmitrovis (*25G*) related sealing properties of waxes to molecular arrangement.

There have been further reports on analysis of petroleum waxes for trace quantities of polycyclic aromatic hydrocarbons in relation to potential health hazards. Lijinsky and coworkers (*20G, 21G*) continued their work in this field. Haenni and his coworkers (*14G, 15G*) used dimethyl sulfoxide as a selective solvent to concentrate aromatics from petroleum waxes and white oils. Helberg (*16G*) developed a paper chromatographic method for detecting polycyclic aromatics. Prokhorova and Znamenskii (*33G*) reported a method for estimation of benzo(*a*)pyrene in waxes.

The increasing use of additives in waxes has stimulated work on their quantitative determination. Bluestein, Hindman, and Bluestein (*3G*), Eells and Brunson (*9G*), and Fox (*11G*) reported methods for estimating the polyethylene content of wax. Braithwaite and Penketh (*4G*) developed a method for determining antioxidants in white oil which should be applicable to waxes.

Dannenbrink and Walker (*6G*) reviewed the standard tests for petroleum waxes. Barras, Boyle, and Grainger (*2G*) reported a method for determination of oil in wax by wide-line nuclear magnetic resonance. Mal'nev and Mikhlina (*24G*) used infrared absorption for the same purpose. Lizogub *et al.* (*22G*) reported a method for determination of wax in petroleum products similar to the ASTM method for oil content of wax. Shirdzhanov (*34G*) published an ultrasonic method for the solidification point of petroleum products of high paraffin content. Gerner and Rudnitskaya (*12G*) reported a method for solidification point similar to the ASTM congealing point method. Presting, Steinbach, and Foehse (*32G*) published a method for testing the thermal stability of waxes. Noll and Grogan (*30G*) developed an improved instrument for determining the sealing strength of waxed paper.

Asphalt

John A. Wronka

Cities Service Oil Co.,
Cranbury, N. J.

During the period covered by this report, an important documentation covering the utilization of asphalt for paving purposes was made available with the completion of a multimillion dollar American Association of State Highway Officials road test (*51H–56H*). A second important contribution consisted of a series of papers and discussions by 123 authors presented in connection with an international conference on the structural design of asphalt pavements (*69H, 70H, 71H*).

The final volume of a revised edition of a standard asphalt reference was presented by Abraham (*1H*) and Barth (*5H*) published an authoritative treatise on asphalt.

A series of papers considered the relationship of the absolute viscosity of asphalt to problems of construction and performance of bituminous highways. Levy (*96H*) surveyed methods and instruments used to measure asphalt viscosity; Griffith and Puzinauskas (*40H*) described the apparatus, procedure, relative precision, and results pertaining to the measurement of absolute viscosity at 140° F. and the relationship of viscosity data to empirical tests; Welborn, Halstead, and Olsen (*156H*) reported on the effect of asphalt viscosity on the stability of asphalt-aggregate mixes; Griffin, Izatt, and Lettier (*38H*) discussed the use of asphalt viscosity measurements in hot-plant mixing, spreading, and compaction of paving mixes and in the service performance of pavements; and Gzemski (*43H*) reviewed the relationships of the flow properties of asphalts and paving mixes to shear rate, asphalt type, service conditions, and the lack of correlation between penetration and viscosity-temperature properties.

Griffith (*39H*) discussed methods for measuring the absolute viscosity of asphalt and the possibility of establishing specifications based on viscosity and Welborn and Halstead (*155H*) considered methods and problems associated with the use of absolute viscosity as a specification control for asphalt binders. Halstead and Zenewitz (*46H*) determined changes in viscosity during the thin-film oven and microfilm durability tests for 11 asphalts. Martin (*102H*) used microviscometer data to predict the durability of roofing asphalts and Heithaus (*48H*) reported that the resistance to accelerated weathering for five asphalts increased with increasing viscosity of their maltene constituents measured at 77° to 180° F. but failed to correlate with viscosity, viscosity-temperature susceptibility, or elastic modulus of the original sample. On the basis of microfilm viscometer data, Tolson (*144H*) concluded that air blowing generally does not improve the quality of a paving asphalt and may reduce adhesive characteristics and Traxler (*147H, 148H, 145H*) used viscosity data and chemical analysis to study hardening of asphalt films; effects of polymerization, volatilization, and photochemical hardening; oxidation and photooxidation of asphalt cements; and the correlation of asphalt rheology and composition with pavement durability.

Evans and Griffin (*28H*) reported modifications in sample plates and apparatus of the sliding plate viscometer and Schmidt and Santucci (*130H*) described a falling plunger viscometer for determining asphalt viscosity at low temperatures. Oglio, Zenewitz, and Halstead (*113H*) compared the Zeitfuchs cross-arm and Saybolt-furol viscometers for measuring the viscosity of asphalt cutbacks. Sakanoue (*128H*) measured the rheological properties of asphalt using a vibrating, double cylinder type rheometer and Aida (*2H*) measured viscoelastic properties with a cone and plate type viscometer and reported apparent activation energies of 30 to 40 kcal. per mole for calculated viscosity coefficients. Carre and Laurent (*20H*) reported that bitumens having the same penetration have the same viscosity for a given shear rate regardless of the rheological type, and Carre (*19H*) discussed examples where this relationship can be used. Barrall, Schmidt, and Johnson (*4H*) described a thermal expansion apparatus and determined the glass softening point of several asphalts. The glass transition temperature of asphalts was also studied by Iijima (*66H*) and Lenz, Barrante, and Andrews (*94H*). Jones (*76H*) determined changes in the brittle point temperature of asphalts during weathering to predict durability. Spurr (*139H*) reported that creep and rebound curves of a bitumen containing asbestos fell on a common curve.

Winniford (*158H*) presented electron microscope, ultracentrifuge, and solution viscosity measurements as evidence for association of asphaltenes in dilute solutions. Electron and/or nuclear magnetic resonance methods were used by Jeffries-Harris and Poindexter (*72H*) to investigate asphaltenes from a Greeley, Calif., crude; asphalt solutions and deuterated asphalt solutions by Poindexter (*122H*); the structure of asphaltenes from naturally occurring, air-blown, and thermal cracked sources by Winniford and Bersohn (*159H*); structure of petroleum asphaltenes by Yen and Erdman (*170H*); the nature of free radicals in petroleum asphaltenes by Yen, Erdman, and Saraceno (*171H*); and with x-ray and composition analysis by Erdman and Dickie (*27H*) to demonstrate a mild thermal alteration of asphaltic crude and asphaltic crude fractions.

Wright and Minesinger (*166H*) concluded that asphaltene electrophoretic mobility in nitromethane supported the colloidal structure of asphalt and that asphaltene movement results from electrophoresis and not from electroosmosis or other nonelectrophoretic effects. Eldib (*26H*) studied the solvation and electrophoretic properties of asphaltenes in petroleum. Gorshenina, Kuperman, and Mikhailov (*34H*) reported on an electron-microscopic study of the structure of asphalt-polymer blends. Newman (*110H*) described a point-projection x-ray microscopic technique applicable to asphalts and asphalt mixes, and Newman and Greenfeld (*111H*) used this technique to reveal defects in various asphalt specimens. Iijima (*65H*) considered the relationship of asphalt viscosity to composition and concluded that the colloidal structure of asphalt changes at the softening point, and Iijima and Koma (*67H*) related asphalt composition to temperature susceptibility and softening point.

Knoterus and Krom (*86H*) concluded that wax separated from seven bitumens consisted chiefly of long paraffinic chains which were often bound to saturated or aromatic homocyclic or to sulfur, oxygen, or nitrogen heterocyclic rings on the basis of ndM and x-ray diffraction analyses. Wales and VanderWaarden (*152H*) determined molecular weights of asphaltenes by centrifugation, diffusion, sedimentation rate, ebullioscopic, and intrinsic viscosity methods and concluded that asphaltenes are associated in solution and that the association is affected by temperature. Corbett (*23H*) described a densimetric method to characterize straight reduced asphalts and Bodan and Sklyar (*9H*) reported a cryoscopic method for determining the molecular weight of bitumens. Thelen (*143H*) correlated the Sternberg-Rostler composition of an asphalt with initial viscosity, staining tendency, and distribution of relaxation times and Fischer and Schram (*32H*) concluded that the properties of various bitumens are primarily influenced by the dissolving power of the oil component which determines the size of the colloidal micelles.

Bestougeff and Gendrel (*7H*) classified asphaltenes from several crudes on the basis of sulfur atoms per asphaltene molecule and attempted to correlate asphaltene composition with geologic age. Maksimova (*100H*) developed methods for the determination of Cl, Br, and I in organic compounds in asphalt and Hara and Takahashi (*47H*) determined the concentrations of Ni and V in various asphalt fractions. Florovskaya and Klyuev (*33H*) studied the luminescence spectra of asphaltenes and oil fractions and Kapkova (*81H*) used luminescence to determine the nature of the change in the group composition of bitumen during reaction with various mineral powders.

Constantinides, Arich, and Lomi (*22H*) reported an improved spectrophotometric method for the determination of the porphyrin content in petroleum residues and bitumens. Chelton and Traxler (*21H*) studied the compositions of typical asphalts using chromatography and thermal diffusion and Padovani, Berti, and Prinetti (*116H*) investigated the properties and structures of thermocatalytic and primary asphaltenes using ring analysis and infrared absorption spectra methods. Kossowicz (*88H*) determined the physical, chemical, and rheological properties of asphaltic extracts from cresol and furfural refining and Rodionova, Shishenina, and Kanakina (*126H*) studied the saponification, elemental, and structural-group analysis of bituminous resins.

Ziehmann (*173H*) characterized several bitumen fractions by nuclear magnetic resonance, paramagnetic electron resonance, infrared, mass spectrometer, ultraviolet, and elementary analyses and ebullioscopic molecular weight determinations.

Boyd and Montgomery (*11H*) reported the structural group analysis of Athabasca bitumen fractions and Yatsenko, Gutsalyuk, and Rafikov (*169H*) investigated crude oil fractions using infrared absorption spectra. Prinzler, Klimke, and Dorn (*123H*) reported C, H, N, and S and molecular weights for fractions of a Romashkino topped residue.

Gundermann and Kloss (*42H*) studied the resistance of petroleum asphalts to acids and alkali and Jones and Moote (*74H*) reported that maltene molecules from Athabasca tar are smaller and have a less complex ring structure than those of native or petroleum asphalts. Zakar and Mozes (*172H*) compared the properties of asphalts from Hungarian Nagylengyel crude with Mexican, Venezuelan, and North American asphalts. Leibnitz, Mueller, and Gundermann (*93H*) compared the specific heat, electrical breakdown potential gradient, electrical conductivity, and dielectric constants of brown coal tar pitch and petroleum asphalt. Halstead, Oglio, and Olsen (*45H*) compared the properties of an 85/100 penetration asphalt cement and a tar binder prepared by the high temperature reaction of finely divided coal with tar and tar oils.

Herrin and Jones (*49H*) analyzed the behavior of an asphalt in terms of the absolute rate theory.

Changes in the infrared absorption spectra of asphalt were used to identify oxygen-containing products formed by air or photooxidation and the ozonization of asphalt flux by Campbell and Wright (*13H*, *14H*); to determine the effect of oxidation in the presence of ozone and the photooxidation of asphalts in the presence of ozone by Wright and Campbell (*163H*, *164H*); to determine the effect of intensity of carbon-arc radiant energy on the rate of oxidation by Wright, Campbell, and Fridinger (*165H*); to determine the effect of temperature and humidity on the oxidation of air-blown asphalts by Campbell, Wright, and Bowman (*15H*); and to compare oxidation rates using carbon and xenon arcs and solar radiation by Martin (*103H*). Smith, Schuetz, and Hodgson (*135H*) predicted the weatherability of roofing asphalts using infrared absorption spectroscopy.

Illman and Deahl (*68H*) alkylated asphaltenes which could be blended to form roof coating grade asphalts with improved durability and concluded that high quality roofing asphalts contain high molecular weight asphaltenes which exhibit a high tolerance for saturates and have a low C/H ratio. Jones (*78H*) described a modification for determining asphaltene dispersibility and noted some exceptions in using this property to predict weatherability. Oliensis (*115H*) discussed the relative homogeneity of asphalts as disclosed by the "spot" test as well as other asphalt properties and testing methods with respect to the performance of roofing asphalts and Jones (*75H*) described techniques for the preparation of roofing membranes and tensile specimens and reported some engineering properties of built-up roofing. Knight, Platt, and Smith (*85H*) reported that the excessive production of voids in excess of 220 microns in roofing shingles determines the intensity of blistering and Corbett and Swarbrick (*24H*) used a separation and reconstitution technique to relate asphalt composition to physical properties, method of manufacture, and weathering.

Wronka (*167H*) described a method to measure the tendency of oxidized asphalts to decrease in softening point and increase in penetration or "fallback" during storage at elevated temperatures and described some characteristics of this tendency. Kurnosenkova and Slepchenko (*91H*) proposed a method for asphaltene determination using isooctane and Wetzel and Peter (*157H*) and Rajeswaran and Kirk (*125H*) described the use of paper chromatograms for the characterization of bituminous materials. Psalomshchikova (*124H*) presented a procedure for the separation of bitumens into naphthene-methane hydrocarbons, aromatic hydrocarbons, and resins and oxygen compounds.

Varma and Sheffert (*150H*) used a commercially available cellulose membrane in place of the asbestos mat specified in ASTM Method D 165. Methods for determining the neutralization value or acid components in asphalt were presented by Caro (*16H, 17H*), Arato (*3H*), and Mathews, Brown, and Welch (*104H*). Methods for determining the paraffin content of bitumen were presented by Nishikawa, Yamamoto, and Toyoguchi (*112H*) and Kannisto, Harva, and Laiho (*80H*). Nelson (*107H*) described the Oliensis spot test and Fink (*31H*) presented a statistical comparison of softening point data obtained using ASTM Method D 36-26 with a proposed revision using a single bath liquid and thermometer. Hirose and Wada (*58H*) described an improved penetrometer for routine testing of asphaltic bitumens.

Greenfeld (*37H*) studied the effects of variations in temperature, agitation, air rate, and asphalt flux during blowing on the durability of coating grade asphalts and Hirose, Tsuyuki, and Wada (*57H*) made dilatometric, dynamic elastic, and dielectric measurements and creep tests on seven asphalts blown to different stages. Hughes (*62H*) studied variables in asphalt oxidation and concluded that the data are readily correlated in terms of a peroxide-catalyzed chain polymerization mechanism. Gundermann (*41H*) reported the effects of composition, air volume, reaction time, and air distribution on the physical properties of 11 asphalts during blowing and Sergienko and Garbalinskiĭ (*132H*) analyzed gaseous products formed during asphalt oxidation to show that 70 to 90% of oxygen consumed was used for dehydrogenation and that the product quality depended on the intensity of oxygen uptake. Chromatographic analyses by Penchev, Andreev, and Davidova (*118H*) showed that paraffinic-naphthenic fractions were altered only slightly during asphalt oxidation but the aromatics became oxidized to resins and the latter to asphaltenes and that resins produced by oxidation differed from natural resins. Levinter and Galiakbarov (*95H*) studied the use of pressure in asphalt oxidation on a laboratory scale and Gorshenina and Mikhailov (*35H*) reported a beneficial effect on the rheological properties of an asphalt which was air-blown using a cavitation technique.

Microorganisms belonging to the genera *Pseudomonas* and *Chromobacter* were detected on asphalt-coated roofs and highways (*105H*) and were reported to be capable of biodegrading asphalt. Traxler (*146H*) isolated these microorganisms and a bacillus from asphalt roofing, etc., and Phillips and Traxler (*119H*) studied the effect of the atmosphere, pH, and asphalt composition on degradation. Bergmann (*6H*) and Yanysheva, Kireeva, and Serzhantova (*168H*) reported the determination of 3,4-benzopyrene in petroleum bitumens.

Nelson and Patel (*108H*) presented a method for predicting the asphalt content of a crude and Nelson (*106H*) constructed a graph showing the approximate relationship between the percentage of asphalt, initial boiling point of the asphalt fraction on the distillation curve, and the API gravity. Farley, Guffy, Leonard, and Vasse (*30H*) described an improved procedure for the determination of the salt content of heavy crude oils and asphalt using a phenol-chloroform diluent followed by extraction with a sodium or ammonium nitrate solution. Mapstone (*101H*) determined the UOP characterization factor for asphalts from their API gravities from a nomogram developed by extrapolation methods. Smith and Schweyer (*136H*) determined the heat of reaction for the air-blowing of asphalt and reported that the amount of heat liberated decreased with increasing softening point, and Kaganov, Levinter, and Medvedeva (*79H*) determined the kinetics of the coking of asphaltenes and proposed a mechanism consistent with experimental data obtained.

Horowitz and Mandel (*60H*) proposed a Soxhlet extraction for the determination of the bitumen content in joint fillers and Wirth (*161H*) distinguished between tar and asphalt by boiling a sample in ethanol and observing a yellow color with tar and a clear or slightly fluorescent solution with asphalt. Szatkowski (*140H*) estimated the total unvulcanized rubber content of a rubberized bitumen from the iodine value of a carbon tetrachloride solution of the acetone-insoluble fraction of the sample and Winters (*160H*) discussed results of bleeding and peel tests as well as performance characteristics of butadiene-styrene rubberized asphalts.

Bohn (*10H*) correlated the setting time of bitumen emulsions with evaporation velocity and Egorov and Natanzon (*25H*) used the change in electrical conductivity of an emulsion film on stone to measure the breaking time of asphalt emulsions. Karius and Dalton (*84H*) described an asphalt cutback coating and stripping test for bituminous emulsions; Skog and Zube (*134H*) studied the effect of water action on bituminous mixtures using a dye stripping test; and Ewers (*29H*) described an accelerated aging test for cutback and fluxed road asphalts. Hunter (*63H*) described test methods to evaluate asphalt and other types of coatings.

Gotolski, Ciesielski, and Kofalt (*36H*) evaluated the low temperature service of four asphalt cements by using the standard and microductility tests and discussed possible reasons for relationships. Rostler and White (*127H*) reported changes in composition after mixing and aging for 119 asphalts and Kolbanovskaya and Golovkina (*87H*) correlated the chemical composition and quality of road asphalts obtained from a Tuimasy crude using different refinery methods.

Speer, Brunstrum, Sisko, Ott, and Evans (*138H*) studied the relationship of asphalt viscosity to pavement performance and Speer (*137H*) summarized the effect of temperature, tire pressure, asphalt hardness, and asphalt content on pavement performance using miniature test tract data. Halstead (*44H*) used ductility-penetration curves of thin film residues to facilitate the selection of paving asphalts and Lammiman, Les, and Way (*92H*) studied hardening tendencies and performance of seven asphalts. VandenBurgh, Bouwman, and Steefelaar (*149H*) reported that penetration changes furnished the best indication of aging for asphalts. Lottman, Sonawala, and Al-Habboobi (*98H*) studied the change of asphalt viscosity during mixing with hot aggregates and Bissett and Ford (*8H*) concluded that large decreases in asphalt penetration or ductility during service will result in pavement cracking.

Jones (*77H*) compared the spark gap tester for asphalt films specified in ASTM Method D 1670-59T with an instrument using a higher voltage, lower current, and multiple probes. Tajima and Fushima (*141H, 142H*) reported the formation of carbon dioxide, water, and water-soluble products consisting primarily of aromatic compounds and resins during accelerated weathering of blown asphalts and correlated durability with asphalt properties.

Carpenter (*18H*) suggested the use of statistical data analysis for specification limits for bituminous materials; Shook (*133H*) discussed the statistical significance of test results obtained from random samples of asphaltic highway materials; and Warden and Sandvig (*153H*) suggested a method of comparing specification limits with actual variations in the properties of bituminous paving mixtures. Krom and Dormon (*90H*) discussed the performance requirements for road bitumens and their expression in specifications. Nevitt (*109H*) presented a rational approach to fewer asphalt cutback grades.

Pignataro (*120H*) studied the effect of test temperature on Marshall stability and presented an empirical formula to express the stability at the test temperature in terms of a standard stability at 60° C.; Vokac (*151H*) reported the repeatability of the Marshall test by analysis of factorial experiment data; Wojdanowicz (*162H*) showed that the compressive strength of a bituminous mix was a function of the penetration of the bitumen and the amount present; Oksala (*114H*) studied the effects of aggregate size and distribution, voids, etc., on the stability of surface mixes; and Schuhbauer (*131H*) showed some relationships between Marshall stability and methods of sample preparation.

Hveem, Zube, and Skog (*64H*) described the apparatus and procedures for determining "setting" characteristics of bituminous concrete; Santucci and Schmidt (*129H*) discussed the effect of filler and aggregate on the setting rate of asphaltic concrete; Holl (*59H*) studied effects of asphalt and filler variables on properties of mixes; and Please and Hardman (*121H*) determined the spreading characteristics of bituminous mixtures by plotting the maximum screeding force *vs.* temperature. McCoy and Coyne (*99H*) described a wet-track abrasion test for measuring the performance of slurry seal mixtures and Kari, Coyne, and McCoy (*82H*) reported the interrelation between asphalt, aggregate, and climate with respect to seal coat durability.

Livneh and Shklarsky (*97H*) reported that the design and control of asphalt mixtures can be effected by means of a splitting test used in conjunction with Marshall stability and flow and Busching (*12H*) compared stability values of gyratory compacted specimens with those produced by the California kneading compactor. Herrin, Manke, and George (*50H*) proposed a vacuum-saturation method for soaking soil-asphalt specimens prior to testing.

Jimenez and Gallaway (*73H*) reported on the design and operation of a deflectometer to determine the resistance of coarse sheet-asphalt mixtures to repeated loads; Kari and Santucci (*83H*) described an apparatus to measure the air permeability of asphaltic concrete; and Hughes (*61H*) reported the use of a surface density probe for determining the density of bituminous concrete. Warner and Moavenzadeh (*154H*) studied relationships between air permeability and compaction characteristics as well as other variables. Kraemer (*89H*) presented equations for calculating the amount of water and binder required for optimum stability of soils and Parr (*117H*) outlined quality control practices used by the Michigan State Highway Department in bituminous construction.

Hydrocarbons and Hydrocarbon Types

R. W. King
Sun Oil Co.,
Marcus Hook, Pa.

INSTRUMENTAL methods continue to dominate recent contributions dealing with the analysis of complex mixtures of hydrocarbons. In particular, the application of gas chromatography and mass spectrometry to petroleum fractions boiling below 200° C. has received a great deal of attention. However, it is obvious that the breadth of usefulness of these methods is gradually being expanded to encompass the analysis of hydrocarbon mixtures of higher and higher boiling point. When a single instrumental technique proves incapable of providing the necessary data, it has become commonplace to resort to either in-line or isolated combinations of instrumentation. Both mass spectrometry and infrared and NMR spectroscopy are being used with considerable success for the hydrocarbon-type analysis of the heavier petroleum fractions. Nevertheless, there is ample evidence that physical property methods will continue to be of importance in the routine examination of viscous oils and the classification of crudes because of their rapidity and economy. Despite the fact that methods based upon physical and optical principles have become the most widely used tools for the analysis of hydrocarbons, chemical methods continue to play a role in the determination of unsaturation, in the detection and estimation of certain species of aromatics, and in the analysis of relatively simple hydrocarbon mixtures.

Prinzler (*150J*) has reviewed the problem of the analysis of the heavier petroleum fractions and has outlined the methods used at the Technical Institute for Chemistry at Merseberg, Germany (*149J*). The American Society for Testing and Materials (*5J*) has published an informative and useful "Manual on Hydrocarbon Analysis," which describes the methods commonly practiced for the routine analysis of a broad spectrum of petroleum products and should prove invaluable in the average refinery laboratory.

Sidorov (*183J*) has proposed a procedure for the compound-class analysis of aromatic hydrocarbons boiling below 270° C. The method is based upon the use of standard separation techniques augmented by calculations of R_T and R_A, using the *n-d-M* method. The concentrations of benzenes, naphthalenes, tetrahydronaphthalenes, indans, cyclohexylbenzenes, and biphenyls are reported. Veselov (*206J*) uses the difference in aniline point between a mixture of paraffin-naphthene hydrocarbons and the aniline point for average paraffins of the same molecular weight to estimate the percentage of naphthenic carbons in the average molecule. For the detailed examination of petroleum fractions boiling below about 200° C., the Russians rely heavily on combinations of separation techniques, physical properties, dehydrogenation, oxidation, and Raman and infrared spectroscopy. A typical example has been reported by Sergienko and coworkers (*178J*), who used adsorption chromatography, distillation, catalytic dehydrogenation, refractive index and density relations, and Raman and infrared spectra to identify about 50 hydrocarbons in a gas condensate from the Kara Kum formation. Dehydrogenation, in combination with other methods, was used by Areshidze and Khechinashvili (*7J*) and by Pishnamazzade and others (*146J*) to study the five- and six-membered cyclanes in straight-run fractions boiling up to 200° C. The composition of the aromatic hydrocarbons in a Bibieibat naphtha was determined by Ashumov and coworkers (*9J*) using adsorption, distillation, absorption spectroscopy, and picrate formation. Dimitrov, Stoilova, and Petsev (*44J*) used similar techniques augmented by oxidation to study the aromatics in a coker naphtha. A detailed study of the olefinic hydrocarbons in a shale oil naphtha has been reported by Eisen and Rang (*50J*). They used silica gel chromatography, analytical distillation, physical constants, infrared spectroscopy, and gas chromatography to identify 34 olefins. Twenty-nine of these were determined quantitatively. Rang (*156J*) also reports the use of similar techniques to analyze the paraffin-cycloparaffin portion of a thermal naphtha. The concentrations of 31 hydrocarbons are reported.

Analyses of the heavier fractions have also been reported by a number of investigators. Rodionova, Korchagina, and Il'inskaya (*162J*) isolated aromatic hydrocarbon fractions from several dispersed bitumens by silica gel chromatography. The aromatic portions were then further fractionated on activated alumina. These fractions were analyzed by structural group methods to determine the average number and type of aromatic rings. Similar adsorptive fractionation procedures were used by Rodionova, Il'inskaya, and Nadel'shtein (*161J*) to analyze the paraffin-cycloparaffin portion of a number of crude oils and bitumens. Physical separation methods, including adsorption chromatography and extraction, were used by Ashumov, Nasirov, and Kurkovskaya (*10J*) to characterize a high molecular weight asphaltic fraction from Artemovsk crude. The resinous components of Athabasca bitumen have been studied in some detail by Boyd and Montgomery (*28J*). The resins were isolated by adsorption on activated fuller's earth and then further fractionated using a series of solvents of increasing polarity. Infrared spectroscopy was used to estimate the number of methyl and methylene groups and the number of aromatic carbon atoms. The fractions were also characterized by application of the structural group methods of van Krevelen and by two new methods devised by the authors. The minor constituents in lignite hard paraffins were characterized by Prinzler and Doescher (*151J*). The wax obtained by the alcoholic extraction of Saxon lignite was separated into a number of fractions by silica gel chromatography. The fractions were analyzed by physical, chemical, and spectroscopic methods, and then added back to the refined paraffin to assess their effect on color stability. The fractions containing aromatic hydrocarbons with olefinic chains had the most adverse effect on ultraviolet stability.

The application of combinations of physical separation and instrumental methods has resulted in the identification of several branched paraffins in the higher-boiling fractions of petroleum. Bendoraitis, Brown, and Hepner (*20J*) used mass spectrometry and infrared and proton magnetic resonance spectroscopy to identify seven methyl-branched alkanes in fractions separated from an East Texas gas oil by preparative scale gas chromatography. Similar techniques were used by Mair, Krouskop, and Mayer (*117J*) to identify 2,6,10-trimethylundecane and 2,6,10-trimethyldodecane in the light gas oil fraction of the API representative petroleum.

A substantial number of references attest to the continued use of adsorption chromatography as a tool for the separation and analysis of petroleum. The chromatographic separation of hydrocarbons was reviewed by Srinivasan (*196J*) and by Bestougeff (*24J*). A chromatographic technique that seems to show considerable promise for hydrocarbons was described by Karr, Childers, and Warner (*94J*). They use a system for adsorption chromatography in which the apparatus and operating conditions are analogous to those used in gas-solid chromatography. The column consists of 25 feet of 0.25-inch-diameter copper tubing packed with 80- to 100-mesh activated alumina containing about 4% water. Cyclohexane is used as the eluent. Compounds are detected spectrophotometrically as they emerge from the column and the concentration *vs.* eluent volume is automatically recorded. The retention volumes are highly reproducible and can serve for the identification of components. Snyder (*191J*) has continued to investigate the pertinent variables in linear elution adsorption chromatography. His latest studies are concerned with further elucidation of the role of the eluent in separations over alumina. Nagai and Funakubo reported the results of several fundamental investigations of the column chromatography of polynuclear aromatic hydrocarbons. They measured the adsorption isotherms of a number of aromatic hydrocarbons on alumina and from these data were able to predict certain features of columnar behavior (*130J*). They also studied the behavior of mixed eluents using anthracene as the adsorbed species (*62J*), and the effect of the coexistent solute in the column chromatography of binary mixtures of aromatics (*131J*).

Driatskaya and Zhmykhova (*48J*) recommend the use of equal amounts of silica gel and activated alumina in the upper and lower portions of a column, respectively, for separating hydrocarbon types in the 200° to 450° C. boiling range. This combination of adsorbents is more effective than silica gel alone. The separation of dinuclear from mononuclear aromatics, and of sulfur compounds from hydrocarbons, is sharper. Vamos and Kovats (*204J*) reported the use of silica gel to isolate the aromatic hydrocarbons from a urea-dewaxed fraction of Tuimazy gas oil. The aromatics were then separated according to the number of rings by chromatography over activated alumina. An attempt to separate the isoparaffin-cycloparaffin portion into discrete hydrocarbon groups by percolation over activated carbon was largely unsuccessful.

Mirskiĭ, Mitrofanov, and Dorogochinskiĭ (*124J*) describe a method for the selective removal of normal paraffins from a hydrocarbon mixture using Molecular Sieves with a 1.9 ± 0.1 SiO_2:Al_2O_3 ratio. Rozenberg and coworkers (*165J*) find liquid chromatography using activated charcoal a practical way to determine individual *n*-alkanes in petroleum fractions. The charcoal is treated with nitric acid, washed until neutral, and vacuum-dried. Samples are introduced to the carbon column in isooctane solution and eluted with *n*-hexane. Adsorption chromatography using activated alumina is the basis of a method proposed by Dolejs (*46J*) for the routine determination of resins in mineral oils. The sample is diluted with *n*-hexane and percolated over alumina. The nonresinous portion is eluted with additional hexane. The resins are desorbed with a 1 to 1 benzene-ethanol mixture followed by acetone, and weighed after removal of solvents. The maximum error is reported as 2%.

Descriptions of apparatus for column chromatography are few, evidently because relatively simple equipment suffices for most analytical applications. However, Finke and Heinze (*53J*) have detailed the construction and operation of an 18-meter stainless steel column designed specifically for the elution chromatography of high-boiling hydrocarbon oils.

The primary applications of paper and thin layer chromatography to hydrocarbon mixtures have been in the search for polycyclic aromatic hydrocarbons in certain petroleum products. Raha (*154J*) has reviewed previous analytical work on this problem. Helberg (*74J*) describes a method that permits the detection of 0.03 μg. of polycyclic aromatic hydrocarbons in paraffin and microcrystalline waxes. The procedure involves nitromethane extraction of a cyclohexane solution of the wax followed by chromatography on silica gel paper impregnated with dimethyl formamide. Methylcyclohexane is used as a developer and the polycyclic hydrocarbon spots are located by their fluorescence under ultraviolet illumination. This method forms the basis of the analytical test described in recent West German legislation establishing the purity requirements of certain chewing gum additives, including waxes. Paper chromatographic methods for the separation and estimation of polycyclic aromatics have been described by Malý (*119J*) and by Lijinsky and others (*110J*). The latter authors also report that high temperature gas chromatography with electron-capture detection appears promising. It is rapid, sensitive, and applicable to all of the common polynuclear hydrocarbons through benzo[*g,h,i*]perylene. Ryder and Sullivan (*167J*) have applied ascending paper chromatography for the analysis of the nonvolatile material in solvent hexane. Thin-layer chromatography has been used for the separation of polycyclic aromatics by Badger, Donnelly, and Spotswood (*12J*). They work with plates prepared from partially acetylated cellulose and develop the chromatograms by the ascending technique using a 4:4:1 methanol-ether-water solvent system.

Several applications of thin-layer and paper chromatography not primarily aimed at the polynuclear aromatic problem are worth noting. Wetzel and Peter (*215J*) use paper chromatography to separate bituminous materials into asphaltenes, petroleum resins, and mineral oil types. They claim that semiquantitative results are possible. Rajeswaren and Kirk (*155J*) describe a modification of conventional paper techniques that they call "evaporation chromatography" and give examples of its application for the identification of gasolines, waxes, lubricants, and asphalts. Thin-layer chromatography of aromatic hydrocarbons on silica gel and activated alumina plates has been described by Kucharczyk, Fohl, and Vymetal (*103J*). They detect the spots corresponding to the separated aromatics by spraying with formaldehyde–sulfuric acid reagent, or with tetracyanoethylene, or simply by observing the plates under ultraviolet illumination. Alumina plates were also used by Ognyanov (*136J*) to separate a number of aromatics. The spots were detected by ozonizing and spraying with aqueous indigo solution. Huber (*80J*) uses paper chromatography to determine the concentration of certain individual olefins and diolefins quantitatively in complex mixtures. The olefins are chromatographed as their mercurous acetate complexes. The spots can be detected by exposing the developed chromatogram to HCl vapor and spraying with ammonium sulfide or dithizone.

Urea adduction methods for the separation and determination of normal paraffins in kerosine and higher-boiling petroleum fractions have been described by Frehden and Lăzărescu (*60J*), Yata (*218J*), Negoro, Yagi, and Kuraku (*133J*), Matishev and Bogatova (*121J*), Schmiedel and Teubel (*173J*), and Prohaska, Čubranić, and Bastijanić (*152J*).

Petroleum applications of gas chromatography have continued to grow at an unabated pace during the past two years. Martin (*120J*) has reviewed the analyses performed by gas chromatography in a modern petroleum laboratory and Buzon (*35J*) and Huyten (*83J*) have written brief, but fairly comprehensive, reviews of its application to petroleum products. The use of gas chromatography for the determination of traces of impurities in hydrocarbons and hydrocarbon mixtures been covered by Vigdergauz, Afanas'ev, and Gol'bert (*207J*).

A number of papers have dealt with the effect of temperature, the nature of the support, the nature and amount of the stationary phase, and the gas velocity and outlet pressure on the efficiency of separation and on the time required for analysis. Ashley and coworkers (*8J*) have explored the effect of column loading and have compared the relative efficiency of some common porous and nonporous supports. Waksmundzki, Suprynowicz and Pietrusinska (*211J*) reported the effect of stationary phase composition on the separation of complex mixtures of saturated hydrocarbons. The effect of temperature on the order of elution of certain saturated hydrocarbons has been discussed by Blaustein, Zahn, and Pantages (*26J*). They conclude that for certain hydrocarbons that elute close together, the column temperature may be the critical factor in determining whether or not the compounds will be separated. An interesting review covering the effect of temperature on the gas chromatographic separation of hydrocarbons has been prepared by Vitzthum (*210J*). The problem of choosing suitable conditions for the separation of complex hydrocarbon mixtures has been treated in a systematic and orderly fashion by Bigdergauz and Gol'bert (*25J*).

Several compilations of the retention times of hydrocarbons have been published. Csicsery and Pines (*39J*) tabulate relative retention times for 141 C$_1$ to C$_8$ hydrocarbons on six different stationary phases at temperatures from 25° to 125° C. Doering and Hauthal (*45J*) list relative retention times at 25° to 50° C. for 56 C$_5$ to C$_6$ paraffins, cycloparaffins, monoolefins, and diolefins on eight stationary phases. An extremely comprehensive and useful collection of gas chromatographic data has been made available by the American Society for Testing and Materials (*4J*). Relative retention times are given for over 2000 compounds and involve over 200 stationary phases. This is a reference work that will prove invaluable in helping select the proper stationary phase and conditions to accomplish a given separation.

The satisfactory expression of column performance is still somewhat of a problem. Theoretical plate numbers are cumbersome and often misleading. Hurrell and Perry (*82J*) have proposed the concept of "effective peak number" that appears to have some merit. EPN is related in a simple way to the peak resolution, r, and represents the maximum number of peaks that can be resolved under a given set of operating conditions. It can be calculated for any type of column, and it is immaterial whether or not the operation is isothermal or programmed.

Despite the fact that light hydrocarbon analysis was one of the first problems in the petroleum industry to which gas chromatography was successfully applied, there are still a great many reports in the literature that deal with this subject. Landault and Guiochon (*105J*) and Znamenskaya and Korol (*222J*) describe the high-speed separation of light hydrocarbon mixtures using gas-liquid and gas-solid chromatography. Good separation of the C$_1$ to C$_4$ and some of the C$_5$ hydrocarbons can be achieved with total elution times as short as 50 to 125 seconds. The application of gas chromatography to utility gas analysis has been summarized by Kniebes (*99J*). Methods for the analysis of natural gas, reformed gas, and liquefied petroleum gas that will eventually become ASTM standard methods of test are described. Koons and Walker (*101J*) report that by using $^1/_8$ inch packed columns with a hydrogen flame detector they are able to analyze C$_1$ to C$_5$ paraffin-olefin mixtures in 20 to 48 minutes. If there are no olefins present, the analysis time can be reduced to as little as 3.5 minutes. Liquefied petroleum gas is analyzed in 3 minutes using a $^1/_8$-inch silver nitrate–benzyl cyanide column. Various columns and conditions for the analysis of mixtures of light hydrocarbons have been detailed by Miquel and Benard (*123J*), Zizin and Sokolova (*221J*), and Lulova *et al.* (*115J*). The application of gas chromatography for the estimation of small amounts of C$_4$ hydrocarbons in a methane-hydrogen fraction has been described by Tulupov (*200J*). A method using an internal standard and precut column has been recommended by Hara, Tokai, and Fujii (*73J*) for the determination of the individual C$_1$ to C$_5$ hydrocarbons in crude oils.

The chromatographic determination of small amounts of low-boiling acetylenes and dienes in admixture with other light hydrocarbons was reported by Kontorovich and coworkers (*100J*), by Vigdergauz and his associates (*208J*), and by Heuschkel, Wolny, and Skoczowski (*77J*). The determination of trace impurities in polymerization grade ethylene has been detailed by Zaichenko, Lityaeva, and Markosov (*219J*). Gas chromatography has been successfully applied for the determination of the purity of isobutylene by Dement'eva, Naumova, and Prokopenko (*41J*). Averill and Ettre (*11J*) have successfully separated light hydrocarbon gases using coated capillary columns at room temperature. They claim that their results show that extremely low temperatures are not mandatory for the successful application of capillaries to light gas analysis. Innes, Bambrick, and Andreatch (*85J*) have developed a method for the type analysis of petroleum gases and hydrocarbon vapors. By using parallel chemical absorbers with dual flame ionization detectors, they are able to determine the total content of olefins, acetylenes, paraffins, and aromatics.

The application of gas chromatography for the analysis of gasoline-boiling-range hydrocarbons received a great deal of attention. Mazitova, Virobyants, and Ermakova (*122J*) report the concentrations of 25 C$_3$ to C$_7$ hydrocarbons in crudes from eight fields. They prepared fractions of straight-run gasoline by distillation and analyzed these by gas chromatography using a 1-chloronaphthalene column. Sojak, Gregorik, and Kurcova (*193J*) examined the gasoline portion of Romashkino crude using a similar technique. However, they recommend the use of a column of 3% squalane on silica gel for the separation of the C$_1$ to C$_6$ hydrocarbons. Uhlmann and Prinzler (*202J*) report the analysis of a highly olefinic 90° to 150° C. fraction from the Fischer-Tropsch reaction. Distillation was combined with gas chromatography to produce fairly detailed composition data. Berezkin and Polak (*21J*) are able to report the concentrations of approximately 35 C$_7$ to C$_{12}$ hydrocarbons using a poly(methylphenylsiloxane) oil as the stationary phase. LoChang and Karr (*114J*) identified and determined the concentration of seven normal paraffins, seven 2-methylalkanes, and seven α-olefins of 10 to 16 carbon atoms in a low temperature coal tar. They used displacement chromatography on silica gel to isolate fractions of saturated, unsaturated, and aromatic hydrocarbons. These fractions were then analyzed by gas chromatography using a *m*-di-(*m*-phenoxyphenoxy)benzene column. Albert (*2J*) has described a gas chromatographic apparatus for the simultaneous determination of aromatics, olefins, and saturates, and the individual C_5 to C_{11} normal paraffins in gasoline.

The determination of individual aromatic hydrocarbons in gasoline and naphtha has been described by Shopov, Kotsev, and Georgieva (*182J*) and by Miyake and Mitooka (*125J*). The former use a packed column of fluorene picrate, while the latter determine all 36 C_6 to C_{10} aromatics by successively chromatographing the sample on a squalane and then a polypropylene glycol capillary column. Wingerter (*217J*) reports the use of a packed column of di-*n*-propyl tetrachlorophthalate to resolve all of the C_9 aromatics in a sample of reformer gasoline prepared from Romashkino crude. On the other hand, Smeykal, Hauthal, and Engler (*186J*) found it necessary to use adsorption, distillation, gas chromatography, and infrared spectroscopy to determine the concentration of individual C_9 aromatics in reformate. Mortimer and Gent (*128J*) recommend the use of silicone oil–modified Bentone 34 for the separation of C_6 to C_9 aromatics. Spencer (*195J*) has reported that commercial xylene mixtures may be completely separated in 12 minutes using a 6 foot × $^3/_{16}$ inch column containing 5% Bentone 34 and 5% diisodecyl phthalate on 60 to 80-mesh Chromosorb W.

The complete resolution of all seven of the isomers of *n*-octene was achieved by Bendel and coworkers (*19J*) by using a β, β'-oxydipropionitrile–silver nitrate column followed by a diethylene glycol–silver nitrate column. Columns of silver nitrate and ethylene glycol have been used by Smith and Ohlson (*188J*) and by Shabtai, Herling, and Gil-Av (*76J*, *180J*) to separate monoolefins, diolefins, cyclic olefins, and acetylenes. Cvetanovic, Duncan, and Falconer (*40J*) report the separation of a number of deuterium-substituted olefins using packed columns with a stationary phase of ethylene glycol–silver nitrate. Benzene and perdeuterobenzene have been separated by Liberti, Cartoni, and Bruner (*108J*) using a glass capillary column coated with squalane.

Gas chromatography is being applied for the analysis of the higher-boiling petroleum fractions with reasonable success. The problem of the determination of naphthalene and its alkyl homologs has received the attention of Kawanishi, Funakubo, and Matsumoto (*95J*), Garilli, Lombardo, and Zerbo (*63J*), Gonikberg and coworkers (*67J*) and Walker and Ahlberg (*214J*). The latter authors were able to separate all of the C_{11} through C_{12} alkylnaphthalenes using a 200 foot × 0.01 inch capillary column coated with Ucon 2000. However, 2,6- and 2,7-dimethylnaphthalene were not resolved well enough from each other to allow a good quantitative determination of their individual concentrations. The use of gas chromatography to identify the components of mixtures of alkylphenanthrenes obtained from the degradation of natural products was reported by Solo and Pelletier (*194J*). They present a tabulation of the relative retention times of 60 alkylphenanthrenes on several stationary phases. Subtractive gas chromatography using Molecular Sieves has been used by Schenck and Eisma (*171J*) to determine the concentration of the individual *n*-alkanes in small samples of oil extracted from rock. They found that a certain quantity of isoalkanes is also removed by the sieves and can introduce appreciable error, especially when submilligram samples are used. The problem of the gas chromatographic determination of the concentration of individual *n*-paraffins in petroleum waxes has been discussed in some detail by Hildebrand, Peper, and Dahlke (*78J*). They discovered that if a pour point depressant of the Paraflow type is topped to 300° C. at 3 mm., it provides an excellent stationary phase that is stable at temperatures up to 300° C. A number of other partitioning liquids were tested, but none were as satisfactory.

The rapid separation of relatively high-boiling hydrocarbons at temperatures considerably below their boiling points has been described by Frederick. Miranda, and Cooke (*59J*), Malafeev and coworkers (*118J*) and Bresler, Dobychin, and Popov (*30J*). Glass beads, glass pieces, porous glass, or conventional porous supports coated with a very light loading of stationary phase seem to work reasonably well in the hands of these authors.

The use of capillary column gas chromatography has undoubtedly continued to grow during the past several years. However, few new examples of the practical application of capillaries for the routine analysis of hydrocarbon mixtures have been forthcoming. The use of capillary columns to analyze the complex mixtures encountered in the petroleum industry has been reviewed by Bruderreck (*32J*) and by Walker (*213J*). The quantitative aspects of open tubular columns were discussed by Ettre, Cieplinski, and Brenner (*51J*). Jentzsch and Hövermann (*89J*) have published a critical evaluation of capillary cloumns and have delineated applications where they may be used with maximum effectiveness. Data on the response of the flame ionization detector to different functional groups have been published by Perkins and his coworkers (*142J*, *143J*). Schwartz and Brasseaux (*174J*) describe the use of a capillary coated with a mixed phase of hexadecane and a fluorocarbon oil to effect the complete resolution of a 39-component blend approximating the composition of the 28° to 114° C. fraction of petroleum.

The preparation and use of capillary columns containing a thin film of finely divided solid on their interior walls have been reported by Halasz and Horvath (*71J*, *72J*) and by Schwartz, Brasseaux, and Shoemake (*175J*). Carbon black, ferric oxide, firebrick, alumina, and colloidal silica have all been successfully deposited on capillary tubing. These columns may be used without further treatment to accomplish a wide variety of hydrocarbon separations. They have the advantage that no stationary phase bleeding can take place when high temperatures are employed. They may also be coated with a partitioning liquid, and will hold 10 to 50 times as much stationary phase as conventional capillaries without corresponding increases in film thickness, HETP, and pneumatic resistance. Both of these features make them useful for trace analysis. The use of aluminum capillary tubing containing an integral layer of oxide on the interior wall has also been disclosed by Petitjean and Leftault (*145J*).

Temperature-programmed gas chromatography has become almost indispensable for the quantitative analysis of the wide-boiling mixtures often encountered in petroleum research. Ettre and Kabot (*52J*) have published a critical study of the quantitative reproducibility of an instrument designed for use with capillary columns. McEwen (*116J*) has compared the relative merits of temperature-programmed capillaries and temperature-programmed packed columns for hydrocarbon separations, and concludes that each may have certain advantages depending upon the application. Golay, Ettre, and Norem (*66J*) have developed a nomograph for predicting elution data in a linearly programmed system. An instrument that permits programming from −75° to 400° C. has been developed by Baumann, Klaver and Johnson (*17J*). Samples that contain gases, or are particularly wide-boiling, are easily analyzed and the retention times of individual compounds are reproducible. Temperature programming has been used with considerable success by Lijinsky, Domsky, and Mason (*109J*) for the determination of aromatic hydrocarbons in tars and pyrolysis residues. They work with a column containing 0.1% SE-30 silicone rubber on glass beads and program from 100° to 220° C. at a rate of 3.5° C. per minute.

The use of retention times for qualitative identification purposes has continued to receive some attention. Smith, Ohlson, and Larson (*189J*) describe the identification by boiling point and hydrocarbon type of C_2 through C_7 saturated and unsaturated hydrocarbons from their retention times on several stationary phases. Huguet (*81J*) and Dhont (*43J*) discuss the use of Kovats' "retention index" for the qualitative analysis of hydrocarbon mixtures.

Casu and Cavallotti (*37J*), in an entirely different approach, describe an apparatus which exposes reagent-impregnated strips to the effluent from a gas chromatograph. By this means the class of organic compound represented by the emerging peaks may be established. Oxidation, hydrogenation, hydrogenolysis reactions, or thermal cracking patterns may also be used to aid in the identification of unknown peaks. Keulemans and Perry (*96J*) have produced additional evidence that the gas chromatographic patterns that result when a sample is thermally cracked in a quartz microreactor at 500° C. are reproducible and can be used for identification purposes. Beroza and Sarmiento (*23J*) describe the use of catalytic hydrogenolysis and gas chromatography to aid in the identification of unknown compounds. Smith, Ohlson, and Olson (*190J*) identify monoolefins, diolefins, and acetylenes by trapping the desired peak as it emerges from the chromatograph ozonizing, cleaving, and identifying the fragments by paper chromatography of their dinitrophenylhydrazones. Smith, Larson, and Rydén (*187J*) also apply microhydrogenation to trapped fractions in order to establish the presence of certain hydrocarbons containing olefinic double bonds. Lille (*111J*) uses a two-stage chromatograph in which a short column containing a nickel hydrogenation catalyst is connected in series with the analysis column. This arrangement permits *in situ* hydrogenation. The chromatographic separation of the resulting saturated hydrocarbons provides data that can be interpreted in terms of the carbon number distribution of the original mixture and the types of olefins present.

When the mixtures to be analyzed are extremely complex, or suitable reference standards are not obtainable, packed column or capillary chromatography is often combined with other instrumental methods of analysis. Archer, Shively, and Francis (*6J*) used a combination of gas chromatography and NMR spectroscopy to analyze a narrow-boiling fraction of C_7 olefins. The components were identified by correlating retention time data for a nonpolar Apiezon L column and a highly polar benzyl cyanide–silver nitrate column with NMR data for small fractions collected from the polar column. Of the 15 peaks resolved, six were identified as C_6 olefins, eight as C_7 olefins, and one as a C_8 olefin. The major components were 2-methyl-1-hexene and 2-ethyl-1-pentene. Francis and Archer (*58J*) used similar methods to identify four triisobutylene isomers and six tetraisobutylene isomers in butene polymer. The analysis of complex mixtures of C_6 to C_9 olefins produced by the polymerization or copolymerization of propylene and butene has been described by Leonard and Loewenguth (*106J*). By using fractional distillation, liquid-solid and gas-liquid chromatography, catalytic hydrogenation, and infrared spectroscopy, they were able to identify the major constituents of these materials. A combination of pyrolytic gas chromatography and NMR spectroscopy was employed by Barrall, Porter, and Johnson (*13J*) to investigate the structure and composition of polymers made from pure isobutylene or isobutylene containing various amounts of propylene, 1-butene, 2-butenes, or 2-pentenes. Szynagel and Kecki (*198J*) report the use of gas chromatography and Raman spectroscopy for the analysis of gasolines. They claim that combining these techniques makes the use of standard reference samples unnecessary.

The in-line combination of a gas chromatograph and a rapid-scanning mass spectrometer is becoming a fairly common device for the rapid analysis of complex mixtures. Magnetic-field mass spectrometer–gas chromatograph combinations have been described bv Lindeman, Beach, and LeTourneau (*112J*), Dorsey, Hunt, and O'Neal (*47J*) and Henneberg and Schomburg (*75J*). Klaver and LeTourneau (*98J*) give details of a mass spectrum digitizer that will handle up to five peaks per second and is particularly useful for quantizing data produced by a gas chromatograph–magnetic field mass spectrometer system. Lindeman and LeTourneau (*113J*) have used this combination of instruments to identify 14 individual mono- and bicycloparaffins in a California naphtha. Twelve of these hydrocarbons had not previously been identified in any petroleum. The application of a gas chromatograph-time-of-flight–mass spectrometer combination to reaction studies has been described by Levy, Miller, and Beggs (*107J*). Gaylor and coworkers (*64J*) have used this combination of instruments for the rapid analysis of crude oil fractions with boiling points as high as 385° C.

A number of papers that report novel stationary phase or adsorbent compositions have appeared during the past few years. Several of these hold interesting implications for hydrocarbon analysis. Zhdanov, Kiselev, and Yashin (*220J*) have used porous glass particles for the rapid separation of hydrocarbon mixtures by gas-solid chromatography. They prepare the "adsorbent" by the selective treatment of borosilicate glass. Bombaugh (*27J*) has found that the efficiency of Molecular Sieve columns is greatly improved if the sieve material is ground to a flour and coated on 60 to 80-mesh red Chromsorb. The potential of linear gas-solid chromatography is commencing to receive some scrutiny. Scott (*177J*) has been able to produce linear isotherms (and consequently, symmetrical peaks) by modifying the surface of activated alumina with sodium hydroxide. Huber and Keulemans (*79J*) report that a column packed with uncoated, thermally treated silica gel yielded linear isotherms up to high hydrocarbon concentrations. The separation equaled that of a good gas-liquid column. The advantages of the gas-solid method are the nonvolatility of the stationary material at high temperatures and its usefulness for trace analysis with ionization detectors. Its potential in the latter application has been demonstrated by Huyten, Rijnders, and van Beersum (*84J*), who used a column of deactivated alumina with a sensitive flame ionization detector to determine 0.01 to 100 p.p.m. of lower-boiling hydrocarbons without prior sample concentration. In another interesting paper, Rogers and Altenau (*163J*) describe the preparation of tailored adsorbents for gas chromatography. They prepare these by evaporating volatile substances from crystals so as to avoid collapse of the crystal framework. The preparation of polar stationary liquids of tailored selectivity, by the successive replacement of $[(R_2)Si\text{-}O]_y$ groups along a silicone polymer backbone with $[(R)(N\text{-}C\text{-}R)Si\text{-}O]_x$ groups has been reported by Rotzsche (*164J*).

A number of reports that deal with apparatus modifications and detector design appear to have some import for petroleum analysis. Villalobos and Turner (*209J*) describe several useful valving arrangements that permit the regrouping of gas chromatographic peaks to allow presentation of the analysis in terms of the total concentration of materials of like structure, carbon number, or reactivity. Nuss (*134J*) has given examples of the utility of these systems for group-type and trace analyses. Myers (*129J*) has designed a device that prevents nonvolatile viscous materials from accumulating in the sample introduction system. The apparatus is particularly useful when predominantly nonvolatile materials are analyzed by gas chromatography for traces of low-boiling constituents. Designs for several new detectors have been reported. Perkins and Folmer (*141J*) and Folmer, Yang, and Perkins (*57J*) have modified the catalytic combustion detector so that the signal depends upon ion formation, rather than on heat of combustion. This results in improved selectivity and sensitivity. They claim that the selective characteristics of the catalytic combustion-ion detector should make it useful for the analysis of petroleum mixtures where the aromatic and paraffin peaks are not completely resolved. Schay, Sze'kely, and Traply (*170J*) reported on the construction and evaluation of a very sensitive and efficient catalytic combustion cell. By using a platinum filament coated with a platinum-palladium catalyst, they obtained complete combustion of hydrocarbons at wire temperatures of 150–200° C. The response was linear, and the sensitivity was only one or two orders of magnitude less than that of conventional flame ionization detectors. Varadi and Ettre (*205J*) reported on the use of the QQ detector first described several years ago. This device combines an ionization detector and a mass analyzer in a single unit and permits simultaneous quantitative measurement and qualitative identification. It can be used effectively for the analysis of mixtures that do not exceed its somewhat limited mass range.

Despite the popularity of gas chromatography, infrared, ultraviolet, fluorescence, Raman, and NMR spectroscopy continue to be the methods of choice for the identification or characterization of individual compounds, for the routine analysis of certain simple mixtures, or for the structural analysis of the heavier petroleum fractions. The analytical applications of near-infrared spectroscopy have been discussed by Moore, Gordon, and Eiffert (*127J*). deRuiter (*166J*) has described a rapid method for the determination of the ratio of aromatic to aliphatic hydrogen using near-infrared absorption spectra. The method involves measuring the peak areas of the second overtones of the aromatic and aliphatic C-H stretching vibrations at 1.15 and 1.2 microns, respectively. Lafaix (*104J*) has found that for a series of asphaltene- and resin-free oils, the ratios of the intensities of the infrared bands near 1600 and 720 cm.$^{-1}$ increased regularly from 0.64 to 38.5 on going from paraffinic to highly aromatic samples. He suggests that this ratio may therefore be useful for the classification of crude oils. Glebovskaya (*65J*) uses infrared spectra obtained using a single-beam instrument to characterize petroleum oils according to their aromatic, paraffin, and carbonyl contents. Aromatic and paraffin contents derived from the infrared data exhibited good agreement with data obtained by chemical classification methods. The infrared structural group analysis of the heavier fractions has been the subject of several reports. Methods for the determination of the content of methyl and of methylene groups in saturated fractions have been described by Krasnova and coworkers (*102J*) and by Iogansen and Broun (*86J*). The method proposed by the latter authors is also applicable to the determination of methyl groups in alkylbenzenes. Finkel (*54J*) has developed an infrared method for the analysis of mixtures of alkanes for CH_3, CH_2, and CH groups, and quaternary carbon atoms. Siryuk and Iogansen (*184J*) devised an infrared method for the determination of the aromatic carbon content of petroleum oils. For oils containing less than 10% aromatic carbons, the method gives results that compare favorably with those obtained by hydrogenation. Above 10% aromatic carbons, the hydrogenation method is more accurate. Goodspeed and Montgomery (*68J*) have used infrared spectroscopy to determine the numbers of methyl and methylene groups per molecule in the oil and resin fractions isolated from Athabasca bitumen.

A number of papers testify to the fact that infrared spectroscopy is still useful for the analysis of mixtures of gasoline-boiling-range hydrocarbons. Densham and Parkyns (*42J*) use infrared to establish the benzene content of crude benzole samples. Rashkes (*157J*) and Ishida (*87J*) have developed infrared methods for the analysis of mixtures of alkylbenzenes. Pop and Stănciulescu (*148J*) use infrared spectroscopy to determine the concentrations of benzene toluene, ethylbenzene, and styrene in material produced by the dehydrogenation of ethylbenzene. The error is ±1% over the full concentration range of 0 to 100%. An infrared method for determining the concentration of α-olefins in wide-boiling-range fractions prepared from ethylene by the Ziegler process has been described by Saier, Cousins, and Basila (*168J*). The concentrations of the various olefin impurities are also accessible using this method.

Pobiner, Wallace, and Hofmann (*147J*) have suggested that hydrocarbons and carboxylic acids can be determined in systems containing base, water, and solvent by extraction, ion exchange, and infrared absorption or by extraction and ultraviolet absorption. The former method is preferred for aliphatic and the latter for aromatic hydrocarbons, and their carbanion oxidation products. The ultraviolet determination of the isomeric xylenes and ethylbenzene has been described by Przybylski (*153J*) and by Jokl and Knizek (*90J*). The latter authors use a six-component matrix which is solved by machine computation. The analysis requires one hour and the errors are ±1% or less for each C_8 isomer. Ishida (*88J*) has developed an ultraviolet method for the determination of the concentrations of *o*-, *p*-, and *m*-ethyltoluene in mixtures of the three isomers. The error is less than 2% for each component. Shabalin, Kruglov, and Vaisberg (*179J*) report an ultraviolet method for the determination of naphthalene, 1-methylnaphthalene, 2-methylnaphthalene, dimethylnaphthalenes, and trimethylnaphthalenes in catalytic gas oils. Siryuk and Zimina (*185J*) use an ultraviolet method to estimate the concentration of naphthalenes, phenanthrenes, and anthracenes in virgin and cracked stocks with boiling points up to about 400° C. A useful method for the determination of traces of C_4 and C_5 acetylenes in hydrocarbons has been described by Scoggins and Price (*176J*). Acetylenes are hydrated to carbonyls with mercuric sulfate–sulfuric acid catalyst, the carbonyls are converted to their 2,4-dinitrophenylhydrazones, and the concentration of the latter is determined by measuring the ultraviolet absorbance at 340 mμ.

Fluorescence methods have received some attention for the determination of polynuclear aromatics. Dubois and Monkman (*49J*) describe a scheme of fluorescence analysis that can be used to determine a number of the commonly encountered polycyclic hydrocarbons. An internal standard method for the determination of 3,4-benzpyrene by fluorescence spectroscopy has been reported by Personov (*144J*). Bergmann (*22J*) used silica gel chromatography and fluorescence spectroscopy to examine a commercial paraffinum liquidum for the presence of this hydrocarbon. He found the concentration to be well below 2×10^{-9} weight %. In connection with geological studies, Alekseeva (*3J*) has employed liquid chromatography and fluorescence methods to estimate the content of certain polycyclic aromatics in oils obtained from the extraction of rock samples.

A fairly comprehensive review of Raman spectroscopy has been compiled by Green (*69J*). Recent advances and applications for structure determination and the analysis of hydrocarbon mixtures are emphasized. A method for the analysis of mixtures of isomeric butenes by Raman spectroscopy has been reported by Nakamura (*132J*). Burova and Khodzhaev (*33J*) used Raman spectroscopy in combination with other methods to analyze Uzbekistan crudes from three different producing formations in considerable detail. The relative amounts of 43 paraffins, 32 naphthenes, and 37 aromatic hydrocarbons in the gasoline boiling range were reported.

The integration of NMR proton absorption bands was used by Jungnickel and Forbes (*92J*) to determine the apparent percentage of chemically different hydrogen types in 24 organic compounds. The standard deviation from the theoretical values was 0.3%. Moniz and Dixon (*126J*) have determined the relaxation times of a number of hydrocarbons over a range of temperatures using the spin-echo method. They claim that these data provide significant information concerning molecular motion in liquid hydrocarbons. A new method for the characterization of the the heavier petroleum aromatic fractions has been developed by Williams and Chamberlain (*216J*). The density, elemental analysis, molecular weight, and NMR hydrogen distribution are determined. A modification of van Krevelen's densimetric method is used to determine the number of aromatic rings and the number of naphthene rings in the average molecule. The rings are assumed to be kata-condensed. The NMR data then permit calculation of the number of methyl groups on aromatic rings and the number, average length, and amount of branching of the side chains. NMR also indicates the distribution of aromatic ring hydrogens between condensed and noncondensed rings and permits the identification of certain individual ring systems. Carbon-13 nuclear magnetic resonance has been used by Friedel and Retcofsky (*61J*) to identify tri- and tetra-substituted olefins and to determine the total number of olefinic carbon atoms in complex hydrocarbon mixtures. A novel electron spin resonance method for the detection and estimation of some polynuclear hydrocarbons has been reported by Flockhart and Pink (*56J*). They claim that less than 10^{-10} mole of anthracene, perylene, dimethylanthracene, and naphthacene can be detected by adding a strongly dehydrated silica-alumina cracking catalyst to a benzene or carbon disulfide solution of the hydrocarbon and measuring the concentration of the resulting stable radicals by ESR.

Mass spectral methods for hydrocarbon analysis are being applied to materials of increasingly higher molecular weight primarily because of the availability of instruments with the necessary resolving power and the development of practical high temperature inlet systems. The most significant reports that deal with the analysis of gasolines and naphthas describe the combined use of gas chromatography and mass spectrometry, and have already been cited. A high temperature sample introduction device has been described by Oshima and Katsumata (*139J*). In a companion paper, Oshima (*138J*) reports the development of a method for the type analysis of mixtures of C_{12} to C_{22} normal and isoparaffins made possible by use of this high temperature inlet. Low ionizing voltage mass spectrometry was used by Sharkey, Schultz, and Friedel (*181J*) to determine the carbon number and compound type distribution of neutral oils derived from coal. The analytical possibilities of medium-voltage (15 to 25 volts) mass spectrometry have been pointed out by Joly (*91J*). He claims that the use of intermediate voltages leads to more accurate results and allows the determination of a larger number of components without the use of a preliminary separation. Examples of the application of this technique to gasolines, jet fuels, and gas oils are given. Oshima and Katsumata (*140J*) suggest that intermediate voltages are potentially useful for the quantitative analysis of butene and pentene isomers. A rearrangement technique for the analysis of C_9 to C_{42} monoalkylbenzene mixtures has been developed by Boyer, Hamming, and Ford (*29J*). The method takes advantage of the consistency of rearrangement phenomena and employs sensitivity data that are independent of volume or pressure measurements. The mass spectrometric analysis of heavy reformates and hydrodealkylated products in the 350° to 550° F. boiling range has been described by Snyder, Howard, and Ferguson (*192J*). The concentrations of eight hydrocarbon types and of alkylbenzothiophenes can be reported along with a carbon number distribution of alkylnaphthalenes. The accuracy is about ±1%, and the analysis requires less than 2 man-hours per sample.

Bartz and his associates (*14J*) and Aczel and coworkers (*1J*) have used mass spectrometry along with catalytic microdehydrogenation and infrared, ultraviolet, and NMR spectroscopy to characterize the aromatic hydrocarbons in a 622 to 625° F. distillate fraction from catalytic cycle stock. The aromatics were first fractionated by elution chromatography over alumina. The C_nH_{2n-12} series consisted of naphthalenes and dibenzothiophenes. The C_nH_{2n-14} series consisted chiefly of tetrahydroanthracenes, tetrahydrophenanthrenes, benzindans, and the corresponding ketones. The -16 series was composed primarily of alkylfluorenes. The -18 series contained methylanthracenes and phenanthrenes. Cornu, Copet, Ulrich, and Bedague (*38J*) used similar techniques to analyze a catalytic reformer residue. They found that the principal aromatic types were fluorenes and phenanthrenes. Carlson, Andre, and O'Neal (*36J*) have used molecular distillation, adsorption chromatography, and high resolution mass spectrometry to analyze fractions of a residual oil with average molecular weights as high as 780. Hydrocarbon analyses were made by parent-peak and fragment-peak methods. Cycloalkanes containing up to seven rings and aromatic subtypes containing up to nine rings were found in the heaviest fraction.

Chemical methods for hydrocarbon analysis have been chiefly concerned with the determination of olefins, although several reports dealing with the quantitative determination or qualitative identification of aromatics have appeared. The problem of the determination of olefinic unsaturation has been reviewed by Fleet (*55J*). Coulometric bromination procedures have been described by Baumann and Gilbert (*16J*), Walisch and Ashworth (*212J*), and Roberts (*159J*). An automatic end point detector for coulometric titrations has been designed by Roberts and Brejcha (*160J*). A scheme for the determination of other olefinic impurities in mixtures of normal α-olefins has been reported by Suatoni (*197J*). An olefinic mixture in the C_{12} to C_{18} range is hydrobrominated in the presence of lauroyl peroxide. Normal α-olefins are converted to normal primary bromides, which are separated from other alkylbromides by adsorption on 5A Molecular Sieves. The weight of the unadsorbed bromide is a measure of the impurity. A rapid, precise hydrogenation procedure for the determination of olefinic unsaturation has been developed by Brown, Sivasankaran, and Brown (*31J*). The catalyst is prepared *in situ* by the reaction of platinum metal salts with sodium borohydride. Hydrogen is also evolved *in situ* from the borohydride, which is automatically introduced into the reaction vessel through a special valve. Hydrogenation is complete within 1 to 2 minutes after sample introduction. The unsaturation is calculated from the volume of borohydride solution consumed.

According to Karpov (*93J*), tertiary pentenes can be determined in a mixture of C_5 olefins by hydrochlorination and hydrolysis. Buzlanova, Kozhikohova, and Polyanskiĭ (*34J*) attack the same problem by adding a known excess of standard mercuric sulfate and back-titrating with ammonium thiocyanate. They claim that the relative error is ±5%. Chemical methods for the determination of acetylene have been proposed by Usami (*203J*) and Tur'yan and Romanov (*201J*). The former reacts acetylene with aqueous mercuric acetate and determines the concentration of the addition compound polarographically, while the latter authors use an amperometric titration based on the reaction of acetylene with mercurous nitrate in ammonia solution.

Gronsberg (*70J*) has developed a scheme for the analysis of certain C_6 to C_9 mixtures of aromatics by selective nitration. Reynolds and Wild (*158J*) use nitration and polarography to determine small amounts of benzene in toluene. A chemical procedure for the determination of phenanthrene in mixtures with other aromatics has been suggested by Bednov, Sukhorukova, and Novikov (*18J*). The method involves successive treatment with formaldehyde, iodine pentoxide, sodium bisulfite, and *o*-phenylenediamine hydrochloride, requires 3 hours, and is accurate to within 3% relative.

Color reactions are of some importance for the quantitative determination or qualitative detection of hydrocarbons containing unsaturated carbon-carbon linkages. Schenk, Santiago, and Wines (*172J*) have studied the pi complexes that tetracyanoethylene forms with phenols, aromatic hydrocarbons, and aromatic ethers and conclude that quantitative analyses using this reagent are feasible. Bauer (*15J*) has demonstrated that mono-, di-, tri-, and tetra-substituted unconjugated olefins can be differentiated by the spectra of their pi complexes with tetracyanoethylene. In these spectra a band appears in or near the visible and its position is strongly dependent on the degree of substitution at the double bond. Sawicki, Stanley, and Elbert (*169J*) report a large number of electrophilic color reagents that are suitable for the spectrophotometric determination of azulene. All of the reagents listed can detect as little as 1 μg. of this hydrocarbon.

Several qualitative detection methods for unsaturated organic compounds have been described. Okuda (*13J7*) finds that benzaldehyde and sulfuric acid will produce a color with compounds containing a carbon-carbon double bond with at least one hydrogen, compounds that readily form such a bond when brought in contact with a mineral acid, and compounds with a triple bond. Saturated, aromatic, and heterocyclic compounds do not undergo the color reaction. Ognyanov (*135J*) is able to detect unsaturated aliphatic and arylaliphatic compounds that have been separated by thin-layer or paper chromatography by ozonizing and spraying with a solution of indigosulfonic acid. The reaction is also sensitive to phenols, aromatic alcohols, aliphatic and aromatic aldehydes, and polynuclear aromatic hydrocarbons. The spots are white on a blue ground. Khin and Szasz (*97J*) detect aromatic compounds on paper chromatograms by exposing the chromatogram to bromine vapor and spraying with an alcoholic solution of dimethyl yellow. Red spots on a yellow background are obtained. Talsky (*199J*) has reported additional work on the use of aluminum chloride as a reagent for the group analysis of organic compounds. He finds the colors produced by the reaction of a chloroform solution of aluminum chloride with aromatic ring compounds, polynuclear aromatics, and heterocyclic compounds may be related to the structure of the compounds that undergo the color reaction.

Sulfur and Sulfur Compounds

J. C. Morris and W. E. Haines

Bureau of Mines, U. S. Department of the Interior,
Laramie, Wyoming

Improvements in total sulfur determinations by combustion methods were the principal feature of several publications. More rapid combustion of large amounts of sample was achieved by Jordan (*32K*), who substituted a small-bore oxyhydrogen burner in the Wickbold apparatus. Methods to improve combustion were reported by Takens (*63K*), Radmacher and Hoverath (*51K*), and the International Conference of Benzole Producers (*31K*). By choosing appropriate absorber solutions, Hanykova (*29K*) was able to determine the amount of both SO_2 and SO_3 in combustion products. Sima, Taslar, and Juklova (*57K*) designed special apparatus for determining sulfur in the parts per million range in liquid fuels using combustion followed by sulfate determination. Malissa and Machherndl (*43K*) evaluated the effectiveness of the Schoeniger flask combustion technique for various types of sulfur compounds. Application of the Schoeniger technique to the determination of sulfur in hydrocarbons was described by Liddell (*40K*), Gomoryova (*25K*), Fabre (*19K*), and Levaggi and Feldstein (*38K*). Boisselet and Baron (*7K*) and Pentelow (*48K*) recommended titration of the sulfate ion in the combustion products with barium perchlorate. The use of a new highly sensitive indicator for barium in sulfate determinations was described by Novikova and Basargin (*44K*). Koga, Kajikawa, and Nonaka (*36K*) precipitated the sulfate with excess barium (ethylenedinitrilo)-tetraacetate, then titrated the liberated (ethylenedinitrilo)-tetraacetic acid with standard barium chloride solution, using an indicator. Ciola (*9K*) added excess barium perchlorate to precipitate the sulfate ion, then titrated the excess barium with standard (ethylenedinitrilo)-tetraacetic acid potentiometrically. As little as 1 p.p.m. of sulfur in benzene was determined by Ozaki and Yasuda (*46K*), using lamp combustion and a turbidimetric measurement of the precipitated barium sulfate.

Several workers determined sulfur by reduction of the sample and subsequent analysis of the hydrogenated products. Okuno, Morris, and Haines (*45K*) reduced the sample catalytically in a stream of hydrogen and, by comparison of the amounts of methane and hydrogen sulfide in the products, calculated the sulfur content of the sample. A modification of the Raney nickel reduction method of Granatelli permitted Peter (*49K*) to determine microgram quantities of sulfur with greater precision. Reed (*52K*) found the preparation of the catalyst to be a critical factor in the determination of sulfur in toluene by Raney nickel reduction. The reduction method of Schluter was modified by Kato, Fujishima, and Takeuchi (*33K*) to determine microgram quantities of sulfur in organic materials.

The use of x-ray methods in the determination of sulfur in petroleum fractions has received increasing interest. Nondispersive analysis of the fluorescence spectrum of x-rays generated by tritium β-radiation was used by Burckhardt, Gruetter, and Stoessel (*8K*) to determine sulfur in petroleum fractions. The Sub-panel on Sulphur by X-Ray Absorption, Institute of Petroleum (*30K*), recommended a 2-curie tritium-titanium or tritium-zirconium target for their procedure, discussed factors affecting accurate counting, and included a calibration procedure. Rowley (*54K*) determined sulfur in hydrocarbon using a tritium-zirconium source at 8 k.e.v., an x-ray-sensitive Geiger-Müller tube, and an amplifier-recording system. Tritium or iron-55 was the source used in a method developed by Csanyi, Horanyi, and Levay (*12K*). The sulfur content of motor fuels was determined in methods reported by Dahn and Brunner (*15K*) and Khachaturov (*34K*), using iron-55 as the radiation source. Afonin and coworkers (*2K*) described a method and apparatus for continuous automatic monitoring of the sulfur content of a diesel fuel stream. Levay, Csanyi, and Kralik (*39K*) pointed out that, for x-rays of wavelength 0.5 to 5.0 A., the mass-absorption coefficient of sulfur is several orders of magnitude greater than that of carbon and hydrogen; a method of sulfur determination based on this information was described. A method and the required apparatus for continuous determination of sulfur in hydrocarbons using a promethium-147–aluminum brehmsstrahlung source were described by Rhodes, Florkowski, and Cameron (*53K*). Gilpin and Franks (*22K*) used a promethium-zirconium source to determine sulfur in petroleum products. Sahores (*55K*) developed apparatus to determine sulfur in gas streams at pressures up to 500 atm.

Sulfur isotope ratios have been used as clues in the studies of the age and origin of petroleum. Eremenko and Pankina (*16K*) studied the relative amounts of sulfur isotopes in various Russian crude oils and in the associated natural gases; greater variations in the isotope ratios were found in the gases.

The same authors (*17K*) concluded that differences in the isotope ratios found in the Pashiisk crude oils were related to the genesis of the oil.

A scheme of analysis for the types of sulfur compounds in gasoline, kerosine, and gas oil was developed by Luk'yanitsa (*41K*); free sulfur, hydrogen sulfide, thiols, sulfides, and ring sulfur were determined. Prezler and Oehler (*50K*) studied the chromatographic behavior of various types of sulfur compounds on silica gel and suggested alterations in the gel and in the procedure to give better separations. The types of sulfur compounds occurring in fractions of Mukhanovsk crude boiling below 350° were determined by Gordash and coworkers (*26K*), who found sulfide and residual sulfur to be the abundant types. A fractional combustion technique in a temperature-programmed oven followed by analysis of the products was used by Pell and Malissa (*47K*) to study the character of carbon-sulfur bonds in a petroleum fuel. Thompson and coworkers (*65K*) developed a microhydrogenation technique for identifying thiols, sulfides, thiophenes, and benzothiophenes in petroleum fractions. In a modification of the method described by Thompson and coworkers, Beroza (*5K*) characterized the hydrocarbons obtained by hydrogenation of thiols, cyclic and paraffinic sulfides, and disulfides. Szepesvary (*62K*) proposed a polarographic method, using a Novak cell and a mercury lower electrode, to determine elemental sulfur, thiols, and thiophenes in crude oils. Organic, pyritic, and sulfate sulfurs in Green River oil shale were differentiated in work reported by Smith, Young, and Lawlor (*58K*). Glebovskaya and Bol'shakov (*23K*) discussed the use of infrared spectroscopy in studying the sulfur compounds present in petroleum products.

Hydrogen sulfide and organic sulfur compounds in natural gas were determined by Whitehead and Austin (*66K*) with an iodine-bromine titration in an automatic system suitable for stream monitoring as well as spot analyses. Claytor and Jones (*10K*) developed a calibration system and studied the reproducibility and accuracy of the methylene blue method for hydrogen sulfide determination. Fredericks and Harlow (*21K*) determined thiols in sour natural gases by separation on a gas-liquid chromatographic system with an automatic microcoulometric titration cell as the sensing device. Luk'yanitsa and Gal'pern (*42K*) reported a polarographic method for determining thiols, hydrogen sulfide, and free sulfur on a single sample. Sporek and Danyi (*60K*) identified thiols by converting them to disulfides by oxidation with iodine in ether solution followed by gas chromatographic separation and identification of the disulfides. Dahmen, Dijkstra, and Verjaal (*14K*) converted thiols to dinitrophenyl thio ethers, which were separated chromatographically and measured spectrophotometrically. The determination of thiols in hydrocarbons by potentiometric titration with alcoholic silver nitrate was described by Hammerich and Gondermann (*28K*). A method for monitoring the thiol content of natural gas streams using radioactive silver nitrate was reported by Stout and Early (*61K*). Substituted aromatic thiols were analyzed by Croitoru and Freedman (*11K*), who treated the thiol mixture with acetic anhydride and separated the resulting esters by gas chromatography.

Organic sulfides in petroleum fractions boiling below the heavy gas oil range were determined by Koga, Kajikawa, and Kawaguchi (*35K*), using an adaptation of the sulfur-iodine complexing method. Thompson and coworkers (*64K*) identified several alkyl cycloalkyl sulfides in Wasson, Tex., crude oil by microhydrogenation and gas chromatography. The resolution of sulfides and thiols by isothermal and programmed temperature gas chromatography was examined by Baumann and Olund (*4K*). A two-stage gas chromatographic system was used by Adreen and Kniebes (*1K*) to determine alkyl and cyclic sulfides, and thiols in natural gas streams. Zabrodina (*67K*) described a bromimetric microdetermination of organic sulfides.

Curtis and Phillips (*13K*) separated 26 thiophene derivatives by thin-layer chromatography; detection was by the ultraviolet fluorescence of the isatin-treated plate. Dibenzothiophenes were separated from fractions of Lacq petroleum by Espagno and Poquet (*18K*), using distillation followed by crystallization from ethanol. Thiophenes, benzothiophenes, and dibenzothiophenes were identified in petroleum fractions by Bestougeff (*6K*), who employed both chemical and spectral techniques to analyze a concentrate of sulfur compounds. Snyder, Howard, and Ferguson (*59K*) identified alkylbenzothiophenes in a kerosine distillate using mass spectra. Krol, Rozanova, and Rozhdestvenskaya (*37K*) separated benzothiophene and cyclic sulfides from transformer oils.

Several useful reviews and comparisons have been published recently. Methods for microdetermination of sulfur in petroleum fractions were compared by Gulyaeva and coworkers (*27K*). Comparison of ranges of applicability, precision, and analysis time for numerous methods for the determination of sulfur in petroleum products, ranging from gases to bitumens, was given by Franks and Gilpin (*20K*). The reproducibility of results in the analyses of various sulfur types by published methods was evaluated by Golynets and Ponomareva (*24K*). The chemical thermodynamic properties of 100 alkane thiols, sulfides, and disulfides were included in work reported by Scott and McCullough (*56K*). A group of papers that covered many aspects of the chemistry and reactions of organic sulfur compounds, especially as they relate to analytical and processing problems of petroleum and its products, formed a part of a symposium (*3K*) on the sulfur and nitrogen compounds in petroleum.

Nitrogen and Nitrogen Compounds

D. R. Latham and W. E. Haines

Bureau of Mines, U.S. Department of the Interior,
Laramie, Wyoming

A MICRO-KJELDAHL method for the determination of total nitrogen in petroleum was described by Battles and coworkers (*5L*); the advantages of the method are elimination of fume problems, less time for analysis, smaller samples, and less space for equipment. Mann (*28L*) reported a method for determining the nitrogen from a regular Kjeldahl digest by the reaction of ammonia and phenol in the presence of base to form the "phenol-indophenol" complex; the absorptivity of the colored complex is then measured spectrophotometrically. Hashmi, Ehsan, and Umar (*18L*) determined the nitrogen from the Kjeldahl digest by adding sodium hypobromite, potassium iodide, and sulfuric acid and titrating the liberated iodine with sodium thiosulfate. The reaction of ammonia with indantrione hydrate to form the blue ammonium iodide complex was used by Jacobs (*22L*) to determine nitrogen in organic compounds; the absorption maximum of the complex was measured spectrophotometrically and the nitrogen content of the sample calculated from standard curves. Recent modifications of the Kjeldahl method were reviewed by Ashraf and Shah (*3L*).

Determination of nitrogen in petroleum products boiling above 300° C. by Dumas combustion and gas chromatographic analysis of the gases was described by Dubois and Leonard (*14L*). Mizukami and Miyahara (*29L*) developed an apparatus for multiple determinations by the micro-Dumas method. Farley, Guffy, and Winkler (*15L*) adapted the Coleman analyzer to the determination of nitrogen in lubricating oils and additives at the 20-p.p.m. level. The analysis time for the Dumas method was reduced by the use of NiO as the packing in the combustion tube according to Abramyan, Kocharyan, and Karapetyan (*1L*). Block, Morgan, and Siggia (*10L*) applied differential reaction rate techniques to modify the micro-Dumas method. Hozumi (*20L*) burned the sample in the presence of copper gauze and BaO-MgO; the gaseous nitrogen was measured by displacement of mercury. Vecera (*45L*) used Co_3O_4-CuO as the catalyst for the sample combustion. A high frequency induction furnace was used by Parsons, Pennington, and Walker (*34L*) to burn the sample; the nitrogen gas was analyzed by gas chromatography on 5A Molecular Sieves. Sternglanz and Kollig (*41L*) evaluated a commercial nitrogen analyzer and found it to be superior to the standard Dumas procedure; these authors developed a modification for refractory compounds such as purines and pyrimidines that utilizes V_2O_5 as a combustion aid. Nitrogen and oxygen were determined in the same sample by Terent'ev and coworkers (*43L*); the sample was burned in the presence of a specially prepared "nickelized" carbon black and the reaction products were analyzed by gas chromatography.

The simultaneous determination of carbon, hydrogen, and nitrogen by the dry combustion technique was studied by several workers using a variety of methods to analyze the product gases. Frazer (*17L*) measured the volatile components—CO_2, H, and N—volumetrically in a gas buret. Walisch (*46L*) used a system of catharometers to measure the same gases. Kirsten, Hozumi, and Nirk (*26L*) measured the N at reduced pressure, while the CO_2 and H_2O were frozen out instead of being absorbed in KOH solution. Gas chromatography on Molecular Sieve was used by Nightingale and Walker (*33L*) to measure N, CO_2, and C_2H_2 produced. Hozumi and Kirsten (*21L*) calculated the H_2O from the difference in the volumes of the gases at room temperature and 192°; CO_2 was determined by measuring the shrinkage in gas volume upon treatment with alkali; the volume remaining, due to N, was measured directly. Thuerauf and Thiemann (*44L*) absorbed and weighed separately the CO_2 and H_2O and determined the N volumetrically.

A mixture of chromic and sulfuric acids was used by Terent'ev and Luskina (*42L*) in the determination of total nitrogen in organic compounds by a wet oxidation method. Shah and Quadri (*38L*) decomposed the sample with sulfuric acid in a sealed tube; the $(NH_4)_2SO_4$ in the digest was converted to NH_4 I on an anion exchange resin and determined by the Leipert amplification method.

Gas chromatographic separation of nitrogen-containing heterocyclic compounds was studied by several investigators using various liquid substrates and solid supports. Rezl (*36L*) determined the specific elution volumes of 15 quinoline derivatives using a column of glycerol on Celite. Pyridine homologs were separated by Kametani and Kubota (*24L*) on a column of C-22 firebrick coated with a mixture of 10% ethanolamine and 5% *o*-phenylphenol. Kirsten and Andren (*25L*) found that Tween 20 (polyoxyethylene sorbitan monolaurate) as the liquid phase on Embacel gave good separation of pyridines. Mossini and Vitali (*31L*) reported good separation of indole and substituted indoles using a column of Silicone Oil 550 on Celite. A commercial dye, Rhodamine B Base, was used as the liquid phase by Ferrin, Latham, and Haines (*16L*) to separate nitrogen compounds from aromatic hydrocarbons and to separate nitrogen types from each other.

Snyder and Buell (*39L*) developed a method for the routine determination of alkylindoles, carbazoles, and benzcarbazoles in cracked gas oils; linear elution adsorption chromatography was used to separate the compounds; the types were determined from their ultraviolet spectra. Bezinger, Abdurakhmanov, and Gal'pern (*9L*) quantitatively determined pyridines and quinolines by potentiometric titration with $HClO_4$ in acetic anhydride solution before and after treatment with anhydrous formic acid. A method to determine the chemical structure of amines and amides with chain lengths up to C_9 was developed by Beroza (*6L*) using catalytic hydrogenation to produce the paraffin or cycloparaffin, which is analyzed by gas chromatography. The method was extended by Beroza and Sarmiento (*7L*) to determine the structure of compounds with carbon chains up to C_{20}. Munday and Eaves (*32L*) discussed the use of ion exchange resin in the separation of pyrrolic-type compounds, organic acids, and bases from petroleum distillates.

Jewell and Hartung (*23L*) identified the following types of basic nitrogen compounds in a cracked, heavy gas oil: quinolines, benzoquinolines, indolo- and carbazoloquinolines, hydroxybenzoquinolines, 1,10-phenanthroline, and tetrahydrocarbazoloemines. Bezinger, Abdurakhmanov, and Gal'pern (*8L*) investigated the nonbasic nitrogen compounds in a number of Russian oils and concluded that these compounds are mostly amides of the *N*-substituted lactam type. The same authors (*9L*), in an investigation of the basic nitrogen compounds, found quinolines, acridines, and dihydroacridines to be present.

Rein, Miville, and Fainberg (*35L*) separated nitrogen and oxygen gases using a gas chromatographic column of Silicone Oil 200 on Chromosorb P. A method to analyze the equilibrium vapor and liquid phases of a hydrocarbon-nitrogen system was worked out by Roberts and McKetta (*37L*), who used a multiple column arrangement consisting of Molecular Sieve, Ansul ether on firebrick, and SE-30 on firebrick.

The various methods of separating porphyrins from petroleum were the subject of a review by Corwin (*11L*). Costantinides, Arich, and Lomi (*13L*) reported an improved spectrophotometric method for the determination of the porphyrin content of petroleum residues. Stefanescu (*40L*) studied a number of Romanian crude oils whose porphyrin content was found to vary from 94 to 881 p.p.m. Several Japanese crude oils, studied by Hodgson and coworkers (*19L*), were found to contain from 0.02 to 200 p.p.m. of vanadyl and nickel porphyrins and porphyrin carboxylic acids. Ball and Rall (*4L*) reported information on the metal and porphyrin content of Wilmington, Calif., crude oil. Corwin and Baker (*12L*) in a recent study of the same oil, reported that all of the vanadyl porphyrins isolated were of the phyllo series. A study of the porphyrins in several crude oils from the Dnieper-Donets depression by Makovet'skii (*27L*) indicated that all of these oils are of related origin; spectra suggested that the porphyrins were of plant origin or constituted mixtures in which chlorophyll derivatives predominated. Morandi and Jensen (*30L*) found that the porphyrins in shale oil were predominantly of the etio type; the average compound consisted of the porphine ring with nine carbon atoms of methylene constituents.

A symposium (*2L*) on petroleum-type nitrogen and sulfur compounds included papers on the occurrence, separation, concentration, and identification of nitrogen compounds in petroleum.

Oxygen and Oxygen Compounds

W. E. Haines

Bureau of Mines, U.S. Department of the Interior, Laramie, Wyoming

Methods for the determination of total oxygen included modifications of older techniques and application of newer tools. Salzer (*46M*) suggested adding conductometric end point detection to Drekopf and Braukmann's modification of the Unterzaucher procedure. Haraldson (*19M*) showed that the Unterzaucher procedure gives erroneous results for samples containing sulfur. Ratkovics and Szepesvary (*44M*) evaluated the Unterzaucher and Novak methods for oxygen determinations in heavy petroleum products and synthesis gas, respectively. Mlinko (*32M*) studied the ter Meulen method in detail and suggested several modifications to reduce the errors. Terent'ev and coworkers (*56M*) determined oxygen and nitrogen in the same sample by reduction at 900° with "nickelized" carbon black followed by gas chromatography of the reaction products. Procedures for the determination of total oxygen by neutron activation analysis were described by Stallwood, Mott, and Fanale (*51M*) and Anders and Briden (*3M*).

Oxygen dissolved in organic liquids was determined by Novak (*37M*); the oxygen was expelled by a stream of nitrogen or argon into a special cell in which it was reduced electrolytically; the current was proportional to the amount of oxygen. Natelson and Stellate (*35M*) described a system that permits anaerobic sampling, release of dissolved gases under vacuum, and injection of released gases into a gas chromatographic column.

Water in hydrocarbon oils and gases continues to receive the attention of analytical chemists. Engel and Moebes (*15M*) used the direct Karl Fischer titration for the determination of very low water concentrations in insulating oils. Special apparatus for the Karl Fischer titration was suggested by Petukhova and Fomenko (*43M*) and Dirscherl and Erne (*14M*). Muroi (*34M*) reported that water in gaseous samples could be accurately determined by passing the gas through anhydrous methanol, which is then titrated. Two coulometric procedures were described; one, by Onuska (*38M*), involves the reaction of water with P_2O_5, while the other, by Swensen and Keyworth (*53M*), measures concentrations less than 10 p.p.m. by electrolytic generation of iodine for the Karl Fischer determination. Apparatus for water determinations based on changes in dielectric constant as a function of water content were described by Honorat (*21M*) and by Korchagina and coworkers (*28M*, *29M*). Neuvirt and Ksandr (*36M*) measured traces of water in organic solvents by a differential infrared technique based on the different absorption bands that water exhibits in active and inactive solvents. Critchfield, Meeker, and Bishop (*11M*) measured the absorption band near 1.9 microns: the dried sample was used as the reference solution. Baumann (*5M*) studied the application of amperometric hygrometers to the determination of concentrations of water below 10 p.p.m. in gases. Penther and Notter (*41M*) described a process-type analyzer for determining water in liquid hydrocarbons; the analyzer combines chromatographic separation and electrolysis-cell detection. Tarasov (*54M*) reported that less than 0.04% water in petroleum products may be determined by centrifuging at 5000 r.p.m. in a calibrated test tube. The volume of acetylene evolved upon heating a sample with calcium carbide is the basis of a method by Aliev (*2M*). Berezkin, Mysak, and Polak (*6M*) suggested a chromatographic determination of the products from the reaction of the sample with $NaAlH_4$. Stoll, Rembold, and Weiss (*52M*) determined small amounts of dissolved water in oils by sweeping the oils with nitrogen, collecting the gases in methanol, and determining the methanol by gas chromatography.

Alcohols produced by the oxidation of aliphatic hydrocarbons were determined by Kotel'nikov, Prokhorova, and Gerasimova (*30M*) using the absorbance of their nitrites at 357 and 370 $m\mu$. Kamzolkin, Bashkirov, and Lodzik (*25M*) used a method involving chromatography on silica gel to study the composition of alcohols obtained from the direct oxidation of light petroleum. Trace amounts of primary and secondary alcohols, through C_{12}, were determined by Scoggins (*48M*) using ultraviolet spectrophotometry of their *p*-nitrobenzoyl chloride esters. Robinson (*45M*) used gas chromatography with Silicone E-301 to analyze a mixture of alcohols up to C_{20}. Sokolov and Kolesnikova (*50M*) studied the use of cetyl alcohol or tritolyl phosphate as stationary phases in the gas chromatographic separation of the C_1 to C_5 aliphatic alcohols. Kolesnikova, Kamzolkin, and Khotimskaya (*27M*) determined the composition of isomeric heptanols and methylcyclohexanols by gas chromatography with diglycerol as the liquid substrate. Perkins, Laramy, and Lively (*42M*) found that the flame response in the quantitative determinations of high-molecular-weight alcohols by gas chromatography is additive with C number; values for alcohols are about $^1/_2$ C number less than the corresponding paraffin. The infrared spectrophotometric determination of α,α-dimethylbenzyl alcohol in the presence of α-methylstyrene and cumene was described by Terent'ev, Shaburov, and Ivanova (*55M*). Moore, Gordon, and Eiffert (*33M*) suggested that quantitative determination of different OH types could be made by analysis of the near-infrared absorption bands due to overtones of OH stretching vibrations.

The differentiation of phenol groups by fractional chromatography on silica gel treated with ethanol was reported by Klesment (*26M*). Freedman and Croitoru (*17M*) quantitatively measured phenols by the gas liquid chromatography of their trimethylsilyl ethers. Palocz and Nadasy (*39M*) extracted mono- and bicyclic phenols with methanol, water, and CCl_4; determination was made by the Kopescharr and lead acetate methods. Crump (*12M*) identified trace amounts of phenol in kerosine by forming their stable azo dyes, which were then separated by chromatography, on paper impregnated with formamide.

Trace amounts of carbonyl compounds were determined by Jordan and Veatch (*24M*) by spectrophotometric examination of the 2,4-dinitrophenylhydrazones. Baranova, Kalyazin, and Makarov (*4M*) formed the 2,4-dinitrophenylhydrazones and extracted them with pentane; spectrophotometric absorbance and paper chromatography on the extract were used to obtain quantitative and qualitative data. Kyazimov, Khaldei, and Khar'kovskii (*31M*) determined furfural by a method based on its condensation with aniline in the presence of concentrated acids. Harrison (*20M*) described the effect of temperature on aniline acetate reagents used in the detection and analysis of furfural. Sawicki, Noe, and Stanley (*47M*) described a color test with 2,2'-dinitrobiphenyl in dimethylformamide to detect aralkyl and dialkyl ketones.

The determination and differentiation of the acids in petroleum have been the object of several studies. A sensitive test for naphthenic acids developed by Pelcik (*40M*) depends upon the color produced in an oxidized aqueous solution of *N*,*N*-diethyl-*p*-phenylenediamine sulfate. Fritz and Marple (*18M*) found that *tert*-butyl alcohol was a good solvent for the potentiometric titration of phenols and weak acids. Sobolev, Popova, and Rubinshtein (*49M*) reported that carboxylic and sulfonic acids in sulfur-containing petroleums can be differentiated by potentiometric titration with potassium hydroxide in benzene-alcohol solvent. Izmailov and Aleksandrova (*22M*) studied the behavior of glass electrodes in the potentiometric determination of the acidity of petroleum products in a benzene-ethanol solution. A coulometric method of determining organic acids in a benzene-ethanol system was described by Crisler and Conlon (*10M*). Cerchez and Vladeanu (*9M*) separated aliphatic acids from naphthenic acids in the C_{15} to C_{50} range by crystallization with urea. Fester and Robinson (*16M*) used the ion exchange reaction with calcium acetate coupled with distillation to determine the carboxyl content of insoluble oil shale kerogen.

Total oxygen impurities (O, H_2O, CO, CO_2, alcohols, etc.) in liquids or gases were determined by Jarzynska and Mirecka (*23M*) by the use of a standard solution of potassium 2-phenyl isopropylate. Waclawik and Waszak (*58M*) developed an apparatus for continuous colorimetric determination of small quantities of oxygen in gases based on the reversible reaction of oxygen with sodium β-anthraquinone sulfonate in alkaline solution.

A series of oxygenated compounds, probably ketonaphthenonaphthalenes, was found in light catalytic cycle oil by

Aczel and coworkers (*1M*), using spectrometric techniques on chromatographic fractions. Csicsery and Pines (*13M*) reported relative retention times on different gas-liquid chromatographic columns for hydrocarbons, ketones, alcohols, H_2O, CO, and CO_2. Beroza (*7M*) and Beroza and Sarmiento (*8M*) modified and extended the microhydrogenation method of Thompson and coworkers (*57M*); this method for the identification of oxygen compounds depends upon the gas chromatographic identification of the hydrocarbons resulting from catalytic hydrogenation of the sample.

Other Elements

G. H. Patterson

E. I. du Pont de Nemours and Co.,
Wilmington, Del.

The determination of trace elements in petroleum and petroleum products has become of increasing importance in modern technology To aid in this work, Isbell and coworkers (*42N*) at the National Bureau of Standards established stable, oil-soluble salts of 24 elements for use as analytical standards. Il'ina (*40N*) surveyed the 1946–60 literature on spectrographic techniques for determining metallic constituents of lubricating or fuel oils. A critical review of fluorescent x-ray spectrographic techniques for determining trace elements in petroleum and petroleum products was made by Campbell and Thatcher (*15N*). Gunn (*36N*) studied the effects of absorption in x-ray fluorescence measurement of elements in oil. Rowe and Yates (*72N*) developed a xylenesulfonic acid ashing x-ray fluorescence method which was more rapid than conventional wet ashing and less likely to introduce interfering elements. A porous electrode was used by Boisselet and Baron (*8N*) for determination of metals in crude oils by emission spectrography coupled with x-ray fluorescence. Nash (*61N*) employed a controlled argon atmosphere d.c. arc for quantitative determination of trace metals. Eckardt, Keil, and Rast (*27N*) determined the various elements by both chemical and instrumental techniques. Polarography formed the basis for Krotky and Kozakova's (*50N*) method for metal analysis in used mineral oils from aircraft engines. Bruno (*12N*) reported the high-frequency complexometric titration of metals with EDTA. Zul'fugarly and Umakhanova (*82N*) determined the distribution of trace elements in Dagestan oils, while Makovets'kiĭ and Serdyuk (*58N*) reported data on 22 elements in petroleum from the Dnieper-Donets trough. Blagova and Lipshtein (*7N*) presented a systematic analysis of ash from petroleum products and deposits from boiler tubes and turbine blades. Hammerich and Gondermann (*37N*) described a simplified procedure for quantitative analysis of combustion chamber deposits using a pH meter and electric and flame photometers. Emission spectrography of refinery water was used to monitor corrosion (*64N*).

Balint (*3N*) determined hydrogen in petroleum products by measurement of β-radiation absorption with ionization chambers and investigated the factors contributing to the accuracy of radiation absorption measurements. Kakabadse and Manohin (*47N*) claimed a 12-minute microdetermination of carbon and hydrogen using MgO-CuO catalytic combustion. Modified Pregl combustion was used by Rosenberg, Downer, and Riber (*71N*) for a 30-minute determination. Chang, Huang, and Chang (*18N*) modified Koerbl's catalytic method with Korshun's pyrolysis procedure to speed up the determination. Vecera (*77N*) described an apparatus for rapid automatic microdetermination of carbon and hydrogen based on changes of thermal conductivity in a carrier gas after separate combustion for each element. Carbon determinations were accelerated by capillary connection of combustion apparatus to CO_2-absorption system in a weighing room, according to Vecera (*79N*). Cacace and coworkers (*14N*) continuously determined the carbon-hydrogen ratio of gas chromatography effluents by quantitative conversion to CO_2 and hydrogen and subsequent measurement on an auxiliary column. Critical conditions for water absorption were given by Newman and Tomlinson (*62N*) to obviate high hydrogen results when external nitrogen dioxide absorbents are used in carbon and hydrogen determinations. Kainz and Mayer (*46N*) investigated the carbon dioxide retention by MnO_2 and PbO_2 used as absorbents for nitrogen oxides in carbon and hydrogen analyses. Simultaneous determination of carbon, hydrogen, and nitrogen was obtained by Nightingale and Walker (*63N*) by rapid combustion of sample in helium and treatment of combustion products to form carbon dioxide, nitrogen, and acetylene which were measured by gas chromatography. Nitrogen was determined in automatic apparatus by Vecera (*78N*) by oxidation of sample with Co_3O_4 and measurement of nitrogen after reduction of oxides and removal of halides and water. Samples were burned in a flame with oxygen without oxidation aids in a method described by Radmacher and Hoverath (*69N*) for determining carbon, hydrogen, and sulfur.

Electron paramagnetic resonance provided a rapid and precise determination of vanadium in petroleum products and a means for continuous control in flow processes, according to Il'yasov (*41N*). Buncak (*13N*) determined vanadium spectrographically after decomposing the sample on silica containing a cobalt salt. Neutron irradiation was used by Aoki and Okada (*2N*) in rapid determination of vanadium. Stefanescu (*75N*) used the color reaction of vanadium salts and pyrocatecholsulfonic acid for determination in crude oil. Vanadium was assayed in mineral-oil ash by Piazzi (*67N*) by specific color formation by reaction with acetic anhydride, hydrochloric acid, and methyl salicylate. Janousek (*44N*, *45N*) leached the vanadium from fuel oil ash with acid, removed interfering ions with cation exchange resin, and determined the element spectrophotometrically after reaction with Xylenol Orange. Oil samples were ashed by Faulhaber, Vogt, and Moll (*31N*) in the presence of benzene, hydroxyquinoline, and dithizone to retain vanadium quantitatively and determine it colorimetrically with catechol. Borzova and coworkers (*11N*) ashed oil residues slowly, with solid CO_2 present initially, for quantitative retention of vanadium and subsequently determined the element colorimetrically as a phosphate-tungstate complex. The intensity of the red color developed by oxidation of *p*-phenetidine citrate in the presence of vanadium was the basis of analysis described by Bonchev (*9N*). An indirect EDTA titration for microdetermination of vanadium was reported by de Sousa (*24N*). The vanadium content of heavy gas oils down to 0.1 p.p.m. was determined by an electron paramagnetic resonance stream analyzer and the nickel level calculated on the basis of the vanadium-nickel ratio in the crude stock (*19N*). Hara and Yamazaki (*38N*) determined absorption curves of porphyrin complexes of vanadium and nickel and used the curve of the vanadium complex for analysis. Ball and Rall (*4N*) found 26% of the vanadium and nickel in Wilmington crude present as porphyrin complexes. Direct x-ray emission spectrographic determination of vanadium, nickel, and iron in oils and catalysts was reported by Dwiggins (*26N*). Agazzi and coworkers (*1N*) incinerated oils in the presence of sulfur to prevent loss of metals and determined vanadium, nickel, and iron by sensitive spectrophotometric methods.

Barrall and Ballinger (*5N*) determined the five methylethyllead alkyls in gasoline using gas chromatographic separation on a 1,2,3-tris(2-cyanoethoxy)propane column and electron-capture detection. Bonelli and Hartmann (*10N*) employed the same column and detection system but added a silver nitrate scrubber column to remove halogenated scavengers. Dawson's (*22N*) electron-capture chromatograph utilized successive columns of SE-30 silicone rubber, and silver nitrate in Carbowax 400 for the lead alkyl analysis. The critical operating parameters and limitations of electron-capture ionization detectors were reviewed by Lovelock and Gregory (*54N*). Parker and Hudson (*65N*) simplified their gas chromatographic method for separation and identification of mixed lead alkyls in gasoline for field application, using an Apiezon M column but no detector; identification was made based on retention times determined by calibration. Modifications in the chromate method for determination of tetraethyllead in gasoline were suggested by Ryabova, Belova, and Sharapov (*73N*). Bellomo and D'Amore (*6N*) devised an instrument for measurement of organic lead compounds in fuels and lubricants wherein the lead is converted to the ionic form and determined by automatic conductometric high-frequency titration with potassium chromate. Loroue and Paul (*52N*, *53N*) assayed rapidly the tetraethyllead in gasoline or jet fuel by extraction of the lead with potassium chlorate in nitric acid and determination colorimetrically as lead sulfide by comparison with known standards. Bromination of tetraethyllead in gasoline and subsequent titration with EDTA were used for rapid analysis by Koyama, Taguchi, and Eguchi (*49N*). A similar procedure using Chelaplex (III) titration was described by Gelius and Preussner (*32N*) in a review of methods for determining tetraethyllead. Farkas and Csanyi (*30N*) claimed good accuracy and speed in measuring tetraethyllead in gasoline by tritium radiation. Conditions and limitations for x-ray fluorescence determination of tetraethyllead in gasoline and lead in lubricants were given by Campo (*17N*).

Hexaethyldilead was determined in tetraethyllead by Tagliavini, Belluco, and Riccoboni (*76N*) using coulometric iodination at constant current with a dead-stop end point. Gol'dshtein, Lapisova, and Shtifman (*34N*) used high-frequency permanganimetric titration for the analysis. Slawson and Russel (*74N*) reported a silicone contaminant occasionally present in tetramethyllead.

Mattarella and O'Neil (*59N*) analyzed the lead compounds in spark plug deposits by x-ray diffraction and spectrographic methods. Gorusch (*35N*) investigated losses of lead, antimony, chromium, iron, and zinc as volatile chlorides during dry ashing in the presence of inorganic chlorides.

Phosphorus was determined in heavy petroleum products and additives in one fifth to one tenth the normal time by Makarov and coworkers (*56N*) by dry ashing the sample in the presence of magnesium nitrate, single-stage precipitation of phosphorus, and volumetric finish. Di Pietro, Kramer, and Sassaman (*25N*) modified the micro-Carius method to obtain complete combustion of phosphorus compounds. Gel'man and Shanina (*33N*) used Schöniger oxygen combustion followed by spectrophotometric determination of phosphorus as molybdenum blue. The Schöniger method was employed also by Liddell (*51N*) to determine phosphorus, sulfur, and chlorine in petroleum products using volumetric and colorimetric finishes. The "rotrode" spectrographic technique was followed by de Clippelier (*23N*) to assay phosphorus, barium, calcium, and zinc additives in new oils.

Pforr, Mikkeleit, and Hauptmann (*66N*) determined zinc and calcium in engine oils by excitation of oil in a high-tension spark using a rotating carbon electrode and a counter electrode of electrolytic copper. Zinc was quantitatively retained by ashing samples with $Ca(H_2PO_4)_2 \cdot H_2O$, according to Campen and Dumoulin (*16N*), who measured zinc spectrophotometrically as dithizonate. Makarov and Pershina (*57N*) reported complexometric titration of zinc in zinc dialkyl thiophosphate. Collins and Pearson (*21N*) analyzed oil-field brines for calcium and magnesium by EDTA titration, and Collins (*20N*) measured the potassium, lithium, strontium, barium, and manganese content of the brines with flame photometry.

Zul'fugarly and Umakhanova (*81N*) surveyed the copper content of Dagestan oils using the *o*-tolidine method for analysis. Howard and Spauschus (*39N*) found the neocuproine method simpler and faster but the carbamate more sensitive for determination of copper in mineral oils. Faerman (*28N*) used Complexone III and Faerman and Chigrin (*29N*) employed Trilon B to determine copper in petroleum products.

Arsenic was determined in Schöniger combustion products by titration with lead nitrate by Püschel and Stefanac (*68N*), and by the arsenomolybdenum blue reaction by Merz (*60N*). The trace Gutzeit arsine was caught by mercuric bromide paper and determined by x-ray fluorescence in a method described by Jackwerth and Kloppenburg (*43N*). The arsine was absorbed in a pyridine solution of silver diethyldithiocarbamate and measured spectrophotometrically by Kashiwagi and coworkers (*48N*).

Yao and Harrison (*80N*) determined chlorine, iron, sulfur, and titanium at the parts per million level in hydrocarbons by x-ray fluorescence. Maeck and coworkers (*55N*) extracted submilligram quantities of boron as the tetrabutylammonium boron tetrafluoride from aqueous solutions into methyl isobutyl ketone and determined boron by flame photometry on the organic phase. Romanov (*70N*) determined the salt content of desalted crude oil by measuring the electrical conductance of aqueous extracts.

Catalysts

Joseph Walker and June E. Walker

The Pure Oil Co., Crystal Lake, Ill.

DEVICES including an electrolytic buret for liquids, an electrical manometer of the resistance type, a spring beam microbalance for use in adsorption measurements, and a portable microcathetometer were reported by Vasserberg (*27P*) for use in the standardization and automation of laboratory processes for preparing and testing catalysts.

In a thermographic method for determining the activity of natural and synthetic silica-alumina catalysts Zul'fugarov, Aliev, *et al.* (*30P*) established a close correlation between the decolorizing and cracking activity of catalysts and the initial endothermic drop in their thermal analysis curves. The variations in catalytic activity of fluorine-promoted alumina (1.6 to 23% F) catalysts were determined by Holm and Clark (*9P*) for propylene polymerization, *o*-xylene isomerization, and *n*-octane cracking, and their isosteric heats of adsorption were calculated from ammonia adsorption studies.

Paul and Krishnan (*19P*) reported a volumetric method for determining aluminum and nickel in Raney nickel catalyst and its alloys based on the greater stabilities of aluminum triethanolamine and nickel cyanide complexes, compared with complexes of (ethylenedinitrilo) tetraacetic acid. Mott and Rhodes (*14P*) described the determination of silicon, aluminum, and silicon-aluminum ratios in catalytic material and rocks by neutron activation and γ-ray spectrometry. Kazanskiĭ and Pecherskaya (*11P*) noted changes in the appearance of a narrow signal in the ESR spectrum of chromia-alumina-silica ethylene polymerization catalysts when treated with water, ethylene, heptane, and cyclohexane, showing that Cr^{+5} ions, formed during the high temperature activation of the catalyst, are adsorption centers for water, ethylene, and solvents. Vergnaud (*28P*) described a method for determining the amount of catalytic metal deposited on the support of a catalyst by measuring densities with helium and using apparatus similar to that used for measuring the total surfaces in the BET method. Townsend (*26P*) devised universal standards for x-ray spectrographic analysis of SiO_2 and Al_2O_3 base catalysts by diluting the oxides with Li_2CO_3, fusing with borax, and casting into disks. To determine approximately 0.3% platinum in an alumina catalyst, Tan, Tan, and Li (*25P*) used a spectrograph with an a.c. arc source and photographic plates Nikfi TIP-1; Al_2O_3 was used as a standard sample and Pt 2998–Mo 3132 A. were used as analysis lines.

Weidenbach and Fuerst (*29P*) described methods for measuring the active platinum surfaces of supported reforming catalysts. The alumina-supported catalyst (previously heated 2 hours in a nitrogen stream) was saturated at room temperature with oxygen from an air or oxygen stream, the adsorbed oxygen was allowed to react at room temperature with a known amount of hydrogen, and the excess hydrogen was determined with a thermal conductivity cell; an alternative gas chromatographic method measured the retention times of hydrogen in an oxygen-free nitrogen stream, and allowed comparative measurements of the active surfaces of various catalysts.

The amount of potassium in catalysts used for dehydrogenation of hydrocarbons was determined radiometrically by Poddubnyi and Kolechkova (*20P*). Osipov, Semenova, and Rogov (*18P*) determined the relative efficiencies of hydrofining catalysts by comparing the hydrodesulfurization activity of an unknown catalyst with that of a standard alumina–cobalt oxide–molybdena catalyst. The relation between the physical properties (electrical conductivity and crystal structure) and the activity of certain catalysts (MoO_2-Al_2O_3, Pt-Al_2O_3, ZnO + promoters) in the aromatization and dehydrogenation of hydrocarbons was studied by Nicolescu (*17P*).

The product yield from the laboratory cracking (using a fixed-bed glass tubular reactor with a SiO_2-Al_2O_3 catalyst) of petroleum fractions with high paraffin and cycloparaffin hydrocarbon content was studied by Sergienko, Taimova, and Garbalinskii (*23P*) using various cracking temperatures and LHSV's. The catalytic effects of Askana clays and askangel on gasoline formation and on the gasoline-coke ratio were reported by Romankova, Remizov, Maidebor, and Golovenko (*21P*). Gas-phase chromatography was used by de Mourgues (*16P*) to study such catalytic processes as the cracking of isooctane and cumene as well as the physical adsorption of hydrocarbons on activated carbon and benzene on platinized Al_2O_3. In a study of metal contaminants in catalytic cracking, Grane, Connor, and Masologites (*8P*) determined the contaminant-coke-producing effect of measured additions of Fe, V, or Ni to catalysts of different surface areas, the change in the contaminating metal's effect made by alternating oxidizing and reducing atmospheres, and the effect of catalyst residence time on the production of inherent catalytic coke and contaminant coke.

The catalytic properties of bentonite clay from the Chardar'insk deposit was tested by Battalova, Likerova, and Sharipov (*4P*) in the cracking of a diesel fuel and a standard gas oil using inactivated clay samples and clay samples activated by treatment with H_2SO_4. Barras and Helwig (*3P*) discussed the various techniques available for the rapid determination of trace metals concentrations in gas oil feedstocks with emphasis on the application of atomic absorption spectroscopy with the use of a multipass optical system for the determination of iron, copper, and nickel. Garcia and Hermana (*7P*) described a method for measuring the acidity of petroleum cracking catalysts based on a catalyzed reaction of an acid and base (acetic acid–ethyl alcohol) under controlled conditions. Time of flight mass spectrometry and gas chromatography were applied to reaction studies in the catalytic cracking of nonane over SiO_2-Al_2O_3 by Levy, Miller, and Beggs (*13P*).

A survey by Stone (*24P*) of the physical methods of examining catalysts covered the determination of surface area and pore size by gas adsorption; phase identification and particle-size determination by microscopy, x-ray techniques, and electron diffraction; special methods—e.g., magnetic techniques and adsorption spectroscopy—and methods based on chemisorption and catalyst-adsorbate interaction, with emphasis on the study of the catalyst in relation to the intended reaction. He also discussed the utility of infrared spectroscopy, colorimetry, and other methods. Rubinshtein and Klyachko-Gurvich (*22P*) reported two simple and rapid methods for determining the surface area of catalysts, one involving periodical weighing of the adsorbent during the adsorption of benzene, carbon tetrachloride, or any other highly volatile organic substance at room temperature from a mixture of its vapor with nitrogen as a carrier gas, and the other involving measurement of surface area by adsorption of air on the catalyst at liquid nitrogen temperature, and compared the results obtained from the two methods with data obtained by the BET and other methods. Mottlau and Fisher (*15P*) reported improvements in the method of Innes for the measurement of pore volume by a titration technique, reporting particularly that 2-methoxyethanol is the most generally useful solvent. Klevleev, Orochko, Skoblo, and Levinson (*12P*) studied the effect of solid particle diameter, viscosity of the liquid phase, layer height, specific gravity of the suspension, and ratio of the diameter of the vertical column to the diameter of the outlet opening on the flow and movement of wet fine-grain materials, using spherical aluminosilicate catalyst.

In a review of adsorption methods for measuring the specific surface and pore structure of catalysts, Karnaukhov (*10P*) covered the conditions under which the BET method can be used for determining the surface area of solids; the importance of selecting the proper adsorbate for such determinations; the area occupied by the adsorbate as a monolayer on the catalyst surface; the evaluation of the pore structure of catalyst particles; measurement of the surface of composite and supported catalysts; and the various types of adsorption units used for measuring the specific surface and pore structure. Ettre, Brenner, and Cieplinski (*6P*) described and evaluated the Perkin-Elmer Sorptometer for surface area measurements by the continuous flow method. Clement (*5P*) surveyed methods for measuring the specific surface areas of petroleum processing catalysts by adsorption techniques.

Barker and Everett (*2P*) theorized on the adsorption of a dilute gas by a solid at high temperatures and presented a new method of determining surface areas which, in its precise form, determines the surface area entirely from adsorption data and was applied to the adsorption of helium, neon, and argon on carbon black. Atkins (*1P*) described a new technique for the rapid and precise measurement of surface areas based on that of Nelson and Eggertsen and involving adsorption of nitrogen from its mixture with helium at liquid nitrogen temperatures and measurement of the amount of nitrogen desorbed from the sample on removal of liquid nitrogen.

Pollution

G. R. Meador

Richfield Oil Corp., Wilmington, Calif.

THE Air Pollution Control Association's Petroleum Committee TI-3 (*1Q*) published a general review of control methods being used for particulate and gaseous emissions including sulfur oxides, hydrocarbons, nitrogen oxides, mercaptans, hydrogen sulfide, phenolic compounds and naphthenic acids, organic sulfides and nitrogen bases, aldehydes, and carbon monoxide. A critical review by Hanykova (*26Q*) covered published methods of sampling flue gases, volumetric determination of sulfur dioxide, complexiometric determination of sulfur trioxide, and determination of total sulfur by titration with sodium hydroxide after absorption in hydrogen peroxide.

Rostenbach and Kling (*51Q*) developed a coulometric method for the quantitative determination of nitrogen dioxide. Hummel and Ebert (*32Q*) modified the Okometer photometer for the separate determinations of nitrogen dioxide, chloride, and sulfate in effluent gases by light absorption. The measuring range for nitrogen dioxide is 0 to 0.5%, for chloride 0 to 0.1%, and for sulfate 0 to 0.5%. A gas chromatographic method was reported by Grune, Chueh, and Gutermuth (*25Q*) for the determination of the oxides of nitrogen and sulfur. The literature of stack gas components is reviewed critically.

Herrmann (*28Q*) devised a portable apparatus for determining sulfur dioxide in the air based on the pararosaniline method. The measuring range is 0.025 to 2.0 mg per cu. meter for a sampling period of 8 minutes. Pate, Lodge, and Neary (*49Q*) used potassium bicarbonate–impregnated membrane filters to collect sulfur dioxide in the air. In the range of 1 to 10 p.p.m., the absorption efficiency is greater than 95%. For the same purpose, Huygen (*33Q*) used a mixture of potassium hydroxide with glycerol or triethanolamine to impregnate filter papers, which at a relative humidity of greater than 25% showed an efficiency of better than 95%. A detector gel consisting of purified silica gel mixed with iodine solution and tetramethyldiaminodiphenylmethane in a glass detector tube was used by Kobayashi (*36Q*) to determine sulfur dioxide by measuring the length of the colored portion after sample aspiration. The mean relative error of the method is below ±5%. Brousaides and Hommel (*11Q*) described an apparatus for laboratory or field monitoring of SO_2 in air. The SO_2 is absorbed in aqueous $NaClO_2$, releasing ClO_2, which in turn releases Kr^{85} from its clathrate with $C_6H_4(OH)_2$. The Kr^{85} passes through a counter which is correlated to indicate parts per million of SO_2. Zurlo (*60Q*) described the titration of SO_4 with thorium nitrate. Interferences are evaluated by titration of a blank. A method was presented by Goksoeyr and Ross (*19Q*) for the determination of SO_3 in flue gases after condensation as H_2SO_4 on cooled surfaces. The volume sampled and hence the sampling period are determined by the quantity of acid needed for titration. An improved method for the detection and estimation of micron-sized sulfate particles was advanced by Lodge and Parbhakar (*42Q*). Reaction spots on an impregnated membrane filter were used to estimate the sulfate concentration. As little as 2×10^{-15} gram of SO_4 can be detected. Hissink (*30Q*) described a portable instrument designed for simultaneous determination of SO_3 and SO_2 in flue gases by two acid titrations after collection of the sample. The performance of the instrument on synthetic gas mixtures showed good agreement when the SO_3 content was in the range of 5 to 40 p.p.m. by volume, while 600 to 1000 p.p.m. of SO_2 was present.

Kozlayeva and Vorokhobin (*38Q*) reported a linear colorimetric method for the quantitative determination of toluene vapors in air utilizing a Type UG-2 portable gas analyzer. The total time of determination is 4 to 7 minutes and error is less than 10% of the upper limit of the scale. An ultraviolet spectrophotometric method for the determination of benzene in air samples adsorbed on silica gel was described by Elkins, Pagnotto, and Comproni (*18Q*). The method gives accurate results even when benzene is present as a component of petroleum naphtha containing toluene and other aromatics. Altshuller, Bellar, and Clemons (*3Q*) found silica gel, at the temperature of solid CO_2–acetone, to be a satisfactory adsorbent for concentrating hydrocarbons and compounds containing sulfur prior to the gas chromatographic analysis of samples.

Sand (*53Q*) described a modified method for determination of lead in air using a commercial lead-in-air detector.

The Research Committee of the Water Pollution Control Federation reviewed the literature of 1961 (*56Q*) and 1962 (*57Q*) concerning waste-water and water pollution control. Malz (*44Q*) lists new physicochemical methods for waste-water analysis, including photometric determination of ions and compounds with an average sensitivity of 0.01 mg. per liter; direct gas chromatographic analysis of the water without prior extraction of hydrocarbons or phenols; and polarographic and paramagnetic resonance measurements on the oxygen content of activated sludge. Three methods for the determination of the total biological oxygen demand (T_bOD) were presented by Busch, Hiser, and Grady (*13Q*), each having specific precision capability and hence specific applicability. Organic pollution may be measured in as short a time as 4 hours. Chenouard, Dirian, and Gabilly (*14Q*) presented a method for the continuous determination of dissolved oxygen in water down to 0.002 p.p.m. The limit of sensitivity is less than 10 μg. of oxygen per liter, and the precision is within ±3%. A dissolved oxygen sampler described by Hirsch (*29Q*) consists of a 1-gallon polyethylene jar having inflow and outflow tubes for filling the bottle and permitting the escape of air. Apte, Dabadghao, and Deshpande (*5Q*) compared colorimetric and polarographic methods for the determination of dissolved oxygen with the conventional titrimetric Winkler method. Colorimetric and polarographic methods proved more rapid than titrimetric and avoided the personal error in judging the end point. The polarographic method can also be used for turbid samples. Roumy (*52Q*) classified and reviewed methods for the determination of oxygen dissolved in water and their sensitivity and accuracy in measurements of low concentrations. A method for the continuous automatic determination of oxygen in water based on the oxidation of leuco-safranine was presented by Aleskovskiĭ, Kovaltsov, Fedorov, and Tsyplyatnikov (*2Q*). Morgan and Bewtra (*48Q*) developed a polarographic method for the determination of oxygen uptake rate for use where high uptake rates in a full-scale aeration system make it impossible to use the chemical titration method. An error of ±7% is expected in applying the method to liquids with high oxygen uptake rates. Bouquiaux (*9Q*) evaluated several aspects of the BOD test as presented in Belgian Standard Method IBN 407. He includes comments on equipment, incubation, reagents, preliminary treatment, dilution ratio, and manipulation in detail. Results of precision and interlaboratory tests are quoted and discussed. Greenberg and Lennette (*24Q*) suggested rapid procedures to permit selection of optimum dilutions for BOD determinations. A table was presented to cover sample dilutions based on the results of these procedures. The differences between the total-oxygen demand and the values obtained by the BOD and permanganate-demand determinations, and the development and adoption of the dichromate procedure as a standard method were discussed by Beuthe (*8Q*). Mancy, Westgarth, and Okun (*45Q*) described an oxygen electrode in which the generated current is proportional to oxygen concentration at the cathode in discussing the application of the galvanic cell oxygen analyzer to waste control programs. Edeline (*17Q*) described two methods for measuring "polluting power" of an effluent that are more suitable because of increased precision than using BOD values: the methods of Köppen (*37Q*), intended only to give a rapid characterization of the degree of pollution, and Busch *et al.* (*12Q*), a short-term total-oxygen demand test which gives increased precision in 36 hours. To adapt the Winkler oxygen method to very low concentrations, Smit and Ehrenburg (*55Q*) modified the technique, the reagents, and the apparatus to minimize inherent oxygen contamination.

Ladendorf (*40Q*) presented a method for hydrocarbon and water, with a reproducibility of 0.1 mg. per liter on samples containing 0.3 mg. per liter. The oil is extracted with tetrabromoethane and a density determination is made on the dried extraction solution. The method is comparable in accuracy to infrared analysis. Trials of the official Belgian and American methods for determination of oil in waters and of a modification of the American method are reported by Bouquiaux and Mertens (*10Q*). Full working details of the preferred modified method are given. Michelsen and Märki (*46Q*) presented a method for the detection of anionic detergents in water by extracting the detergent–methylene blue complex into $CHCl_3$. The method can be adapted to field work by comparing the color of the $CHCl_3$ phase with a scale of standard colors. The application of gas chromatography to direct waste stream analysis and a procedure for analysis of extracted organic matter are discussed by Gorlas (*23Q*). A rapid method for determining the petroleum products content of waste waters, reported by Poplavskaya and Vorobeva (*50Q*), is used for detection of petroleum products with a boiling point of less than 140° F. For very light petroleum products a correction factor must be introduced. Lure and Panova (*43Q*) presented a method for the determination of small amounts of aromatic hydrocarbons in waste waters. The method is based on Nastyukov's reaction, in which the hydrocarbons form a brown product on treatment with a mixture of formaldehyde and sulfuric acid. This product is analyzed with a colorimetric method. The benzene, toluene, and styrene contents are determined from calibration curves. The relative error is about 3%.

Wayman (*58Q*) used neutron-activation analysis for the determination of total sulfur in water. If chloride and phosphorus are initially removed to avoid interference, concentrations of sulfur as low as 0.01 p.p.m. can be detected and determined. A method for the amperometric determination of sulfides in the presence of sulfates, thiosulfates, sulfites, and carbonates in alkaline petroleum wastes was presented by Lenskaya and Terekhova (*41Q*). Whiston and Cherry (*59Q*) reported a method for the determination of thiocyanate in the effluents of coal-carbonizing plants. A colorimetric determination of 0 to 60 p.p.m. of thiocyanate with $FeCl_3$ is used.

Kaplin and Fesenko (*35Q*) presented a method for the determination of phenols in water with amidopyrene. Babkin and Voloskovets (*6Q*) reported a method for the determination of phenols in waste water by nitration. By reference to calibration curves the concentrations of phenol, *o*-cresol, *m*-cresol, and xylenols within the limits of 5 to 100 mg. per liter, nitrated and alkalized, can be determined as functions of absorbance with the application of colorimetry. A rapid method for the determination of phenols in oil and water was presented by Kanibalodskaya, Sidorskaya, and Shevlyakov (*34Q*). The method is similar to that reported by Babkin and Voloskovets (*6Q*) and has an error of determination of 1 to 2 mg. per liter, and a minimum determinable concentration of 5 mg. per liter. Hrivnak and Schiessl (*31Q*) determined chlorinated cresols in waste water by use of a gas chromatograph with a 1.6-meter column of 4-mm. diameter filled with Celite 545 and containing 12% poly(propylene adipate) and 1% H_3PO_4. The method was found reliable and sufficiently precise. A colorimetric determination of phenols in waste materials from oil refineries is described by Mitkalev, Avferenok, and Kozorezov (*47Q*). Small amounts of phenols (volatile and nonvolatile with steam) are determined using the photoelectric colorimeter, FEK-M. In comparison tests with the bromometric method, the new method proved rapid, simple, and accurate. Another method for the colorimetric determination of phenolic materials in refinery waste waters was presented by Gordon (*22Q*), in which he modifies the 4-aminophenazone method to eliminate the phenol distillation stage. Schwabe (*54Q*) reported an amperometric method for the determination of phenols in waste water, in which phenols and other organic compounds that act as depolarizers on graphite electrodes are determined by measurement of electrical current with rotating graphite electrodes.

A colorimetric method for the determination of nitrobenzene in oil refinery waste waters is described by Golubeva (*20Q*). Its sensitivity is 0.1 mg. per liter of water and the error of determination is ±5%. Benzene does not interfere, but the method is not specific in the presence of other aromatic nitro compounds. Dyatlovitskaya and Potemkina (*16Q*) determined nitrobenzene in industrial waste by indophenol and polarographic methods. Nitrophenols are determined directly by colorimetry and nitrophenols and chloronitrobenzenes are determined polarographically. By comparison of the results of the two methods it is possible to distinguish between the two compounds.

Goodman (*21Q*) described the preparation and use of a reagent in a simplified nitrite test. Dobremyslova, Dvorak, and Novobilsky (*15Q*) report a flame-photometric method for determination of calcium and sodium in waste effluents. The relative error of a single determination is 3%. A rapid method for the determination of Fe(III) in polluted waters by direct titration with EDTA was presented by Hellwig and van Steenderen (*27Q*). Zn and Cu in concentrations of about 5 and 0.2 p.p.m., respectively, interfere and Fe(II) must be oxidized prior to beginning the analysis. From 1 to 100 p.p.m. of Fe was titrated.

Bahensky, Zika, and Lastovkova (*7Q*) detailed the preparation and use of a reagent for the rapid detection of sexivalent chromium in waste waters.

American Petroleum Institute's Division of Refining (*4Q*) published a new manual in a series concerning disposal of solid refinery wastes. A chromatographic method for the determination and estimation of mineral oils in soil was presented by Kreiger (*39Q*). Chromatograms of crude oil, heavy fuel oil, light fuel oil, light petroleum, and mixtures of mineral and tar oils were prepared. The method is rapid and valuable for determining petroleum product leakage, its extent, and in many cases, the exact product.

Process Analytical Instruments

R. E. Farrell and J. D. Beardsley

The Standard Oil Co. (Ohio), Cleveland, Ohio

BECAUSE process stream analyzers are expensive in initial cost and upkeep, they must be selected with extreme care. Nootbaar (*25R*) describes a stream analyzer task force whose purpose is to promote, evaluate, and coordinate analytical applications for the refining division. Out of this approach has evolved an expanding nucleus of successful analytical installations, including chromatographs, end point and flash point analyzers, acid analyzers, viscosity analyzers, oxygen analyzers, and moisture monitors. Berridge, Pearce, and White (*2R*) give a general review of the incentives for and the problems involved in the installation and effective use of continuous plant analyzers. The case for setting up a specialist organization and the type of personnel required are discussed.

The latest developments in instrumentation and automation of the petroleum industry are discussed by Compain (*10R*). Oleinikov and Zotov (*26R*) describe automatic control of technical processes in the petroleum and petroleum-chemical industry. The quality control instruments for recording ASTM distillation characteristics, dew point and vapor pressure, rheological equipment, flash point recorder, radiation detectors, a turbidity meter, near-infrared analyzer, paramagnetic oxygen analyzers, visible and ultraviolet colorimetry, flame photometry, chromatographs, and possible future developments are covered by Crespin (*11R*), Fraade (*15R*), and Marten (*23R*). Wallis (*37R*) describes developments in control engineering and instrumentation and how their application to the control of refining processes offers improved operation and permits more economic plant design. The improved operation and consequent financial savings to be realized from proper installation and use of process analyzers as direct process control elements are illustrated by descriptions of an initial boiling point analyzer and end point analyzer by Tyler (*33R*). Two case histories are presented by Fritz and Davis (*17R*) to show the advantages of stream analyzers on process units over laboratory instrumentation.

Gas chromatography continues to find wide usage in process control applications. Righi (*30R*) describes three methods of process monitoring by continuous gas chromatography: (1) a two-column gas chromatographic method to determine H_2, CH_4, C_2H_2, C_2H_4, C_2H_6, C_3H_6, C_3H_8, C_4H_8, and iso-C_4H_{10} in C_2H_4 plant gas streams; (2) a method for separating and determining C_3H_6, iso-C_4H_{10}, *n*-C_4H_{10}, iso-C_4H_8, *trans*-2-C_4H_8, and *cis*-2-C_4H_8 in petroleum gas fraction streams; and (3) a two-column method to determine CO_2, Cl, O, N, and H in impure Cl gas streams. The operation and maintenance of two gas chromatographs are described by Maciw (*22R*). For two years these gas chromatographs have analyzed process streams containing H_2S (trace), CH_4, C_2H_4, C_2H_6, CO_2, and C_2H_2. Zhukhovitskiĭ and Turkel'taub (*38R*) developed a stepped (frontal) gas chromatography theory and experimentally confirmed it as a variant suitable for automatic regulation because it gave a simple and strong signal and did not require complete separation of components. Several variations of concentration-development (elution) gas chromatography were developed, the simplest of which was "vacancy" chromatography. The use of chromatographs for controlling industrial chemical processes in the USSR has been only partly successful, largely because of deficiencies in the apparatus. Aronovich, Galitskiĭ, and Zavertkin (*1R*) give several examples. Villalobos and Turner (*35R*) describe a gas chromatography system which obtains groupings of components similar in some respects (boiling point, molecular weight, chemical type, etc.). The net result is an entire group at a single symmetric peak with a height representative of the total. This technique is particularly useful in process analyzer applications. Two applications of plant-type chromatographic analyzers in determining the trace impurities (isobutene, MeCl) in liquid petroleum gas, and MeCl in butylene, are described by Brister (*4R*). A semiautomatic process analysis system in the form of a hybrid chromatograph was developed by Serne and Sinek (*31R*) for analyzing whole crude and crude unit side streams to 600° F. boiling point.

Keller and Burk (*21R*) have developed a total analysis digital system (TADS) which automatically reduces the chromatographic signal and presents the results on a logged digital record. Three applications are described for the chromatographic analysis of pilot plant streams.

The principles of construction of the Du Pont 800 photometric analyzer, the general type of applications requiring such an infrared analyzer, and the handling of interferences are described by Trippeer and Riggle (*34R*). A typical sample cell is described briefly. *Canadian Chemical Processing* (*6R*) reports that Wright and Daykin have developed a Fresnel-zone-plate far-infrared spectrometer which may be applicable in the process control analysis of cyclic hydrocarbons. *Chemical and Engineering News* (*7R*) describes a continuous analyzer (the Hymonitor) which monitors hydrogen in gaseous mixtures. The Hymonitor can measure varying hydrocarbon concentrations in any gas stream which is free of oxygen and saturated hydrocarbons, and materials that attack palladium. A special monitor for a continuous instrument for determining the concentration of aromatic hydrocarbons is described by Sukhomlinov (*32R*). The apparatus operates on the principle of determining the dielectric constant proportional to the content of aromatic hydrocarbons. It can provide continuous control of hydrocarbon fractions, determine the interface of products pumped in pipelines or stored in tanks, and record the volume flow rate of oil production. Continuous process analysis by magnetic resonance is described by Nelson (*24R*). Examples are given of how two process instruments (NMR and EPR) are used on liquid flow systems where all or part of a process stream is passed, at a constant rate, through the cavity or probe and a continuous reading of the output is produced. The EPR vanadium analyzer is described. Two examples of how the continuous vanadium analyzer has been successfully applied by the petroleum industry in controlling processes which prepare feedstocks for catalytic cracking or other catalytic processes where trace metals poison the catalyst are described by Coggeshall, Nelson, Doolen, and Baker (*9R*) and Donaldson, Murphy, McBride, and Story (*13R*).

Briot (*3R*) surveys the industrial applications of the dew point hygrometer. The survey covers the design of standard recording hygrometers for continuous dew point measurement and control and of special types for use in explosive atmospheres. A device which obtains bottom sediment and water on stream continuously and accurately has been developed to treat and separate bottom sediment and water continuously from a crude oil sample collected by an automatic sampler. Jasek (*20R*) describes the components, installation, operation, and testing of the instrument Clark (*8R*) reports that continuous boiling point analyzers prove accurate and reliable. Haselhorst (*19R*) describes an automatic apparatus for continuously determining the incipient wax separation point of mineral oils, which has been in use since early 1962. An apparatus for the automatic control of the melting point of petroleum products in a process stream is described by Gerzhevan-Lati (*18R*). An automatic analyzer for the continuous determination and recording of the ignition temperature of flowing petroleum products is described by Votlokhin (*36R*) and Pokrovskiĭ and Sabitov (*29R*).

Dahn and Brunner (*12R*) propose a method for the continuous determination of sulfur in the production of motor fuel, based on the difference in the absorption coefficients among C, H, and S for radiation from radioactive isotopes. How a continuous electrophotometric analyzer for acidic or basic components of process gas streams has successfully monitored hydrocarbon chloride levels from 0.1 to over 300 p.p.m. by volume is described by Burnett and Klaver (*5R*). Forney, Gulbrandsen, and Borup (*14R*) describe how the conductometric determination of salt in crude oils has been extended to on-stream analysis. A completely automatic apparatus will sample the stream, analyze, and record the salt content at predetermined intervals. Orlov, Krikunova, and Starkova (*27R*) employ a photocolorimeter in the automatic determination of the carbon disulfide content of crude benzene, toluene, and xylene fractions.

Fraade (*16R*) gives an example of a continuous sampling system in connection with a sulfur-recovery system. *The Petroleum Refiner* (*28R*) describes a sampling system in which the sample lines run from the distillation unit's catalytic cracking unit, etc., to the central analytical laboratory. Return lines provide for constant circulation of the product.

Laboratory Apparatus and Instruments

R. E. Farrell and J. D. Beardsley

The Standard Oil Co. (Ohio),
Cleveland, Ohio

PROHASKA (*37S*) relates experiences in the construction and application of some apparatus for the structural analysis of higher petroleum fractions. Apparatus are described for laboratory vacuum distillation, continuous solvent extraction, thermal diffusion, ebullioscopy, and elution chromatography. The British Standards Institution (*6S*) has published a specification which includes revised versions of apparatus (including flasks and receivers) for the determination of distillation range. A controller for maintaining a constant rate of vaporization in fractional distillation is described by Kuehner and Leslie (*29S*). Blumer (*5S*) reports on a new still designed for the efficient rectification of milligram quantities of materials in the molecular weight range of 200 to 850.

Ethyl Corp. has a new controller for the ASTM knock testing engine which adjusts the compression ratio and maximum knock air-fuel ratio in a minimum of steps. Its operation and components are described by Jones (*24S*). Details on a semiautomatic apparatus for the cryoscopic determination of the molecular weights of petroleum products are given by Dukhovnyi (*15S*). *Chemical and Engineering News* (*13S*) reports on an automatic osmometer with built-in recorder which carries out molecular weight tests in about 10 minutes by use of a null-seeking mechanism to balance the pressures across the membrane dividing the solvent sample cell. It is intended for use in high polymer research. A newly developed apparatus for the high-pressure sampling of gasoline, including gases from stabilizing plants, and for accurately determining sulfur, is described by Pomykala (*36S*). Weir (*46S*) describes a compact igniter and safety shield for the improved Schoniger apparatus. *Chemical and Engineering News* (*14S*) reports that an oxyhydrogen burner has been developed for combustion analysis which has good flame characteristics, disassembles into four easily cleaned components, and resists chemical attack, thus overcoming many of the limitations of the Wickbold quartz burner. Weitkamp and Korte (*47S*) describe an automatic instrument with percentage printer for the simultaneous determination of C, H, and N. Caines (*10S*) describes electronic instruments for testing petroleum products, covering the principles of operation and examples of automatic chemical or physical analyzers and automatic versions of standard tests. Khaet, Belova, and Bobrov (*26S*) give data on the performance of quality analyzers for the determination of salts and water in crude oil after desalting and dehydration, and for the determination of the flash point of petroleum products. Smith and Merritt (*43S*) explain a magnetic ion gas analysis technique by which oxygen in nitrogen, butane, and butadiene, and H_2O in nitrogen are detected in a three-electrode gas ionization chamber. Trace amounts of electrophores in gas streams whose primary constituents remain un-ionized may be measured quantitatively. A diagram and a description are given by Zaidman and Orechkin (*49S*) of an automatic apparatus for determination of flash point of petroleum products. Wotring and Keeney (*48S*) describe a galvanic cell-type oxygen analyzer for use with liquid and gas samples. A conventional null-type recording balance is described by Hurd (*19S*) for use in controlled pressure, controlled atmospheric thermogravimetry.

Sablina, Leont'ev, and Sazonov (*41S*) report on the use of a electron microscope in the investigation of some properties of hydrocarbon fuels. The high separation power of elution gas chromatography can be realized in a countercurrent process in which, by selection of suitable sorption materials, particular selectivities can be utilized. The continuous separation of various mixtures under these conditions is described by Pichler, Schulz, Firnhaber, and Rao (*35S*).

Recent applications of gas chromatography to the analysis of petroleum products are surveyed by Buzon (*9S*). Its use in combination with other analytical methods, preparative chromatography, and semicontinuous and continuous automatic analyzers is also covered. Carter and Golbayegani (*11S*) describe a rotary gas liquid chromatography sampling valve which performs satisfactorily in taking 0.5-ml. samples of diesel oil combustion products. Merritt and Walsh (*31S*) describe a simultaneous, dual-column gas chromatography apparatus which is suitable for isothermal or programmed temperature work and permits qualitative analyses by means of retention volume constants. Rapid and quantitative hydrocarbon determination in the refinery can be conveniently accomplished by the use of capillary columns in conjunction with flame-ionization detectors. Bruderreck (*7S*) describes this combination, which permits the use of an automatic peak integrator. Petrocelli (*33S*) describes a modified thermal conductivity detector with a modified cavity for capillary columns which furnishes curves comparable to those obtained on ionization detector instruments. Future developments in gas chromatography will probably include the replacement of chart recorders with electronic measurement circuits, an increase in the speed of analysis to 2.5 μseconds for easily separated mixtures, and the analysis of samples with vapor pressures as low as 10^{-5} mm. of Hg, predicts Purnell (*38S*). Chromatographic separation followed by stream splitting, differential chemical absorption, and flame ionization detection can be used for the simultaneous and separate determination of olefinic and paraffinic hydrocarbon components. Innes and Bambrick (*20S*) illustrate the method by application to analysis of auto exhaust, cigarette smoke, and gases from catalytic cracking. The Beckman Megachrom gas chromatograph is described by Rath (*39S*). The maximum quantity which can be separated on one occasion is 15 to 20 ml., and the process gives the amount of a component sufficient for an infrared or ultraviolet spectral investigation or other physicochemical determinations. An apparatus for high pressure gas chromatography above critical temperatures has been constructed by Klesper, Corwin, and Turner (*28S*). Johnson (*22S*) describes an automatic computation system for gas chromatographic data which overcomes the problem of peakbase drift and peak sensing. It is used for the evaluation of gas chromatographs containing single resolved peaks.

Barber *et al.* (*2S*) have been studying gas phase chemical reactions using a mass spectrometer with an ion source incorporating a differentially pumped electron beam filament chamber, and line-of-sight access from the sampling pinhole to the ionization region. Beckey and Wagner (*3S*) report on analytical applications of the field ionization mass spectrometer. The most promising application is the qualitative and quantitative analysis of complicated hydrocarbon mixtures, because quantitative analysis of an *n*-component mixture requires only a single calibration spectrum, whereas electron impact mass spectrometry requires no calibration spectra. Other differences between field ionization and electron impact mass spectrometry are discussed. A mass spectrum digitizer which will handle up to 5 peaks per second was developed primarily to meet the needs of a gas chromatograph–mass spectrometer combination. Klaver and LeTourneau (*27S*) list the specifications of the digitizer and document its performance. An example of the use of the GC-MS combination in conjunction with the digitizer in analyzing a petroleum fraction rich in C_9 cyclopentanes is given. Pattillo and Young (*32S*) describe a device for injecting liquid hydrocarbon samples in the gasoline boiling point range into a mass spectrometer. Akramkhodzhaev, Amirkhanov, and Alavutdinov (*1S*) describe the use of mass spectrometric analysis in oil geology.

In the analysis of petroleum products, Stemberger (*45S*) states that the application of infrared spectrometry has developed chiefly in three directions: (1) identification of unknown compounds; (2) identification of characteristic groups in unknown compounds; and (3) quantitative analysis of mixtures. The use of organic solvents in limited-area flame spectrometry is investigated by Buell (*8S*). Robinson (*40S*) makes some observations in atomic absorption spectroscopy.

During the last 5 years, x-ray fluorescence spectrography has taken its place in industry as a method of routine analytical analysis as well as a valuable research tool. Jenkins (*21S*) gives a short description of the production and characteristics of x-ray and of the equipment employed. Application of the technique in routine control determination of low atomic number elements such as phosphorus, sulfur, and chlorine is discussed. *Petroleum* (*34S*) reports on the new Fluorovac x-ray fluorescence spectrometer which will determine lead in gasoline, sulfur in fuel oils, and additives in lubricating oils as well as examine sludges and deposits. Lafay (*30S*) discusses principles of x-ray fluorescence spectroscopy in the petroleum industry, the equipment required, and the use of this technique for qualitative and quantitative analysis. The electron probe microanalyzer can be of great value to the oil industry, as exemplified by Bird (*4S*), who describes its use in investigations of bearing failure, the scaling of a heat-resistant alloy, the distribution of impurities in aluminum particles, and the distribution of the active element in a catalyst.

Neutron activation analysis has become an attractive, competitive means of elemental analysis available to almost all laboratories. Guinn, Johnson, and Mull (*18S*) describe a rapid and comprehensive activation analysis method using fast and slow neutrons and give applications in the petroleum and petrochemical industries. Johnson (*23S*) discusses the use of an activation analysis system as a standard instrumental method complementing other methods in meeting the demands for elemental analysis. Siering (*42S*) reviews the uses of neutron activation analysis and of isotopically labeled materials in the petroleum industry. Gray and Cartwright (*17S*) discuss the economics and individual applications of activation analysis. Gilmore, Hull, and Fries (*16S*) describe the use of automatic calculation procedures in the analysis of petroleum stocks by neutron activation. The automatic program is illustrated in the analysis of compounded motor lubricants for six of the elements commonly present in materials added to such oils: N, O, P, Cl, Zn, and Ba. The application of nuclear magnetic resonance of protons to the solution of analytical problems in petroleum refining research is discussed by Chamberlain, Neureiter, Saunders, and Williams (*12S*). Snyder (*44S*) presents the results of some studies in magnetic nonequivalence. NMR spectra of two-spin and two-spin-group systems are evaluated. Jungnickel and Forbes (*25S*) discuss quantitative measurement of hydrogen types by integrated nuclear magnetic resonance intensities.

Miscellaneous

Ralph O. Clark

Gulf Research & Development Co., Pittsburgh, Penna.

THIS section directs attention to published papers on physical and chemical properties, correlative methods, stability studies, and certain other selected investigations on petroleum or its products. No attempt has been made to present either a comprehensive or critical review; selection was made on the basis of potential interest in the subject material.

Considerable work continues on the measurement of viscosity of a wide range of petroleum products. Hardy (*24T*) has reported on ten oils in the range of 0.02 to 450 poises provided by the NBS for calibration of relative viscometers. Kinoshita and Onoyama (*28T*) have studied the kinetic energy correction of capillary viscometers having circular and elliptical cross sections. Viscosity of the lower saturated hydrocarbons at different temperatures and pressures has been shown by Agaev and Golubev (*3T*) to follow a generalized curve, and they have described the design and construction of their apparatus that can be used up to 500 atm. and 275° C. (*1T*, *2T*). Sanin and coworkers (*41T*) have measured the viscosity of C_{24} and C_{28} polycyclic hydrocarbons from $-40°$ to 150° C. and have made some observations on the effect of substituent groups on viscosity and its temperature dependence. A modification of the Pinkevich viscometer has been proposed by Stepanov and Stul'ginskaya (*44T*) to improve the reliability of the resulting data.

A portable, direct-reading viscometer based upon the change in frequency of a vibrating element has been described (*48T*). A method for predicting the viscosity of normal paraffin mixtures has appeared (*4T*) and a viscosity correlation has been developed for nonassociating liquids; the correlation is not valid at low temperatures (*13T*).

A simple, falling-coaxial-cylinder viscometer has been described by Santucci and Schmidt (*42T*) for routine application to asphalts at low temperatures. Fink (*19T*) has published a statistical comparison of a modified softening-point method with the ASTM ring and ball method. Carre and Laurent (*11T*) have developed a relationship between the penetration and viscosity of bitumen that is useful when the amount of sample is limited.

An equation has been proposed for determining the viscosity of oil-water emulsions based upon the kinematic viscosity of the oil itself and a coefficient whose value is obtained from a previously prepared graph (*38T*). The behavior of rheologically anomalous petroleum products has been investigated (*34T*) and an equation derived for using the falling-ball viscometer with low viscosity crude oils (*12T*). A report has appeared (*40T*) on newly developed empirical formulas useful in the viscosity-temperature-pressure relation of lubricating oils and their correlation with chemical constitution.

A graphic method was described by Lyashkevich (*30T*) for determination of the purity of organic substances from cryoscopic measurements; increased accuracy over other methods is claimed. Gunn (*22T*) has devised a simple melting-curve technique in which 10^{-5} mole fraction impurity can be observed on a 0.3-ml. sample. The purity and heat capacity of the substance are calculated from simple equations, and the heat of fusion is determined by means of graphical integration.

A semimicropycometer, based on the design covered by ASTM D 941, suitable for 0.3- to 0.5-ml. samples has been described (*5T*). The densities of C_2 through C_4 paraffins and mixtures thereof have been measured by a simple hydrometer method (*23T*), and the effect of the error in temperature corrections on the density of eastern USSR and Baker crude oils has been presented (*33T*).

The ratios of true vapor pressure to Reid pressure have been collected for cracked gasolines, natural gasolines, and crude oils (*35T*). Nelson (*36T*) has also shown how an approximate Reid vapor pressure of debutanized naphthas and gasolines can be deduced as a function of cut point and crude gravity. The Frost-Kalkwarf vapor-pressure equation has been used by Bondi and McConaughy (*10T*) as a basis for the estimation of the vapor pressure of high molecular weight hydrocarbons, and by Pasek and Thodos (*37T*) for some 34 naphthenic hydrocarbons. Bell and Groszek (*6T*) have utilized a flame ionization detector in a flowing system to measure the vapor pressure of mineral oils within the range of 10^{-1} to 10^{-4} mm. of mercury, and over a wide range of temperature, with an error of about 10%. The determination of vapor pressure by differential thermal analysis, using a modification of Vassallo and Harden's boiling point technique, was demonstrated by Krawetz and Tovrog (*29T*).

Comparative studies on the surface tension of a variety of liquids were reported by Schatt and Pietsch (*43T*); the pressure or rise in capillaries and by Averbach's and Altenburg's correlations of surface tension and ultrasonic velocity showed general agreement. Ben'kovskii and associates (*7T*) investigated the effect of temperature on the surface tension of *n*-alkanes by capillary rise and maximum bubble pressure in a Segden apparatus.

Wickey and Chittenden (*47T*) obtained the flash point of petroleum products by the use of a flash point index. The index was calculated from the flash point by an empirical equation developed from extensive experimental data. Ivanov and Gurkina (*27T*), on the other hand, found a good correlation for diesel fuels based on the arithmetic average of the initial and 10% Engler distillation points.

An isothermal distillation method, using 10- to 30-mg. samples, was used by Gerber *et al.* (*21T*) for molecular weight measurements of petroleum oils; the method is not applicable to materials having a boiling point below 250°

C. Using a differential ebullioscopic technique Trowell, Taylor, and Brown (*45T*) with an a.c. bridge were able to obtain values that were precise to within about 0.8% for materials having a molecular weight less than 500. A semiautomatic cryoscopic method was employed by Dukhovnyi (*17T*) to measure the molecular weight of petroleum products in 10 to 12 minutes; the detector signal was recorded with a potentiometer.

Physical properties based on microanalytical techniques have been reviewed by Berthold (*8T*), who with coworkers (*9T*) has described micromethods for molecular weight, density, and viscosity. Dixon (*15T*) presented empirical structure-property equations and generalizations used in predicting the values of the physical properties of unknown complex polyaromatics and polyalicyclics. White (*46T*) has noted the effects of degree of branching and of molecular weight on the physical properties of polymer acyclic oils. A report covering the correlation among high-voltage discharge, chemical stability, and composition of mineral oils was issued by Foldiak (*20T*). The hydrogen number (an empirically derived relation) appears to be characteristic of the chemical composition of mineral oils and can be correlated with other properties—e.g., specific gravity, refractive index, viscosity index, etc.

Kirk continued his studies, in cooperation with Rajeswaran (*39T*), to extend knowledge useful in forensic identifications. Ascending paper chromatography was applied to wax, grease, asphalt, and gasoline; better resolution was achieved than by conventional chromatography. Ettling (*18T*) was able to characterize residues by infrared examination of the material separated on a column of activated alumina.

An interlaboratory study was reported by Donlan (*16T*) on distillation methods ASTM D-86 and D-1078. No deviation between these methods was noted for mineral spirits, VM&P naphtha, and lacquer diluent naphtha if emergent stem corrections were applied to D-86 results, or if the thermometer used in the D-1078 test was substituted for the one specified by D-86.

A correlation was established by Hollinghurst (*25T*) for aniline point from viscosity and specific gravity, and from viscosity and viscosity index. The gravity-viscosity relationship for kerosines, gas oils, lubricating oils, bright stocks, cylinder stocks, and additive-containing oils showed a standard deviation of 3.5° from experimentally determined aninine points; the other, on lubricating oils, bright stocks, and oils containing additives, of 4.7° C.

Lyn (*31T*) compared diesel smoke measurements by the BP-Hartridge, Bosch, and Volvo smoke meter results with Bacharach disks, and with absolute values (weight of carbon cubic foot). And Malschaert (*32T*) found some correlation between a smoke meter, based upon photoelectric monitoring of the blackening of a paper filter, and another meter in which a light beam was attenuated by the smoke in a tube. The latter device was used to make long-term measurements and under road-test conditions.

A rapid method for determining mineral oil in liquid oxygen has appeared (*26T*). After evaporation of the liquid oxygen the residue is taken up in cyclohexane and the absorbance of the solution measured. The precision of measurement is 0.01 mg. when less than 1 mg. of oil is present, and about one hour is required for the analysis.

Cranston and Gammon (*14T*) have presented a new approach on the construction of specification and related testing of finished petroleum products. Often specification tests are used that do not uniquely characterize important performance properties. Criteria were devised for developing a set of specification tests that usually will evaluate the performance of a product with a reduced laboratory testing schedule.

International Standardization

R. R. Wright

American Petroleum Institute, New York, N. Y.

THE formal channel for adoption of international standards for petroleum products is through the activities of the International Organization for Standardization (ISO). ISO proposals in this field are delegated to Technical Committee No. 28, Petroleum Products. ISO members are the national standardizing bodies in the various countries. The U. S. member is the American Standards Association.

Since June 1952, ISO/TC 28 has held four meetings. A fifth meeting has been called for June 2 to 4, 1965, at the headquarters of the American Society for Testing and Materials (ASTM) in Philadelphia, Pa. Since 1952, the work of ISO/TC 28 has resulted in the adoption of the ASTM-IP Petroleum Measurement Tables (ASTM D-1250; IP-200) (*2V*) as International Recommendation R 91. The June meeting will call for final action on other draft ISO recommendations: Knock Characteristics of Motor Fuels by the Motor Method (ASTM D-357) and Knock Characteristics of Motor Fuels by the Research Method (ASTM D-908), and their companion methods for testing motor fuels above 100 octane number, ASTM D-1948 and ASTM D-1656, respectively.

An informal channel for international standardization of methods of test for petroleum products stems from the acceptance and use of the ASTM standards (*1V*) prepared by Committee D-2 on Petroleum Products and Lubricants. Many of these standards are used unchanged or as adaptations in such countries as Canada, Mexico, Argentina, Australia, New Zealand, United Kingdom, Norway, Sweden, Denmark, The Netherlands, Belgium, Germany, and France. Standards developed by the Institute of Petroleum (IP) also enjoy wide use in the United Kingdom and in commonwealth countries.

ASTM D-2 and the IP have been active since 1963 on a program to develop joint methods. As a result, 32 ASTM and IP test methods (listed below) have been combined and issued (*1V, 3V*). See Table I.

Other joint ASTM-IP projects include a proposed manual on significance of tests and the development of standard methods on the precision of test methods, the design of cooperative test programs, the analysis of test data, and the expression of results. In this latter effort significant cooperation is also being given by the standardization bodies of France, Bureau de Normalization du Pétrole (BNP), and Germany, Fachausschuss Mineralol- und Brennstoff-normung (FAM).

The national standardizing bodies of Common Market countries and the eight countries that belong to the customs union on the Common Market periphery participate in the work of the Committee on European Standardization (CEN). Test methods for petroleum products are recommended by CEN Working Group 19. The product of Working Group 19 is CEN "Unification Documents," which ultimately may be referred to ISO/TC 28. ASTM-IP Joint Methods are also subjects for CEN and ISO/TC 28 consideration.

Acknowledgment

This review has been prepared through the cooperative effort of a number of analytical chemists in the petroleum industry. It was undertaken this year as a cooperative project of the Division of Petroleum Chemistry of the American Chemical Society. The contributions of others in suggestions, in time, and in effort have been of great assistance in coordinating and completing the assignment. In particular, grateful acknowledgment is given to the authors of each subject section, and to E. B. Tucker, Research and Development Department, Whiting Laboratories, American Oil Co., Whiting, Ind.; J. Walker, Research Center, Pure Oil Co., Crystal Lake, Ill.; E. N. Davis,

Table I. ASTM and IP Test Methods

ASTM D-86;	IP-123	Distillation of Petroleum Products
ASTM D-92;	IP-36	Flash and Fire Point by Cleveland Open Cup
ASTM D-97;	IP-15	Cloud and Pour Points
ASTM D-127;	IP-133	Melting Point of Petrolatum and Microcrystalline Wax
ASTM D-129;	IP-61	Sulfur in Petroleum Products by the Bomb Method
ASTM D-130;	IP-154	Copper Strip Corrosion by Petroleum Products
ASTM D-189;	IP-13	Conradson Carbon Residue of Petroleum Products
ASTM D-216;	IP-191	Distillation of Natural Gasoline
ASTM D-287;	IP-192	API Gravity of Petroleum Products, Hydrometer Method
ASTM D-322;	IP-23	Dilution of Gasoline Engine Crankcase Oils
ASTM D-381;	IP-131	Existent Gum in Fuels by Jet Evaporation
ASTM D-445;	IP-71	Kinematic Viscosity
ASTM D-482;	IP-4	Ash from Petroleum Oils
ASTM D-524;	IP-14	Ramsbottom Carbon Residue of Petroleum Products
ASTM D-525;	IP-40	Oxidation Stability of Gasoline (Induction Period Method)
ASTM D-526;	IP-96	Lead Antiknock Compounds in Gasoline (Gravimetric Method)
ASTM D-566;	IP-132	Dropping Point of Lubricating Greases
ASTM D-664;	IP-177	Neutralization Number by Potentiometric Titration
ASTM D-721;	IP-158	Oil Content of Petroleum Waxes
ASTM D-873;	IP-138	Oxidation Stability of Aviation Fuels (Potential Residue Method)
ASTM D-874;	IP-163	Sulfated Ash from Lubricating Oils and Additives
ASTM D-937;	IP-179	Penetration of Petrolatum
ASTM D-938;	IP-76	Congealing Point of Petrolatums and Petroleum Waxes
ASTM D-942;	IP-142	Oxidation Stability of Lubricating Grease (Oxygen Bomb Method)
ASTM D-943;	IP-157	Oxidation Characteristics of Inhibited Steam Turbine Oils
ASTM D-974;	IP-139	Neutralization Number by Color Indicator Titration
ASTM D-1019;	IP-145	Olefinic Plus Aromatic Hydrocarbons in Petroleum Distillates
ASTM D1250;	IP-200	ASTM-IP Petroleum Measurement Tables
ASTM D-1266;	IP-107	Sulfur in Petroleum Products and LPG (Lamp Method)
ASTM D1317;	IP-118	Chlorine in New and Used Lubricants (Sodium Alcoholate Method)
ASTM D-1405;	IP-193	Estimation of Net Heat of Combustion of Liquid Petroleum Products
ASTM D-1833;	IP-185	Odor of Petroleum Wax

Sinclair Research Laboratories, Harvey, Ill.; and F. D. Tuemmler, Shell Development Co., Emeryville, Calif., who performed the literature search and assisted in the coding of the references selected; and to A. H. Batchelder, California Research Corp., Richmond, Calif., and J. S. Ball, U. S. Bureau of Mines, Bartlesville, Okla., Chairman and Chairman Elect, respectively, of the Division of Petroleum Chemistry (ACS), who were of significant help in the change of sponsorship of the project to the ACS from the API.

LITERATURE CITED

Preface

(AA) Gambrill, C. M., *et al.*, *Anal. Chem.* **35,** 111R–42R (1963).

Introduction

(1A) Aleksandrov, A. N., Dement'eva, M. I., Shmulyakovskiĭ, Ya. E., eds., *Metody Issled. Produktov Neftepereabotki i Neftekhim. Sinteza* (Gostoptekhizdat., Leningrad), **1962.**

(2A) Am. Soc. Testing Materials, Philadelphia, Pa., "First Supplement to the Molecular Formula List of Compounds, Names, and References to Published Infrared Spectra," **STP 331-A.**

(3A) *Ibid.*, "Index of Mass Spectral Data," **STP 356.**

(4A) *Ibid.*, "Manual for Hydrocarbon Analysis," **STP 332.**

(5A) *Ibid.*, "Molecular Formula List of Compounds, Names, and References to Published Ultraviolet and Visible Spectra," **STP 357.**

(6A) *Ibid.*, "Report on Available Standard Samples, Reference Samples, and High-Purity Materials for Spectro-Chemical Analysis," **STP 58-E.**

(7A) Bobbith, J. M., "Thin-Layer Chromatography," Reinhold, New York, 1963.

(8A) British Non-Ferrous Metals Research Association, London, "Proceedings of a Conference on Analytical Methods," 1962.

(9A) Bruderreck, H., *Erdoel Kohle* **16,** 847–50 (1963).

(10A) Hakala, N. V., *Oil Gas Intern.* **3,** No. 6, 94–6 (1963).

(11A) Hara, A., *Bunko Kenkyu* **8,** 169–83 (1960).

(12A) Jenkins, J., *J. Inst. Petrol.* **48,** No. 464, 246–56 (1962).

(13A) Killer, F. C. A., *Inst. Petrol. Rev.* **17,** No. 198, 223 (1963).

(14A) Milner, O. I., "Analysis of Petroleum for Trace Elements," Pergamon Press, New York, 1963.

(15A) Prinzler, H., *Z. Chem.* **2,** 136–45 (1962).

(16A) Reilley, C. N., *Advan. Anal. Chem. Instr.* **2,** (1963).

(17A) Stemberger, Vida, *J. Inst. Petrol.* **49,** No. 478, 211A (1963).

(18A) Szepesy, L., Simon, J., *Chem. Tech.* (*Berlin*) **14,** No. 5, 377 (1962).

Crude, Condensates, and Shale Oil

(1B) Adel'berg, I. M., Dvali, M. F., *Tr. Vses. Neft. Nauchn.-Issled. Geologorazved. Inst.* **1961,** No. 174, 186.

(2B) Areshidze, K. I., Khechianshvili, E. P., *Soobsch. Akad. Nauk Gruz. SSR* **28,** 401 (1962).

(3B) Bailly, F. H., *World Oil* **155,** 75 (1962).

(4B) Ball, J. S., Haines, W. E., Helm, R. V., *Proc. 5th World Petrol. Congr.* **5,** 175 (1960).

(5B) Baxa, J., *Ropa Uhlie* **4,** 196 (1962).

(6B) Bendoraitis, J. G., Brown, B. L., Hepner, L. S., *Proc. 6th World Petrol. Congr.* **5,** paper 15 (1963).

(7B) Boyd, M. L., Montgomery, D. S., *J. Inst. Petrol.* **49,** 345 (1963).

(8B) Budec, B., *Nafta* **13,** 354 (1962).

(9B) Dean, R. A., Whitehead, E. V., *Proc. 6th World Petrol. Congr.* **5,** paper 9, (1963).

(10B) Dobryanskiĭ, A. F., *Nizkotemperaturnye Kataliticheskie Prevraschen. Uglevodorodov, Leningr. Gos. Univ.* **1962,** 12.

(11B) Drabkin, A. E., *Tr. Vses. Nauchn.-Issled. Inst. Pererabotki i Ispol'z. Topliva* **1961,** No. 10, 91.

(12B) Drabkin, A. E., Babin, I. N., *Ibid.*, **1961,** 96.

(13B) Eisen, O. G., *Goryuch. Slantsy, Khim. i Tekhnol.* **1961,** 213.

(14B) Eisen, O. G., Rang, S. A., *Ibid.*, **1961,** No. 4, 200.

(15B) Eisen, O. G., Rang, S. A., Arumeel, E. K., *Khim. i Tekhnol. Topliv i Masel* **8,** 38 (1963).

(16B) Garilli, F., Lombardo, G., Zerbo, G., *Ann. Chim.* **52,** 849 (1962); *J. Appl. Chem.* **13,** 54 (1963).

(17B) *Ibid.*, **53,** 1244 (1963).

(18B) Goodspeed, F. E., Montgomery, D. S., *Can. Dept. Mines Tech. Surv., Mines Branch Res. Rept.* **R98** (1962).

(19B) Gordash, Y. T., Sergienko, S. R., Semyachko, R. Y., Rekunova, E. A., *Dokl. Akad. Nauk Belorussk. SSR* **5,** 112 (1961).

(20B) Gutyrya, V. S., Kvitkovskiĭ, L. N., Kupriyanova, L. A., Fadeicheva, A. G., *Neft. i Gaz. Prom., Nauchn.-Tekhn. Sb.* **1962,** No. 3, 51.

(21B) Hildebrand, G., Teubel, J., Peper, C., Dahlke, B., *Chem. Tech.* **15,** 482 (1963).

(22B) Kalantar, N. G., Glazunov, V. I., Mannafova, V. S., *Khim. Seraorgan. Soedin. Soderzhasch. v Neft. i Nefteprod* **5,** 203 (1963).

(23B) Kalde, L. Y., *Khim. i Tekhnol. Goryuch. Slantsev i Produktov ikh Pererabotki* **1962,** No. 11, 290.

(24B) Kerenyi, E., *Acta Chim. Acad. Sci. Hung.* **31,** 113 (1962).

(25B) Kerenyi, E., Zakar, P., Mozes, G., *Magy. Asvanyolaj Foldgaz Kiserl. Int. i Kozlemen* **2,** 32 (1961).

(26B) Koten, V. G., Stoyanovskaya, L. I., *Tr. Turkmensk. Filiala Vses. Neft. Nauchnn.-Issled. Inst.* **1961,** No. 3, 88.

(27B) Kuiv, K. A., Gubergrits, M., *Goryuch. Slantsy, Khim. i Tekhnol.* **1961,** No. 4, 46.

(28B) Kuntz, L. E., Remke, M. A., *Oil Gas J.* **61,** 144 (1963).

(29B) Lagoutte, P., de Saint-Palais, P., *Rev. Inst. Franc. Petrole Ann. Combust. Liquides* **18,** 967 (1963).

(30B) Le, B., Urmancheev, F. A., Baranenko, S. E., Novikova, E. F., Bukharaeva, R. G., Lamanova, I. A., Kurzhunova, Z. Z., *Izv. Akad. Nauk SSSR, Ser. Khim.* **1963**, No. 10, 1809.

(31B) Levchenko, E. S., Bobkova, E. N., *Tr. Groznensk. Neft. Nauch.-Issled. Inst.* **1961**, No. 11, 11.

(32B) Lille, Y., Murd, A., Kundel, K., *Khim. i Tekhnol. Goryuch. Slantsev i Produktov ikh Pererabotki* **1963**, No. 12, 142.

(33B) Lindeman, L. P., LeTourneau, R. L., *Proc. 6th World Petrol. Congr.* **5**, Paper 14 (1963).

(34B) McKinney, C. M., Ferrero, E. P., *U. S. Bur. Mines, Rept. Invest.* **6266** (1963).

(35B) Makovetskiĭ, P. S., Serdyuk, D. P., *Dopovidi Akad. Nauk Ukr. RSR* **1962**, 1086.

(36B) Martin, R. L., Winters, J. C., Anal. Chem. **35**, 1930 (1963).

(37B) Martin, R. L., Winters, J. C., Williams, J. A., *Proc. 6th World Petrol. Congr.* **5**, 231 (1963).

(38B) Mazitova, F. N., Virobyants, R. A., Ermakova, S. K., *Izv. Akad. Nauk SSSR, Otd. Khim. Nauk* **1962**, No. 9; 1546.

(39B) Menez, F., *Rev. Inst. Franc. Petrole* **17**, 842 (1962).

(40B) Moiseikov, S. F., Dragunskaya, V. S., Tolstenev, V. S., *Izv. Akad. Nauk Turkm. SSR, Fiz.-Tekhn., Khim. i Geol. Nauk* **1962**, No. 3, 47.

(41B) Mukashev, Z. A., Shmais, I. I., *Uch. Zap. Kazakhsk. Gos. Univ., Geol. i Geogr.* **37**, 140 (1958).

(42B) Muradova, G. A., Sergienko, S. R., Korotkiĭ, A. G., *Izv. Akad. Nauk Turkm. SSR, Ser. Fiz.-Tekhn., Khim. i Geol. Nauk* **1963**, No. 2, 16.

(43B) Musayev, I. A., Rosenberg, L. M., Sanin, P. I., Nifontova, S. S., Galpern, G. D., Kusakov, M. M., Ushakova, I. B., Suchkova, A. A., *Proc. 6th World Petrol. Congr.* **5**, 31 (1963).

(44B) Nasirov, A. B., Ashumov, G. G., *Azerb. Neft. Khoz.* **42**, 46 (1963).

(45B) Nezhevenko, V. F., Smirnova, I. V., *Tr. Kuibyshevsk. Gos. Nauchn.-Issled. Inst. Neft. Prom.* **1961**, No. 9, 82.

(46B) Pashaev, P. A., *Tr. Azerb. Gos. Ped. Inst.* **8**, 23 (1959).

(47B) Pelcik, I., *Collection Czech. Chem. Commun.* **27**, 1503 (1962).

(48B) Polovoi, Y. N., *Nizkotemperaturnye Kataliticheskie Prevrashchen. Uglevodorodov, Leningrad, Gos. Univ.* **1962**, 119.

(49B) Rang, S. A., Arumeel, E. K., Eisen, O. G., *Khim. i Tekhnol. Topliv i Masel* **6**, 40 (1961).

(50B) Robinson, W. E., Lawlor, D. L., Cummins, J. J., Fester, I. I., *U. S. Bur. Mines, Rept. Invest.* **6166** (1963).

(51B) Rodionova, K. F., Il'inskaya, V. V., Nadel'shtein, I. V., *Geol. Nefti i Gaza* **6**, 52 (1962).

(52B) Rodionova, K. F., Il'inskaya, V. V., Nadel'shtein, I. V., *Tr. Vses. Nauchn.-Issled. Geologorazved. Neft. Inst.* **1962**, No. 33, 181.

(53B) Rodionova, K. F., Korchagina, Y. I., Il'inskaya, V. V., *Geol. Nefti i Gaza* **7**, 33 (1963).

(54B) Rozmanek, J., *Ropa Uhlie* **4**, 141 (1962).

(55B) Sadykhov, I. D., Frankfurt, Y. M., Abdullaev, N. D., *Azerb. Khim. Sh.* **1962**, No. 2, 59.

(56B) Sanin, P. I., Druzhinina, N. K., *Neftekhimiya* **1**, 725 (1961).

(57B) Sattarzade, A. D., Farzaliev, V. M., Sattarzade, I. S., *Uch. Zap. Azerb. Gos. Univ., Ser. Fiz. Mat. i Khim. Nauk* **1**, 69 (1962).

(58B) Serbanescu, A., *Petrol Gaze (Bucharest)* **13**, 97 (1962).

(59B) Sergienko, S. R., Garbalinskiĭ, V. A., *Akad. Nauk Turkm. SSR, Ser. Fiz.-Tekhn., Khim. i Geol. Nauk* **3**, 45 (1963).

(60B) Spiovskiĭ, G. V., Kamp, R., *Khim. i Tekhnol. Goryuch. Slantsev i Produktov ikh Pererabotki* **12**, 215 (1963).

(61B) Skidanova, N. I., Chernozhukov, N. I., *Neftekhim., Akad. Nauk Turkm. SSR* **1963**, 91; *CA* **57**, 3691*d*.

(62B) Smith, J. W., *U. S. Bur. Mines Rept. Invest.* **5932** (1962).

(63B) Thomas, A., Lagoutte, P., *Chim. Anal.* **44**, 261 (1962).

(64B) Tisot, P. R., *J. Chem. Eng. Data* **7**, 405 (1962).

(65B) Yatsenko, E. A., Gutsalyuk, V. G., *Izv. Akad. Nauk kaz. SSR, Ser. Khim.* **1962**, No. 2, 77.

Gases (LPG)

(1C) Aliev, Ya. Yu., Aminova, R. I., *Issled. Mineral'n. i Rast. Syr'ya Uzbekistana, Akad. Nauk Uz. SSR, Inst. Khim.* **1962**, 106–15; *CA* **58**, 12341*e* (1963).

(2C) Am. Soc. Testing Materials, Philadelphia, Pa. "ASTM Standards 1964," Part 18, p. 885.

(3C) *Ibid.*, p. 956.

(4C) Baranenko, S. E., *Tr. Gas. Vses. Inst. po Proektir. i Nauchn.-Issled. Rabotam "Yuzhgiprotsement" (Yuzhnyi Gos. Inst. po Proektir. Tsementn. Predpriyatii) Sb.* **2**, 109–23 (1961); *CA* **58**, 7765*b* (1963).

(5C) Baranenko, S. E., Burnykh, N. M., *Novosti Neft. i Gaz. Tekhn., Gaz. Delo* **1962** (8), 30–3; *CA* **60**, 2697*c* (1964).

(6C) Cieplinski, E. W., Averill, W., Ettre, L. S., *J. Chromatog.* **8**, 550–4 (1962).

(7C) Colson, E. R., Brown, G. M., *J. Inst. Fuel* **35** (260), 399–404 (1962).

(8C) Demenkov, I. A., *Tr. Groznensk. Neft. Nauchn.-Issled. Inst.*, No. **15**, 351–2 (1963); *CA* **60**, 9069*g* (1964).

(9C) Dement'eva, M. I., Naumova, T. I., Prokopenko, N. A., *Neftekhimiya* **2** (6), 892–6 (1962); *CA* **59**, 4548*d* (1963).

(10C) Erickson, E. L., *Instr. Control Systems* **33**, 1362 (1960).

(11C) Genkin, A. N., *Fiz.-Khim. Metody Analiza i Issled. Produktov Proizv. Sintetich. Kauchuka, Vses. Nauchn. Issled. Inst. Sintetich. Kauchuka* **1961**, 32–54; *CA* **57**, 14414*i* (1962).

(12C) Graf, Laszlo, Toth, Jozsef, *Abhandl. Deut. Akad. Wiss. Berlin, Kl. Chem., Geol. Biol.* **1962**, No. 1, 145–61; *CA* **59**, 7275*e* (1963).

(13C) Guiochon, G., Landault, C., Annual Meeting, *Soc. Chim. France* Pau, May 17, 1963; *Bull. Soc. Chim. France*, No. **8–9**, 1526 (1963) Abstr.

(14C) Heuschkel, G., Wolny, J., Skoczowski, S., *Erdoel Kohle* **13**, 98–9 (1960).

(15C) Innes, W. B., Bambrick, W. E., Andreatch, A. J., Anal. Chem. **35**, 1198–203 (1963).

(16C) Jarzynska, M., Mirecka, T., *Chem. Anal., Warsaw* **8** (1), 91–4 (1963); *CA* **58**, 13106*e* (1963).

(17C) Kienitz, Hermann, *Z. Anal. Chem.* **192**, 160–89 (1963).

(18C) Kipping, P. J., Jeffery, P. G., *Res. Develop. Ind.*, No. 9, 46–9 (1962).

(19C) Kniebes, D. V., *Inst. Gas Technol., Tech. Rept.* **4**, (1962).

(20C) Koons, P. D., Walker, J. Q., *Hydrocarbon Process Petrol. Refiner* **42**, No. **4**, 133–5 (1963).

(21C) Lenac-Lukacevic, N., *Nafta (Zagreb)* **13**, 376 (1962); *J. Inst. Petrol.* **49**, No. 478, 211A (1963) Abstr.

(22C) Lulova, N. I., Tarasov, A. I., Kudryavtseva, N. A., Zemskova, E. I., *Tr. Komis, po Analit. Khim., Akad. Nauk SSSR, Inst. Gzokhim. i Analit. Khim.* **13**, 238–46 (1963); *CA* **59**, 9701*f* (1963).

(23C) Mamedova, V. M., Shnulin, A. N., Portyanskiĭ, A. E., *Neftekhimiya* **3** (4), 620–1 (1963); *CA* **59**, 12181*g* (1963).

(24C) Markosov, P. I., Zaichenko, V. N., Lityaeva, Z. A., *Zavodsk. Lab.* **27**, 285–7 (1961); *CA* **57**, 7906*d* (1962).

(25C) Mazitova, F. N., Ermakova, S. K., Virobyants, R. A., *Khim. i Tekhnol. Topliv i Masel* **7**, No. 4, 66–9 (1962); *CA* **57**, 5285*f* (1962).

(26C) Miller, A. J., *Rev. Inst. Franc. Petrole* **17**, (11), 1317–27 (1962).

(27C) Mueller, E. P., Freund, W., *Z. Angew. Geol.* **8**, 304–7 (1962); *CA* **57**, 12781*c* (1962).

(28C) Penchev, N. P., Pencheva, E. N., *Izv. Geol. Inst. Bulgar. Akad. Nauk* **12**, 257–66 (1963); *CA* **60**, 9069*e* (1964).

(29C) Pribyl, M., Soucek, J., *Collection Czech. Chem. Commun.* **26**, 1793–8 (1961); *CA* **57**, 1556*b* (1962).

(30C) Sarycheva, L. A., Pinchevskaya, S. I., *Novosti Neft. i Gaz. Tekhn., Neftepererabotka i Neftekhimiya*, **1962**, No. 1, 26–8; *CA* **59**, 2551*f* (1963).

(31C) Schubert, S., *Gas u. Wasserfach*, **102**, 1011–14 (1961); *CA* **56**, 2635*f* (1961).

(32C) Schuster, F., *Brennstoff-Chem.* **43**, 112–13 (1962).

(33C) Seiyama, T., Kato, A., *et al.*, Anal. Chem. **34**, 1502–3 (1962).

(34C) Skarvada, Ales, Tesarik, Karel, *Prace Vyzkum. Ustavu Cs. Naftovych Dolu, Publ.* **19**, No. 84–91, 115–19 (1962); *CA* **59**, 3690*e* (1963).

(35C) Smith, V. N., Merritt, E. J., Anal. Chem. **34**, 1476–82 (1962).

(36C) Sojak, L., *Ropa Uhlie* **4** (5), 136–9 (1962); *CA* **57**, 11448*f* (1962).

(37C) Spencer, C. F., *J. Chromatog.* **11**, 108–9 (1963).

(38C) Torocheshnikov, N. S., Semenova, V. A., *Vestn. Tekhn. i Ekon. Inform. Nauchn.-Issled. Inst. Tekhn.-Ekon. Issled. Gos. Komiteta Sov. Min. SSSR po Khim.* **1960**, No. 10, 55–7; *CA* **57**, 3695*a* (1962).

(39C) Tung, K'un-Nien, Yu, Wei-Le, *Shih'yu Lien Chih* **1960**, No. 11, 36–7; *CA* **59**, 355*d* (1963).

(40C) Van Luik, F. W., Jr., Rippere, R. E., Anal. Chem. **34**, 1617–20 (1962).

(41C) Vigdergauz, M. S., Gol'bert, K. A., *Neftekhimiya* **2**, 825–30 (1962); *CA* **57**, 2825*c* (1962).

(42C) Yurovskiĭ, Yu. M., *Razved. i Promysl. Geofiz., Vses. Nauchn.-Issled. Inst. Geofiz. Metodov Razvedki* **1959**, No. 30, 71–3 (1959); *CA* **57**, 3694*i* (1962).

(43C) Zhdanov, S. P., Kalmanovskiĭ, V. I., Kiselev, A. V., Fiks, M. M., Yashin, Ya. I., *Zh. Fiz. Khim.* **36**, 1118–20 (1962); *CA* **57**, 6581*h* (1962).

(44C) Zhukhovitskiĭ, A. A., Turkel'taub, N. M., Shlyakhov, A. F., *Khim. i Tekhnol. Topliv i Masel* **7**, No. 6, 7–11 (1962); *CA* **57**, 7879*h* (1962).

(45C) Zhukhovitskiĭ, A. A., Turkel'taub, N. M., *et al., Zavodsk. Lab.* **29**, No. 1, 8–13 (1963); *CA* **59**, 2145*b* (1963).

(46C) *Ibid.*, pp. 14–18; *CA* **59**, 5753*d* (1963).

(47C) Zielinski, Emil, *Chem. Anal. (Warsaw)* **8** (3), 399–404 (1963); *CA* **60**, 2320*b* (1964).

(48C) Zizin, V. G., Sokolova, V. I., *Khim. i Tekhnol. Topliv i Masel* **7**, No. 9, 27–29 (1962); *CA* **60**, 15147*h* (1964).

Gaseous and Liquid Fuels

(1D) Abel, K., de Schmertzing, H., Anal. Chem. **35**, 1754–6 (1963).

(2D) Adams, W. E., McCullough, J. D., *Proc. Am. Petrol. Inst.*, **40**(III), 64–70 (1960).

(3D) Agaev, N. A., Golubev, I. F., *Gaz. Prom.* **8**(5), 45–50 (1963).
(4D) *Ibid.*, **8**(7), 50–3 (1963).
(5D) Albert, D. K., ANAL. CHEM. **35,** 1918–21 (1963).
(6D) Beggi, C., *Riv. Combust.* **16**(10), 411–22 (1962).
(7D) Bellomo, A., D'Amore, G., *Chem. Tech.* (*Berlin*) **14**(5/6), 341–3 (1962).
(8D) Bentur, S., Babitz, M., Rocker, A., *Bull. Res. Council Israel*, Sect. C, **11**(2), 219–24 (1962).
(9D) Bentur, S., Babitz, M., Rocker, A., Skurnik, S., *J. Inst. Petrol.* **49**(473), 146–9 (1963).
(10D) Boyadzhiev, L., *Compt. Rend. Acad. Bulgare Sci.* **15**(2), 147–50 (1962).
(11D) Budanov, I. P., *Khim. i Tekhnol. Topliv i Masel* **8**(5), 66–8 (1963).
(12D) Caffo, E., *Riv. Combust.* **16**(7/8), 323–33 (1962).
(13D) Campo, C., *Chim. Anal.* (*Paris*) **45**(7), 343–7 (1963).
(14D) Clarkson, A., Gooderham, W. J., *Chem. Ind.* (*London*) **9,** 402–3 (March 3, 1962).
(15D) Claytor, E. E., Jones, J. H., *Oil Gas J.* **61,** 82, 85–6 (May 27, 1963).
(16D) Colson, E. R., Brown, G. M., *J. Inst. Fuel* **35**(260), 399–403 (1962).
(17D) Criqui, A., *Proc. 6th World Petrol. Congr.* **5,** Paper 23 (1963).
(18D) Dawson, H. J., Jr., ANAL. CHEM. **35,** 542–5 (1963).
(19D) Donlan, T. R., *Mater. Res. Std.* **2**(10), 820–4 (1962).
(20D) Funakubo, E., Taniguchi, H., Uejima, A., *Brennstoff-Chem.* **43**(9), 260–3 (1962).
(21D) Futterer, E., Muenz, G., *Ibid.* **44**(6), 187–91 (1963).
(22D) Gardner, L., Topol, G., *Mater. Res. Std.* **1**(2), 112–15 (1961).
(23D) Gelius, R., Preussner, K. R., *Chem. Tech.* (*Berlin*) **15**(5), 290–4 (1963).
(24D) George, M. E., *U. S. Aerospace Medical Research Labs., AMRL Memo* **M-26** (1963) 6 pp. (AD-296089).
(25D) Gol'dshtein, A. L., Lapisova, N. P., Shtifman, L. M., *Z. Analit. Khim.* **17**(1), 143–4 (1962).
(26D) Gross, W. R., Jackson, H. R., *et al.*, *Soc. Automotive Engrs., Natl. Fuels and Lubricants Mtg., Paper 610A*, Philadelphia, October 1962.
(27D) Harris, D. N., Coker, G. T., *Proc. 5th World Petrol. Congr.* **8,** 123–43 (1960).
(28D) Hawks, W. R., Edgington, P. G., *Ibid.*, **6,** 15–27 (1963).
(29D) Hoffmann, H. F., Chapin, R. H., Kelly, H. S., *Natl. Petrol. Refiners Assoc., Tech. Paper 62–17* San Antonio, April, 1962.
(30D) Hoggan, D., Battles, W. R., ANAL. CHEM. **34,** 1019–23 (1962).
(31D) Jenkins, G. I., Scruton, M. R., *J. Inst. Petrol.* **49**(474), 176–9 (1963).
(32D) Jentzsch, H., *Erdoel Kohle* **13,** 964–9 (1960).
(33D) Jones, J. T., Ludt, W. C., Kellogg, H. W., *Oil Gas J.* **61,** 112–14, 116 (Feb. 4, 1963).
(34D) *J. Inst. Petrol.* **48**(467), 388–90 (1962).
(35D) Krumbholz, K., Jaechel, G., *Chem. Tech.* (*Berlin*) **15**(5), 304 (1963).
(36D) Lapisova, N. P., Gol'dshtein, A. L., *Zh. Analit. Khim.* **16**(4), 508–9 (1961).
(37D) Lavrent'ev, V. I., *Novosti Neft. i Gaz. Tekhn. Neftepererabotka i Neftekhim.* (**9**), 24–8 (1962), *CA* **60,** 15653*g*.
(38D) Likhachev, A. D., *Zavodsk. Lab.* **29**(11), 1302–4 (1963).
(39D) Loomer, D. M., Graham, A. L., *Oil Gas J.* **61,** 82–4 (Jan. 14, 1963).
(40D) Loroue, D., Paul, G., *Bull. Assoc. Franc. Techniciens Pétrole* **157,** 127–38 (1963).
(41D) Loroue, D., Paul, G., *Rev. Inst. Franc. Pétrole Ann. Combust. Liquides* **17**(6), 830–41 (1962).
(42D) *Mater. Res. Std.* **2**(6), 494–7 (1962).
(43D) Morris, T. A., Shively, J. H., Constantin, C. S., ANAL. CHEM. **33,** 1556–8 (1961).
(44D) Nagypataki, G., Tamási, Z., *Magy. Kém. Lapja* **17**(3), 140–3 (1962).
(45D) Nelson, W. L., *Oil Gas J.* **61,** 100 (Aug. 26, 1963).
(46D) Nottes, E. G., *Erdoel Kohle* **14** (8), 616–20 (1961).
(47D) Ogawa, T., *Nenryo Kyokaishi* **41,** 99–103 (1962).
(48D) *Oil Gas Intern.* **3**(11), 58–60 (1963).
(49D) Parker, W. W., Hudson, R. L., ANAL. CHEM. **35,** 1334–5 (1963).
(50D) Parker, W. W., Smith, G. Z., Hudson, R. L., *Ibid.*, **33,** 1170–1 (1961).
(51D) Pedinelli, M., *Chim. Ind.* (*Milan*) **44**(6), 651–2 (1962).
(52D) Pom̃ykala, Z., *Nafta* (*Katowice*) **18,** 250–2 (1962).
(53D) Powell, H., *J. Appl. Chem.* **6,** 488–94 (1956).
(54D) Randi, M., *Atti Accad. Roveretana Agiati* **210** (*Ser. VI, 2B*), 91–5 (1961).
(55D) Reznikov, V. D., *Khim. i Tekhnol. Topliv i Masel* **7**(9), 61–3 (1962).
(56D) Rogers, J. D., Krynitsky, J. A., Churchill, A. V., *Soc. Automotive Engrs., Natl. Aeron. Space Eng. Mfg. Mtg., Paper 583C*, Los Angeles, October 12, 1962.
(57D) Rozhkov, I. V., Marunin, M. P., *Novosti Neft. i Gas. Tekhn., Neftepererabotka i Neftekhim.* **1,** 45–7 (1962); *CA* **58,** 8832*c*.
(58D) Ruf, H., *Schweiz. Archiv. Angew. Wiss. Tech.* **29,** 428–44 (1963).
(59D) Ruf, H., Rüetschi, W., Zimmermann, P., Mosca, C., *Mitt. Gebiete Lebensm. Hyg.* **54**(2), 114–24 (1963).
(60D) Rybakov, P. A., Zhukov, N. A., Isaeva, N. S., *Khim. i Tekhnol. Topliv i Masel* **7**(11), 68–71 (1962).
(61D) Sablina, Z. A., Fomina, A. M., Churshkov, E. S., Sakodynskaya, T. P., *Ibid.*, **8,** 57–61 (1963).
(62D) Schubert, S., *Gas u. Wasserfach* **102,** 1011–14 (1961).
(63D) Sharapov, J. F., Fomina, A. M., *Khim. i Tekhnol. Topliv i Masel* **7,** 64 (1962).
(64D) Shimonaev, G. S., Stepanova, L. S., *Ibid.*, **7**(9), 67–70 (1962).
(65D) Stone, C. T., Soc. Automotive Engrs., Natl. Aeron. Space Eng. Mfg. Mtg., *Paper 583D*, Los Angeles, October 1962.
(66D) Tagliavini, G., Belluco, U., Riccoboni, L., *Ric. Sci. Rend.* Sez. A. **1,** 338–45 (1961).
(67D) Taylor, J. H., *Mater. Res. Std.* **1**(8), 626 (1961).
(68D) Wolf, W., *Brennstoff-Chem.* **43**(9), 257–60 (1962).
(69D) Zabryanskiĭ, E. I., Losaev, K. N., Shchegol, B. B., Aronov, G. M., Zarubin, A. P., *Khim. i Tekhnol. Topliv i Masel* **8**(6), 65–9 (1963).

Lubricating Oil

(1E) Am. Soc. Testing Materials, Philadelphia, Pa., "Engine Test Sequences for Evaluating Lubricants for API Services," **STP 315.**
(2E) Bor, G., *Magyar Asvanyolaj Foldgaz Kiserl. Int. Kozlem.* **2,** 143 (1961).
(3E) Bornemann, P., Finke, M., Heinze, G., *Chem. Tech.* (*Berlin*) **14,** 292 (1962).
(4E) Brewer, P. I., *J. Inst. Petrol.* **48,** 277, 287 (1962).
(5E) Cummins, R. A., Mason, P. R., *Ibid.*, **48,** 237 (1962).
(6E) Delves, R. B., *Ibid.*, **48,** 283 (1962).
(7E) Dichter, M., Ogradowska, J., *Chem. Tech.* (*Berlin*) **14,** 569 (1962).
(8E) Dornenburg, E., Serboli, G., *Brown Boveri Rev.* **49,** 590 (1962).
(9E) Drescher, K., Teubel, J., *Freiberger Forschungst.* **A296,** 431 (1963).
(10E) Eckardt, H., Keil, G., Rentrop, K. H., *Chem. Tech.* (*Berlin*) **14,** 305 (1962).
(11E) Ellis, E. G., *Sci. Lubrication* **14,** 14 (1962).
(12E) Engel, M., *Elektrie* **4,** 119 (1962).
(13E) Engel, M., Moebes, R., *Ibid.*, **5,** 157 (1962).
(14E) Faerman, D. V., Chigrin, D. S., *Khim. i Tekhnol. Topliva i Masel* **8,** 64 (1963).
(15E) Finke, M., Heinze, G., *J. Prakt. Chem.* **20,** 304 (1963).
(16E) *Ibid.*, **21,** 250 (1963).
(17E) Fisher, W., *J. Inst. Petrol.* **48,** 290 (1962).
(18E) Florkowski, T., Gilewicz, J., *Nucleonika* **7,** 793 (1962).
(19E) Hall, C. A., Martin, E. A., *ASTM, 4th Pacific Area Natl. Meeting,* Paper 145, 1962.
(20E) Hall, D. A., "Turbine Oils," *Am. Soc. Testing Materials,* **STP 321,** 107 (1962).
(21E) Hasegawa, K., Kajikawa, M., Kawaguchi, M., *Bull. Japan Petrol. Inst.* **5,** 82 (1963).
(22E) Hensley, A. L., Bergmann, J. G., *Anal. Chem.* **35,** 1318 (1963).
(23E) Keil, G., Eckardt, H., Milowsky, L., *Freiberger Forschungsh.* **A250,** 87 (1962).
(24E) Keil, G., Milowsky, L., Saxe, H., *Chem. Tech.* (*Berlin*) **14,** 274 (1962).
(25E) Kendall, P. F., Rimmer, A., *Chem. Ind.* **43,** 1864 (1962).
(26E) Khalupovskiĭ, M. D., *Zavodsk. Lab.* **28,** 206 (1962).
(27E) Khudominskaya, L. S., Aksenova, N. N., *Tr. Vses. Nauchn.-Issled. Inst. Pererabotki i Ispol'z. Topliva* **11,** 304 (1962).
(28E) LaSell, J. H., *Develop. Appl. Spect.* **2,** 239 (1962).
(29E) Lock, J. K., Mason, P. R., *Nature* **199,** 652 (1963).
(30E) Lopez de Azcona, J. M., Sanz de la Rosa, J., 8th International Colloquium on Spectroscopy, Lucerne, Switzerland, p. 129, 1960.
(31E) Loveland, J. W., *et al.*, *Proc. Am. Petrol. Inst.* **40,** 410 (1960).
(32E) McLaughlin, E. J., *ASTM, 4th Pacific Area Natl. Meeting,* Los Angeles, Paper 146, 1962.
(33E) Manley, L. W., Henrikson, K. G., *ASTM, 65th Annual Meeting,* New York, Preprint 85f, 1962.
(34E) Mashireva, L. G., Zimina, K. I., *Novosti Neft. i Gaz. Tekhn. Neftepererabotka i Neftekhim.* **8,** 15 (1961).
(35E) Milz, W. C., Bunting, J. T., *ASTM, 65th Annual Meeting,* New York, Preprint 85c, 1962.
(36E) Palovcikova, M., *Ropa Uhlie* **4,** 114 (1962).
(37E) Papock, K. K., *et al.*, *Proc. 6th World Petrol. Congr. Sec.* **IV,** 303 (1963).
(38E) Rost, R., *Brennstoff Chem.* **43,** 341 (1962).
(39E) Schreiber, T. P., Ottolini, A. C., Johnson, J. L., *Appl. Spectry.* **17,** 17 (1963).
(40E) *Sci. Lubrication* **14,** 23 (1962).
(41E) Serboli, G., Golgowski, L., *Elektrotech. Z.* **83,** 530 (1962).
(42E) Siegfriedt, R. K., DeWoody, O. A., *Proc. Am. Petrol. Inst.* **40,** 416 (1960).
(43E) Stannett, A. W., *Proc. Inst. Elec. Engrs.* **109A,** 80 (1962).
(44E) Tennyson, T. A., *Lubrication Eng.* **18,** 514 (1962).

Greases

(1F) Bird, R. B., Turian, R. M., *Chem. Eng. Sci.* **17,** 331–4 (May 1962).
(2F) Brunstrum, L. C., *NLGI Spokesman* **27,** 144 (August 1963).
(3F) Carter, C. F., Bauman, F., *Ibid.,* **27,** 213–14 (October 1963).
(4F) Cox, D. B., *Nature (London)* **193,** 670 (1962).
(5F) Criddle, D. W., *NLGI Spokesman* **27,** 108–11 (July 1963).
(6F) Ewbank, W. J., Dye, J., Gargaro, J., Doke, K., Beattie, J., *Ibid.,* **27,** 75–82 (June 1963).
(7F) Febles, R., Bresciano, N., Maccoll Piriz, C. R., *Ind. Quim. (Buenos Aires)* **20,** 618–27 (1960); *J. Appl. Chem. (London)* **12,** ii-225 (September 1962) (abstract).
(8F) Gabrysh, A. F., Eyring, H., *et al., Rev. Sci. Instr.* **33,** 670–82 (June 1962).
(9F) Herold, R., *Erdoel Kohle* **16,** 957 (September 1963) (abstract).
(10F) Jenkins, G., Soc. Automotive Engrs., International Summer Meeting, Montreal, Paper 709B, June 1963.
(11F) Krym, K. S., Meshchaninov, S. M., Zabelina, N. P., *Proizv. Smaz. Materialov* No. **9,** 34–59 (1962).
(12F) Pope, C. L., *NLGI Spokesman* **27,** 221 (October 1963).
(13F) Rooney, G., Bird, R. J., *J. Inst. Petrol.,* **49** (476), 256–8 (1963).
(14F) Smithson, L. D., Matta, M. S., U. S. Dept. Comm. Office Tech. Services, Rept. **AD 287756** (August 1962).

Waxes

(1G) Barmby, D. S., Bostwick, L. G., Huston, J. A., Jr., *Proc. 6th World Petrol. Congr.* **6,** 161 (1963).
(2G) Barras, R. C., Boyle, J. F., Grainger, C. F., Tech. Assoc. Pulp Paper Ind., Spec. Tech. Assoc. Publ. **2,** 43 (1963).
(3G) Bluestein, B. R., Hindman, T. D., Bluestein, C. K., *Ibid.,* **2,** 146 (1963).
(4G) Braithwaite, B., Penketh, G. E., *Analyst* **49,** 297 (1963).
(5G) Brown, D. S., Lewis, W. E., Smith, A. C., Jr., Tech. Assoc. Pulp Paper Ind., Spec. Tech. Assoc. Publ. **2,** 135 (1963).
(6G) Dannenbrink, R. W., Walker, D. I., reprint from *Paper, Film and Foil Converter* (February and March 1963).
(7G) Dietz, W. A., Starnes, P. K., Brown, R. A., Tech. Assoc. Pulp Paper Ind., Spec. Tech. Assoc. Publ. **2,** 33 (1963).
(8G) Edwards, R. T., *Ibid.,* **2,** 95 (1963).
(9G) Eells, R., Brunson, M. O., *Ibid.,* **2,** 176 (1963).
(10G) Ferris, S. W., *Ibid.,* **2,** 1 (1963).
(11G) Fox, R. C., *Ibid.,* **2,** 160 (1963).
(12G) Gerner, M. M., Rudnitskaya, E. A., *Zavodsk. Lab.* **29,** 733 (1963).
(13G) Guseva, A. N., Ashkinadze, L. D., Leifman, I. E., *Izv. Akad. Nauk SSSR, Ser. Fiz.* **27,** No. 1, 104 (1963).
(14G) Haenni, E. O., *et al., J. Assoc. Offic. Agr. Chemists* **45,** 59 (1962).
(15G) *Ibid.,* p. 67.
(16G) Helberg, I. D., *Deut. Lebensm. Rundschau* **58,** 321 (1962).
(17G) Hildebrand, G., Peper, C., Dahlke, B., *Chem. Tech. (Berlin)* **15** (3), 147 (1963).
(18G) Levy, E. J., Galbraith, F. J., Melpolder, F. W., Advances in Mass Spectrometry, Proceedings of 2nd Conference, Oxford, 1961, Vol. **2,** 395, 1963.
(19G) Levy, E. J., Paul, D. G., 9th Proceedings of National Instrumentations Symposium, Houston, Tex., **1963,** p. 105.
(20G) Lijinsky, W., Domsky, I., Mason, G., Ramahi, H. Y., Satavi, T., *Anal. Chem.* **35,** 952 (1963).
(21G) Lijinsky, W., Domsky, I., Raha, C. R., *J. Assoc. Offic. Agr. Chemists* **46,** 725 (1963).
(22G) Lizogub, A. P., Sklyar, V. T., Krasnova, S. I., *Nauchn. Zap., Gos. Nauchn.-Issled. i Proektn. Inst. Ugol'n., Rudn., Neft. i Gaz. Prom.* **1962,** No. 8, 18.
(23G) McMahon, G. S., Wood, J. A., Tech. Assoc. Pulp Paper Ind., Spec. Tech. Assoc. Publ. **2,** 78 (1963).
(24G) Mal'nev, A. F., Mikhlina, I. M., *Ukr. Fiz. Zh.,* **6,** 859 (1961).
(25G) Moyer, H. C., Zmitrovis, R. P., Tech. Assoc. Pulp Paper Ind., Spec. Tech. Assoc. Pub. **2,** 121 (1963).
(26G) Negoro, K., *Bull. Chem. Soc. Japan* **35,** 375 (1962).
(27G) *Ibid.,* p. 916.
(28G) Negoro, K., *Kogyo Kagaku Zasshi* **64,** 295 (1961).
(29G) Negoro, K., *Nippon Kagaku Zasshi* **82,** 229 (1961).
(30G) Noll, R. G., Grogan, P. M., Tech. Assoc. Pulp Paper Inc. Spec. Tech. Assoc. Publ. **2,** 112 (1963).
(31G) Phillips, J., *Ibid.,* **2,** 20 (1963).
(32G) Presting, W., Steinbach, K., Foehse, C., *Chem. Tech. (Berlin)* **15,** 159 (1963).
(33G) Prokhorova, E. K., Znamenskii, N. N., *Vopr. Onkol.* **9** (8), 72 (1963).
(34G) Shirdzhanov, N., *Tr. Turkmensk. Filiala Vses. Neft. Nauchn.-Issled. Inst.* **1962,** (5), 205.
(35G) Templin, P. R., Tech. Assoc. Pulp Paper Ind., Spec. Tech. Assoc. Publ. **2,** 51 (1963).
(36G) Teubel, J., Roesner, H., Leschner, O., *Chem. Tech. (Berlin)* **14,** 320 (1962).

Asphalt

(1H) Abraham, H., "Asphalts and Allied Substances," 6th ed., Vol. V, Van Nostrand, New York, 1962.
(2H) Aida, H., *J. Japan Petrol. Inst.* **4,** 845 (1961).
(3H) Arato, J., *Banyasz. Kutato Intezet Kozlemenyei* **6,** 337 (1961).
(4H) Barrall, E. M., II, Schmidt, R. J., Johnson, J. F., Division of Petroleum Chemistry, 147th Meeting, ACS, Philadelphia, Pa., 1964, Preprints **9,** No. 2, B27.
(5H) Barth, E. J., "Asphalt, Science and Technology," Gordon and Breach Science Pub., New York, 1962.
(6H) Bergmann, G., *Erdoel Kohle* **15,** No. 8, 612 (1962).
(7H) Bestougeff, M. A., Gendrel, P., Division of Petroleum Chemistry, 147th Meeting, ACS, Philadelphia, Pa., 1964, Preprints **9,** No. 2, B51.
(8H) Bissett, J. R., Ford, M. C., Jr., *Proc. Assoc. Asphalt Paving Technol.* **31,** 329 (1962).
(9H) Bodan, A. N., Sklyar, V. T., *Nauchn. Zap., Gos. Nauchn.-Issled. i Proekten. Inst. Ugol'n., Rudn., Neft. i Gaz. Prom.* **1962,** No. 8, 58.
(10H) Bohn, A. O., *Proc. Assoc. Asphalt Paving Technol.* **32,** 353 (1963); *Bitumen, Teere, Asphalte, Peche* **14,** No. 9, 454 (1963).
(11H) Boyd, M. L., Montgomery, D. S., *Fuel* **41,** No. 4, 335 (1962).
(12H) Busching, H. W., Mid-Continent Regional Meeting, National Petroleum Refiners Association, Wichita, Kan., June 1963, Tech. Paper **63.**
(13H) Campbell, P. G., Wright, J. R., Division of Petroleum Chemistry, 147th Meeting, ACS, Philadelphia, Pa., 1964, Preprints **9,** No. 2, B89; *J. Res. Natl. Bur. Stds.* **C68,** 115 (1964).
(14H) Campbell, P. G., Wright, J. R., Division of Petroleum Chemistry, 148th Meeting, ACS, Chicago, Ill., 1964, Preprints **9,** No. 3, 147.
(15H) Campbell, P. G., Wright, J. R., Bowman, P. B., Division of Petroleum Chemistry, 142nd Meeting, ACS, Atlantic City, N. J., 1962, Preprints **7,** No. 3, 123; *Mater. Res. Std.* **2,** No. 12, 988 (1962).
(16H) Caro, J. H., *Erdoel-Z.* **1962,** No. 7; *Bitumen, Teere, Asphalte, Peche* **13,** No. 11, 526 (1962) (Cond.).
(17H) Caro, J. H., *J. Inst. Petrol.* **49,** No. 471, 85 (1963).
(18H) Carpenter, C. A., 66th Annual Meeting, Am. Soc. Testing Materials, Atlantic City, June 1963, Preprint **79e.**
(19H) Carre, G., *Bull. Assoc. Franc. Techniciens Petrole.* No. **160,** 491 (1963).
(20H) Carre, G., Laurent, D., *Ibid.,* **157,** 3 (1963).
(21H) Chelton, H. M., Traxler, R. N., *Proc. 5th World Petrol. Congr.* **5,** 247 (1960).
(22H) Constantinides, G., Arich, G., Lomi, C., *Ibid.,* **5,** 131 (1960).
(23H) Corbett, L. W., Division of Petroleum Chemistry, 147th Meeting, ACS, Philadelphia, Pa., 1964, Preprints **9,** No. 2, B81.
(24H) Corbett, L. W., Swarbrick, R. E., "Recent Research on Bituminous Materials," Am. Soc. Testing Materials, **STP 347,** 39 (1963).
(25H) Egorov and Natanzon, *Avtomobil'yne Dorogi* **1962,** No. 10; *Bitumen, Teere, Asphalte, Peche* **14,** No. 2, 63 (1963) (abstract).
(26H) Eldib, I. A., Division of Petroleum Chemistry, 141st Meeting, ACS, Washington, D. C., 1962, Preprints **7,** No. 1, 31.
(27H) Erdman, J. G., Dickie, J. P., Division of Petroleum Chemistry, 147th Meeting, ACS, Philadelphia, Pa., 1964, Preprints **9,** No. 2, B69.
(28H) Evans, C. C., Griffin, R. L., *Proc. Assoc. Asphalt Paving Technol.* **32,** 64 (1963).
(29H) Ewers, N., Hungarian Chemists' Association, Hungarian Academy of Science Petroleum Conference, Budapest, April 1962; *Chem. Tech. (Berlin)* **14,** No. 5–6, 377 (1962) (abstract).
(30H) Farley, L. L., Guffy, J. C., Leonard, Vasse, L., Division of Petroleum Chemistry, 147th Meeting, ACS, Philadelphia, Pa., 1964, Preprints **9,** No. 2, B15.
(31H) Fink, D. F., *Mater. Res. Std.* **3,** No. 1, 14 (1963).
(32H) Fischer, K. A., Schram, A., *Proc. 5th World Petrol. Congr.* **5,** 259 (1960).
(33H) Florovskaya, V. N., Klyuev, Y. A., *Dokl. Akad. Nauk Belorrusk. SSR* **5,** No. 10, 438 (1961).
(34H) Gorshenina, G. I., Kuperman, M. E., Mikhailov, N. V., *Kolloidn. Zh.* **26,** No. 2, 165 (1964).
(35H) Gorshenina, G. I., Mikhailov, N. V., *Dokl. Akad. Nauk SSR* **154,** No. 4, 929 (1964).
(36H) Gotolski, W. H., Ciesielski, S. K., Kofalt, J. A., 67th Annual Meeting, ASTM, Chicago, June 1964.
(37H) Greenfeld, S. H., *Ind. Eng. Chem. Prod. Res. Develop.* **3,** 158 (1964).
(38H) Griffin, R. L., Izatt, J. O., Lettier, J. A., ASTM, "Symposium on Fundamental Viscosity of Bituminous Materials," **STP 328,** 67 (1962).
(39H) Griffith, J. M., *Asphalt Inst. Quart.* **14,** No. 3, 4 (1962).
(40H) Griffith, J. M., Puzinauskas, V. P., Am. Soc. Testing Materials, **STP 328,** 20 (1962).
(41H) Gundermann, E., *Chem. Tech. (Berlin)* **14,** No. 5–6, 324 (1962).
(42H) Gundermann, E., Kloss, B., *Ibid.,* **16,** No. 1, 41 (1964).
(43H) Gzemski, F. C., Am. Soc. Testing Materials, **STP 328,** 80–94 (1962).
(44H) Halstead, W. J., *Proc. Assoc. Asphalt Paving Technol.* **32,** 247 (1963).

(45H) Halstead, W. J., Oglio, E. R., Olsen, R. E., *Public Roads* **32,** 88 (1962).
(46H) Halstead, W. J., Zenewitz, J. A., *Ibid.,* **31,** 211 (1961).
(47H) Hara, A., Takahashi, S., *et al., J. Japan Petrol. Inst.* **4,** 197 (1961) *Bull. Japan Petrol. Inst.* **4,** 68 (1962) (abstract).
(48H) Heithaus, J. J., *Ind. Eng. Chem. Prod. Res. Dev.* **1,** 149 (1962).
(49H) Herrin, M., Jones, G. E., *Proc. Assoc. Asphalt Paving Technol.* **32,** 82 (1963).
(50H) Herrin, M., Manke, P. G., George, Z., 66th Annual Meeting ASTM, Atlantic City, June 1963, Preprint **79c.**
(51H) Highway Research Board, Div. Eng. and Ind. Research, Spec. Rept. **61A,** "AASHO Road Test. History and Description of Project," National Academy Sciences–National Research Council, Washington, D. C. Pub. **816** (1961).
(52H) *Ibid.,* **61B,** "AASHO Road Test, Rept. 2, Materials and Construction," Publ. **951** (1962).
(53H) *Ibid.,* **61C,** "AASHO Road Test, Rept. 3, Traffic Operations and Pavement Maintenance," Publ. **952** (1962).
(54H) *Ibid.,* **61D,** "AASHO Road Test Rept. 4 Bridge Research," Publ. **953** (1962).
(55H) *Ibid.,* **61E,** "AASHO Road Test, Rept. No. 5, Pavement Research," Publ. **954** (1962).
(56H) *Ibid.,* **61F,** "AASHO Road Test, Rept. 6, Special Studies," Publ. **955** (1962).
(57H) Hirose, H., Tsuyuki, U., Wada, Y., *Oyo Butsuri* **32,** 29 (1963).
(58H) Hirose, H., Wada, Y., *Japan J. Appl. Phys.* **1,** 174 (1962).
(59H) Holl, A., *Bitumen, Teere, Asphalte, Peche* **14,** No. 4, 188, 192 (1963).
(60H) Horowitz, E., Mandel, J., *Mater. Res. Std.* **3,** No. 9, 723 (1963).
(61H) Hughes, C. S., *Proc. Assoc. Asphalt Paving Technol.* **31,** 400 (1962).
(62H) Hughes, F. J., *Ind. Eng. Chem., Prod. Res. Dev.* **1,** 290 (1963).
(63H) Hunter, J. N., Jr., *Mater. Protect.* **3,** No. 2, 50, 56, 58 (1964).
(64H) Hveem, F. N., Zube, E., Skog, J., *Proc. Assoc. Asphalt Paving Technol.* **32,** 271 (1963).
(65H) Iijima, H., *J. Japan Petrol. Inst.* **8,** 559 (1962).
(66H) *Ibid.,* p. 564.
(67H) Iijima, H., Koma, H., *Ibid.,* **8,** 568 (1962).
(68H) Illman, J. C., Deahl, T. J., Am. Soc. Testing Materials, **STP 347,** 21–28 (1963).
(69H) International Conference on Structural Design of Asphalt Pavements Proceedings, University of Michigan, Ann Arbor, Mich., August 1962.
(70H) *Ibid.,* Preprint Volume.
(71H) *Ibid.,* Preprint Volume, Supplement.
(72H) Jeffries-Harris, M. J., Poindexter, E. H., *J. Chem. Phys.* **38,** No. 1, 148 (1963).
(73H) Jimenez, R. A., Gallaway, B. M. *Proc. Assoc. Asphalt Paving Technol.* **31,** 477 (1962).
(74H) Jones, J. H., Moote, T. P., Division of Petroleum Chemistry, 144th Meeting ACS, Los Angeles, Calif., 1963, Preprints **8,** No. 2, A29.
(75H) Jones, P. M., Am. Soc. Testing Materials, **STP 347,** 70–80 (1963).
(76H) Jones, P. M., Division of Petroleum Chemistry, 147th Meeting, ACS, Philadelphia, Pa., 1964, Preprints **9,** No. 2, B107.
(77H) Jones, P. M., *Mater. Res. Std.,* **3,** No. 4, 293 (1963).
(78H) Jones, T. K., Am. Soc. Testing Materials, **STP 347,** 48–57 (1963).
(79H) Kaganov, S. A., Levinter, M. Kh., Medvedeva, M. I., *Khim. i Tekhnol. Topliv i Masel* **7,** No. 7, 38 (1962).
(80H) Kannisto, P., Harva, O., Laiho, A., *Valtion Tek. Tutkimuslaitos, Julkaisu,* **1960,** No. 54, 1.
(81H) Kapkova, E. I., *Tr. Khar'kovsk Avtomob.-Dor. Inst.* **1961,** No. 26, 76.
(82H) Kari, W. J., Coyne, L. D., McCoy, P. E., *Proc. Assoc. Asphalt Paving Technol.* **31,** 1 (1962).
(83H) Kari, W. J., Santucci, L. E., *Ibid.,* **32,** 148 (1963).
(84H) Karius, H., Dalton, G. L., *J. Inst. Petrol.* **50,** No. 481, 1 (1964).
(85H) Knight, R. S., Platt, R. B., Smith, D. J., Am. Soc. Testing Materials, **STP 347,** 82 (1963).
(86H) Knoterus, J., Krom, C. J., Division of Petroleum Chemistry, 147th Meeting, ACS, Philadelphia, Pa., 1964, Preprints **9,** No. 2, B39.
(87H) Kolbanovskaya, A. S., Golovkina, O. K., *Wiss. Z. Hochsch. Bauwesen, Leipzig* **8,** No. 2, 107 (1962).
(88H) Kossowicz, L., *Proc. 5th World Petrol. Congr.* **5,** 411 (1960).
(89H) Kraemer, P., *Bitumen, Teere, Asphalte, Peche* **15,** No. 2, 68; No. 4, 160 (1964).
(90H) Krom, C. J., Dormon, G. M., 6th World Petrol. Congr., Frankfurt a. M., June 1963, Sect. VI, Paper **14.**
(91H) Kurnosenkova, S. A., Slepchenko, L. G., *Neftepererabotka i Neftekhim., Nauchn. Tekhn. Sb.,* No. **7,** 14 (1963).
(92H) Lammiman, K. A., Les, T., Way, P. J., *J. Appl. Chem. (London)* **12,** No. 11, 510 (1962).
(93H) Leibnitz, E., Mueller, K., Gundermann, E., *Acta Chim. Acad. Sci. Hung.* **31,** 145 (1962).
(94H) Lenz, R. W., Barrante, J. R., Andrews, R. D., *J. Appl. Polymer Sci.* **7,** No. 5, S45 (1963).
(95H) Levinter, M. Kh., Galiakbarov, M. F., *Khim. i Tekhnol. Topliv i Masel* **9,** No. 3, 32 (1964).
(96H) Levy, D. F., Am. Soc. Testing Materials, **STP 328,** 3–19 (1962).
(97H) Livneh, M., Shklarsky, E., *Proc. Assoc. Asphalt Paving Technol.* **31,** 457 (1962).
(98H) Lottman, R. P., Sonawala, S. K., Al-Habboobi, M., *Ibid.,* **32,** 1 (1963).
(99H) McCoy, P., Coyne, L. D., 67th Annual Meeting, Am. Soc. Testing Materials, Chicago, Ill., June 1964.
(100H) Maksimova, S. N., *Geokhim. Nefti i Neft. Mestorozhd. Akad. Nauk. SSSR, Inst. Geol. i Razrabotki Goryuch Iskop.,* **1962,** 5.
(101H) Mapstone, G. E., *Hydrocarbon Process., Petrol. Refiner* **42,** No. 6, 149 (1963).
(102H) Martin, K. G., Division of Petroleum Chemistry, 147th Meeting, ACS, Philadelphia, Pa., 1964, Preprints **9,** No. 2, B115.
(103H) Martin, K. G., Division of Petroleum Chemistry, 148th Meeting, ACS, Chicago, Ill., 1964, Preprints **9,** No. 3, 175.
(104H) Mathews, D. H., Brown, J. R., Welch, T. R., *J. Appl. Chem. (London)* **12,** 64 (1962).
(105H) *Nachr. Chem. u. Tech.,* No. **1,** (1963); *Bitumen, Teere, Asphalte, Peche* **15,** No. 6, 293 (1964) (abstract).
(106H) Nelson, W. L., *Oil Gas J.* **61,** No. 48, 159 (1963).
(107H) *Ibid.,* **62,** No. 19, 149 (1964).
(108H) Nelson, W. L., Patel, S., *Ibid.,* **62,** No. 7, 120 (1964).
(109H) Nevitt, H. G., *Proc. Assoc. Asphalt Paving Technol.* **31,** 90 (1962).
(110H) Newman, S. B., 21st Annual Meeting Electron Microscope Society, Denver, August 1963.
(111H) Newman, S. B., Greenfeld, S. H., *Mater. Res. Std.* **3,** No. 11, 893 (1963).
(112H) Nishikawa, E., Yamamoto, H., Toyoguchi, M., *J. Japan Petrol. Inst.* **3,** 547 (1960).
(113H) Oglio, E. R., Zenewitz, J. A., Halstead, W. J., *Public Roads* **31,** 248 (1962).
(114H) Oksala, N., *Valtion Tek. Tutkimuslaitos Julkaisu* **72**; *Bitumen, Terre, Asphalte, Peche* **15,** No. 4, 173 (1964) (cond.).
(115H) Oliensis, G. L., Am. Soc. Testing Materials, **STP 347,** 58–69 (1963).
(116H) Padovani, C., Berti, V., Prinetti, A., *Proc. 5th World Petrol. Congr.* **5,** 273 (1960).
(117H) Parr, W. K., Annual Meeting, Natl. Petrol. Refiners Assoc., San Antonio, April 1962, Tech. Paper **62-18.**
(118H) Penchev, V., Andreev, A., Davidova, N., Hungarian Chemists Assoc., Hungarian Academy of Science, Petroleum Conference (Budapest, April 1962); *Bitumen, Teere, Asphalte, Peche,* **13,** No. 10, 493 (1962) (abstract).
(119H) Phillips, U. A., Traxler, R. W., *Appl. Microbiol.* **11,** 235 (1963).
(120H) Pignataro, L. J., *Proc. Assoc. Asphalt Paving Technol.* **31,** 563 (1962).
(121H) Please, A., Hardman, R., *Chem. Ind. (London)* **1962,** 1610.
(122H) Poindexter, E. H., *J. Chem. Phys.* **37,** No. 3, 463 (1962); **39,** No. 10, 2772 (1963).
(123H) Prinzler, H., Klimke, R., Dorn, N., *Chem. Tech. (Berlin)* **14,** No. 12, 739 (1962).
(124H) Psalomshchikova, K. I., *Tr. Vses. Neft. Nauch.-Issled. Geologorazved. Inst.* **1961,** 199.
(125H) Rajeswaran, P., Kirk, P. L., *Microchem. J.* **6,** No. 1, 21 (1962).
(126H) Rodionova, K. F., Shishenina, E. P., Kanakina, M. A., *Tr. Vses. Nauchn.-Issled. Geologorazved. Neft. Inst.* No. **33,** 211 (1962).
(127H) Rostler, F. S., White, R. M., *Proc. Assoc. Asphalt Paving Technol.* **31,** 35 (1962).
(128H) Sakanoue, S., *Nippon Kagaku Zasshi* **84,** No. 5, 384 (1963).
(129H) Santucci, L. E., Schmidt, R. J., *Bull. Highway Res. Board* **333,** 1 (1962).
(130H) Schmidt, R. J., Santucci, L. E., 66th Annual Meeting, Am. Soc. Testing Materials, Atlantic City, N. J., June 1962, Preprint **79a.**
(131H) Schuhbauer, A., *Bitumen, Teere, Asphalte, Peche* **13,** No. 6, 273 (1962).
(132H) Sergienko, S. R., Garbalinskiĭ, V. A., Hungarian Chemists Association, Hungarian Academy of Science, Petroleum Conference Budapest, April 1962; *Chem. Tech. (Berlin)* **14,** No. 5–6, 377 (1962) (abstract).
(133H) Shook, J. F., 66th Annual Meeting, Am. Soc. Testing Materials, Atlantic City, N. J., June 1963, Preprint **79d.**
(134H) Skog, J., Zube, E., *Proc. Assoc. Asphalt Paving Technol.* **32,** 380 (1963).
(135H) Smith, C. D., Schuetz, C., Hodgson, R. S., Division of Organic Coatings and Plastics Chemistry, 145th Meeting, ACS, New York, N. Y., 1963, Abstracts of Papers, p. 11R.
(136H) Smith, D. B., Schweyer, H. E., *Ind. Eng. Chem., Process Design Develop.* **2,** 209 (1963).
(137H) Speer, T. L., *Proc. Asphalt Paving Technol.* **31,** 507 (1962).
(138H) Speer, T. L., Brunstrum, L. C., Sisko, A. W., Ott, L. E., Evans, J. V., *Ibid.,* **32,** 236 (1963).
(139H) Spurr, R. T., *J. Appl. Polymer Sci.* **7,** 2211 (1963).
(140H) Szatkowski, W., *J. Appl. Chem. (London)* **13,** No. 2, 64 (1963).

(141H) Tajima, E., Fushima, A., *J. Japan Petrol. Inst.* **3,** 888 (1960); *Bull. Japan Petrol. Inst.* **4,** 62 (1962) (abstract).

(142H) Tajima, E., Fushima, A., *J. Japan Petrol. Inst.* **4,** 761 (1961); *Bull. Japan Petrol. Inst.* **4,** 63 (1962) (abstract).

(143H) Thelen, E., Division of Petroleum Chemistry, 147th Meeting, ACS, Philadelphia, Pa., 1964, Preprints **9,** No. 2, B5-14.

(144H) Tolson, R. G., Mid-Continent Regional Meeting, Natl. Petrol. Refiners Assoc., Wichita, June 1962, Tech. Paper **62-29.**

(145H) Traxler, R. N., 66th Annual Meeting, Am. Soc. Testing Materials, Atlantic City, N. J., June 1963, Preprint **79.**

(146H) Traxler, R. W., Division of Microbial Chemistry and Technology, 142nd Meeting, ACS, Atlantic City, N. J., 1962, Abstracts of Papers, p. 9P.

(147H) Traxler, R. N., *Proc. Assoc. Asphalt Paving Technol.* **32,** 44 (1963).

(148H) *Ibid.*, p. 229.

(149H) VandenBurgh, A. J. P., Bouwman, J. P., Steefelaar, G. M. A., *Schr. R. Rijkswaterst*, No. **3** (1962); *Bitumen, Teere, Asphalte, Peche* **14,** No. 1, 47 (1963) (abstract).

(150H) Varma, M. M., Sheffert, P. C., *Mater. Res. Std.* **3,** No. 7, 560 (1963).

(151H) Vokac, R., *Proc. Assoc. Asphalt Paving Technol.* **31,** 533 (1962).

(152H) Wales, M., VanderWaarden, M., Division of Petroleum Chemistry, 147th Meeting, ACS, Philadelphia, Pa., 1964, Preprints **9,** No. 2, B21.

(153H) Warden, W. B., Sandvig, L. D., 66th Annual Meeting, Am. Soc. Testing Materials, Atlantic City, N. J., June 1963, Preprint **79f.**

(154H) Warner, D. B., Moavenzadeh, F., 67th Annual Meeting, Am. Soc. Testing Materials, Chicago, Ill., 1964.

(155H) Welborn, J. Y., Halstead, W. J., *Public Roads* **31,** 243 (1962).

(156H) Welborn, J. Y., Halstead, W. J., Olsen, R. E., Am. Soc. Testing Materials, **STP 328,** 48 (1963); *Public Roads* **32,** 145 (1963).

(157H) Wetzel, H., Peter, S., *Z. Anal. Chem.* **190,** No. 1, 208 (1962).

(158H) Winniford, R. S., *J. Inst. Petrol.* **49,** No. 475, 215 (1963).

(159H) Winniford, R. S., Bersohn, M., Division of Fuel Chemistry, 142nd Meeting, ACS, Atlantic City, N. J., 1962, Abstracts of papers, p. 1K.

(160H) Winters, W. F., *Proc. Assoc. Asphalt Paving Technol.* **32,** 584 (1963).

(161H) Wirth, H., *Bitumen, Teere, Asphalte, Peche* **14,** No. 9, 448 (1963).

(162H) Wojdanowicz, S., *Drogownicto*, No. **9** (1959); *Bitumen, Teere, Asphalte, Peche* **14,** No. 6, 316, 318 (1963) (cond.).

(163H) Wright, J. R., Campbell, P. G., Division of Petroleum Chemistry, 147th Meeting, ACS, Philadelphia, Pa., 1964, Preprints **9,** No. 2, B99.

(164H) *Ibid.*, No. 3, 161.

(165H) Wright, J. R., Campbell, P. G., Fridinger, T. L., Am. Soc. Testing Materials, **STP 347,** 3 (1963); *Natl. Bur. Std. Tech. News Bull.* **47,** No. 7, 110–11 (1963); *J. Appl. Chem.* (*London*) **14,** No. 1, 30 (1964).

(166H) Wright, J. R., Minesinger, R. R., Division of Petroleum Chemistry, 142nd Meeting, ACS, Atlantic City, N. J., 1962, Preprints **7,** No. 3, 113; *J. Colloid Sci.* **18,** No. 3, 223, No. 8, 802 (1963).

(167H) Wronka, J. A., Am. Soc. Testing Materials, **STP 347,** 96 (1963).

(168H) Yanysheva, N. Ya., Kireeva, I. S., Serzhantova, N. N., *Gigiena i Sanit.* **28,** No. 11, 71 (1963).

(169H) Yatsenko, E. A., Gutsalyuk, V. G., Rafikov, S. R., *Izv. Akad. Nauk SSSR, Ser. Fiz.* **27,** No. 1, 107 (1963).

(170H) Yen, T. F., Erdman, J. G., Division of Petroleum Chemistry, 142nd Meeting, ACS, Atlantic City, N. J., 1962, Preprints **7,** No. 3, 99.

(171H) Yen, T. F., Erdman, J. G., Saraceno, A. J., Anal. Chem. **34,** 694 (1962).

(172H) Zakar, P., Mozes, G., *Bitumen, Teere, Asphalte, Peche* **13,** No. 6, 284 (1962).

(173H) Ziehmann, G., *Ibid.*, **10,** 377 (1959).

Hydrocarbons and Hydrocarbon Types

(1J) Aczel, T., Bartz, K. W., Lumpkin, H. E., Stehling, F. C., Anal. Chem. **34,** 1821 (1962).

(2J) Albert, D. K., *Ibid.*, **35,** 1918 (1963).

(3J) Alekseeva, T. A., *Tr. Vses. Nauch-Issled. Geologorazved. Neft. Inst.*, No. **33,** 271 (1962).

(4J) Amer. Soc. Testing Materials, "Compilation of Gas Chromatographic Data," **STP 343** (1963).

(5J) Am. Soc. Testing Materials, "Manual on Hydrocarbon Analysis," **STP 332** (1963).

(6J) Archer, E. D., Shively, J. H., Francis, S. A., Anal. Chem. **35,** 1369 (1963).

(7J) Areshidze, Kh. I., Khechinashvili, E. P., *Soobshch. Akad. Nauk Gruz. SSR* **28,** 401 (1962).

(8J) Ashley, J. W., Reilley, C. N., Hurwitz, P., Rogers, L. B., Anal. Chem. **34,** 1537 (1962).

(9J) Ashumov, G. G., Nasirov, A. B., Agamirova, S. I., Agaeva, R. M., *Azerb. Neft. Khoz.* **41,** 43 (1962).

(10J) Ashumov, G. G., Nasirov, A. B., Kurkovskaya, S. I., *Azerb. Khim. Zh.*, No. **6,** 13 (1959).

(11J) Averill, W., Ettre, L. S., *Nature* **196,** 1198 (1962).

(12J) Badger, G. M., Donnelly, J. K., Spotswood, T. M., *J. Chromatog.* **10,** 397 (1963).

(13J) Barrall, E. M., Porter, R. S. II, Johnson, J. F., *Ibid.*, **11,** 177 (1963).

(14J) Bartz, K. W., Aczel, T., Lumpkin, H. E., Stehling, F. C., Anal. Chem. **34,** 1814 (1962).

(15J) Bauer, R. H., *Ibid.*, **35,** 107 (1963).

(16J) Baumann, F., Gilbert, D. D., *Ibid.*, **35,** 1133 (1963).

(17J) Baumann, F., Klaver, R. F., Johnson, J. F., "Gas Chromatography 1962," M. van Swaay, ed., pp. 152–61, Butterworths, London, 1962.

(18J) Bednov, B. M., Sukhorukova, E. A., Novikov, V. N., *Zavodsk. Lab.* **29,** 806 (1963).

(19J) Bendel, E., Kern, M., Janssen, R., Steffan, G., *Angew. Chem.* **74,** 905 (1962).

(20J) Bendoraitis, J. G., Brown, B. L., Hepner, L. S., *Proc. Sixth World Petrol. Congr.*, Sect. V, 1963, p. 13.

(21J) Berezkin, V. G., Polak, L. S., *Tr. Komis. po Analit. Khim. Akad. Nauk SSSR, Inst. Geokhim. i Analit. Khim.* **13,** 205 (1963).

(22J) Bergmann, G., *Erdoel Kohle* **15,** 612 (1962).

(23J) Beroza, M., Sarmiento, R., Anal. Chem. **35,** 1353 (1963).

(24J) Bestougeff, M. A., *Journees Intern. Etude Methodes Separation Immediate Chromatog., Paris,* **1961,** 123.

(25J) Bigdergauz, M. S., Gol'bert, K. A., *Neftekhimiya* **1,** 706 (1961).

(26J) Blaustein, B. D., Zahn, C., Pantages, G., *J. Chromatog.* **12,** 104 (1963).

(27J) Bombaugh, K. J., *Nature* **197,** 1102 (1963).

(28J) Boyd, M. L., Montgomery, D. S., Can. Dept. Mines Tech. Surv., Mines Branch Res. Rept. **R88** (1961).

(29J) Boyer, E. W., Hamming, M. C., Ford, H. T., Anal. Chem. **35,** 1168 (1963).

(30J) Bresler, S. E., Dobychin, D. P., Popov, A. G., *Zh. Prikl. Khim.* **36,** 66 (1963).

(31J) Brown, H. C., Sivasankaran, K., Brown, C. A., *J. Org. Chem.* **28,** 214 (1963).

(32J) Bruderreck, H., Deut. Ges. Mineraloelwiss. u. Kohlechem., Annual Meeting, Oct. 10–13, 1962; *Brennstoff Chem.* **43,** 344 (1962) (abstract).

(33J) Burova, E. G., Khodzhaev, G. Kh., *Izvest. Akad. Nauk SSSR, Ser. Fiz.* **27,** 100 (1963).

(34J) Buzlanova, M. M., Kozhikohova, N. A., Polyanskiĭ, N. G., *Zh. Analit. Khim.* **18,** 1125 (1963).

(35J) Buzon, J., *Bull. Soc. Chim. France,* **1963,** 526.

(36J) Carlson, E. G., Andre, M. L., O'Neal, M. J., *Advan. Mass Spectrometry, Proc. 2nd. Conf. Oxford, 1961* **2,** 377 (1963).

(37J) Casu, B., Cavallotti, L., Anal. Chem. **34,** 1514 (1962).

(38J) Cornu, A., Copet, A., Ulrich, J., Bedague, P., *Proc. Sixth World Petrol. Congr.*, Sect. V, 1963, p. 191.

(39J) Csicsery, S. M., Pines, H., *J. Chromatog.* **9,** 34 (1962).

(40J) Cvetanovic, R. J., Duncan, F. J., Falconer, W. E., *Can. J. Chem.* **41,** 2095 (1963).

(41J) Dement'eva, M. I., Naumova, T. I., Prokopenko, N. A., *Neftekhimiya* **2,** 892 (1962).

(42J) Densham, A. B., Parkyns, N. D., *Chem. Ind.* (*London*) **1963,** No. 23, 948.

(43J) Dhont, J. H., *Nature* **198,** 990 (1963).

(44J) Dimitrov, Khr., Stoilova, Kr., Petsev, N., *Godishnik Sofiiskiya Univ., Fiz.-Mat. Fak. Khim.* **55,** 23 (1960–61).

(45J) Doering, C. E., Hauthal, H. G., *J. Prakt. Chem.* **19,** 17 (1962).

(46J) Dolejs, P., *Ropa Uhlie* **3,** 81 (1961).

(47J) Dorsey, J. A., Hunt, R. H., O'Neal, M. J., Anal. Chem. **35,** 511 (1963).

(48J) Driatskaya, Z. V., Zhmykhova, N. M., *Khim. i Tekhnol. Topliva i Masel* **7,** 9 (1962).

(49J) Dubois, L., Monkman, J. L., Division of Water and Waste Chemistry, 142nd Meeting, ACS, Atlantic City, N. J., 1962, Abstracts of Papers, p. 18V.

(50J) Eisen, O., Rang, S. A., *Goryuch. Slantsy, Khim. i Tekhnol. Akad. Nauk Est. SSR, Inst. Khim.*, No. **4,** 200 (1961).

(51J) Ettre, L. S., Cieplinski, E. W., Brenner, N., *ISA Trans.* **2,** 134 (1963).

(52J) Ettre, L. S., Kabot, F. J., Anal. Chem. **34,** 1431 (1962).

(53J) Finke, M., Heinze, G., *Chem. Tech.* (*Berlin*) **14,** 299 (1962).

(54J) Finkel, A. G., *Uch. Zap. Saratovsk. Gos. Univ.* **69,** 171 (1960).

(55J) Fleet, B., *Ind. Chemist* **39,** 601 (1963).

(56J) Flockhart, B. D., Pink, R. C., *Talanta* **9,** 931 (1962).

(57J) Folmer, F., Yang, K., Perkins, G., Jr., Anal. Chem. **35,** 455 (1963).

(58J) Francis, S. A., Archer, E. D., *Ibid.*, **35,** 1363 (1963).

(59J) Frederick, D. H., Miranda, B. T., Cooke, W. D., *Ibid.*, **34,** 1521 (1962).

(60J) Frehden, O., Lăzărescu, G., *Rev. Chim. Bucharest* **13,** 491 (1962).

(61J) Friedel, R. A., Retcofsky, H. L., 13th Annual Conference on Analytical Chemistry and Applied Spectroscopy, Pittsburgh, Pa., March 5–9, 1962.

(62J) Funakubo, E., Nagai, T., *Z. Physik. Chem.* **222,** 161 (1963).

(63J) Garilli, F., Lombardo, G., Zerbo, G., *Ann. Chim.* (*Rome*) **52,** 849 (1962).

(64J) Gaylor, V. F., Jones, C. N., Landerl,

J. H., Hughes, E. G., *Proc. Sixth World Petrol. Congr.*, Sect. VI, 1963, p. 201.
(65J) Glebovskaya, E. A., *Tr. Vses. Neft. Nauchn.-Issled. Geologorazved Inst., No.* **212**, 220 (1963).
(66J) Golay, M. J. E., Ettre, L. S., Noreur, S. D., "Gas Chromatography 1962," M. van Swaay, ed., pp. 139–51, Butterworths, London, 1962.
(67J) Gonikberg, M. G., Dorogochinskiĭ, A. Z., Gavrilova, A. E., Komanenkova, R. A., Mitrofanov, M. G., Kupriyanov, V. A., *Neftekhimiya* **3**, 916 (1963).
(68J) Goodspeed, F. E., Montgomery, D. S., Can. Dept. Mines Tech. Surv., Mines Branch Res. Rept. **R98** (1962).
(69J) Green, J. H. S., *Research (London)* **15**, 515 (1962).
(70J) Gronsberg, E. Sh., *Novoe v Oblasti Sanit.-Khim. Analiza (Raboty po Prom.-Sanit. Khim.)* **1962**, 124.
(71J) Halasz, I., Horvath, C., ANAL. CHEM. **34**, 499 (1963).
(72J) Halasz, I., Horvath, C., *Nature* **197**, 71 (1963).
(73J) Hara, A., Tokai, H., Fujii, A., *J. Japan Petrol. Inst.* **4**, 293 (1961).
(74J) Helberg, I. D., *Deut. Lebensm.-Rundschau* **58**, 321 (1962).
(75J) Henneberg, D., Schomburg, G., "Gas Chromatography 1962," M. van Swaay, ed., pp. 191–202, Butterworths, London, 1962.
(76J) Herling, J., Shabtai, J., Gil-Av, E., *J. Chromatog.* **8**, 349 (1962).
(77J) Heuschkel, G., Wolny, J., Skoczowski, S., *Erdoel Kohle* **13**, 98 (1960).
(78J) Hildebrand, G., Peper, C., Dahlke, B., *Chem. Tech. (Berlin)* **15**, 147 (1963).
(79J) Huber, J. F. K., Keulemans, A. I. M., "Gas Chromatography 1962," M. van Swaay, ed., pp. 26–35, Butterworths, London, 1962.
(80J) Huber, W., *Mikrochim. Acta* **1960**, 44.
(81J) Huguet, M., *Journees Intern. Etude Methodes Separation Immediate Chromatog., Paris*, **1961**, 69.
(82J) Hurrell, R. A., Perry, S. G., *Nature* **196**, 571 (1962).
(83J) Huyten, F. H., *Chem. Weekblad* **59**, 457 (1963).
(84J) Huyten, F. H., Rijnders, G. W. A., van Beersum, W., "Gas Chromatography 1962," M. van Swaay, ed., pp. 335–45, Butterworths, London, 1962.
(85J) Innes, W. B., Bambrick, W. E., Andreatch, A. J., ANAL. CHEM. **35**, 1198 (1963).
(86J) Iogansen, A. V., Broun, E. V., *Tr. Komis. po Analit. Khim., Akad. Nauk SSSR, Inst. Geokhim. i Analit. Khim.* **13**, 367 (1963).
(87J) Ishida, K., *Bunko Kenkyu* **9**, 27 (1960).
(88J) Ishida, K., *Ibid.*, **9**, 152 (1961).
(89J) Jentzsch, D., Hövermann, W., "Gas Chromatography 1962," M. van Swaay, ed., pp. 204–15, Butterworths, London, 1962.
(90J) Jokl, J., Knizek, J., *Acta Chim. Acad. Sci. Hung.* **33**, 17 (1962).
(91J) Joly, D., *Proc. Sixth World Petrol. Congr.*, Sect. V, 1963, p. 173.
(92J) Jungnickel, J. L., Forbes, J. W., ANAL. CHEM. **35**, 938 (1963).
(93J) Karpov, O. N., *Zhur. Anal. Khim.* **17**, 1029 (1962).
(94J) Karr, C., Jr., Childers, E. E., Warner, W. C., ANAL. CHEM. **35**, 1290 (1963).
(95J) Kawanishi, I., Funakubo, E., Matsumoto, B., *Coal Tar, Japan* **13**, 534 (1961).
(96J) Keulemans, A. I. M., Perry, S. G., "Gas Chromatography 1962," M. van Swaay, ed., pp. 356–67, Butterworths, London, 1962.
(97J) Khin, L., Szasz, G., *J. Chromatog.* **11**, 416 (1963).
(98J) Klaver, R. F., LeTourneau, R .L. 28th API Div. Refining Midyear Meeting, Philadelphia, Pa., May 13–16, 1963.
(99J) Kniebes, D. V., Inst. Gas Technol., Tech. Rept. **4** (1962).
(100J) Kontorovich, L. M., Iogansen, A. V., Levchenko, G. T., Semina, G. N., Bobrova, V. P., Stepanova, V. A., *Zavodsk. Lab.* **28**, 146 (1962).
(101J) Koons, P. D., Walker, J. Q., *Hydrocarbon Process. Petrol. Refiner* **42**, 133 (1963).
(102J) Krasnova, S. I., Mal'nev, A. F., Puchkivs'ka, G. O., Sklyar, V. T., *Isv. Akad. Nauk SSSR, Ser. Fiz.* **27**, 98 (1963).
(103J) Kucharczyk, N., Fohl, J., Vymetal, J., *J. Chromatog.* **11**, 55 (1963).
(104J) Lafaix, A., *Bull. Soc. Chim. France*, No. **8–9**, 1639 (1962).
(105J) Landault, C., Guiochon, G., *Ibid.*, No. **11**, 2433 (1963).
(106J) Leonard, R., Loewenguth, J. C., *Proc. Sixth World Petrol. Congr.*, Sect. V, 1963, Paper 1.
(107J) Levy, E. J., Miller, E. D., Beggs, W. S., ANAL. CHEM. **35**, 946 (1963).
(108J) Liberti, A., Cartoni, G. P., Bruner, F., *J. Chromatog.* **12**, 8 (1963).
(109J) Lijinsky, W., Domsky, I., Mason, G., Division of Analytical Chemistry, 144th Meeting, ACS, Los Angeles, Calif., 1963, Abstracts of Papers, p. 21B.
(110J) Lijinsky, W., Domsky, I., Mason, G., Ramahi, H. Y., Safavi, T., ANAL. CHEM. **35**, 952 (1963).
(111J) Lille, Yu., *Khim. i Tekhnol. Goryuch. Slantsev i Produktov ikh Pererabotki*, No. **12**, 134 (1963).
(112J) Lindeman, L. P., Beach, J. Y., LeTourneau, R. L., *Ninth Intern. Colloq. Spectros., Lyons 1961* **3**, 637 (1962).
(113J) Lindeman, L. P., LeTourneau, R. L., *Proc. Sixth World Petrol. Congr.*, Sect. V, 1963, p. 281.
(114J) LoChang, Ta-Chuang, Karr, C., Jr., *Anal. Chim. Acta* **26**, 410 (1962).
(115J) Lulova, N. I., Tarasov, A. I., *et al.*, *Khim. i Tekhnol. Topliva i Masel* **7**, 14 (1962).
(116J) McEwen, D. J., Division of Analytical Chemistry, 144th Meeting, ACS, Los Angeles, Calif., 1963, Abstracts of Papers, p. 19B.
(117J) Mair, B. J., Krouskop, N. C., Mayer, T. J., *J. Chem. Eng. Data* **7**, 420 (1962).
(118J) Malafeev, N. A., Yudina, I. P., Nevskaya, E. M., Zhavoronkov, N. M., *Khim. Prom.* **1962**, 320.
(119J) Malý, E., *J. Chromatog.* **7**, 422 (1962).
(120J) Martin, R. L., "Gas Chromatography," L. Fowler, ed., pp. 127–38, Academic Press, New York, 1963.
(121J) Matishev, V. A., Bogatova, L. S., *Tr. Mosk. Inst. Neftekhim. i Gaz. Prom.*, No. **44**, 275 (1963).
(122J) Mazitova, F. N., Virobyants, R. A., Ermakova, S. K., *Izv. Akad. Nauk SSSR, Otd. Khim. Nauk*, No. **9**, 1546 (1962).
(123J) Miquel, R., Benard, C., *Compt. Rend.* **256**, 940 (1963).
(124J) Mirskiĭ, Ya. V., Mitrofanov, M. G., Dorogochinskiĭ, A. Z., *Tr. Groznensk. Neft. Nauchn.-Issled. Inst.*, No. **12**, 84 (1963).
(125J) Miyake, H., Mitooka, M., *Bull. Japan Petrol. Inst.* **5**, 58 (1963).
(126J) Moniz, W. B., Dixon, J. A., *Proc. Sixth World Petrol. Congr.* Sect. V, 1963, Paper 18.
(127J) Moore, R. J., Gordon, R. J., Eiffert, R. C., *Proc. Fifth World Petrol. Congr.*, Sect. V, 1959, p. 13.
(128J) Mortimer, J. V., Gent, P. L., *Nature* **197**, 789 (1963).
(129J) Myers, L. J., ANAL. CHEM. **35**, 119 (1963).
(130J) Nagai, T., Funakubo, E., *Kogyo Kagaku Zasshi* **64**, 643 (1961).
(131J) *Ibid.*, p. 1432.
(132J) Nakamura, S., *Nippon Kagaku Zasshi* **83**, 1081 (1962).
(133J) Negoro, K., Yagi, S., Kuraku, H., *Kogyo Kagaku Zasshi* **64**, 1316 (1961).
(134J) Nuss, G. R., "Gas Chromatography," L. Fowler, ed., pp. 119–25, Academic Press, New York, 1963.
(135J) Ognyanov, I., *Compt. Rend. Acad. Bulgare Sci.* **16**, 161 (1963).
(136J) *Ibid.*, p. 265.
(137J) Okuda, T., *Chem. Pharm. Bull. (Tokyo)* **10**, 96 (1962).
(138J) Oshima, S., *Bunseki Kagaku* **11**, 854 (1962).
(139J) Oshima, S., Katsumata, A., *Ibid.*, **11**, 850 (1962).
(140J) Oshima, S., Katsumata, A., *J. Japan Petrol. Inst.* **4**, 358 (1961).
(141J) Perkins, G., Jr., Folmer, O. F., Jr., "Gas Chromatography," L. Fowler, ed., pp. 193–202, Academic Press, New York, 1963.
(142J) Perkins, G., Jr., Laramy, R. E., Lively, L. D., ANAL. CHEM. **35**, 360 (1963).
(143J) Perkins, G., Jr., Rouayheb, G. M., Lively, L. D., Hamilton, W. C., "Gas Chromatography," N. Brenner, J. E. Callen, M. D. Weiss, eds., pp. 269–85, Academic Press, New York, 1962.
(144J) Personov, R. I., *Zh. Analit. Khim.* **17**, 506 (1962).
(145J) Petitjean, D. L., Leftault, C. J., *J. Gas Chromatog.* **1**, 18 (1963).
(146J) Pishnamazzade, B. F., Ismailzade, I. G., Kosheleva, L. M., Eibatova, Sh. E., Mamedov, F. A., *Azerb. Khim. Zh.*, No. **6**, 27 (1961).
(147J) Pobiner, H., Wallace, T. J., Hofmann, J. E., ANAL. CHEM. **35**, 680 (1963).
(148J) Pop, A., Stănciulescu, E., *Stud. Cercet. Chim., Cluj* **12**, 159 (1961).
(149J) Prinzler, H., *Ropa Uhlie* **5**, 42 (1963).
(150J) Prinzler, H., *Z. Chem.* **2**, 136 (1962).
(151J) Prinzler, H., Doescher, L., *Wiss. Z. Tech. Hochsch. Chem. Leuna-Merseberg* **4**, 143 (1961–62).
(152J) Prohaska, B., Čubranić, A., Bastijanić, A., *Nafta, Zagreb* **13**, 49 (1962).
(153J) Przybylski, Z., *Chem. Anal. (Warsaw)* **8**, 779 (1963).
(154J) Raha, C. R., *J. Proc. Inst. Chemists (India)* **34**, 80 (1962).
(155J) Rajeswaran, P., Kirk, P. L., *Microchem. J.* **6**, 21 (1962).
(156J) Rang, S. A., *Goryuch. Slantsy, Khim. i Tekhnol., Akad. Nauk Est. SSR, Inst. Khim.*, No. **4**, 182 (1961).
(157J) Rashkes, Ya. V., *Zh. Anal. Khim.* **17**, 627 (1962).
(158J) Reynolds, G. F., Wild, J., *J. Polarog. Soc.* **8**, 62 (1962).
(159J) Roberts, C. B., Proceedings of Symposium on Titrimetric Methods, Ontario, Can., 1961, p. 21.
(160J) Roberts, C. B., Brejcha, H. A., ANAL. CHEM. **35**, 1104 (1963).
(161J) Rodionova, K. F., Il'inskaya, V. V., Nadel'shtein, I. V., *Geol. Nefti i Gaza* **6**, 52 (1962).
(162J) Rodionova, K. F., Korchagina, Yu. I., Il'inskaya, V. V., *Ibid.*, No. **1**, 33 (1963).
(163J) Rogers, L. B., Altenau, A. G., ANAL. CHEM. **35**, 915 (1963).
(164J) Rotzsche, H., "Gas Chromatography 1962," M. van Swaay, ed., pp. 111–17, Butterworths, London, 1962.
(165J) Rozenberg, L. M., Ushakova, I. B., Shchekin, V. V., Genekh, I. S., *Neftekhimiya* **3**, 472 (1963).
(166J) Ruiter, E. de, *Erdoel Kohle* **15**, 616 (1962).

(167J) Ryder, J. W., Sullivan, G. P., *J. Am. Oil Chemists' Soc.* **39,** 263 (1962).

(168J) Saier, E. L., Cousins, L. R., Basila, M. R., Anal. Chem. **35,** 2219 (1963).

(169J) Sawicki, E., Stanley, T. W., Elbert, W., *Microchem. J., Symp. Ser* **2,** 633 (1962).

(170J) Schay, G., Sze'kely, Gy., Traply, G., "Gas Chromatography 1962," M. van Swaay, ed., pp. 178–84, Butterworths, London, 1962.

(171J) Schenck, P. A., Eisma, E., *Nature* **199,** 170 (1963).

(172J) Schenk, G. H., Santiago, M., Wines, P., Anal. Chem. **35,** 167 (1963).

(173J) Schmiedel, R., Teubel, J., *Chem. Tech.* (*Berlin*) **15,** 142 (1963).

(174J) Schwartz, R. D., Brasseaux, D. J., Anal. Chem. **35,** 1374 (1963).

(175J) Schwartz, R. D., Brasseaux, D. J., Shoemake, G. R., *Ibid.*, **35,** 496 (1963).

(176J) Scoggins, M. W., Price, H. A., *Ibid.*, **35,** 48 (1963).

(177J) Scott, C. G., "Gas Chromatography 1962," M. van Swaay, ed., pp. 36–48, Butterworths, London, 1962.

(178J) Sergienko, S. R., Atameredova, K. R., Petrova, A. A., Chirova, E. V., *Izv. Akad. Nauk Turkm. SSR, Ser. Fiz.-Tekhn., Khim. i Geol. Nauk*, No. **5,** 26 (1962).

(179J) Shabalin, I. I., Kruglov, E. A., Vaisberg, K. M., *Khim. i Tekhnol. Topliva i Masel* **7,** 25 (1962).

(180J) Shabtai, J., Herling, J., Gil-Av, E., *J. Chromatog.* **11,** 32 (1963).

(181J) Sharkey, A. G., Schultz, J. L., Friedel, R. A., *Fuel* **41,** 359 (1962).

(182J) Shopov, D., Kotsev, N., Georgieva, K., *Izv. Inst. po Obshcha Neorg. Khim., Org. Khim. Bulgar. Akad. Nauk* **9,** 231 (1962).

(183J) Sidorov, R. I., *Tr. Vost. Sibirsk. Filiala, Akad. Nauk SSSR, Ser. Khim.*, No. **38,** 125 (1961).

(184J) Siryuk, A. G., Iogansen, A. V., *Tr. Komis, po Analit. Khim., Akad. Nauk SSSR, Inst. Geokhim. i Analit. Khim.* **13,** 393 (1963).

(185J) Siryuk, A. G., Zimina, K. I., *Khim. i Tekhnol. Topliva i Masel* **8,** 52 (1963).

(186J) Smeykal, K., Hauthal, H. G., Engler, W., *Chem. Tech.* (*Berlin*) **14,** 732 (1962).

(187J) Smith, B., Larson, G., Ryden, J., *Acta Chem. Scand.* **17,** 313 (1963).

(188J) Smith, B., Ohlson, R., *Ibid.*, **16,** 351 (1962).

(189J) Smith, B., Ohlson, R., Larson, G., *Ibid.*, **17,** 436 (1963).

(190J) Smith, B., Ohlson, R., Olson, A.-M., *Ibid.*, **16,** 1463 (1962).

(191J) Snyder, L. R., *J. Chromatog.* **8,** 178 (1962).

(192J) Snyder, L. R., Howard, H. E., Ferguson, W. C., Anal. Chem. **35,** 1676 (1963).

(193J) Sojak, L., Gregorik, M., Kurcova, J., *Ropa Uhlie* **5,** 289 (1963).

(194J) Solo, A. J., Pelletier, S. W., Anal. Chem. **35,** 1584 (1963).

(195J) Spencer, S. F., *Ibid.*, **35,** 592 (1963).

(196J) Srinivasan, M., *Chem. Age India* **13,** 39 (1962).

(197J) Suatoni, J. C., Anal. Chem. **35,** 2196 (1963).

(198J) Szynagel, P., Kecki, Z., *Nafta* (*Katowice*) **19,** 45 (1963).

(199J) Talsky, G., *Z. Anal. Chem.* **195,** 171 (1963).

(200J) Tulupov, P. E., *Zavodsk. Lab.* **28,** 1430 (1962).

(201J) Tur'yan, Ya. I., Romanov, V. F., *Ibid.*, **28,** 1066 (1962).

(202J) Uhlmann, L., Prinzler, H., *Chem. Tech.* (*Berlin*) **14,** 351 (1962).

(203J) Usami, S., *Japan Analyst* **10,** 769 (1961).

(204J) Vamos, E., Kovats, E., *Magy. Asvanyolaj Foldgaz Kiserl. Int. Kozlemen.* **3,** 52 (1962).

(205J) Varadi, P. F., Ettre, K., Anal. Chem. **34,** 1417 (1962).

(206J) Veselov, V. V., *Tr. Nauchn.-Issled. Inst. Sintetichn. Zhirozamenitelei i Moyushchikh Sredstv*, No. **1,** 81 (1960).

(207J) Vigdergauz, M. S., Afanas'ev, M. I., Gol'bert, K. A., *Usp. Khim.* **32,** 754 (1963).

(208J) Vigdergauz, M. S., Gol'bert, K. A., Savina, I. M., Afanas'ev M. I., Zimin, R. A., Bakhareva, N. I., *Zavodsk. Lab.* **28,** 149 (1962).

(209J) Villalobos, R., Turner, G. S., "Gas Chromatography," L. Fowler, ed., pp. 105–18, Academic Press, New York, 1963.

(210J) Vitzthum, O., *Z. Anal. Chem.* **189,** 66 (1962).

(211J) Waksmundzki, A., Suprynowicz, A., Pietrusinska, T., *Chem. Anal.* (*Warsaw*) **7,** 1043 (1962).

(212J) Walisch, W., Ashworth, M. R. F., *Mikrochim. Acta* **1959,** 497.

(213J) Walker, J. Q., *Oil Gas J.* **61,** 78 (1963).

(214J) Walker, J. Q., Ahlberg, D. L., Anal. Chem. **35,** 2028 (1963).

(215J) Wetzel, H., Peter, S., *Z. Anal. Chem.* **190,** 208 (1962).

(216J) Williams, R. B., Chamberlain, N. F., *Proc. Sixth World Petrol. Congr* Sect. V, 1963, p. 217.

(217J) Wingerter, K. H., *Acta Chem. Acad. Sci. Hung.* **37,** 177 (1963).

(218J) Yata, N., *Kogyo Kagaku Zasshi* **62,** 801 (1959).

(219J) Zaichenko, V. N., Lityaeva, Z. A., Markosov, P. I., *Azerb. Khim. Zh.*, No. **6,** 127 (1961).

(220J) Zhdanov, S. P., Kiselev, A. V., Yashin, Ya. I., *Neftekhimiya* **3,** 417 (1963).

(221J) Zizin, V. G., Sokolova, V. I., *Khim. i Tekhnol. Topliva i Masel* **7,** 27 (1962).

(222J) Znamenskaya, N. B., Korol, A. N., *Tr., Nauchn.-Issled. Inst. Avtomatiz. Proizv. Protsessov Khim. Prom. i Tsvetn. Met.* No. **10,** 70 (1962).

Sulfur

(1K) Adreen, B. H., Kniebes, D. V., *Proc. Operating Sect., Am. Gas Assoc.* **CEP-62-13** (1962).

(2K) Afonin, A. T., Drobiz, A. M., Klinov, B. M., Ivanov, P. G., Salakhov, Kh. N., Stepanenko, G. S., Khachaturov, Yu. G., Chernetsov, V. V., *Neftepererabotka i Neftekhim., Nauchn.-Tekhn. Sb.* **1963** (4), 39–42.

(3K) Am. Petrol. Inst., "Symposium on Chemistry and Properties of Petroleum-Type Sulfur and Nitrogen Compounds," *Proc. Am. Petrol. Inst.*, **42,** Sect. VIII 15–138 (1962).

(4K) Baumann, F., Olund, S. A., *J. Chromatog.* **9,** 431–8 (1962).

(5K) Beroza, M., Anal. Chem. **34,** 1801–11 (1962).

(6K) Bestougeff, M., *Proc. 5th World Petrol. Congr., N. Y., 1959* **5,** 143–64 (pub. 1960).

(7K) Boisselet, L., Baron, G., Delion, R., Favere, J., Parc, G., Poussin, A., *Ibid.*, **5,** 165–74 (1960).

(8K) Burckhardt, Ch., Gruetter, A., Stoessel, H., *Nature* **195,** 825–6 (1962).

(9K) Ciola, R., *Anais Assoc. Brasil. Quim.* **20,** 7–18 (1961).

(10K) Claytor, E. E., Jr., Jones, J. H., *Oil Gas J.* **61,** (21), 82, 85–6 (1963).

(11K) Croitoru, P. P., Freedman, R. W., Anal. Chem. **34,** 1536–7 (1962).

(12K) Csanyi, P. F., Horanyi, G., Levay, B., *Energia Atomtech.* **14** (11), 504–7 (1961).

(13K) Curtis, R. F., Phillips, G. T., *J. Chromatog.* **9,** 366–8 (1962).

(14K) Dahmen, E. A. M. F., Dijkstra, R., Verjaal, A. J., *Erdoel Kohle* **16,** (7), 768–75 (1963).

(15K) Dahn, E., Brunner, G., *Chem. Tech.* (*Berlin*), **16** (2), 112–13 (1964).

(16K) Eremenko, N. A., Pankina, R. G., *Geol. Nefti i Gaza* **6** (9), 43–8 (1962).

(17K) Eremenko, N. A., Pankina, R. G., *Neftegaz. Geol. i Geofiz., Nauchn.-Tekhn. Sb.* **1963** (5), 50–2.

(18K) Espagno, L., Poquet, B., *J. Chim. Phys.* **59,** 509 (1962).

(19K) Fabre, P., *Ann. Pharm. France* **20** (6), 563–4 (1962).

(20K) Franks, M. C., Gilpin, R. L., *J. Inst. Petrol.* **48,** 226–36 (1962).

(21K) Fredericks, E. M., Harlow, G. A., Anal. Chem. **36,** 263–6 (1964).

(22K) Gilpin, R. L., Franks, M. C., *Proc. Am. Petrol. Inst.* **43,** Sect. III, 258 (1963).

(23K) Glebovskaya, E. A., Bol'shakov, G. F., *Zavodsk. Lab.* **29** (2), 172–5 (1963).

(24K) Golynets, Yu. F., Ponomareva, L. I., *Tr. Komis. po Analit. Khim. Akad. Nauk SSSR, Inst. Geokhim. i Analit. Khim.* **13,** 137–8 (1963).

(25K) Gomoryova, A., *Ropa Uhlie* **4,** 177–8 (1962).

(26K) Gordash, Yu. T., Shevchik, A. M., Laryutina, E. A., Pavlyuchenko, K. V., *Dokl. Akad. Nauk Belorussk. SSR* **6,** 442–4 (1962).

(27K) Gulyaeva, L. I., Vinogradova, A. P., Khyanina, A. P., Karpovskaya, R. R., *Neftekhimiya* **3,** 296–302 (1963).

(28K) Hammerich, T., Gondermann, H., *Erdoel Kohle* **17** (1), 20–2 (1964).

(29K) Hanykova, M., *Paliva* **42,** (6), 174–5 (1962).

(30K) Institute of Petroleum, Sub-Panel on Sulphur by X-Ray Absorption, *J. Inst. Petrol.* **48,** 405–13 (1962).

(31K) International Conference of Benzole Producers, Anal. Chem. **36,** 339–42 (1964).

(32K) Jordan, J. H., *Hydrocarbon Process, Petrol. Refiner* **41** (11), 258–9 (1962).

(33K) Kato, M., Fujishima, I., Takeuchi, T., *Bunseki Kagaku* **11** (2), 178–82 (1962).

(34K) Khachaturov, Yu. G., *Radioizotopnye Metody Avtomat. Kontrolya, Akad. Nauk Kirg. SSR, Tr. Rasshiren, Soveshch., Vses. Seminara po Primeneniyu Radioaktivn. Izotopov v Izmeritel'n. Tekhn. i Priborostr.* **2,** 81–94 (1961) (pub. 1962).

(35K) Koga, Y., Kajikawa, M., Kawaguchi, M., *Sekiyu Gakkai Shi* **3,** 397 (1960).

(36K) Koga, Y., Kajikawa, M., Nonaka, K., *Ibid.*, **3,** 641 (1960).

(37K) Krol, B. B., Rozanova, Z. I., Rozhdestvenskaya, A. A., *Khim. i Tekhnol. Topliv i Masel* **8** (5), 26–31 (1963).

(38K) Levaggi, D. A., Feldstein, M., *J. Air Pollution Control. Assoc.* **13** (8), 380, 387 (1963).

(39K) Levay, B., Csanyi, P. F., Kralik, V. Z., *Magy. Kem. Folyoirat* **69** (3), 136–8 (1963).

(40K) Liddell, C., *J. Inst. Petrol.* **48,** 221–5 (1962).

(41K) Luk'yanitsa, V. G., Hungarian Chemists Association, Hungarian Academy of Science Petroleum Conference, Budapest, April 1962; *Chem. Tech.* (*Berlin*) **14,** No. 8, 502 (1962) Abstract.

(42K) Luk'yanitsa, V. G., Gal'pern, G. D., *Khim. Sera-i Azotorgan. Soedin. Soderzhashch. v Neft. i Nefteprod., Akad. Nauk SSSR, Bashkirsk. Filial* **3,** 121–9 (1960).

(43K) Malissa, H., Machherndl, L.,

Mikrochim. Ichnoanal. Acta **1962**, 1089–94.
(44K) Novikova, N. F., Basargin, N. N., *Tr. Komis. po Analit. Khim. Akad. Nauk SSSR, Inst. Geokhim. i Analit. Khim.* **13**, 27–32 (1963).
(45K) Okuno, I., Morris, J. C., Haines, W. E., Anal. Chem. **34**, 1427–31 (1962).
(46K) Ozaki, S., Yasuda, H., *Kogyo Kagaku Zasshi* **65**, 350–3 (1962).
(47K) Pell, E., Malissa, H., *Talanta* **9**, 1056–8 (1962).
(48K) Pentelow, Joyce, Benzole Producers, Ltd. (London), Res. Paper **4**, (1961).
(49K) Peter, I. S., *Chem. Tech. (Berlin)* **14**, 755–6 (1962).
(50K) Prezler, H., Oehler, R., *Chem. Tech. (Leipzig)* **15**, (1), 31–3 (1963).
(51K) Radmacher, W., Hoverath, A., *Brennstoff-Chem.* **41**, 304–8 (1960).
(52K) Reed, R. H., *Analyst* **88**, 735–6 (1963).
(53K) Rhodes, J. R., Florkowski, T., Cameron, J. F., At. Energy Research Establ. (Gt. Brit.), Rept. **R3925** (1962).
(54K) Rowley, T. B., *Radio-Electron. Engr.* **26** (4), 298–304 (1963).
(55K) Sahores, J., *Rev. Inst. Franc. Petrole et Ann. Combustibiles Liquides* **18** (11), 1632–47 (1963).
(56K) Scott, D. W., McCullough, J. P., *U. S. Bur. Mines Bull.* **595** (1962).
(57K) Sima, J., Taslar, Z., Juklova, V., *Sb. Praci Vyzkumu Chem. Byuziti Uhli, Dehtu Ropy* **1960** (1), 161–5.
(58K) Smith, J. W., Young, N. B., Lawlor, D. L., Anal. Chem. **36**, 618–22 (1964).
(59K) Snyder, L. R., Howard, H. E., Ferguson, W. C., *Ibid.*, **35**, 1676 (1963).
(60K) Sporek, K. F., Danyi, M. D., *Ibid.*, **35**, 956–8 (1963).
(61K) Stout, J. W., Jr., Early, E., *Proc. Operating Sect., Am. Gas Assoc.* **CEP-62-2** (1962).
(62K) Szepesvary, P., *Magy. Asvanyolaj Foldgaz Kiserl. Int. Kozlemen.* **2**, 50–8, (1961).
(63K) Takens, W., *Gas (The Hague)* **83** (12), 250–1 (1963).
(64K) Thompson, C. J., Coleman, H. J., Hopkins, R. L., Rall, H. T., *J. Chem. Eng. Data* **9**, 293–6 (1964).
(65K) *Ibid.*, U. S. Bur. Mines Rept. Invest. **6096** (1962).
(66K) Whitehead, A. L., Austin, R. R., ISA, *Proc., Symp. Instr. Methods Anal.* **6**, F3 (1960).
(67K) Zabrodina, K. S., *Izv. Akad. Nauk SSSR, Otd. Khim. Nauk* **1963** (5), 941–3.

Nitrogen

(1L) Abramyan, A. A., Kocharyan, A. A., Karapetyan, A. G., *Izv. Akad. Nauk Arm. SSR, Khim. Nauk* **15** (3), 225–30 (1962).
(2L) Am. Petrol. Inst., "Symposium on Chemistry and Properties of Petroleum-Type Sulfur and Nitrogen Compounds," *Proc. Am. Petrol. Inst.*, **42**, Sect. VIII, 15–138 (1962).
(3L) Ashraf, M., Shah, R. A., *Pakistan J. Sci. Res.* **15** (2), 47–57 (1963).
(4L) Ball, J. S., Rall, H. T., *Proc. Am. Petrol. Inst.*, **42**, Sect. III, 128–45 (1962).
(5L) Battles, W. R., Ellertson, W. P., Hoggan, G. D., Walker, H. B., Wattenbarger, C. W., *J. Inst. Petrol.* **49**, 340–4 (1963).
(6L) Beroza, Morton, Anal. Chem. **34**, 1801–11 (1962).
(7L) Beroza, Morton, Sarmiento, Rafael, *Ibid.*, **35**, 1353–7 (1963).
(8L) Bezinger, N. N., Abdurakhmanov, M. A., Gal'pern, G. D., *Neftekhimiya* **1**, 583–8 (1961).
(9L) *Ibid.*, pp. 589–98.
(10L) Block, J., Morgan, E., Siggia, S., Anal. Chem. **35**, 573–5 (1963).
(11L) Corwin, A. H., *Proc. 5th World Petrol. Congr. N. Y. 1959* **5**, 119–29 (1960).
(12L) Corwin, A. H., Baker, E. W., Division of Petroleum Chemistry, 147th Meeting, ACS, Philadelphia, Pa., 1964, Preprints 9, No. 1, 19.
(13L) Costantinides, G., Arich, G., Lomi, C., *Proc. 5th World Petrol. Congr., N. Y., 1959*, **5**, 131–42 (pub. 1960).
(14L) Dubois, C., Leonard, R., *Bull. Soc. Chim. France* **1963** (8–9), 1526.
(15L) Farley, L. L., Guffy, J. C., Winkler, R. A., Anal. Chem. **36**, 1061–3 (1964).
(16L) Ferrin, C. R., Latham, D. R., Haines, W. E., Division of Petroleum Chemistry, 139th Meeting, ACS, St. Louis, Mo., 1961, Preprints **6**, No. 2B, 23–6.
(17L) Frazer, J. W., *Mikrochim. Ichnoanal. Acta* **1962**, 993–9.
(18L) Hashmi, M. H., Ehsan, A., Umar, M., Anal. Chem. **34**, 988–90 (1962).
(19L) Hodgson, G. W., Ushijima, N., Taguchi, K., Shimada, I., *Sci. Rept. Tohoku Univ.* **8** (3), 483–513 (1963).
(20L) Hozumi, Kuichiro, Anal. Chem. **35**, 666–9 (1963).
(21L) Hozumi, K., Kirsten, W. J., *Ibid.*, **35**, 1522–7 (1963).
(22L) Jacobs, S., *9th Colloq. Spectros. Intern., Lyons, 1961* **3**, 247–62 (pub. 1962).
(23L) Jewell, D. M., Hartung, G. K., *J. Chem. Eng. Data* **9**, 297–304 (1964).
(24L) Kametani, F., Kubota, S., *Yakugaku Zasshi* **82**, 659–91 (1962).
(25L) Kirsten, W. J., Andren, R. G., *J. Chromatog.* **8**, 531 (1962).
(26L) Kirsten, W. J., Hozumi, K., Nirk, L., *Z. Anal. Chem.* **191**, 161–71 (1962).
(27L) Makovet'skii, P. S., *Dopovidi Akad. Nauk Ukr. RSR* **1963** (5), 642–4.
(28L) Mann, Lewis T., Jr., Anal. Chem. **35**, 2179–82 (1963).
(29L) Mizukami, S., Miyahara, K., *Mikrochim. Acta* **1962**, 705–16.
(30L) Morandi, J. R., Jensen, H. B., Division of Fuel Chemistry, 147th Meeting, ACS, Philadelphia, Pa., 1964, Preprints **8**, No. 1, 293.
(31L) Mossini, F., Vitali, T., *Ric. Sci., Rend. Sez.* **A1**, 244–6 (1961).
(32L) Munday, W. A., Eaves, A., *Proc. 5th World Petrol. Congr. N.Y., 1959*, **5**, 103–17 (pub. 1960).
(33L) Nightingale, C. F., Walker, J. M., Anal. Chem. **34**, 1435–7 (1962).
(34L) Parsons, M. L., Pennington, S. N., Walker, J. M., *Ibid.*, **35**, 842–4 (1963).
(35L) Rein, H. T., Miville, M. E., Fainberg, A. H., *Ibid.*, **35**, 1536 (1963).
(36L) Rezl, V., *Chem. Prumysl.* **12** (5), 246–7 (1962).
(37L) Roberts, L. R., McKetta, J. J., *J. Gas Chromatog.* **1**, 14–7 (1963).
(38L) Shah, R. A., Quadri, A. A., *Talanta* **10**, 1083–6 (1963).
(39L) Snyder, L. R., Buell, B. E., Anal. Chem. **36**, 767–73 (1964).
(40L) Stefanescu, A., *Petrol Gaze (Bucharest)* **13** (2), 53–6 (1962).
(41L) Sternglanz, P. D., Kollig, H., Anal. Chem. **34**, 544–7 (1962).
(42L) Terent'ev, A. P., Luskina, B. M., *Zh. Analit. Khim.* **17** (2), 227–30 (1962).
(43L) Terent'ev, A. P., Turkel'taub, A. M., Bondarevskaya, E. A., Domochkina, L. A., *Dokl. Akad. Nauk SSSR* **148** (6), 1316–9 (1963).
(44L) Thuerauf, W., Thiemann, W., *Brennstoff-Chem.* **44** (9), 275–80 (1963).
(45L) Vecera, M., *Mikrochim. Acta* **1962**, 896–912.
(46L) Walisch, Walter, *Trans. N. Y. Acad. Sci.* **25** (7), 693–704 (1963).

Oxygen

(1M) Aczel, T., Bartz, K. W., Lumpkin, H. E., Stehling, F. C., Anal. Chem. **34**, 1821–8 (1962).
(2M) Aliev, D. A., *Neftepererabotka i Neftekhim., Nauchn.-Tekhn. Sb.* **1963** (1), 25–7.
(3M) Anders, O. U., Briden, D. W., Anal. Chem. **36**, 287–92 (1964).
(4M) Baranova, N. M., Kalyazin, E. P., Makarov, V. I., *Zh. Analit. Khim.* **19** (3), 398–9 (1964).
(5M) Baumann, F., Anal. Chem. **34**, 1664–5 (1962).
(6M) Berezkin, V. G., Mysak, A. E., Polak, L. S., *Neftekhimiya* **4**, 156–9 (1964).
(7M) Beroza, Morton, Anal. Chem. **34**, 1801–11 (1962).
(8M) Beroza, M., Sarmiento, R., *Ibid.*, **35**, 1353–7 (1963).
(9M) Cerchez, V. Th., Vladeanu, Al., *Petrol Gaze (Bucharest)* **13**,(10), 453–8 (1962).
(10M) Crisler, R. O., Conlon, R. D., *J. Am. Oil Chemists' Soc.* **39** (11), 470–3 (1962).
(11M) Critchfield, F. E., Meeker, R. L., Bishop, E. T., Division of Analytical Chemistry, 142nd Meeting, ACS, Chicago, Ill., 1962, Abstracts of Papers, 3B.
(12M) Crump, G. B., *J. Chromatog.* **10**, 21–8 (1963).
(13M) Csicsery, S. M., Pines, H., *Ibid.*, **9**, 34–43 (1962).
(14M) Dirscherl, A., Erne, F., *Mikrochim. Acta* **1962**, 794–802.
(15M) Engel, M., Moebes, R., *Elektrie* **16**, 157–9 (1962).
(16M) Fester, J. I., Robinson, W. E., Anal. Chem. **36**, 1392–4 (1964).
(17M) Freedman, R. W., Croitoru, P. P., *Ibid.*, **36**, 1389–90 (1964).
(18M) Fritz, James S., Marple, L. W., *Ibid.*, **34**, 921–4 (1962).
(19M) Haraldson, Lars, *Mikrochim. Acta* **1962**, 650–70.
(20M) Harrison, R. B., *Analyst* **88**, 644–6 (1963).
(21M) Honorat, R., *Ind. Petrole* **30** (6), 40–1, 43 (1962).
(22M) Izmailov, N. A., Aleksandrova, A. M., *Uch. Zap. Khar'kovsk. Univ.*, No. **110**, *Tr. Khim. Fak. i Nauchn.-Issled. Inst. Khim., Khar'kov. Gos. Univ.*, No. **17**, 121–4 (1961).
(23M) Jarzynska, M., Mirecka, T., *Chem. Anal. (Warsaw)* **8** (4), 567–73 (1963).
(24M) Jordan, D. E., Veatch, F. C., Anal. Chem. **36**, 120–4 (1964).
(25M) Kamzolkin, V. V., Bashkirov, A. N., Lodzik, S. A., *Neftekhimiya* **1**, 276–9 (1961).
(26M) Klesment, I. R., *Khim. i Tekhnol. Goryuch. Slantsev i Produktov ikh Pererabotki* **1962** (11), 271–81.
(27M) Kolesnikova, L. P., Kamzolkin, V. V., Khotimskaya, M. I., *Neftekhimiya* **2**, 355–8 (1962).
(28M) Korchagina, V. I., Ginzburg, S. A., Fin'ko, A. A., Rutman, L. I., Davydov, I. V., Lavrinovich, D. A., *Neft. i Gaz. Prom., Nauchn.-Tekhn. Sb.* **1962** (2), 51–6.
(29M) Korchagina, V. I., Ginzburg, S. A., Fin'ko, A. A., Rutman, L. I., Davydov, I. V., Lavrinovich, D. A., *Novosti Neft. i Gaz. Tekhn., Neftepererabotka i Neftekhim.* **1962** (6), 39–43.
(30M) Kotel'nikov, B. P., Prokhorova, Z. A., Gerasimova, N. T., *Tr. Nauchn.-Issled. Inst. Sintetich. Zhirozamenitelei i Moyushchikh Sredstv* **1961** (2), 85–9.

(31M) Kyazimov, A. A., Khaldei, Z. V., Khar'kovskii, Yu. I., *Khim. i Tekhnol. Topliv i Masel* **8** (11), 61–4 (1963).

(32M) Mlinko, S., *Mikrochim. Acta* **1961,** 833–53.

(33M) Moore, R. J., Gordon, R. J., Eiffert, R. C., *Proc. 5th World Petrol. Congr., N. Y., 1959* **5,** 13–20 (pub. 1960).

(34M) Muroi, Kaname, *Bunseki Kagaku* **10** (8), 847–50 (1961).

(35M) Natelson, S., Stellate, R. L., ANAL. CHEM. **35,** 847–51 (1963).

(36M) Neuvirt, J., Ksandr, Z., *Collection Czech. Chem. Commun.* **29** (4), 1068–72 (1964).

(37M) Novak, K., *Chem. Prumysl* **12** (12), 658–60 (1962).

(38M) Onuska, F., *Chem. Zvesti* **17,** 359–64 (1963).

(39M) Palocz, I., Nadasy, M., *Magy. Tud. Akad., Kem. Tud. Oszt., Kozlemen.* **18,** (1), 1–5 (1962).

(40M) Pelcik, J., *Collection Czech. Chem. Commun.* **27** (6), 1503–7 (1962).

(41M) Penther, C. J., Notter, L. J., ANAL. CHEM. **36,** 283–6 (1964).

(42M) Perkins, G., Jr., Laramy, R. E., Lively, L. D., *Ibid.*, **35,** 360–2 (1963).

(43M) Petukhova, L. N., Fomenko, V. A., *Azerb. Khim. Zh.* **1961** (6), 121–5.

(44M) Ratkovics, F., Szepesvary, P., *Magy. Asvanyolaj Foldgaz Kiserl. Int. Kozlemen.* **1,** 19–34 (1960).

(45M) Robinson, J. W., *Anal. Chim. Acta* **27,** 377–80 (1962).

(46M) Salzer, F., *Mikrochim. Acta* **1962,** 835–67.

(47M) Sawicki, E., Noe, J., Stanley, T. W., *Ibid.*, **1960,** 286–90.

(48M) Scoggins, M. W., ANAL. CHEM. **36,** 1152–4 (1964).

(49M) Sobolev, E. P., Popova, E. A., Rubinshtein, I. A., *Khim. i Tekhnol. Topliv i Masel* **8** (2), 56–61 (1963).

(50M) Sokolov, V. A., Kolesnikova, L. P., *Neftekhimiya* **1,** 564–6 (1961).

(51M) Stallwood, R. A., Mott, W. E., Fanale, D. T., ANAL. CHEM. **35,** 6–10 (1963).

(52M) Stoll, P., Rembold, H., Weiss, M., *Schweiz. Arch. Angew. Wiss. Tech.* **29** (7), 225–34 (1963).

(53M) Swensen, R. F., Keyworth, D. A., ANAL. CHEM. **35,** 863–7 (1963).

(54M) Tarasov, O. A., *Zavodsk. Lab.* **29,** 733–4 (1963).

(55M) Terent'ev, V. A., Shaburov, M. A., Ivanova, A. N., *Neftekhimiya* **1,** 567–72 (1961).

(56M) Terent'ev, A. P., Turkel'taub, A. M., Bondarevskaya, E. A., Domochkina, L. A., *Dokl. Akad. Nauk SSSR* **148** (6), 1316–9 (1963).

(57M) Thompson, C. J., Coleman, H. J., Hopkins, R. L., Rall, H. T., U. S. Bur. Mines Rept. Invest. **6096** (1962).

(58M) Waclawik, Jerzy, Waszak, Stefan, *Chem. Anal. (Warsaw)* **8** (4), 633–42 (1963).

Other Elements

(1N) Agazzi, E. J., Burtner, D. C., Crittenden, D. J., Patterson, D. R., ANAL. CHEM. **35,** 332–5 (1961).

(2N) Aoki, F., Okada, M., *Rept. Tokyo Chem. Ind. Res. Inst.* **58,** 49–53 (1963).

(3N) Balint, T., *Acta Chim. Acad. Sci. Hung.* **39** (2), 161–82 (1963).

(4N) Ball, J. S., Rall, H. T., *Proc. Am. Petrol. Inst.* **42,** Sect. III, 128–45 (1962).

(5N) Barrall, E. M., II, Ballinger, P. R., *J. Gas Chromatog.* **1** (8), 7–13 (1963).

(6N) Bellomo, A., D'Amore, G., *Chem. Tech. (Berlin)* **14** (5–6), 341–3 (1962).

(7N) Blagova, T. A., Lipshtein, R. A., *Bor'ba s Korroziei Dvigatelei Vnutr. Sgoraniya i Gazoturbinnykh Ustanovok, Vses. Sov. Nauchn.-Tekhn. Obshchestv* **1962,** 289–94.

(8N) Boisselet, L., Baron, G., *et al.*, *Proc. 5th World Petrol. Congr., New York, 1959* **5,** 165–74 (pub. 1960).

(9N) Bonchev, P. R., *Mikrochim. Acta* **1962,** 577–83.

(10N) Bonelli, E. J., Hartmann, H., ANAL. CHEM. **35,** 1980–1 (1963).

(11N) Borzova, L. D., Torina, I. V., Astaf'eva, N. G., Gallyamov, V. M., Soboleva, L. A., *Energotekhnol Ispol'z. Topliva, Akad. Nauk SSSR, Energ. Inst.* **1962,** No. 2, 192–8.

(12N) Bruno, E., *Rass. Chim.* **14,** 137–40 (1962).

(13N) Buncak, P., *Chem. Anal. (Warsaw)* **7,** 269–82 (1962).

(14N) Cacace, F., Cipollini, R., Perez, G., Possagno, E., *Gazz. Chim. Ital.* **91,** 804–24 (1961).

(15N) Campbell, W. J., Thatcher, J. W., U. S. Bur. Mines Rept. Invest. **5966** (1962).

(16N) Campen, W. A. C., Dumoulin, H., *Chem. Weekblad* **55,** 632–3 (1959).

(17N) Campo, C., *Chim. Anal. (Paris)* **45,** 343–7 (1963).

(18N) Chang, Y., Huang, H., Chang, J., *Hua Hsueh Hsueh Pao* **28** (2), 75–9 (1962).

(19N) *Chem. Eng. News* **40** (40), 48–50 (1962).

(20N) Collins, A. G., U. S. Bur. Mines, Rept. Invest. **6047** (1962).

(21N) Collins, A. G., Pearson, C. A., *Producers Monthly* **27** (10), 18–21 (1963).

(22N) Dawson, H. J., Jr., ANAL. CHEM. **35,** 542–5 (1963).

(23N) De Clippelier, G., Souillard, G., van Rysselberge, J., *8th Intern. Colloq. Spectros., Lucerne, Switz.* **1959,** 130–40 (pub. 1960).

(24N) De Sousa, A., *Microchem. J., Symp. Ser.* **2,** 821–4 (1962).

(25N) Di Pietro, C., Kramer, R. E., Sassaman, W. A., ANAL. CHEM. **34,** 586–7 (1962).

(26N) Dwiggins, C. W., Jr., U. S. Bur. Mines Rept. Invest. **6039** (1962).

(27N) Eckardt, H., Keil, G., Rast, A., *Freiberger Forschungsh.* **A215,** 153–66 (1962).

(28N) Faerman, D. V., *Novosti Neft. i Gaz. Tekhn. Neftepererab. i Neftekhim.* **1961,** No. 4, 25–7.

(29N) Faerman, D. V., Chigrin, D. S., Tr., *Berdyanskii Opytn. Neftemaslozavod*, No. **1,** 31–5 (1963).

(30N) Farkas, M., Csanyi, F. P., *Magy. Kem. Folyoirat* **69** (9), 407–9 (1963).

(31N) Faulhaber, E., Vogt, W., Moll, H., *Monatsber. Deut. Akad. Wiss. Berlin* **3,** 579–83 (1961).

(32N) Gelius, R., Preussner, K. R., *Chem. Tech. (Berlin)* **15** (5), 290–4 (1963).

(33N) Gel'man, N. E., Shanina, T. M., *Zh. Analit. Khim.* **17** (8), 998–1004 (1962).

(34N) Gol'dshtein, A. L., Lapisova, N. P., Shtifman, L. M., *Ibid.*, **17,** No. 1, 143–4 (1962).

(35N) Gorusch, T. T., *Analyst* **86,** 112–15 (1962).

(36N) Gunn, E. L., *Advan. X-Ray Anal.* **6,** 403–16 (1962).

(37N) Hammerich, T., Gondermann, H., *Brennstoff-Chem.* **43** (11), 344–5 (1962).

(38N) Hara, A., Yamazaki, H., *Sekiyu Gakkai* **3** (4), 311–16 (1960).

(39N) Howard, J. M., Spauschus, H. O., ANAL. CHEM. **35,** **1016**–17 (1963).

(40N) Il'ina, E. V., *Zavodsk. Lab.* **29** (11), 1317–21 (1963).

(41N) Il'yasov, A. V., *Khim. i Tekhnol. Topliv i Masel* **7,** No. 9, 63–7 (1962).

(42N) Isbell, H. S., Tipson, R. S., *et al.*, Natl. Bur. Std. (U. S.), Monograph **54** (1962).

(43N) Jackwerth, E., Kloppenburg, H. G., *Z. Anal. Chem.* **186,** 428–36 (1962).

(44N) Janousek, I., *Collection Czech. Chem. Commun.* **27,** 2972–5 (1962).

(45N) Janousek, I., *Ropa Uhlie* **4,** 249 (1962).

(46N) Kainz, G., Mayer, J., *Mikrochim. Ichnoanal. Acta* **1963** (3), 542–51.

(47N) Kakabadse, G. J., Manohin, B., *Analyst* **88** (1051), 816–18 (1963).

(48N) Kashiwagi, H., Tsukamoto, Y., Kan, M., *Takeda Ken-Kyusho Nempo* **22,** 69–75 (1963).

(49N) Koyama, K., Taguchi, Y., Eguchi, S., *Bunseki Kagaku* **12,** 435–8 (1963).

(50N) Krotky, J., Kozakova, V., *Zpravodaj VZLU* **1960** (5), 39–42.

(51N) Liddell, C., *J. Inst. Petrol.* **48** (464), 221–25 (1962).

(52N) Loroue, D., Paul, G., *Bull. Assoc. Franc. Tech. Petrole* **1963,** 127–38.

(53N) Loroue, D., Paul, G., *Rev. Inst. Franc. Petrole Ann. Combust. Liquides* **17** (6), 830–41 (1962).

(54N) Lovelock, J. E., Gregory, N. L., *Gas Chromatog., Intern. Symp.*, **1961 3,** 219–29 (pub. 1962).

(55N) Maeck, W. J., Kussy, M. E., *et al.*, ANAL. CHEM. **35,** 62–5 (1963).

(56N) Makarov, P. T., In'kova, N. M., Klement'eva, N. F., *Khim. i Tekhnol. Topliv. i Masel* **8,** 65–66 (1963).

(57N) Makarov, P. T., Pershina, K. E., *Ibid.*, **8** (10), 62–4 (1963).

(58N) Makovets'kiĭ, P. S., Serdyuk, D. P., *Dopovidi Akad. Nauk Ukr. RSR* **1963** (2), 261–3.

(59N) Mattarella, S. P., O'Neil, R. L., *Gen. Motors Eng. J.* **9,** No. 4, 16–20 (1962).

(60N) Merz, W., *Mikrochim. Acta* **1959,** 640–6.

(61N) Nash, D. L., *Spectrochim. Acta* **18,** 868 (1962).

(62N) Newman, D. G., Tomlinson, C., *Mikrochim. Acta* **1962,** 599–610.

(63N) Nightingale, C. F., Walker, J. M., ANAL. CHEM. **34,** 1435–7 (1962).

(64N) *Oil Gas J.* **60,** No. 10, 98–9 (1962).

(65N) Parker, W. W., Hudson, R. L., ANAL. CHEM. **35,** 1334–5 (1963).

(66N) Pforr, G., Mikkeleit, M., Hauptmann, G., *Chem. Tech. (Berlin)* **14,** 754–5 (1962).

(67N) Piazzi, M., *Riv. Combust.* **16,** 379–83 (1962).

(68N) Püschel, R., Stefanac, Z., *Mikrochim. Acta* **1962** (6), 1108–14.

(69N) Radmacher, W., Hoverath, A., *Brennstoff-Chem.* **41,** 304–8 (1960).

(70N) Romanov, I. S., *Novosti Neft. i Gaz. Tekhn., Neftepererabotka i Neftekhim.* **1961,** No. 6, 39–40.

(71N) Rosenberg, H. M., Downer, T. M., Riber, C. R., Natl. Aeron. Space Admin., NASA Doc. **N62-12084** (1962).

(72N) Rowe, W. A., Yates, K. P., ANAL. CHEM. **35,** 368–70 (1963).

(73N) Ryabova, A. S., Belova, S. R., Sharapov, V. I., *Neftepererabotka i Neftekhim., Nauchn.-Tekhn. Sb.* **1963** (2), 11–12.

(74N) Slawson, W. F., Russel, R. D., *Mikrochim. Ichnoanal. Acta* **1963,** 165–8.

(75N) Stefanescu, P., *Rev. Chim.* **11,** 49–50 (1960).

(76N) Tagliavini, G., Belluco, U., Riccoboni, L., *Ric. Sci. Rend. 1A* (2), 338–45 (1961).

(77N) Vecera, M., *Collection Czech. Chem. Commun.* **26,** 2298–307 (1961).

(78N) *Ibid.*, pp. 2308–15.

(79N) Vecera, M., *Mikrochim. Acta* **1962,** 891–5.

(80N) Yao, T. C., Harrison, R. E., *Appl. Spectry.* **17** (5), 138 (1963).

(81N) Zul'fugarly, D. I., Umakhanova, N. S., *Azerb. Khim. Zh.* **1959,** No. 6, 99–104.

(82N) Zul'fugarly, D. I., Umakhanova, N. S., *Tr. Geol. Inst., Akad. Nauk SSSR Dagestansk. Filial,* **1960**, **2**, 128–36 (pub. 1961).

Catalysts

(1P) Atkins, J. H., Division of Analytical Chemistry, 145th Meeting, ACS, New York, N. Y., 1963, Abstracts of Papers 23B.
(2P) Barker, J. A., Everett, D. H., *Trans. Faraday Soc.* **58**, 1608–23 (1962).
(3P) Barras, R. C., Helwig, J. D., *Proc. Am. Petrol. Inst.* **43**, 223 (1963).
(4P) Battalova, Sh., Likerova, A. A., Sharipov, M. Sh., *Izv. Akad. Nauk Kaz. SSR, Ser. Tekhn. i Khim. Nauk* **1963** (1), 14–20.
(5P) Clement, C., *Rev. Inst. Franc. Petrole Ann. Combust. Liquides* **18**, 420–37 (1963).
(6P) Ettre, L. S., Brenner, N., Cieplinski, E. W., *Z. Physik. Chem.* (*Leipzig*) **219** (1–2), 17–35 (1962).
(7P) Garcia, J. F., Hermana, E., *Anales Real Soc. Espan. Fis. Quim.* **59** (1), 13–22 (1963).
(8P) Grane, H. R., Connor, J. E., Masologites, *Proc. Am Petrol. Inst.* **41**, 241–6 (1961).
(9P) Holm, V. C. F., Clark, A., *Ind. Eng. Chem., Prod. Res. Develop.* **2**, 38–9 (1963).
(10P) Karnaukhov, A. P., *Kinetika i Kataliz* **3**, 583–98 (1962).
(11P) Kazanskiĭ, V. B., Pecherskaya, Yu. I., *Ibid.*, **4**, 244–51 (1963).
(12P) Klevleev, M. A., Orochko, D. I., Skoblo, A. I., Levinson, S. Z., *Tr. Vses. Nauchn.-Issled. Inst. po Pererabotke Nefti i Gaza i Polucheniyu Iskusstv Zhidkogo Topliva* **8**, 115–26 (1959).
(13P) Levy, E. J., Miller, E. D., Beggs, W. S., ANAL CHEM. **35**, 946–9 (1963).
(14P) Mott, W. E., Rhodes, D. F., *Appl. Spectry.* **17**, 144 (1963).
(15P) Mottlau, A. Y., Fisher, N. E., ANAL. CHEM. **34**, 714–15 (1962).
(16P) Mourgues, L. de, *Chim. Anal.* **45**, 103–10 (1963).
(17P) Nicolescu, I. V., *Rev. Chim., Acad. Rep. Populaire Roumaine* **7** (1), 359–67 (1962).
(18P) Osipov, L. N., Semenova, E. S., Rogov, S. P., *Neftepereabotka i Neftekhim., Nauchn.-Tekhn. Sb.* **3**, 5–7 (1963).
(19P) Paul, S. D., Krishnan, M. S., *J. Sci. Ind. Res.* (*India*) **20D**, 333–35 (1961).
(20P) Poddubnyi, I. Ya., Kolechkova, M. F., *Fiz.-Khim. Metody Analiza i Issled. Produktov Proizv. Sintetich. Kauchuka, Vses. Nauchn.-Issled. Inst. Sintetich. Kauchuka,* **1961**, 113–17.
(21P) Romankova, I. K., Remizov, V. G., Maidebor, L. K., Golovenko, A. M., *Tr. Grosnensk. Neft. Nauchn.-Issled. Inst.* **12**, 94–105 (1963).
(22P) Rubinshtein, A. M., Klyachko-Gurvich, A. L., *Kinetika i Kataliz* **3**, 599–601 (1962).
(23P) Sergienko, S. R., Taimova, B. A., Garbalinskii, V. A., *Neftekhim., Akad. Nauk Turkm. SSR,* **1963**, 105–12.
(24P) Stone, F. S., *Chem. Ind.* (*London*), **1963**, 1027.
(25P) Tan, F-C., Tan, S-T., Li, V-C., *Wu Li Hsueh Pao* **15** (6), 331–6 (1959).
(26P) Townsend, J. E., *Appl. Spectry.* **17**, 37–9 (1963).
(27P) Vasserberg, V. E., *Kinetika i Kataliz* **3**, 556–64 (1962).
(28P) Vergnaud, J. M., *Bull. Soc. Chim. France* **1963** (10), 2163–5.
(29P) Weidenbach, G., Fuerst, H., *Chem. Tech.* (*Berlin*) **15**, 589–91 (1963).
(30P) Zul'fugarov, Z. G., Aliev, A. S., *et al.*, *Kinetika i Kataliz* **3**, 565–71 (1962).

Pollution

(1Q) Air Pollution Control Assoc., *J. Air Pollution Control Assoc.* **14**, 30–3 (January 1964)
(2Q) Aleskovskiĭ, V. B., Kovaltsov, V. A., Fedorov, I. N., Tsyplyatnikov, G. P., *Zavodsk. Lab.* **28** (12), 1440–2 (1962).
(3Q) Altshuller, A. P., Bellar, T. A., Clemons, C. A., *J. Am. Ind. Hyg. Assoc.* **23**, 164–6 (1962).
(4Q) Am. Petroleum Inst., Div. of Refining, "Manual on Disposal of Refinery Wastes," Vol. VI, "Solid Wastes," 1st ed., 1963.
(5Q) Apte, V. R., Dabadghao, S. B., Deshpande, W. M., *Environ. Health, India* **4**, 202–6 (1962).
(6Q) Babkin, M. P., Voloskovets, A. L., *Nefteperabotka i Neftekhim. Nauchn.-Tekhns. Sb.* **1963** (3), 7–8.
(7Q) Bahensky, W., Zika, Zd., Lastovkova, O., *Korose Ochrana Mater.* **1962**, 96–7.
(8Q) Beuthe, C. G., *Gesundh. Ing.* **83**, 70–2 (1962).
(9Q) Bouquiaux, M. J., *Trib. Cebedeau* **16**, 124–6 (1963).
(10Q) Bouquiaux, M. J., Mertens, A., *Centre Belge Etude Doc. Eaux, Bull. Mens.*, No. **110**, 42–8 (1960).
(11Q) Brousaides, F. J., Hommel, C. O., *J. Air Pollution Control Assoc.* **12**, 129–37, 146 (1960).
(12Q) Busch, A. W., Grady, L., Jr., Rao, I. S., Swilley, E. L., *J. Water Pollution Control Fed.* **34**, 354–62 (1962).
(13Q) Busch, A. W., Hiser, L. L., Grady, L., Jr., 52nd Natl. Mtg. Am. Inst. Chem. Engrs., Abstract **18***a* (1964).
(14Q) Chenouard, J., Dirian, G., Gabilly, R., *Chim. Anal.* **44** (8), 334–9 (1962).
(15Q) Dobremyslova, I., Dvorak, J., Novobilsky, V., *Chem. Prumysl* **12** (6), 287–9 (1962).
(16Q) Dyatlovitskaya, F. G., Potemkina, S. K., *Gigiena i Sanit.* **28**, No. 1, 38–44 (1963).
(17Q) Edeline, F., *Trib. Cebedeau* **16**, 127–9 (1963).
(18Q) Elkins, H. B., Pagnotto, L. D., Comproni, E. M., ANAL. CHEM. **34**, 1797–801 (1962).
(19Q) Goksoeyr, H., Ross, K., *J. Inst. Fuel* **35**, 177–9 (1962).
(20Q) Golubeva, M. I., *Lab. Delo* **10** (1), 6–9 (1964).
(21Q) Goodman, B. L., *Water Sewage Works* **110** (1), 27–8 (1963).
(22Q) Gordon, G. E., ANAL. CHEM. **32**, 1325–6 (1960).
(23Q) Gorlas, J., *Vortr. Symp. Gas-Chromatog.*, **4** (Leuna, Germany), 71–84 (1963).
(24Q) Greenberg, A. E., Lennette, E. P., *Water Sewage Works* **110**, 166–7 (1963).
(25Q) Grune, W. N., Chueh, C. F., Gutermuth, J. M., Division of Water and Waste Chemistry, 142nd Meeting, ACS, Atlantic City, N. J., 1962, Preprints **59–60**.
(26Q) Hanykova, M., *Erdoel Kohle* **16**, 1212 (December 1963).
(27Q) Hellwig, D. H. R., van Steenderen, R. A., *Water Waste Treat. J.* **9**, No. 3, 118–20 (1963).
(28Q) Herrmann, G., *Chem. Tech.* (*Berlin*) **15** (6), 342–9 (1963).
(29Q) Hirsch, A. A., *Water Sewage Works* **110** (4), 147 (1963).
(30Q) Hissink, M., *J. Inst. Fuel* **36** (272), 372–6 (1963).
(31Q) Hrivnak, J., Schiessl, O., *Vodni Hospodarstvi* **14**, 39–40 (1964).
(32Q) Hummel, H., Ebert, J., *Gas-Wasserfach.* **104** (35), 1019–20 (1963).
(33Q) Huygen, C., *Anal. Chim. Acta* **28** (4), 349–60 (1963).
(34Q) Kanibalodskaya, R. G., Sidorskaya, L. F., Shevlyakov, V. A., *Novosti Neft. i Gaz. Tekhn., Neftepererabotka i Neftekhim.*, No. **2**, 20–1 (1961).
(35Q) Kaplin, V. I., Fesenko, N. G., *Zavodsk. Lab.* **28** (3), 287–8 (1962).
(36Q) Kobayashi, Y., *Bunseki Kogaku* **9** (3), 229–32 (1960).
(37Q) Köppen, *Gesundh.* **78**, No. 13–14 (1957).
(38Q) Kozlayeva, I. M., Vorokhobin, I. G., *Sb. Nauchn. Rabot Inst. Okhrany Truda Vses. T'sentr. Soveta Prof. Sayuzov* **1962** (3), 92–7.
(39Q) Kreiger, H., *Gas-Wasserfach,* **104**, 695–8 (1963).
(40Q) Ladendorf, P., Ges. Deut. Chemiker-Wasserchem. Fachgruppe Meeting, Fulda, May 29–30, 1962.
(41Q) Lenskaya, V. N., Terekhova, R. K., *Izv. Vysshikh Uchebn. Zavedenii, Khim. i Khim. Tekhnol.* **5**, 717–21 (1962).
(42Q) Lodge, J. P., Jr., Parbhakar, K. J., *Anal. Chim. Acta* **29** (4), 372–4 (1963).
(43Q) Lure, Yu. Yu., Panova, V. A., *Zavodsk. Lab.* **29**, No. 3, 293–5 (1963).
(44Q) Malz, F., Ges. Deut. Chem. Fachgruppe Wasserchem. Meeting, Berlin, May 24–25, 1963.
(45Q) Mancy, K. H., Westgarth, W. C., Okun, D. A., Purdue Univ. Eng. Bull., Ext. Ser. No. **112**, 508–23 (1963).
(46Q) Michelsen, E., Märki, E., *Mitt. Lebensmitt. Hyg., Bern* **52** (6), 557–71 (1961).
(47Q) Mitkalev, B. A., Avferenok, S. N., Kozorezov, E. S., *Novosti Neft. i Gaz. Tekhn., Neftepererabotka i Neftkhim.*, No. 12, 20–2 (1961).
(48Q) Morgan, P. F., Bewtra, J. K., *J. Water Pollution Control Fed.* **34**, 363–75 (1962).
(49Q) Pate, J. B., Lodge, J. P., Jr., Neary, M. P., *Anal. Chim. Acta* **28** (4), 341–8 (1963).
(50Q) Poplavskaya, A. V., Vorobeva, E. I., *Novsti Neft. i Gaz. Tekhn., Neftepererabotka i Neftekhim.*, No. **5**, 15–17 (1961).
(51Q) Rostenbach, R. E., Kling, R. E., *J. Air Pollution Control Assoc.* **12**, 459–63 (1962).
(52Q) Roumy, R., *Centre Etudes Nucl. Saclay, Ser. Bibl.*, No. **36** (1963).
(53Q) Sand, Th., *Zentr. Arbeitsmed. Arbeitsschutz* **13**, 157–62 (1963).
(54Q) Schwabe, K., Ger. (East) Patent **22,154** (CL 42L) (Oct. 12, 1961; Appl., Dec. 3, 1958).
(55Q) Smit, G. B., Ehrenburg, J. P., *Mitt. Ver. Grosskesselbesitzer* **78**, 208–14 (1962).
(56Q) Okun, D. A., *et al.*, *J. Water Pollution Control Fed.* **34**, 419–58 (1962).
(57Q) *Ibid.*, **35**, No. 5, 535–86 (1963).
(58Q) Wayman, C. H., ANAL. CHEM. **35**, 768–9 (1963).
(59Q) Whiston, T. G., Cherry, G. W., *Analyst* **87**, 819–23 (1962).
(60Q) Zurlo, N., *Med. Lavoro* (*Milan*) **53**, 325–9 (1962).

Process Analytical Instruments

(1R) Aronovich, V. V., Galitskiĭ, A. Ya., Zavertkin, K. V., *Khim. Prom.* (1962), 30.
(2R) Berridge, S. A., Pearce, C. J., White, E. N., Proc. Sixth World Petroleum Congress, Sec. V, Paper 8, 1963.
(3R) Briot, A., *Chim. Ind.* (*Paris*) **88**, 619 (1962).
(4R) Brister, T. B., *ISA Proc. Symp. Instr. Methods Anal.* **6**, C2 (1960).
(5R) Burnett, R. L., Klaver, R. F., ANAL. CHEM. **35**, 1709 (1963).
(6R) *Can. Chem. Process* **46**, 75 (1962).
(7R) *Chem. Eng. News* **41**, No. 46, 56 (1963).
(8R) Clark, R. D., *Oil Gas J.* **61**, No. 29, 56 (1963).

(9R) Coggeshall, N. D., Nelson, F. A., Doolen, O. K., Baker, G. A., *Proc. Am. Petrol. Inst.* **43**, Sec. III (Refining), 229 (1963).
(10R) Compain, M., *Chem. Tech.* (*Berlin*) **14**, 372 (May-June 1962).
(11R) Crespin, G. T. F., *Inst. Petrol. Rev.* **17**, 224 (1963).
(12R) Dahn, E., Brunner, G., *Chem. Tech.* (*Berlin*) 16 (2), 112 (1964).
(13R) Donaldson, R. E., Murphy, J. R., McBride, W. R., Story, D. O., *Proc. Am. Petrol. Inst.* **43**, Sec. III (Refining), 238 (1963).
(14R) Forney, W. E., Gulbrandsen, A. F., Borup, R. E., *Ibid.*, **43**, Sec. III (Refining), 267 (1963).
(15R) Fraade, D. J., Natl. Petrol. Refiners Assoc. Rocky Mt. Reg. Mtg., Salt Lake City, Paper Tech. **62-36** (September 1962).
(16R) Fraade, D. J., *Petrol. Refiner* **41**, 144 (1962).
(17R) Fritz, I. T., Davis, R. C., *Oil Gas J.* **60**, No. 21, 139 (1962).
(18R) Gerzhevan-Lati, V. N., *Khim. i Tekhnol. Topliv i Masel* **7**, No. 11, 57 (1962).
(19R) Haselhorst, M., *Erdoel Kohle* **16**, No. 3, 207 (1963).
(20R) Jasek, A. W., *Oil Gas J.* **60**, No. 50, 101 (1962).
(21R) Keeler, R. M., Burk, M. C., *Proc. Am. Petrol. Inst.* **43**, Sec. III (Refining) 248 (1963).
(22R) Maciw, B. O., *ISA Proc. Symp. Instr. Methods Anal.* **6**, C3 (1960).
(23R) Marten, J. S., *Inst. Petrol. Rev.* **17**, No. 198, 223 (1963).
(24R) Nelson, F. A., *ISA Proc. Ann. Instr. Autom. Conf. Exhibit* **18**, Pt. 1 (1963).
(25R) Nootbaar, R. F., *Proc. Am. Petrol. Inst.* **43**, Sec. III (Refining), 216 (1963).
(26R) Oleinikov, V. A., Zotov, N. S., *Gos. Nauchn.-Tekhn. Izd. Neft. i Gorno-Toplivn. Prom., Leningr. Otd.* 323 pp., 1962.
(27R) Orlov, M. L., Krikunova, L. I., Starkova, L. S., *Sb. Nauchn. Tr. Ukr. Nauchn.-Issled. Uglekhim. Inst.*, No. 12, 172 (1961).
(28R) *Petrol. Refiner* **42**, 260 (1963).
(29R) Pokrovskiĭ, B. A., Sabitov, Kh. K., *Novosti Neft. i Gaz. Tekhn. Neftepererabotka i Neftekhim.*, No. **9**, 25 (1961).
(30R) Righi, H., *Dechema Monograph* **35**, No. 528–555, 114 (1959).
(31R) Serne, R. W., Sinek, R. L., *ISA Proc. Ann. Instr.-Autom. Conf. Exhibit* **18**, Pt. 1 (1963).
(32R) Sukhomlinov, V. S., *Neft. i Gaz. Prom., Nauchn.-Tekhn. Sb.* **1963** (1), 58–60.
(33R) Tyler, C. M., *Chem. Eng. Progr.* **58**, No. 9, 51 (1962).
(34R) Trippeer, W. M., Riggle, J. W., *Ibid.*, **58**, 56 (1962).
(35R) Villalobos, R., Turner, G. S., *ISA Proc. Ann. Instr. Autom. Conf. Exhibit* **17** (1) (1962).
(36R) Votlokhin, B. Z., *Tr. Groznensk. Neft. Nauchn.-Issled. Inst.*, No. **11**, 253 (1961).
(37R) Wallis, S. W. J., *J. Inst. Petrol.* **49** (479), 329 (1963).
(38R) Zhukhovitskiĭ, A. A., Turkel'taub, N. M., *Neftekhimiya* **2**, 818 (1962).

Laboratory Apparatus and Instruments

(1S) Akramkhodzhaev, A. M., Amirkhanov, Sh. Kh., Alavutdinov, D., *Uzbeksk. Geol. Zh.* **7** (3), 70 (1963).
(2S) Barber, M., Cuthbert, J., *et al.*, *Proc. Roy. Soc.* (*London*) **A274**, No. 1358, 285 (1963).
(3S) Beckey, H. D., Wagner, G., *Angew. Chem.* **75**, No. 18, 862 (1963).
(4S) Bird, R. J., *Proc. Sixth World Petroleum Congress*, Sec. V, p. 79, 1963.
(5S) Blumer, M., ANAL. CHEM. **34**, 704 (1962).
(6S) British Standards Institution, BS 658 (1962).
(7S) Bruderreck, H., *Erdoel Kohle* **16**, 847 (1963).
(8S) Buell, B. E., ANAL. CHEM. **34**, 635 (1962).
(9S) Buzon, J., *Bull. Soc. Chim. France* No. 3, 526, **1963**.
(10S) Caines, A. J., *Inst. Petrol. Rev.* **17**, No. 195, 88 (1963).
(11S) Carter, J. W., Golbayegani, K. H., *Brit. Chem. Eng.* **8**, 571 (1963).
(12S) Chamberlain, N. F., Neureiter, N. P., Saunders, R. K., Williams, R. B., *Proc. 5th World Petrol. Congr., New York 1959* **5**, 93 (1960).
(13S) *Chem. Eng. News* **41**, No. 31, 128 (1963).
(14S) *Ibid.*, No. 35, 129.
(15S) Dukhovnyi, G. E., *Khim. i Tekhnol. Topliv i Masel* **8**, 62 (1963).
(16S) Gilmore, J. T., Hull, D. E., Fries, B. A., Proc. Sixth World Petroleum Congress, Sec. V, Paper 19, 103, 1963.
(17S) Gray, P. R., Cartwright, L. M., *Appl. Spectry.* **17**, No. 5, 143 (1963).
(18S) Guinn, V. P., Johnson, R. A., Mull, G. C., *Proc. Sixth World Petroleum Congress*, Sec. V, Paper 20, p. 91, 1963.
(19S) Hurd, B. G., ANAL. CHEM. **35**, 1468 (1963).
(20S) Innes, W. B., Bambrick, W. E., Division of Analytical Chemistry, *145th Meeting, ACS, New York, N. Y.*, September 1963.
(21S) Jenkins, R., *J. Inst. Petrol.* **48**, 246 (1962).
(22S) Johnson, H. W., Jr., ANAL. CHEM. **35**, 521 (1963).
(23S) Johnson, R. A., *Appl. Spectry.* **17**, No. 5, 143 (1963).
(24S) Jones, J. T., Ludt, W. C., Kellogg, H. W., *Oil Gas J.* **61**, No. 5, 112 (1963).
(25S) Jungnickel, J. L., Forbes, J. W., ANAL. CHEM. **35**, 938 (1963).
(26S) Khaet, M. Z., Belova, L. A., Bobrov, V. N., *Novosti Neft. i Gaz. Tekhn., Neftepererabotka i Neftekhim.*, No. **11**, 35 (1961).
(27S) Klaver, R. F., LeTourneau, R. L., *Proc. Am. Petrol. Inst.* **43**, Sec. III (Refining), 254 (1963).
(28S) Klesper, E., Corwin, A. H., Turner, D. A., *J. Org. Chem.* **27**, 700 (1962).
(29S) Kuehner, E. O., Leslie, R. T., ANAL. CHEM. **34**, 1155 (1962).
(30S) Lafay, *Ind. Petrole* **30**, No. 10, 95 (1962).
(31S) Merritt, C., Jr., Walsh, J. T., ANAL. CHEM. **34**, 908 (1962).
(32S) Pattillo, A. D., Young, H. A., *Ibid.*, **35**, 1768 (1963).
(33S) Petrocelli, J. A., *Ibid.*, **35**, 2220 (1963).
(34S) *Petroleum* (*London*) **26**, No. 3, 123 (1963).
(35S) Pichler, H., Schulz, H., Firnhaber, B., Rao, B. R., *Proc. Sixth World Petroleum Congress*, Sec. V, p. 1, 1963.
(36S) Pomykala, Z., *J. Inst. Petrol.* **49**, No. 479, 230A (1963).
(37S) Prohaska, B., *Ibid.*, **49**, No. 478, 211A (1963).
(38S) Purnell, J., *Can. Chem. Process.* **46**, No. 7, 48 (1962).
(39S) Rath, G., Instr. Meas., Chem. Anal., Elec. Quant., Nucleonics Process Control, Proc. Intern. Conf., Stockholm, Vol. 1, p. 498, 1960.
(40S) Robinson, J. W., *Anal. Chim. Acta* **27**, 465 (1962).
(41S) Sablina, Z. A., Leont'ev, B. I., Sazonov, A. E., *Azerb. Neft. Khoz.* **41**, No. 9, 35 (1962).
(42S) Siering, R., *Atompraxis* **9**, 424 (1963).
(43S) Smith, V. N., Merritt, E. J., ANAL. CHEM. **34**, 1476 (1962).
(44S) Snyder, E. I., *J. Am. Chem. Soc.* **85**, 2624 (1963).
(45S) Stemberger, V., *Nafta* **13**, 383 (1962).
(46S) Weir, H. E., *Microchem. J.* **6** (1), 109 (1962).
(47S) Weitkamp, H., Korte, F., *Chem.-Ingr. Tech.* **35**, 429 (1963).
(48S) Wotring, A. W., Keeney, W. W., *ISA Proc. Natl. Anal. Instr. Symp.* **8**, 207 (1962).
(49S) Zaidman, M. N., Orechkin, D. B., *Novosti Neft. i Gaz. Tekhn. Neftepererabotka i Neftekhim.*, No. **1**, 32 (1961).

Miscellaneous

(1T) Agaev, N. A., Golubev, I. F., *Gazovaya Prom.* **8** (5), 45–50 (1963).
(2T) *Ibid.*, (7), pp. 50–3.
(3T) Agaev, N. A., Golubev, I. F., *Khim. i Tekhnol. Topliv i Masel* **8** (6), 29–30 (1963).
(4T) Albright, L. F., Perkins, T. D., *Petro/Chem. Engr.* **35**, No. 1, 47–9 (1963).
(5T) Bacurova, E., Vrbjarova, G., *Ropa Uhlie* **4**, No. 6, 175–6 (1962).
(6T) Bell, G. H., Groszek, A. J., *J. Inst. Petrol.* **48**, No. 466, 325–32 (1962).
(7T) Ben'kovskii, V. G., Bogoslovskaya, T. M., Kiiko, L. D., Nauruzov, M. Kh., *Neftekhimiya* **3**, 173–6 (1963).
(8T) Berthold, P. H., *Chem. Tech.* (*Berlin*) **14**, 301–5 (1962).
(9T) Berthold, P. H., Teubel, J., Leibnitz, E., *Freiberger Forschungsh.* **A 250**, 9–18 (1962).
(10T) Bondi, A., McConaughy, R. B., *Proc. Am. Petrol. Inst.* Sect. III, **42**, 40–51 (1962).
(11T) Carre, G., Laurent, D., *Bull. Assoc. Franc. Tech. Petrole* **1963**, 3–54.
(12T) Chekalyuk, E. B., *Nauchn.-Tekhn. Sb. po Dobyche Nefti, Vses. Nefte-Gaz. Nauchn.-Issled Inst.*, No. **12**, 66–9 (1961).
(13T) *Chem. Eng. News* **41**, No. 2, 39–40 (1963).
(14T) Cranston, R. W., Gammon, J., *J. Inst. Petrol.* **50**, No. 483, 73–80 (1964).
(15T) Dixon J. A., *Proc. 5th World Petrol. Congr., N. Y., 1959*, **5**, 205–22 (1960).
(16T) Donlan, T. R., *Mater. Res. Std.* **2**, No. 10, 820–4 (1962).
(17T) Dukhovnyi, G. E., *Khim. i Tekhnol, Topliv i Masel* **8**, No. 8, 62–4 (1963).
(18T) Ettling, B. V., *J. Forensic Sci.* **8**, No. 2, 261–7 (1963).
(19T) Fink, D. F., *Mater. Res. Std.* **3**, No. 1, 14–19 (1963).
(20T) Foldiak, G., *Acta Chim. Acad. Sci. Hung.* **31**, 67–75 (1962).
(21T) Gerber, M. I., Dermanova, I. G., Listkova, T. M., Strigaleva, N. V., *Tr. Vses. Neft. Nauchn-Issled. Geologorazved, Inst.* **1961**, No. 174, 210–17.
(22T) Gunn, S. R., ANAL. CHEM. **34**, 1292–96 (1962).
(23T) Hammerich, T., Schmitz, A., *Erdoel Kohle* **14**, 1021–4 (1961).
(24T) Hardy, R. C., *Natl. Bur. Std. Monogr.* **1962**, 55.
(25T) Hollinghurst, R., *J. Inst. Petrol.* **49**, No. 471, 89–90 (1963).
(26T) Inoue, T., *J. Soc. High Pressure Gas. Ind. Japan* **25**, 319–325 (1961).
(27T) Ivanov, P. G., Gurkina, A. I., *Neftyanik* **1959**, No. 12, 16–17.
(28T) Kinoshita, M., Onoyama, N., *J. Japan Petrol. Inst.* **3**, 121 (1960).
(29T) Krawetz, A. A., Tovrog, T., *Rev. Sci. Instr.* **33**, 1465–6 (1962).
(30T) Lyashkevich, N. I., *Neftekhimiya* **1**, 286–91 (1961).

(31T) Lyn, W. T., *Engineer* (*London*) **215,** 661 (1963).
(32T) Malschaert, F. P., *Mech. Power* **58,** 201–5 (1962).
(33T) Melent'eva, N. I., *Khim. i Tekhnol. Topliv i Masel* **8,** No. 2, 62–3 (1963).
(34T) Mozes, G., Fenyi, M., Fehervari, A., Vamos, E., *Acta Chim. Acad. Sci. Hung.* **37,** No. 2, 191–202 (1963).
(35T) Nelson, W. L., *Oil Gas J.* **60,** No. 53, 151 (1962).
(36T) *Ibid.*, **61,** No. 31, 109 (1963).
(37T) Pasek, G. J., Thodos, G., *J. Chem. Eng. Data* **7,** 21–6 (1962).
(38T) Pektemirov, G. K., *Novosti Neft. i Gaz. Tekhn. Neftepromysl. Delo* **1962,** No. 11, 33–4.
(39T) Rajeswaran, P., Kirk, P. L., *Microchem. J.* **6,** No. 1, 21–9 (1962).
(40T) Roelands, C. J. A., Vlugter, J. C., Waterman, H. I., *J. Basic Eng.* **85,** 601–10 (1963).
(41T) Sanin, P. I., Bagrii, E. I., Petrov, A. A., Nikitskaya, E. A., Tsedilina, A. L., *Neftekhimiya* **3,** 835–44 (1963).
(42T) Santucci, L. E., Schmidt, R. J., *Mater. Res. Std.* **3,** No. 5, 395 (1963).
(43T) Schatt, W., Pietsch E., *Chem. Tech.* (*Berlin*) **15,** No. 5, 284–90 (1963).
(44T) Stepanov, L. P., Stul'ginskaya, I. A., *Tr. Vses. Nauchn.-Issed Inst. Metrol.* **1962,** No. 62, 5–23.
(45T) Trowell, F., Taylor, H. G., Brown, W. J., *Analyst* **87,** 677–83 (1962).
(46T) White, H. T., 139th Meeting ACS, Division of Petroleum Chemistry, St. Louis, Mo., Symposium **6,** No. 2C, 9–16 (1961).
(47T) Wickey, R. O., Chittenden, D. H., *Hydrocarbon Process. Petrol. Refiner* **42,** No. 6, 157–8 (1963).
(48T) Zaitsev, I. F., Tsarevskiĭ, A. F., *Zavodsk. Lab.* **28,** 1135–7 (1962).

Standards

(1V) Am. Soc. Testing Materials, Philadelphia, Pa., "Book of Standards," Parts 17 and 18, 1964.
(2V) Am. Soc. Testing Materials–Institute of Petroleum, "Petroleum Measurement Tables," Am. Soc. Testing Materials, Philadelphia, Pa., 1952.
(3V) Institute of Petroleum, London, "Standards for Petroleum and Its Products," 1964.

Pharmaceuticals and Related Drugs

D. C. Wimer, J. G. Theivagt, and V. E. Papendick,
Analytical Research Department, Abbott Laboratories, North Chicago, Ill.

This survey includes analytical methods and related topics of pharmaceutical interest that have appeared since the last published review (*197*) and covers the literature to June 1964.

The material has been arranged in the same tabular form as was used in the previous review; however, general papers on pharmaceutical stability and incompatibility are now indexed separately (see Table VI). Stability and degradation studies of individual compounds and/or classes may still be found in the appropriate table under the desired topic.

A number of review articles have appeared which are of interest to analytical chemists working in the drug field.

Papers on thin layer chromatography and its application to pharmaceutical analysis continue to increase in number (*233, 765, 1074, 1122, 1184*). The use of grooved glass plates permits uniform application of silica gel or other materials without the use of elaborate spreading equipment (*406*).

Comprehensive reviews of column partition chromatography (*739*) and gas chromatography (*184*) and their application in pharmaceutical analysis have appeared. Gas chromatography has been used to separate and identify a large number of tranquilizing drugs (*957*).

Tschudi-Steiner and Leupin have reviewed the fluorescence analysis of drugs (*1294*). The possibility of using solid state fluorescence for the control of tablet uniformity was explored by Head (*480*).

Phase solubility analysis may be used for determining the purity of compounds suspected to contain structurally similar impurities (*939*). The slope of the total concentration *vs.* the solubility concentration curve can be related to drug purity. The dissolution behavior and the effect of hydration and solvation on the solubility and rate of solution of drugs have been evaluated from a theoretical standpoint (*492, 1138*).

An evaluation of oral sustained release formulations based on the principle of delayed diffusion has been made (*1153*).

The identification of drugs continues to receive considerable emphasis. A compilation of the infrared, ultraviolet, and visible absorption spectra of some U.S.P. and N.F. reference standards and their derivatives has been published (*478*). Over 200 spectra are included. Sunshine and Gerber have written a book on the Spectrophotometric Analysis of Drugs (*1224*). An atlas of spectra is included. The control applications of ultraviolet and infrared spectrophotometry have been reviewed (*892*).

Microscopic identification of drugs has been studied including some of the factors which influence crystal formation from solutions (*896*).

(*Text continued on page 199 R*)

Table I. Alkaloids

Compound or class	Method or subject	Reference
Alkaloidal amines		
Colchicine	Polarographic, stability	(*961*)
Ephedrine	Chromatographic, gas	(*1369*)
	Chromatographic, ion exchange	(*141*)
	Chromatographic, paper	(*495*)
	Colorimetric	(*1286*)
	Counter-current distribution	(*660*)
	Extractive, misc.	(*172, 173*)
	Identity	(*302, 1361*)
	Volumetric, acid-base	(*141, 240, 546*)
	Volumetric, complexometric	(*1297*)
Muscarine	Chromatographic, paper	(*194*)
Alkaloids, general	Chromatographic, column	(*28, 139, 970*)
	Chromatographic, gas	(*188, 297, 622, 959*)
	Chromatographic, ion exchange	(*1448*)
	Chromatographic, paper	(*291, 297, 332, 707, 956, 1049, 1210, 1293, 1355, 1377, 1393, 1443, 1458*)
	Chromatographic, partition column	(*195*)
	Chromatographic, thin layer	(*318, 367, 683, 690*)

(*Continued*)

Table I. Alkaloids (*Continued*)

Compound or Class	Method or subject	Reference
	Colorimetric	(*79, 231, 332, 444, 893, 1038, 1286, 1413*)
	Electrophoretic	(*32, 956, 1251*)
	Fluorometric	(*722, 734*)
	Identity	(*302, 1038*)
	Polarographic	(*1093, 1094*)
	Spectrophotometric, ultraviolet	(*446*)
	Volumetric, acid-base	(*128, 240, 1023, 1178*)
	Volumetric, complexometric	(*150, 1297*)
	Volumetric, conductometric	(*1230*)
	Volumetric, radiometric	(*1155*)
	Volumetric, colorimetric	(*576*)
Glyoxaline alkaloids		
General	Chromatographic, paper	(*997*)
	Colorimetric	(*444*)
Pilocarpine	Chromatographic, paper	(*1371*)
	Colorimetric	(*723*)
	Identity	(*1006*)
Indole alkaloids		
Ajmaline	Chromatographic	(*755, 1134*)
	Electrophoretic, turbidimetric	(*1134*)
	Polarographic	(*559*)
	Spectrophotometric	(*755*)
	Stereochemistry	(*69*)
Brucine	Chromatographic paper	(*149, 387, 794, 1003, 1098, 1293*)
	Colorimetric	(*231, 365, 1098*)
	Electrophoretic	(*270, 271*)
	Spectrophotometric, ultraviolet	(*983*)
	Stability	(*1098*)
	Volumetric, acid-base	(*220*)
Ergotamine	Chromatographic, paper	(*18, 227, 743*)
	Chromatographic, partition column	(*18, 20, 742*)
	Colorimetric	(*17, 18, 20, 743, 1239, 1366*)
	Countercurrent distribution	(*109*)
	Identity	(*302*)
	Review, colorimetric	(*1366*)
	Stability	(*1183*)
	Volumetric, acid-base	(*1239*)
Ergot alkaloids	Chromatographic, paper	(*19, 638, 743, 778*)
	Chromatographic, partition column	(*742, 1381*)
	Chromatographic, thin layer	(*452*)
	Colorimetric	(*19, 313, 638, 743, 828*)
	Countercurrent distribution	(*109*)
	Detection of clavine in	(*1380*)
	Review, critical	(*464*)
	Stability	(*1208*)
Ergot, hydrogenated	Chromatographic, paper	(*7, 8, 9, 387*)
	Colorimetric	(*8*)
	Fluorometric	(*652, 1414*)
	Spectrophotometric, ultraviolet	(*652*)
	Stability	(*7, 8, 9, 387*)
Indoles, general	Chromatographic, paper	(*1003*)
Physostigmine	Chromatographic, paper; misc.	(*1117, 1377*)
	Identity	(*302*)
	Polarographic	(*961*)
	Stability	(*954, 961*)
Rauwolfia alkaloids	Chromatographic, paper	(*607, 681, 755, 1003*)
	Chromatographic, thin layer	(*755*)
	Review	(*1080*)
	Spectrophotometric, ultraviolet	(*606, 607, 755*)
	Stereochemistry	(*69*)
	Volumetric, acid-base	(*73*)
Reserpine	Colorimetric	(*159, 723*)
	Coulometric	(*597*)
	Fluorometric	(*568*)
	Refractometric	(*918*)
	Spectrophotometric	(*755*)
	Volumetric	(*858*)
Strychnine	Chromatographic, ion exchange	(*1400*)
	Chromatographic, paper	(*149, 794, 1003, 1098, 1293, 1390*)
	Chromatographic, partition column	(*737*)
	Colorimetric	(*71, 231, 270, 454, 1098*)
	Electrophoretic	(*270, 271*)
	Extractive	(*298*)
	Identity	(*302*)
	Spectrophotometric, ultraviolet	(*737, 983*)
	Stability	(*1098*)
	Volumetric	(*150*)
	Volumetric, acid-base	(*220, 365*)
	Volumetric, complexometric	(*1297*)
	Volumetric, radiometric	(*83*)
Strychnos alkaloids	Chromatographic, paper	(*794*)
Yohimbine	Chromatographic, paper; thin layer	(*755*)
	Fluorometric	(*247*)
	Spectrophotometric	(*27, 755*)
	Volumetric	(*150*)
	Volumetric, ox.-red.	(*1447*)
Isoquinoline alkaloids		
Apomorphine	Colorimetric	(*1285*)
Berberine	Chromatographic, ion exchange	(*1397, 1398, 1400*)
	Electrophoretic	(*1420*)
	Volumetric	(*978*)
	Volumetric, complexometric	(*1397*)
	Volumetric, ox-red.	(*1399*)
Chelidonine	Volumetric, acid-base	(*669*)
Codeine	Chromatographic, absorption column	(*28, 995*)
	Chromatographic, gas	(*1369, 1432*)
	Chromatographic, paper	(*289, 361, 564 880, 971, 995, 1213, 1293*)
	Chromatographic, partition column	(*678, 680*)
	Chromatographic, thin layer	(*126, 379, 799, 1236*)
	Colorimetric	(*169, 231, 366, 1013, 1273*)
	Extractive, misc.	(*172*)
	Fluorometric	(*256*)
	Gravimetric	(*1013*)
	Identity	(*900*)
	Refractometric	(*1042*)
	Spectrophotometric, infrared	(*421*)
	Volumetric, acid-base	(*201, 240, 673 982, 1347, 1372*)
	Volumetric, high frequency	(*36, 433*)
	X-ray diffraction	(*1087*)
Cotarnine	Fluorometric	(*529*)
Cryptopine	Nuclear magnetic resonance	(*1072*)
Diacetylmorphine	Chromatographic, paper	(*880*)
	Identity	(*400, 734, 834*)
	Spectrophotometric, infrared	(*834*)
	Spectrophotometric, ultraviolet	(*879*)
Diamorphine	Chromatographic, gas	(*1369*)
Dihydrocodeine	Chromatographic, partition column	(*678, 738*)
	Colorimetric	(*1083, 1084*)
	Spectrophotometric	(*738*)

Table I. Alkaloids *(Continued)*

Compound or class	Method or subject	Reference
Dihydrohydroxycodeinone	Colorimetric	*(1286)*
	Spectrophotometric, ultraviolet	*(724)*
Ethylmorphine	Chromatographic, thin layer	*(1236)*
	Extractive, misc.	*(172)*
	Spectrophotometric, ultraviolet	*(351)*
	Volumetric, acid-base	*(1372)*
Hydrastine	Electrophoretic, gel	*(1420)*
	Volumetric	*(978)*
Morphine	Chromatographic, adsorption column	*(55, 1164)*
	Chromatographic, gas	*(189, 1432)*
	Chromatographic, ion exchange	*(257)*
	Chromatographic, paper	*(289, 361, 460, 564, 668, 880, 1293)*
	Chromatographic, partition column	*(680)*
	Chromatographic, thin layer	*(379, 799)*
	Colorimetric	*(231, 334, 510, 668, 823, 1013, 1039, 1085, 1164 1284)*
	Colorimetric, psuedomorphine in	*(397)*
	Electrophoretic, paper	*(510, 754)*
	Extractive	*(298)*
	Fluorometric	*(179, 256)*
	Gravimetric	*(55, 181, 994, 1013)*
	Identity	*(216, 400, 511, 734, 1446)*
	Nuclear magnetic resonance	*(929)*
	Polarographic	*(1116)*
	Review	*(345)*
	Spectrophotometric, infrared	*(421)*
	Spectrophotometric, ultraviolet	*(222)*
	Volumetric	*(150)*
	Volumetric, ox.-red.	*(1001)*
Narcotine	Chromatographic, adsorption column	*(680)*
	Chromatographic, gas	*(1445)*
	Chromatographic, paper	*(10, 289)*
	Chromatographic, thin layer	*(799, 1012)*
	Colorimetric	*(231, 1013, 1086)*
	Fluorometric	*(256)*
	Gravimetric	*(1013)*
	Identity	*(1446)*
	Stability	*(10)*
	Volumetric	*(978)*
Opium alkaloids	Activation analysis of metals in	*(953)*
	Chromatographic, paper	*(289, 361, 460, 564)*
	Chromatographic, partition column	*(680)*
	Chromatographic, thin layer	*(799, 1012)*
	Colorimetric	*(823, 1039)*
	Determination	*(1040)*
	Fluorometric	*(256)*
	Gravimetric	*(1013)*
	Identity	*(511)*
	Spectrophotometric, infrared	*(421)*
	Stability	*(10)*
Oxymorphine	Colorimetric	*(616)*
Papaverine	Chromatographic, adsorption column	*(28)*
	Chromatographic, gas	*(1445)*
	Chromatographic, paper	*(1202)*
	Chromatographic, thin layer	*(318, 379, 894, 1236)*
	Colorimetric	*(698, 1457)*
	Compatibility	*(1079)*
	Electrophoretic, paper	*(754)*
	Extractive, misc.	*(172)*
	Fluorometric	*(256)*
	Gravimetric	*(251)*
	Polarographic	*(1094)*
	Spectrophotometric, ultraviolet	*(698, 1034, 1143)*
	Stability	*(1033, 1445)*
	Volumetric	*(150, 978)*
	Volumetric, acid-base	*(1025, 1178, 1348)*
	Volumetric, complexometric	*(978, 1452)*
	Volumetric, high frequency	*(36)*
Salsoline	Identity	*(1006)*
Tecodine	Identity	*(1027)*
Thebaine	Chromatographic, adsorption column	*(680)*
	Chromatographic, ion exchange	*(257)*
	Chromatographic, paper	*(361)*
	Chromatographic, thin layer	*(799)*
	Colorimetric; gravimetric	*(1013)*
	Determination	*(1040)*
	Fluorometric	*(256)*
	Nuclear magnetic resonance of cryptopine in	*(1072)*
	Volumetric, acid-base	*(680)*
Isoquinoline, bisbenzyl, alkaloids		
Curare	Stability	*(74)*
Emetine	Chromatographic, partition column	*(679)*
	Colorimetric	*(1423)*
	Fluorometric	*(530)*
	Identity	*(302)*
Ipecacuanha alkaloids	Chromatographic, partition column	*(679)*
	Colorimetric	*(1423)*
	Polarographic	*(810)*
Tubocurarine	Fluorometric	*(264)*
Lupinane alkaloids		
General	Chromatographic, ion exchange; paper	*(447)*
	Electrophoretic, paper	*(887)*
	Nephelometric	*(447)*
Sparteine	Identity	*(302)*
	Volumetric, acid-base	*(1301)*
Pyridine alkaloids		
Hemlock alkaloids	Chromatographic, gas	*(359)*
	Chromatographic, thin layer	*(850)*
Lobelia alkaloids	Autoisomerization	*(509)*
	Chromatographic, paper	*(1151, 1152)*
	Colorimetric	*(950)*
	Spectrophotometric	*(759)*
	Volumetric, acid-base	*(1151)*
Quinoline alkaloids		
Cinchona alkaloids	Chromatographic, paper	*(149, 1229)*
	Volumetric, acid-base	*(1287)*
	Volumetric, thermometric	*(324)*
Quincine	Chromatographic, paper	*(461)*
	Colorimetric, polarographic	*(428)*
Quinidine	Chromatographic, thin layer	*(177, 379)*
	Fluorometric	*(177)*
	Identity	*(154, 463)*
	Volumetric, thermometric	*(324)*
Quinine	Chromatographic, adsorption column	*(28)*
	Chromatographic, ion exchange paper	*(1213)*
	Chromatographic, paper	*(1293)*
	Chromatographic, thin layer	*(177, 379)*
	Colorimetric	*(231, 270, 454)*
	Electrophoretic	*(270, 271, 1420)*
	Fluorometric	*(177)*
	Gravimetric	*(1024, 1372)*
	Identity	*(154, 900, 1014)*
	Spectrophotometric, ultraviolet	*(351, 983)*
	Stability	*(461, 463)*
	Volumetric, acid-base	*(919, 1024, 1169)*
	Volumetric, complexometric	*(1297, 1452)*

(Continued)

Table I. Alkaloids (*Continued*)

Compound or class	Method or subject	Reference
	Volumetric, ox.-red.	(*501*)
Steroidal alkaloids		
Solanaceae alkaloids	Colorimetric	(*444*)
Veratensine	Colorimetric	(*1203*)
Veratrine	Colorimetric	(*448*)
	Countercurrent distribution	(*1234*)
Tropane alkaloids		
Atropine	Chromatographic	(*886*)
	Chromatographic, adsorption column	(*28*)
	Chromatographic, ion exchange	(*1398*)
	Chromatographic, paper	(*300, 632, 668, 1293*)
	Chromatographic, partition column	(*1095*)
	Chromatographic, thin layer	(*543, 1268*)
	Colorimetric	(*231, 668, 1186, 1431*)
	Electrophoretic, paper	(*754, 1431*)
	Extractive	(*991*)
	Fluorometric	(*141, 1439, 1440*)
	Identity	(*658, 797, 990, 1446*)
	Stability	(*99, 100*)
	Volumetric	(*150*)
	Volumetric, acid-base	(*99, 100, 543*)
	Volumetric, complexometric	(*1297*)
	Volumetric, high frequency	(*36*)
Cocaine	Chromatographic, gas	(*1105*)
	Chromatographic, paper	(*1293, 1377*)
	Colorimetric	(*1259*)
	Identity	(*302*)
	Stability	(*1377*)
Homatropine	Chromatographic, paper; turbidimetric	(*792*)
	Chromatographic, thin layer	(*1236*)
	Colorimetric	(*49*)
	Identity	(*302, 797*)
Hyoscine	Chromatographic, ion exchange	(*1398*)
	Chromatographic, partition column	(*1095*)
	Colorimetric, electrophoretic	(*1431*)
Hyoscyamine	Chromatographic, paper	(*300, 557*)
	Chromatographic, partition column	(*1095*)
	Countercurrent distribution	(*1310*)
	Identity	(*557, 633, 797*)
	Extractive	(*991*)
	Stability	(*703, 720*)
Norhyoscyamine	Identity	(*633*)
Scopalamine	Chromatographic, ion exchange	(*1398*)
	Colorimetric	(*1286*)
	Identity	(*302, 633, 797, 990, 1446*)
	Stability	(*720*)
Tropane, general	Chromatographic, paper	(*300, 632, 787, 1126*)
	Chromatographic, partition column	(*1095*)
	Chromatographic, thin layer	(*543, 1268*)
	Colorimetric	(*787, 1121, 1126*)
	Countercurrent distribution	(*1310*)
	Electrophoretic, paper	(*752*)
	Extractive	(*172, 251, 1029*)
	Identity	(*797*)
	Review, critical	(*68*)
	Stability	(*1208*)
Tropic acid, tropine	Stability	(*99, 100*)
	Volumetric, acid-base	(*100*)

Table II. Antibiotics

Compound or class	Method or subject	Reference
Actinoidin	Chromatographic, ion exchange	(*180*)
Actinomycin	Chromatographic, column	(*1113*)
Actinospectacin	Chromatographic, column	(*111*)
	Spectrophotometric, ultraviolet	(*417*)
	Stability	(*417*)
	Volumetric, acid-base	(*111*)
Amphomycin	Chromatographic, ion exchange	(*180*)
Amphotericin	Colorimetric	(*239*)
Bacitracin	Gravimetric	(*809*)
	Stability	(*1306*)
Cephalosporins	Bioautographic	(*833*)
Chloramphenicol	Chromatographic, column	(*267*)
	Colorimetric	(*64, 590*)
	Identification	(*337*)
	Polarographic	(*1075*)
	Spectrophotometric, infrared	(*811*)
	Spectrophotometric, ultraviolet	(*469, 1135*)
	Stability	(*1306*)
	Volumetric, general	(*1430*)
	Volumetric, precipitation	(*45, 736*)
Chlortetracycline	Chromatographic, ion exchange	(*1384*)
	Chromatographic, paper	(*217*)
	Chromatographic, thin layer	(*609*)
	Colorimetric	(*13, 1056, 1311, 1442*)
	Coulometric	(*602*)
	Extractive	(*1383*)
	Identification	(*1270*)
	Oscillographic	(*362*)
	Spectrophotometric, ultraviolet	(*1002*)
	Stability	(*1384*)
Erythromycin	Chromatographic, paper	(*923*)
	Colorimetric	(*353, 923*)
	Identification	(*1314*)
	Spectrophotometric, ultraviolet	(*353*)
	Volumetric, conductometric	(*353*)
General	Chromatographic, pH	(*119, 120, 122*)
	Chromatographic, thin layer	(*15, 542*)
	Identification	(*301*)
	Polarographic	(*1193*)
	Review	(*121, 625*)
	Turbidimetric	(*921*)
Griseofulvin	Chromatographic, partition	(*504*)
	Colorimetric	(*841*)
	Polarographic	(*585*)
	Spectrophotometric, ultraviolet	(*504*)
Kanamycin	Chromatographic, column	(*1388*)
	Chromatographic, ion exchange	(*180*)
	Chromatographic, paper	(*1388*)
	Colorimetric	(*589, 677*)
Magnamycin	Chromatographic, pH	(*539*)
Monomycin	Chromatographic, ion exchange	(*180*)
Nafcillin	Specifications	(*374*)
Neomycin	Chromatographic, ion exchange	(*180*)
	Chromatographic, thin layer	(*190*)
	Colorimetric	(*204*)
	Identification	(*719*)
	Polarographic	(*204*)
Novibiocin	Chromatographic, paper	(*1133*)
Nystatin	Colorimetric	(*814*)
	Identification	(*941, 1313*)

(*Continued*)

Table II. Antibiotics *(Continued)*

Compound or class	Method or subject	Reference
Oleandomycin	Spectrophotometric, ultraviolet	(*398*)
Oxytetracycline	Chromatographic, column	(*593*)
	Chromatographic, paper	(*217, 1246*)
	Chromatographic, thin layer	(*609*)
	Colorimetric	(*13, 592, 593, 1056, 1311*)
	Coulometric	(*602*)
	Identification	(*1270*)
	Oscillographic	(*362*)
	Polarographic	(*491*)
	Stability	(*847*)
Penicillins	Chromatographic, ion exchange	(*1092*)
	Chromatographic, paper	(*1354, 1401*)
	Chromatographic, pH	(*539*)
	Chromatographic, thin layer	(*915, 916, 917*)
	Colorimetric	(*401, 1174*)
	Review	(*373, 471, 1307*)
	Spectrophotometric, infrared	(*940*)
	Spectrophotometric, ultraviolet	(*885, 1403*)
	Stability	(*885, 1069, 1185, 1306, 1307*)
	Volumetric, acid-base	(*848, 1044*)
	Volumetric, ox.-red.	(*432, 626, 676, 1069*)
Phenethicillin	Stability	(*1118*)
Polymyxin	Gravimetric	(*809*)
Primycin	Identification	(*1245*)
Rifamycin	Volumetric, amperometric	(*405*)
Sparsomycin	Spectrophotometric, ultraviolet	(*191*)
Staphylomycin	Chromatographic, column	(*440*)
	Chromatographic, thin layer	(*440*)
Streptomycin	Chromatographic	(*275*)
	Chromatographic, ion exchange	(*981*)
	Colorimetric	(*1137*)
	Conductometric	(*44*)
	Fluorometric	(*370*)
	Identification	(*635, 1031*)
	Polarimetric	(*1290*)
	Polarographic	(*438*)
	Spectrophotometric, ultraviolet	(*371*)
	Stability	(*65*)
	Volumetric, ox-red.	(*524, 636*)
Streptovitacin	Stability	(*413*)
Syntomycin	Colorimetric	(*116*)
Tetracycline	Chromatographic, paper	(*217, 1318*)
	Chromatographic, thin layer	(*609*)
	Colorimetric	(*13, 591, 1056, 1311*)
	Extractive	(*1383*)
	Fluorometric	(*479*)
	Identification	(*1270*)
	Oscillographic	(*362*)
	Polarographic	(*491*)
	Spectrophotometric, infrared	(*410*)
	Spectrophotometric, ultraviolet	(*335, 410, 1002, 1135, 1383*)
	Stability	(*410, 1306*)
	Volumetric	(*411*)
Trichothecin	Polarographic	(*585*)
Tubercidin	Spectrophotometric, ultraviolet	(*192*)
Tyrocidin	Colorimetric, review	(*809*)
Viomycin	Chromatographic, paper	(*148*)
	Colorimetric	(*922*)
	Gravimetric	(*809*)

Table III. Inorganic and Metal–Organic Compounds

Compound or class	Method or subject	Reference
Aluminum	Volumetric, complexometric	(*685, 686*)
Ammonia	Identification	(*992*)
Antimony	Volumetric, photometric	(*1131*)
	X-ray analysis	(*1123*)
Arsenic	Colorimetric	(*566, 708, 746*)
	Identification	(*137, 274, 1362*)
	Volumetric, ox-red.	(*1141*)
Bismuth	Colorimetric	(*434, 583, 964, 999*)
	Gravimetric	(*1262, 1264*)
	Identification	(*1302*)
	Volumetric, complexometric	(*51, 52, 1132, 1450*)
	Volumetric, ox.-red.	(*327, 456*)
Boron	Colorimetric	(*1071*)
	Volumetric, acid-base	(*964*)
Bromine	Identification	(*863*)
	Radiometric	(*1110*)
Calcium	Colorimetric	(*733*)
	Nephelometric	(*1424*)
	Review	(*1062*)
	Volumetric, complexometric	(*555, 935*)
Chlorine	Gravimetric	(*1263*)
Copper	Colorimetric	(*427*)
	Volumetric	(*507*)
Fluorine	Identification	(*663*)
General, cations	Volumetric, complexometric	(*262*)
Halogens, general	Identification	(*726*)
	Volumetric	(*1226*)
Heavy metals, general	Identification	(*581*)
	Radiometric	(*1109*)
	Volumetric	(*63*)
	X-ray analysis	(*1139*)
Iodine	Colorimetric	(*326*)
	Volumetric, ox.-red.	(*1070*)
Iodine, organic	Volumetric, ox.-red.	(*706, 732, 817, 1103*)
Iron	Review	(*1063*)
	Volumetric	(*507*)
	Volumetric, complexometric	(*260, 261*)
	Volumetric, ox.-red.	(*1088, 1242*)
Lead	Volumetric	(*507*)
Magnesium	Volumetric, complexometric	(*935*)
Mercury	Colorimetric	(*824*)
	Kinetic	(*608*)
	Volumetric, complexometric	(*1064*)
	Volumetric, ox.-red.	(*1255*)
Mercury, organic	Bioassay	(*1026*)
	Chromatographic, paper	(*85*)
	Gravimetric	(*424*)
	Polarographic	(*38, 221*)
	Volumetric, complexometric	(*209, 554*)
Nitrate	Identification	(*558*)
Nitrite	Identification	(*558*)
Phosphorus	Volumetric, complexometric	(*133, 134, 1340*)
Potassium	Gravimetric	(*1129*)
	Identification	(*992*)
Silver	Volumetric, ox.-red.	(*456*)
Sodium	Gravimetric	(*1129*)
	Identification	(*992*)
	Nephelometric	(*548*)
	Volumetric, complexometric	(*319*)
Sulfate	Volumetric, precipitation	(*228*)
Sulfur	Gravimetric	(*1367*)
	Identification	(*1317*)
Zinc	Colorimetric	(*1454*)
	Volumetric	(*507*)
	Volumetric, complexometric	(*964*)

Table IV. Nitrogen-Containing Compounds

Compound or class	Method or subject	Reference
Acridines	Chromatographic, paper	(*1391*)
Amides	Characterization	(*244*)
	Chromatographic, ion exchange	(*468*)
	Colorimetric	(*115, 386, 866, 979, 1376, 1438*)
	Hydrolysis kinetics	(*37, 418*)
	Polarographic	(*578, 624, 924*)
	Spectrophotometric, ultraviolet	(*1167*)
	Volumetric, acid-base	(*340*)
	Volumetric, ox.-red.	(*339*)
Amidines	Volumetric, acid-base	(*91*)
Amines		
Aliphatic amines	Spectrophotometric, ultraviolet	(*798*)
Antihistamines	Chromatographic, adsorption	(*993*)
	Chromatographic, gas	(*232, 777*)
	Chromatographic, paper	(*1356, 1359, 1360*)
	Chromatographic, partition	(*859*)
	Colorimetric	(*781*)
	Gravimetric	(*1326, 1408*)
	Spectrophotometric, ultraviolet	(*178*)
	Thermomicroscopic	(*178*)
	Volumetric, acid-base	(*1343*)
	Volumetric, compleximetric	(*230*)
Aromatic amines	Volumetric, ox.-red.	(*802*)
Morpholines	Chromatographic, paper	(*390*)
	Polarographic	(*287*)
Nitrogenous organic bases	Chromatographic, paper	(*1358*)
	Colorimetric	(*70, 72, 113*)
	Fluorometric	(*531*)
	Spectrophotometric, ultraviolet	(*156, 1364*)
	Volumetric, acid-base	(*378*)
	Volumetric, compleximetric	(*399, 1395, 1418*)
	Volumetric, precipitation	(*575*)
Phenylalkylamines	Chromatographic, ion exchange	(*692, 695*)
	Chromatographic, paper	(*693*)
	Colorimetric	(*430*)
	Gravimetric	(*35*)
	Spectrophotometric, ultraviolet	(*692*)
	Volumetric, acid-base	(*35, 104, 170*)
Piperazines	Gravimetric	(*241*)
	Volumetric, acid-base	(*241, 521, 812, 963*)
Piperidines	Chromatographic, gas	(*851*)
	Chromatographic, paper	(*325*)
	Colorimetric	(*700*)
	Identification	(*323*)
	Polarographic	(*1249*)
	Volumetric, acid-base	(*659, 1288, 1349*)
	Volumetric, ox.-red.	(*659*)
Procaine and analogs	Colorimetric	(*377, 774*)
	Gravimetric	(*42*)
	Identification	(*42*)
	Polarographic	(*1322*)
	Spectrophotometric, infrared	(*42*)
	Spectrophotometric, ultraviolet	(*42, 484, 1091, 1321*)
	Stability	(*392, 1076*)
	Volumetric, acid-base	(*42, 329*)
	Volumetric, amperometric	(*108*)
Pyridines	Chromatographic, paper	(*1223*)
	Colorimetric	(*1102*)
	Electrophoretic	(*1223*)
	Gravimetric	(*1223*)
	Spectrophotometric, ultraviolet	(*474*)
	Volumetric, acid-base	(*813*)
Sympathomimetic amines	Chromatographic, gas	(*186, 187, 282, 958*)
	Identification	(*958, 1154*)
	Spectrophotometric, ultraviolet	(*237, 1271, 1272*)
	Volumetric, amperometric	(*1161*)
Amino acids		
Amino acids (general)	Chromatographic, general	(*210*)
	Chromatographic, ion exchange	(*396, 898*)
	Chromatographic, paper	(*135, 284, 865*)
	Chromatographic, review	(*210*)
	Colorimetric	(*117, 155, 158, 167, 219, 285, 1052*)
	Identification	(*1319, 1460*)
	Polarographic	(*804, 805*)
	Spectrophotometric, ultraviolet	(*146*)
	Stability	(*541*)
p-Aminosalicylic acids and salts	Chromatographic, paper	(*255, 1274*)
	Chromatographic, thin layer	(*1274*)
Glycine	Colorimetric	(*429, 1096*)
Nucleic acids	Spectrophotometric, ultraviolet	(*1338*)
Taurine	Identification	(*375*)
Thyronine and analogs	Biological assay	(*1404*)
	Chromatographic, ion exchange	(*1304*)
	Chromatographic, paper	(*476*)
	Colorimetric	(*856*)
	Electrophoretic, I^{131}	(*784*)
Aminoalcohols		
Ephedrine	Identification	(*1405*)
N-Aminobarbituric acids	Spectrophotometric, infrared	(*288*)
Aminophenols		
Adrenaline and analogs	Chromatographic, paper	(*1389*)
	Chromatographic, thin layer	(*467, 1392*)
	Fluorometric	(*61, 675, 943*)
	Spectrophotometric, chelates	(*465*)
	Stability	(*512, 1374*)
	Volumetric, ox.-red.	(*1158*)
Aminophenol	Colorimetric	(*127, 688, 766, 1196*)
Epinephrine and analogs	Chromatographic, adsorption	(*199*)
	Fluorometric	(*199, 248*)
	Review	(*53*)
	Stability	(*1241*)
	Volumetric, complexometric	(*1300*)
Isoprenaline	Spectrophotometric, chelates	(*466*)
	Stability	(*1411*)
Methyldopa and analogs	Colorimetric	(*789*)
Phenylephrine	Colorimetric	(*684, 1444*)
	Stability (to oxidation)	(*763*)
Azepines	Colorimetric	(*76, 320*)
	Gravimetric	(*320*)
	Identification	(*76, 304, 1337*)
	Spectrophotometric, ultraviolet	(*304*)
	Volumetric, acid-base	(*320*)
	Volumetric, compleximetric	(*320*)
Barbiturates	Chromatographic, gas	(*185, 250, 889, 960*)
	Chromatographic, paper	(*613, 647, 649, 1009, 1201, 1206, 1212, 1435*)

(*Continued*)

Table IV. Nitrogen-Containing Compounds (*Continued*)

Compound or class	Method or subject	Reference
	Chromatographic, ion exchange	(605)
	Chromatographic, thin layer	(341, 1100, 1225)
	Colorimetric	(286, 290, 758)
	Differentiation (microscopic)	(296)
	Identification	(185, 889, 1201, 1225)
	Refractometric	(835, 836, 837)
	Review (methods)	(161)
	Spectrophotometric, infrared	(967)
	Spectrophotometric, ultraviolet	(196, 996, 1278, 1280)
	Spot tests	(30, 475, 644, 645, 646, 648, 650, 651, 757, 1283, 1328)
	Stability and degradation	(1190, 1402, 1453)
	Volumetric, acid-base	(16, 142, 1054, 1238)
	Volumetric, compleximetric	(138, 140, 253, 254, 1041)
	Volumetric conductimetric	(16)
	Volumetric, coulometric	(600)
	Volumetric, photometric	(348)
Barbiturates	Stability	(741)
silver salts	X-ray diffraction	(1104)
Benzoxazines	Spectrophotometric, ultraviolet	(760)
Carbamates	Colorimetric	(112, 881)
	Spectrophotometric, infrared	(223)
	Volumetric, acid-base	(112, 1172)
Ethylenimines	Volumetric, acid-base	(1077)
Guanidines	Colorimetric	(899, 1148)
Hydantoins	Volumetric, compleximetric	(1030)
	Volumetric, precipitation	(599, 1030)
Hydrazides	Colorimetric	(12, 1296, 1461)
	Fluorometric	(853)
	Volumetric, acid-base	(727, 1171)
	Volumetric, compleximetric	(1266, 1267)
	Volumetric, coulometric	(842)
	Volumetric, ox.-red.	(1227)
Hydrazines	Colorimetric	(772)
	Volumetric, coulometric, tablet variation	(416)
	Volumetric, ox.-red.	(902)
Imidazoles	Polarographic	(861, 1130)
	Volumetric, acid-base	(106)
	Volumetric, ox.-red.	(1228)
Imidazolines	Crystallographic	(1407)
	Identification	(1406)
	Spectrophotometric, ultraviolet	(470)
Imides	Colorimetric	(1437)
	Degradation kinetics	(1410)
	Identification	(1107)
	Volumetric, acid-base	(347)
Indantriones		
Ninhydrin	Anomalous reactions	(1111)
Indoles	Colorimetric	(212)
Indolines	Volumetric, ox.-red.	(333)
Isoxazolidones	Colorimetric	(1312)
Nitro	Colorimetric	(277, 344)
	Gravimetric	(203)
	Spectrophotometric, ultraviolet	(132)
	Stability	(1182)
Oxazolidines	Colorimetric	(496)
	Electrophoretic	(41)
	Spectrophotometric, ultraviolet	(41)
N-Oxides	Colorimetric	(1081)
	Volumetric, acid-base	(827, 876)
	Volumetric, ox.-red.	(502, 1081)
Purines and derivatives	Chromatographic, paper	(258)
	Chromatographic, thin layer	(234)
Pyrazines and derivatives	Colorimetric	(376)
Pyrazolidines	Degradation (oxidation)	(46)
	Volumetric, ox.-red.	(527)
Pyrazolones		
Aminopyrine	Gravimetric	(839)
	Identification (microscopic)	(985)
	Review (methods)	(795)
	Spectrophotometric, Raman	(614)
	Stability	(572)
	Volumetric, acid-base	(1345, 1346)
	Volumetric, compleximetric	(556)
Antipyrine	Chromatographic, paper	(459)
	Colorimetric	(114)
	Gravimetric	(721)
	Volumetric, compleximetric	(316)
	Volumetric, ox.-red.	(500, 721)
Pyrazolones (general)	Chromatographic, paper	(1365)
	Gravimetric	(315)
	Identification	(5)
	Spectrophotometric, ultraviolet	(1233)
	Volumetric, ox.-red.	(6, 48, 202)
Pyrimidines	Colorimetric	(3, 4, 882)
	Polarographic	(1160)
	Stability	(884)
Pyrroles	Colorimetric	(385)
Quaternary ammonium compounds	Chromatographic, ion exchange	(694, 1456)
	Chromatographic, paper	(295)
	Colorimetric	(136, 443, 694, 977, 1108, 1173, 1324)
	Identification	(306)
	Polarographic	(1324, 1325)
	Review (methods)	(694)
	Spectrophotometric, infrared	(1043)
	Spectrophotometric, ultraviolet	(563, 1018)
	Stabilization and decomposition	(1231, 1232)
	Volumetric, acid-base	(78, 473, 694, 1168)
	Volumetric, ox.-red.	(193)
Quinolines	Gravimetric	(955)
Sydnones	Spectrophotometric, ultraviolet	(412)
	Stability	(412)
Tetrazoles	Chromatographic, gas	(621)
Uracils	Colorimetric	(200, 989)
	Volumetric, acid-base	(1170)
Urea and derivatives	Colorimetric	(1147)
	Complexes	(157)
	Identification	(58)
	Volumetric, acid-base	(292)
	Volumetric, ox.-red.	(1217)
Xanthenes	Decomposition and stabilization	(904, 905)
Xanthines	Chromatographic, paper	(1021)
	Colorimetric	(571, 768)
	Isotope dilution	(877)
	Volumetric, acid-base	(745, 816)
	Volumetric, amperometric	(174)
	Volumetric, coulometric	(595, 901)
	Volumetric, ox.-red.	(1008, 1032)
	Volumetric, precipitation	(975, 976)

Table V. Oxygen-Containing Compounds

Compound or class	Method or subject	Reference
Acids and derivatives		
Acetyl salicylic acid	Chromatographic, gas	(279)
	Colorimetric	(303)
	Decomposition	(705, 907)
	General	(283)
	Spectrophotometric, infrared	(414)
	Spectrophotometric, ultraviolet	(716)
	Stability	(617, 704, 705, 747, 907)
Carboxylic acids, aliphatic	Chromatographic, gas	(540)
	Chromatographic, ion exchange	(1451)
	Chromatographic, paper	(515, 1035, 1065)
	Colorimetric	(883, 1090)
	Spectrophotometric, infrared	(160)
	Spectrophotometric, ultraviolet	(515, 1089)
	Volumetric, acid-base	(1342)
	Volumetric, ox.-red.	(951)
Carboxylic acids, aromatic	Chromatographic, gas	(540)
	Chromatographic, paper	(515)
	Colorimetric	(761, 1386)
	Coulometric	(603)
	Gravimetric	(33)
	Spectrophotometric, infrared	(268, 1191)
	Spectrophotometric, ultraviolet	(214, 1191)
	Volumetric, precipitation	(1191)
Esters	Colorimetric	(931, 1386)
	Review	(442)
	Stability	(553, 1240)
	Volumetric, acid-base	(550)
	Volumetric, precipitation	(162, 1200)
Carbohydrates	Aquametric	(358)
	Chromatographic paper	(236, 1036)
	Dialysis	(144)
	Microscopic	(642)
	Volumetric, ox.-red.	(972)
Coumarins	Chromatographic, column	(610)
	Chromatographic, paper	(124)
	Chromatographic, thin layer	(1048, 1189)
	Polarographic	(1204)
	Spectrophotometric, ultraviolet	(657)
Dextrin	Solubility analysis	(265)
Ethers	Chromatographic, gas	(532)
Fats and oils	Chromatographic, gas	(50)
	Chromatographic, paper	(1035, 1220)
	Chromatographic, thin layer	(31)
	Peroxide number	(687)
Glycerides	Chromatographic, thin layer	(67, 619, 1387)
	Colorimetric	(1387)
Glycosides		
Aloin and emodins	Chromatographic, paper	(152)
	Chromatographic, thin layer	(152, 423)
	Colorimetric	(360)
	Polarographic	(391)
	Spectrophotometric, ultraviolet	(423)
	Stability	(1329)
Cardiac glycosides	Chromatographic, paper	(1416)
	Colorimetric	(75, 852)
	Electrophoresis	(311)
	Luminescence	(1352, 1353)
Digitoxin	Chromatographic, paper	(838, 1015)
	Chromatographic, thin layer	(1157, 1188, 1464)
	Colorimetric	(838)
	Fluorometric	(567)
	Review	(123, 389, 838)
Flavones	Chromatographic, paper	(381, 912)
	Chromatographic, thin layer	(1197)
	Colorimetric	(60, 131)
	Coulometric	(598)
	Identification	(1145)
	Polarographic	(294, 936)
	Spectrophotometric, ultraviolet	(912)
	Stability	(912)
General	Chromatographic, paper	(775)
	Chromatographic, thin layer	(368, 631, 1037)
	Colorimetric	(369, 612, 775 826, 1073, 1112, 1156, 1382)
	Gravimetric	(514)
	Identification	(1017)
Santonin	Spectrophotometric, ultraviolet	(130, 942)
Saponin	Colorimetric	(664)
Squill glucosides	Chromatographic, paper	(1415)
	Chromatographic, thin layer	(1194)
	Spectrophotometric, ultraviolet	(1194, 1415)
Strophanthin	Chromatographic, paper	(689)
	Chromatographic, thin layer	(498, 767)
	Colorimetric	(422, 689, 1459)
	Polarographic	(1459)
	Stability	(1459)
Hydroxyl, alcoholic		
Chelidonine	Extractive	(584)
Chloral hydrate	Stability	(1298, 1299)
Ethanol	Distillation	(818, 819)
	Identification	(1363)
	Refractometric	(1422)
	Volumetric, ox.-red.	(968)
Ethchlorvynol	Colorimetric	(21)
General	Chromatographic, gas	(535)
	Gravimetric	(878)
4-Hydroxymethyl-1,3-dioxolanes	Gravimetric	(821)
	Volumetric, ox.-red.	(821)
Mephenesin	Fluorometric	(175)
Hydroxyl, phenolic		
Aminophenols	Chromatographic, paper	(1125)
	Colorimetric	(127)
Bithionol	Spectrophotometric, ultraviolet	(844)
Cresols	Chromatographic, gas	(620)
Eugenol	Colorimetric	(868, 1385)
General	Chromatographic, thin layer	(1099, 1124)
	Identification	(1159)
	Review	(1175)
	Spectrophotometric, ultraviolet	(350, 728, 1089)
Hexestrol	Colorimetric	(94, 671)
	Spectrophotometric, ultraviolet	(717)
Phenolphthalein	Colorimetric	(22, 1265)
Quinolinols	Gravimetric	(25)
	Volumetric, complexometric	(932)
Resorcinol	Amperometric	(870)
Thymol	Colorimetric	(1385)
Thyroxine	Volumetric, ox.-red.	(822)
Hydroxyl, polyhydric	Chromatographic, gas	(395)
	Chromatographic, thin layer	(110)
	Colorimetric	(426)
	Polarimetric	(552)
	Review	(913)
	Volumetric, acid-base	(764)
	Volumetric, ox.-red.	(968)
Ketones	Chromatographic, gas	(57)
	Chromatographic, paper	(125, 980)
	Chromatographic, thin layer	(125, 790)
	Polarographic	(1020)
	Refractometric	(1422)
	Spectrophotometric	(522, 1219)
	Spectrophotometric, ultraviolet	(168)
Quinones	Chromatographic, thin layer	(1010)
	Colorimetric	(43, 1265)

Table VI. Stability (General)

Compound or class	Method or subject	Reference
Drug stability	Kinetics of	(*409, 457, 637, 670*)
Parenterals	Incompatibilities of	(*1053*)
	Preservative loss in	(*715*)
Pharmaceuticals	Accelerated stability	(*579*)
	Incompatibilities of	(*342, 846, 965, 966, 1291*)
Rubber closures	Effect on preservatives	(*712*)
Stability	Prediction of	(*269*)
Tablet formulations	Photostability of	(*356, 713, 714*)

Table VII. Steroids and Hormones

Compound or class	Method or subject	Reference
Adrenocorticals		
Aldosterone	Chromatographic, gas	(*641*)
	Chromatographic, thin layer	(*101, 198, 682*)
	Fluorometric	(*483*)
Cortisones	Chromatographic, gas	(*634*)
	Chromatographic, thin layer	(*573, 807*)
	Colorimetric	(*1378*)
	Electrophoretic	(*560*)
	Polarographic	(*702*)
	Spectrophotometric, ultraviolet	(*849*)
	Stability	(*1256*)
Dexamethasone	Chromatographic, thin layer	(*573*)
	Spectrophotometric, infrared	(*96*)
Epicortisol	Chromatographic, paper	(*933*)
	Colorimetric	(*933*)
Fluorocortisone	Identification	(*562*)
General	Chromatographic, paper	(*441, 518*)
	Chromatographic, thin layer	(*801, 1120*)
	Polarographic	(*516, 547, 1207*)
	Review	(*328*)
Hydrocortisones	Chromatographic, paper	(*363, 807*)
	Chromatographic, thin layer	(*573*)
	Polarographic	(*517, 702*)
Prednisolone	Chromatographic, thin layer	(*573*)
	Colorimetric	(*213, 249*)
	Dissolution rates	(*494*)
	Polarographic	(*517, 702*)
	Solubility	(*493*)
Prednisone	Chromatographic, thin layer	(*573*)
	Colorimetric	(*213, 587, 1378*)
	Polarographic	(*517*)
Triamcinolone	Colorimetric	(*1309*)
	Identification	(*562*)
	Polarographic	(*517*)
	Spectrophotometric, infrared	(*96*)
Androgens		
Androsterone	Polarographic	(*547*)
General	Chromatographic, paper	(*226*)
	Chromatographic, thin layer	(*749*)
Methandrostenolone	Fluorometric	(*1279*)
Nandrolone	Colorimetric	(*24*)
	Identification	(*24*)
Testosterones	Chromatographic, column	(*629*)
	Chromatographic, paper	(*225, 869, 1199*)
	Chromatographic, thin layer	(*235*)
	Colorimetric	(*225, 299*)
	Fluorometric	(*1198*)
	Spectrophotometric, ultraviolet	(*629, 869, 1199*)
Estrogens		
Estradiol	Chromatographic	(*791*)
	Chromatographic, column	(*623*)
	Chromatographic, paper	(*807, 1058*)
	Chromatographic, thin layer	(*312*)
	Fluorometric	(*623, 1058*)
	Stability	(*1058*)

Table VII. Steroids and Hormones (*Continued*)

Compound or class	Method or subject	Reference
Estriol	Chromatographic	(*791*)
	Chromatographic, column	(*623*)
	Chromatographic, paper	(*586, 807*)
	Fluorometric	(*623*)
Estrone	Chromatographic	(*791*)
	Chromatographic, column	(*623*)
	Chromatographic, paper	(*807*)
	Chromatographic, thin layer	(*312*)
	Fluorometric	(*623*)
Ethinyl estradiol	Chromatographic, paper	(*1058*)
	Colorimetric	(*86*)
	Fluorometric	(*1058*)
	Stability	(*1058*)
General	Chromatographic, gas	(*776*)
	Chromatographic, paper	(*656, 920, 1289*)
	Chromatographic, thin layer	(*750, 751*)
	Color reactions	(*751*)
Ethisterone	Chromatographic, column	(*629*)
	Spectrophotometric, ultraviolet	(*629*)
Insulin	Bioassay	(*343*)
	Chromatographic, paper	(*508, 1150, 1336*)
	Polarographic	(*343*)
Keto steroids	Chromatographic, gas	(*1334*)
	Chromatographic, gradient elution	(*1370*)
	Chromatographic, paper	(*945, 946, 1192, 1218*)
	Chromatographic, thin layer	(*748*)
	Colorimetric	(*352, 526, 987*)
	Identification	(*523, 945, 1218*)
	Polarographic	(*263*)
	Spectrophotometric, infrared	(*336*)
	Spectrophotometric, ultraviolet	(*416*)
Methods of general application		
Chromatographic, column		(*1119*)
Chromatographic, gas		(*355, 655, 1421*)
Chromatographic, spot detection		(*171, 806*)
Chromatographic, thin layer		(*92, 266, 338, 472, 808, 1162, 1327*)
Color reactions		(*988, 1144*)
Spectrophotometric, ultraviolet		(*1308*)
Miscellaneous		
Bile acids	Volumetric, acid-base	(*455*)
General, steroid hormones	Chromatographic, paper	(*1057*)
	Microsublimation	(*615*)
	Optical properties	(*280, 281*)
	Stability	(*1057*)
Lipid extracts	Chromatographic, column	(*487*)
19-Norsteroids	Chromatographic, thin layer	(*436*)
Sapogenins	Chromatographic, paper	(*1429*)
	Chromatographic, thin layer	(*102*)
	Colorimetric	(*1429*)
Steroid acids	Chromatographic, thin layer	(*1120*)
Oxytocin	Bioassay	(*1222*)
Pregnans	Chromatographic, gas	(*890*)
	Volumetric, acid-base	(*419*)
Progesterone	Chromatographic, paper	(*869, 944, 1257*)
	Chromatographic, partition	(*1426*)
	Spectrophotometric, ultraviolet	(*869*)
Sterols	Chromatographic, gas	(*90, 1060*)
	Chromatographic, paper	(*718*)
	Chromatographic, thin layer	(*103, 118, 895*)

Table VIII. Sulfur-Containing Compounds

Compound or class	Method or subject	Reference
Benzothiadiazines	Colorimetric	(*393, 425, 594, 1323*)
	Spectrophotometric, ultraviolet	(*725, 788*)
	Volumetric, ox-red.	(*627*)
	Volumetric, precipitation	(*667*)
Phenothiazines	Chromatographic, gas	(*26*)
	Chromatographic, paper	(*820, 860, 1209*)
	Chromatographic, partition	(*29, 505*)
	Chromatographic, thin layer	(*205, 346, 969*)
	Colorimetric	(*384, 785*)
	Identification	(*272, 273, 580, 762, 1449*)
	Review	(*147*)
	Spectrophotometric, ultraviolet	(*182, 1211*)
	Stability and degradation	(*145, 164, 165, 166, 519, 973, 998, 1433, 1434*)
	Sustained release, tracer method	(*1059*)
	Volumetric, acid-base	(*963, 1187*)
	Volumetric, coulometric	(*54*)
	Volumetric, ox.-red.	(*364, 1136*)
Sulfonamides	Chromatographic, paper	(*176, 486, 709, 1000, 1176*)
	Chromatographic, thin layer	(*129, 402, 611, 630, 640, 1046*)
	Colorimetric	(*23, 56, 394, 771, 773, 1214*)
	Gravimetric	(*1011*)
	Identification	(*95, 770, 1019, 1067, 1295*)
	Polarographic	(*925, 926, 927, 928*)
	Spectrophotometric, mass	(*1179*)
	Spectrophotometric, ultraviolet	(*453, 825, 830, 831, 843, 1375*)
	Stability	(*1436*)
	Volumetric, acid-base	(*1055, 1205*)
	Volumetric, amperometric	(*1409*)
	Volumetric, compleximetric	(*2, 485*)
	Volumetric, ox.-red.	(*62, 1305, 1339*)
	Volumetric, precipitation	(*1180, 1235, 1247, 1248*)
Sulfur (Miscellaneous)		
Cyclohexylsulfamates	Identification	(*528*)
Disulfiram	Gravimetric	(*1373*)
	Volumetric, compleximetric	(*1373*)
2-Mercaptoimidazole	Polarographic	(*206*)
Sodium camphorsulfonate	Spectrophotometric, ultraviolet	(*34*)
Sodium lauryl sulfate	Stability	(*906*)
Sodium noramidopyrine methanesulfonate	Volumetric, coulometric	(*601*)
	Volumetric, ox.-red.	(*832*)
	Volumetric, precipitation	(*372*)
Sulfinylpyridazines	Volumetric, ox.-red.	(*569*)
Sulfonylureas	Chromatographic, paper	(*238*)
	Volumetric, ox.-red.	(*829*)
Sulfoxides	Chromatographic, paper	(*1276*)
Surface active agents	Stability to ultrasound	(*380*)
Tetramethylthiuram disulfide	Colorimetric	(*14*)
Thioamides	Colorimetric	(*696*)
	Identification	(*437*)
	Polarographic	(*930*)
	Spectrophotometric, ultraviolet	(*697*)
	Volumetric, acid-base	(*1016, 1351*)
	Volumetric, ox.-red.	(*1016*)
	Volumetric, precipitation	(*1016, 1350*)
Thiobarbituric acids	Volumetric, ox.-red.	(*570*)
Thiosemicarbazones	Colorimetric	(*666*)
	Volumetric, precipitation	(*439, 520*)
Thio-TEPA	Review (methods)	(*744*)

Table IX. Vitamins

Compound or class	Method or subject	Reference
Oil-soluble		
Vitamin A	Chromatographic, paper	(*143, 582, 1028, 1050*)
	Chromatographic, partition column	(*871, 872*)
	Chromatographic, thin layer	(*224, 293*)
	Colorimetric	(*1250*)
	Colorimetric, automatic recording	(*845*)
	Polarographic	(*1101, 1252*)
	Review	(*1028, 1101*)
	Spectrophotometric	(*208*)
	Stability	(*872, 1250, 1315*)
Vitamin A_2	Chromatographic, paper	(*582*)
Vitamin A, 9-*cis* isomers	Spectrophotometric	(*489*)
Vitamin D	Chromatographic	(*310*)
	Chromatographic, adsorption column	(*793, 1216, 1253, 1396*)
	Chromatographic, paper	(*143, 729, 1050*)
	Chromatographic, partition column	(*1216*)
	Chromatographic, thin layer	(*224, 293*)
	Colorimetric	(*793, 1050, 1216, 1253*)
	Review, radiometric methods	(*317*)
	Spectrophotometric, infrared; differentiation of D_2 and D_3	(*862*)
Vitamin E	Chromatographic, adsorption column	(*1216*)
	Chromatographic, paper	(*449, 1050*)
	Chromatographic thin layer	(*224, 293*)

(Continued)

Table IX. Vitamins (*Continued*)

Compound or class	Method or subject	References
	Colorimetric	(*730, 1082*)
	Optical activity, resolution of isomers	(*888*)
	Review	(*730*)
	Review, spectrophotometric	(*66*)
	Review, physicochemical	(*661*)
	Spectrophotometric	(*208, 661*)
Vitamin K	Chromatographic, paper	(*143*)
	Chromatographic, thin layer	(*143*)
	Polarographic	(*207, 208, 293*)
Water-soluble		
B_1, Thiamine	Chromatographic, adsorption column	(*1005*)
	Chromatographic, complexometric	(*1297*)
	Chromatographic, ion exchange paper	(*525, 653*)
	Chromatographic, paper	(*93, 151*)
	Colorimetric	(*229*)
	Fluorometric	(*93*)
	Fluorometric; spectrophotometric	(*561*)
	Polarographic	(*1005, 1101, 1277*)
	Review	(*937, 1101, 1292*)
	Spectrophotometric, UV	(*780*)
	Stability	(*11, 462, 561, 1315, 1316, 1333, 1419*)
	Volumetric, complexometric	(*1297*)
Thiamine 35 S	Chromatographic, electrophoretic	(*1068*)
Thiamine, acyl-	Chromatographic, paper; stability	(*803*)
Thiamine, *N*-cyclohexylsulfamate	Stability	(*215*)
Thiamine pyrophosphate	Chromatographic, fluorometric	(*88*)
	Chromatographic, ion exchange paper	(*653*)
B_2, riboflavin	Chromatographic, ion exchange paper	(*525, 653*)
	Chromatographic, paper	(*151, 1292*)
	Colorimetric	(*639*)
	Fluorometric, spectrophotometric	(*561*)
	Polarographic	(*181, 1101*)
	Review	(*1101*)
	Stability	(*561, 1315*)
Riboflavin derivatives	Chromatographic, paper	(*1292*)
Riboflavin, leuco-	Polarographic	(*181*)
B_6, pyridoxine	Chromatographic, complexometric	(*1297*)
	Chromatographic, ion exchange paper	(*525, 653*)
	Chromatographic, paper	(*305, 867*)
	Chromatographic, thin layer	(*914*)
	Colorimetric	(*308, 779*)
	Spectrophotometric	(*208, 780*)
	Stability	(*1315*)
	Volumetric, complexometric	(*1297*)
B_{12}, cyanocobalamine	Chromatographic, ion exchange	(*854, 855*)
	Chromatographic, paper	(*1, 81, 183 840, 854, 855, 857*)
	Chromatographic, thin layer	(*252*)
	Colorimetric	(*1, 183, 840, 854–5, 857*)
	Electrophoretic, paper	(*490*)
	Identity	(*545*)
	Micro-diffusion	(*183, 857*)
	Radiometric	(*97, 98, 855*)
	Review	(*276, 937, 1379*)
	Spectrophotometric	(*77, 80, 218, 252, 276, 780*)
	Stability	(*80, 82, 490, 1379, 1462, 1463*)
Hydrocobalamine	Spectrophotometric	(*77, 80*)
Hydroxycobalamine	Chromatographic, paper	(*218, 276, 420, 796*)
	Chromatographic, thin layer	(*252*)
	Radiometric	(*97*)
	Spectrophotometric	(*218, 252, 276, 420, 796*)
C, ascorbic acid	Chromatographic, ion exchange paper	(*525, 653, 974*)
	Chromatographic, paper	(*153, 451, 911, 1260*)
	Chromatographic, thin layer	(*252*)
	Colorimetric	(*435, 618, 1163, 1215*)
	Complexometric	(*639*)
	Identity	(*1330*)
	Optical activity	(*672*)
	Polarographic	(*1101*)
	Review	(*1101, 1254*)
	Spectrophotometric, ultraviolet	(*779, 911*)
	Stability	(*11, 245, 382, 451, 458, 488, 577, 911, 934, 938, 1163, 1258, 1315, 1316, 1333*)
	Volumetric	(*383, 911, 1177*)
Folic acid	Chromatographic, ion exchange paper	(*525, 653*)
	Colorimetric	(*782*)
Folic acid, tritiated	Chromatographic, paper	(*1061*)
Nicotinamide	Chromatographic, gas	(*1369*)
	Chromatographic, ion exchange paper	(*525, 653*)
	Colorimetric	(*639*)
	Spectrophotometric, ultraviolet	(*779*)
	Stability	(*458, 1315*)
	Volumetric	(*208, 309*)
Nicotinamide, cobalt chloride complex	Colorimetric	(*544*)
Nicotinic acid	Chromatographic paper	(*151*)
Pantothenic acid and salts	Chromatographic, colorimetric	(*947, 948*)
	Chromatographic, ion exchange	(*949*)
Panthenol	Chromatographic, ion exchange paper	(*525, 949*)
	Fluorometric	(*949*)
Vitamin P substances	Chromatographic, paper	(*305*)
	Chromatographic, spectrophotometric	(*307*)

Table X. Formulations, Mixtures, and General Methods

Compound or Class	Method or subject	Reference
A. General methods		
Acids	Chromatographic, gas	(*537*)
Alcohols	Chromatographic, paper	(*707*)
Aldehydes	Chromatographic, gas	(*536*)
Amides	Volumetric, ox.-red.	(*1341*)
Amines	Chromatographic, gas	(*1335*)
	Chromatographic, ion exchange	(*1332, 1441*)
	Chromatographic, thin layer	(*1236*)
	Colorimetric	(*893*)
	Volumetric, acid-base	(*576*)
	Volumetric, complexometric	(*978, 1452*)
	Volumetric, high frequency	(*36*)
Amines, high boiling	Chromatographic, gas	(*1369*)
Amines, quaternary	Volumetric, complexometric	(*978*)
Antacids	Neutralization rate	(*89, 815, 908, 909, 910*)
	Review	(*1106*)
Antipyretics	Chromatographic, gas	(*800*)
	Complexometric	(*314*)
Antituberculins	Identity	(*1066*)
	Volumetric, ox.-red.	(*753*)
Analgesics	Chromatographic, paper	(*1357*)
	Chromatographic, thin layer	(*259, 407*)
	Compatibility	(*463*)
	Complexometric	(*314*)
	Identity	(*407, 1195*)
	Volumetric, acid-base	(*699*)
Anesthetics	Chromatographic, gas	(*246, 431, 731*)
	Spectrophotometric, infrared	(*588*)
Anesthetics, local	Chromatographic, paper	(*707*)
	Chromatographic, thin layer	(*694, 1097*)
	Review	(*674*)
	Volumetric, acid-base	(*662*)
Antibiotics	Identity	(*1066*)
Antihistamines	Chromatographic, paper	(*47, 1355, 1359*)
	Chromatographic, partition column	(*738*)
	Chromatographic, thin layer	(*47*)
Antiseptics	Stability	(*1275*)
Antitussives	Chromatographic, partition column	(*1084, 1095*)
Azulenes	Colorimetric; paper chromatography	(*1303*)
Barbiturates	Chromatographic, gas	(*800, 959, 1335*)
	Chromatographic, ion exchange	(*477, 707*)
	Chromatographic, paper	(*1047*)
	Chromatographic, thin layer	(*1047, 1268*)
	Spectrophotometric, infrared	(*477*)
	Volumetric, acid-base	(*240*)
Chlorinated hydrocarbons	Spectrophotometric, infrared	(*481, 482*)
Dyes	Chromatographic, column	(*59*)
	Chromatographic, paper	(*864*)
	Chromatographic, thin layer	(*379, 1114, 1427*)
	Identity	(*1004*)
Emodins	Colorimetric	(*354*)
Esters	Chromatographic, gas	(*538*)
N-Ethyl groups	Identity	(*1149*)
Ethylene groups	Identity	(*1149*)
General techniques	Chromatographic, gas	(*321, 404, 533, 534, 622, 1166*)
	Chromatographic, paper	(*149, 513, 783*)
	Chromatographic, thin layer	(*84, 126, 683, 765*)
	Colorimetric	(*654, 740*)
	Complexometric	(*654, 740*)
	Countercurrent distribution	(*278*)
	Dissolution rate	(*897*)
	Electrophoretic	(*32*)
	Isotope dilution	(*701, 1282*)
	Melting point	(*445, 1128, 1455*)
	Phase solubility	(*408*)
	Polarographic	(*331, 873*)
	Purity tests	(*594, 1051*)
	Refractometric	(*1244*)
	Review, ultraviolet	(*691*)
	Spectrophotometric, ultraviolet	(*27, 450, 654, 783, 1417*)
	Stability, effect of trace copper	(*1115*)
	Stability, effect of heavy metals	(*720*)
	Thermal analysis	(*1128*)
	Turbidimetric	(*40*)
	Volumetric, acid-base	(*240, 242, 243*)
	X-Ray	(*952, 1140*)
	Zone melting	(*1394*)
Hypnotics	Chromatographic, thin layer	(*341*)
Ketones	Chromatographic, gas	(*536*)
Monamine oxidase inhibitors	Identity	(*1066*)
Narcotics	Chromatographic, gas	(*735*)
	Chromatographic, paper	(*971*)
Phenothiazines	Chromatographic, gas	(*800*)
	Chromatographic, paper	(*47, 707*)
	Chromatographic, thin layer	(*47*)
	Identity	(*1007*)
Sedatives	Chromatographic, paper	(*643*)
	Chromatographic, thin layer	(*407, 903*)
Steroids	Chromatographic, thin layer	(*1268*)
	Chromatographic, paper	(*477*)
	Spectrophotometric, infrared	(*477*)
Sulfonamides	Chromatographic, paper	(*707, 477*)
	Chromatographic, thin layer	(*1268*)
	Fluorometric	(*1294*)
	Spectrophotometric, infrared	(*477*)
	Volumetric, ox.-red.	(*1341*)
Sympathomimetics	Chromatographic, gas	(*959*)
	Countercurrent distribution	(*660*)
Tranquilizers	Chromatographic, gas	(*957, 959*)
	Chromatographic, paper	(*1359*)
	Spectrophotometric	(*1261*)
Vaccines, adsorbed	Catalytic	(*1425*)
Xanthines	Chromatographic, paper	(*707*)

B. Formulations, mixtures and miscellaneous

Compound or formulation	Method or subject	Reference
Acetone	Volumetric, ox.-red.	(*503*)
Acetazolamide	Polarographic	(*1221*)
Acetonitrile	Use as solvent	(*786*)
Acetylsalicylic acid	Chromatographic, gas	(*499*)
	Chromatographic, ion exchange paper	(*1213*)
	Chromatographic paper	(*298, 1237*)
	Chromatographic, thin layer	(*379*)
	Compatibility	(*463*)
	Identity	(*900*)
	Nuclear magnetic resonance	(*506*)
	Refractometric	(*1042*)
	Volumetric, acid-base	(*699*)

(*Continued*)

Table X. Formulations, Mixtures, and General Methods (*Continued*)

Compound or formulation	Method or subject	Reference
N-Acetylsulfanilamide	Chromatographic, thin layer	(*379*)
Adrenaline	Countercurrent distribution	(*660*)
Aminopyrine	Chromatographic, paper	(*1237, 1357*)
	Chromatographic, thin layer	(*126*)
	Extractive; miscellaneous	(*172, 173*)
	Identity	(*710*)
	Miscellaneous	(*107*)
	Refractometric	(*1042*)
	Volumetric, acid-base	(*201, 1025*)
	Volumetric, complexometric	(*1344*)
	Volumetric, high frequency	(*433*)
p-Aminobenzoic acid esters	Volumetric; complexometric	(*1281*)
p-Aminosalicylate, sodium	Coulometric	(*604*)
Amobarbital	Refractometric	(*1042*)
Amylsil	Volumetric, acid-base	(*357*)
Aniline	Refractometric	(*1042*)
	Volumetric; complexometric	(*1281*)
Antipyrine	Extractive; miscellaneous	(*173*)
	Identity	(*710*)
	Miscellaneous	(*792*)
	Refractometric	(*1042*)
	Volumetric; miscellaneous	(*1344*)
Antrenyl	Volumetric, acid-base	(*357*)
Aprophen	Volumetric, acid-base	(*662*)
Aucubin	Chromatographic, thin layer	(*379*)
Balsum, Peru	Extractive; miscellaneous	(*172*)
Barbital	Extractive; miscellaneous	(*172, 173*)
	Identity	(*900*)
	Refractometric	(*1042*)
Barbital, sodium	Extractive; paper chromatographic	(*298*)
Barbamyl	Colorimetric	(*756*)
Benzacine	Volumetric, acid-base	(*662*)
Belladonna extract	Extractive; miscellaneous	(*172*)
2-Benzyl-2-imidazoline hydrochloride	Volumetric, acid-base	(*1022*)
Benzoate, sodium	Chromatographic, paper	(*551*)
	Volumetric, high frequency	(*433*)
Benzyl mandelate	Chromatographic, thin layer	(*403*)
Bismuth subgallate	Extractive; miscellaneous	(*172, 173*)
Boric acid	Refractometric	(*1243*)
Bromcamphor	Refractometric	(*1042*)
Bromisoval	Refractometric	(*1042*)
Butobarbitone	Chromatographic, partition column	(*1412*)
Caffeine	Chromatographic, gas	(*499*)
	Chromatographic, ion exchange	(*403, 1047, 1448*)
	Chromatographic, paper	(*551, 1213, 1357*)
	Chromatographic, thin layer	(*126, 379, 403, 1047*)
	Coulometric	(*596*)
	Extractive; miscellaneous	(*173, 984*)
	Miscellaneous	(*1269*)
	Nuclear magnetic resonance	(*506*)
	Refractometric	(*918*)
	Volumetric, acid-base	(*673, 1022, 1023*)
	Volumetric, high frequency	(*433*)
Calcium bromide	*Beta*-ray	(*1368*)
Camphor	Refractometric	(*1042*)
Cannabis	Identity	(*211*)
Cetran	Spectrophotometric, ultraviolet	(*780*)
Chloramphenicol	Identity	(*900*)
	Miscellaneous	(*1045*)
Chlorguanide	Identity	(*1007*)
Chlorhexidine	Colorimetric	(*1146*)
Chloroform	Chromatographic, gas	(*497*)
Chlorothiazide	Polarographic	(*1221*)
Chlorphenir-amine	Chromatographic, partition column	(*1095*)
Chlorpromazine	Gravimetric	(*962*)
	Identity	(*1007*)
	Miscellaneous	(*216*)
Chlortetracycline	Colorimetric	(*665*)
	Volumetric, complexometric	(*1297*)
Codeine	Chromatographic, paper ion exchange	(*1213*)
	Chromatographic, thin layer	(*126, 379*)
	Extractive; miscellaneous	(*172*)
	Refractometric	(*1042*)
	Volumetric, high frequency	(*433, 1347*)
Cymarine	Chromatographic, thin layer	(*379*)
Dehydrochloric acid	Identity	(*900*)
Demorphan	Volumetric, acid-base	(*1078*)
Dibucaine	Volumetric, acid-base	(*662*)
Dichloroethane	Use as extractive	(*1142*)
Diethazine	Identity	(*1007*)
	Volumetric, acid-base	(*357*)
Diphylline	Extractive; gravimetric	(*962*)
Dipyrone	Volumetric; complexometric	(*1344*)
Dormutil	Chromatographic, paper	(*1428*)
Ephedrine	Extractive; miscellaneous	(*172, 173, 546*)
Ethylaminobenzoate	Colorimetric	(*769, 1146*)
	Extractive; miscellaneous	(*173*)
	Identity	(*900*)
Ethylmorphine	Colorimetric	(*546*)
	Extractive; miscellaneous	(*172*)
Estradiol benzoate	Spectrographic, ultraviolet	(*780*)
Ethionamide	Extractive; gravimetric	(*962*)
Ferric chloride	Complexometric	(*1181*)
	Refractometric	(*1243*)
Ferrous iron	Complexometric	(*1181*)
Ferrous sulfate	Miscellaneous	(*349*)
Gentisic acid	Spectrophotometric, ultraviolet	(*983*)
Glucose	Extractive; miscellaneous	(*172*)
	Refractometric	(*1243*)
Glycerol	Miscellaneous	(*349*)
	Refractometric	(*1243*)
Halothane	Spectrophotometric, infrared	(*588*)
Hemp, Indian	Miscellaneous	(*216*)
Hexamethylenetetramine	Miscellaneous	(*1269*)
	Refractometric	(*1042*)
Hexobarbital	Identity	(*900*)
Homatropine	Miscellaneous	(*792*)
1-Hydrazino phthalazine	Colorimetric	(*769*)
Hydrochloric acid	Refractometric	(*1243*)
Hydrocortisone acetate	Spectrophotometric, ultraviolet	(*780*)
8-Hydroxyquinoline sulfate	Identity	(*1007*)
Hydroxyzine	Coulometric	(*604*)
Infusions	Chromatographic, adsorption column	(*1331*)
Injectables	Stability	(*720*)
Isoniazid	Coulometric	(*604*)
Isonicotinic acid	Refractometric	(*918*)
Kanamycin	Volumetric, ox.-red.	(*858*)
Lemoran	Volumetric, acid-base	(*1078*)
Magnesium sulfate	Miscellaneous	(*349*)
Meperidine hydrochloride	Spectrophotometric, ultraviolet	(*780*)
Meprobamate	Miscellaneous	(*216*)
Methadone hydrochloride	Extractive; paper chromatography	(*298*)

(*Continued*)

Table X. Formulations, Mixtures, and General Methods (*Continued*)

Compound or formulation	Method or subject	Reference
Methapyrilene hydrochloride	Extractive; paper chromatographic	(*298*)
	Spectrophotometric, ultraviolet	(*351*)
Methylphenobarbitone	Chromatographic, partition column	(*1412*)
Morphine hydrochloride	Chromatographic, thin layer	(*379*)
	Extractive, paper chromatographic	(*298*)
Nicotinic acid	Identity	(*1007*)
Nitrofurantoin	Polarographic	(*1221*)
Norcaine	Gravimetric; volumetric	(*330*)
Papaverine hydrochloride	Chromatographic, ion exchange, thin layer	(*379, 403, 1047*)
	Extractive, miscellaneous	(*107, 172, 173, 298*)
	Gravimetric; volumetric	(*330*)
	Nuclear magnetic resonance	(*506*)
	Volumetric, acid-base	(*201, 1025, 1347*)
Pentinol	Chromatographic, paper	(*1428*)
Pentobarbital	Spectrophotometric, ultraviolet	(*196*)
Pentobarbital calcium	Hydration, crystallinity	(*1320*)
Perparine	Miscellaneous	(*792*)
Phenacetin	Chromatographic, gas	(*499*)
	Chromatographic, ion exchange	(*1047*)
	Chromatographic, paper	(*1237*)
	Chromatographic, paper ion exchange	(*1213*)
	Chromatographic, thin layer	(*126, 379*)
	Extractive; miscellaneous	(*173, 984*)
	Identity	(*900*)
Pheniramine	Chromatographic, partition column	(*738*)
Phenobarbital	Chromatographic, paper	(*1237*)
	Chromatographic, partition column	(*1412*)
	Chromatographic, thin layer	(*126*)
	Colorimetric	(*756*)
	Extractive; miscellaneous	(*107, 173*)
	Identity	(*900*)
	Miscellaneous	(*792*)
	Refractometric	(*1042, 1243*)
	Spectrophotometric, ultraviolet	(*196*)
	Volumetric, acid-base	(*875, 1347*)
Phenobarbital sodium	Extractive; miscellaneous	(*172*)
Phenylpropanolamine	Chromatographic, partition column	(*1095*)
Phenylsalicylate	Spectrophotometric, ultraviolet	(*983*)
	Refractometric	(*1042*)
Phenytoin	Chromatographic, partition column	(*1412*)
Pholedrine	Countercurrent distribution	(*660*)
Phthalazol	Colorimetric	(*769*)
Potassium bromide	Extractive; miscellaneous	(*173*)
	Refractometric	(*1243*)
Potassium iodide	Extractive; miscellaneous	(*173*)
	Refractometric	(*1243*)
Preludin	Miscellaneous	(*216*)
Procaine	Chromatographic, thin layer	(*126*)
	Refractometric	(*918*)
	Volumetric, acid-base	(*662*)
	Volumetric, complexometric	(*1297*)
Progesterone	Spectrophotometric, ultraviolet	(*780*)
Pyridine derivatives	Volumetric; complexometric	(*1281*)
Pyrimethamine	Volumetric, acid-base	(*357*)
Pyruvate isoniazone calcium	Hydration, crystallinity	(*1320*)
Quinidine	Chromatographic, thin layer	(*379*)
Quinine	Chromatographic, thin layer	(*379*)
Quinocide	Volumetric, acid-base	(*357*)
Saffron adulterations	Chromatographic, paper	(*891*)
Salicylic acid	Compatibility	(*463*)
	Spectrophotometric, ultraviolet	(*983*)
Salicylate, sodium	Miscellaneous	(*551, 1269*)
	Volumetric, high frequency	(*433*)
Santonin	Identity	(*900*)
Sodium chloride	Refractometric	(*1243*)
Sodium *p*-chlorobenzoate	*Beta*-ray	(*1368*)
Sodium thiosulfate	*Beta*-ray	(*1368*)
K-Strophanthin-B	Chromatographic, thin layer	(*379*)
K-Strophanthoside	Chromatographic, thin layer	(*379*)
Strychnine hydrochloride	Extractive; chromatographic	(*298*)
Succinyl sulfathiazole	Miscellaneous	(*1045*)
Sulfadiamine	Colorimetric	(*769*)
Sulfamethazine	Identity	(*900*)
	Miscellaneous	(*1045*)
Sulfamethylthiazole	Identity	(*900*)
Sulfanilamide	Chromatographic, thin layer	(*379*)
Sulfapyridine	Chromatographic, thin layer	(*379*)
Sulfathiazole	Chromatographic, thin layer	(*379*)
	Identity	(*900*)
	Miscellaneous	(*107*)
Sulfathiourea	Identity	(*900*)
Suppository mixtures	Miscellaneous	(*105, 107*)
Tetracaine hydrochloride	Volumetric, acid-base	(*662*)
Tetracycline	Colorimetric	(*665*)
Terpin hydrate	Refractometric	(*1042*)
Theobromine	Coulometric	(*551, 596*)
	Volumetric, acid-base	(*875, 1023*)
Tetrahydrozoline	Refractometric	(*1243*)
	Volumetric, acid-base	(*1022*)
Theophylline	Chromatographic, ion exchange	(*1448*)
	Coulometric	(*596*)
	Extractive; miscellaneous	(*172, 173*)
	Volumetric, acid-base	(*1023*)
Tofranil	Miscellaneous	(*216*)
Urotropine	Volumetric, high frequency	(*433*)
Vincamine	Polarographic	(*322*)
Water in drugs	Dielectric	(*388, 1127*)
	Hygroscopicity	(*711*)
	Radiometric	(*628*)
Zinc chloride	Refractometric	*1243*)

A review of methods of identification of 47 narcotic drugs is available (*874*).

A comprehensive survey of the use of EDTA in drug analysis has been made (*163*). Indicators, buffers, masking and demasking techniques, and applications are discussed.

Recent developments in pharmaceutical analysis have been discussed (*986*) and the analytical chemistry of pharmaceuticals, antibiotics, and surfactants presented (*574*). The use of tritium-labeled compounds in pharmaceutical research was reviewed by Snell (*1165*).

Beckett and Stenlake have published a book on the quantitative analytical aspects of pharmaceutical chemistry (*87*). Ashworth has compiled an exhaustive bibliography on titrimetric methods for organic compounds (*39*). The subject material is arranged by compound class and also according to the titrant used. Many references to pharmaceutical products are included.

Papers presented at the 1961 Symposium on Titrimetric Methods have been published in book form (*565*). Several papers are devoted to topics of pharmaceutical interest. A section on the basicity of a large number of excipients used in pharmaceutical formulations is included. By choosing the proper solvent for the titration of a formulation, excipient interference may be minimized or eliminated entirely in some instances.

LITERATURE CITED

(1) Abd El Raheem, A. A., Dikhana, M. M., *Z. Anal. Chem.* **189**, 389–96 (1962); *C.A.* **58**, 3879*b* (1963).

(2) Abdine, H., Abdel Sayed, W. S., *J. Pharm. Pharmacol.* **14**, 761–3 (1962); *C.A.* **58**, 4379*c* (1963).

(3) Adachi, T., *Bitamin* **27**, 433–7 (1963); *C.A.* **60**, 2723*b* (1964).

(4) *Ibid.*, pp. 438–41 (1963).

(5) Adamanis, F., Kucharski, S., *Poznan. Towarz. Przyjaciol Nauk, Wydzial Lekar., Prace Komisji Farm.* **2**, No. 2, 89–91 (1962); *C.A.* **59**, 1440*f* (1963).

(6) *Ibid.*, pp. 93–7 (1962).

(7) Adamanis, F., Pawelczyk, E., Plotkowiakowa, Z., *Farm. Polska* **18**, 180–3 (1962); *C.A.* **58**, 4381*f* (1963).

(8) *Ibid.*, pp. 513–51 (1962); *C.A.* **59**, 3718*h* (1963).

(9) Adamanis, F., Pawelczyk, E., Plotkowiakowa, Z., *Poznan. Towarz. Przyjaciol Nauk., Wydzial Lekar., Prace Komisji Farm.* **1**, No. 3, 3–11 (1962); *C.A.* **59**, 3721*g* (1963).

(10) *Ibid.*, pp. 13–22 (1962); *C.A.* **59**, 2869*a* (1963).

(11) Agrawal, D. K., Sen, R., Uprety, M. C., Sen, N., Mohan Rao, V. K., *Indian J. Technol.* **1**, No. 2, 90–3 (1963); *C.A.* **58**, 12373*a* (1963).

(12) Agrawal, K. C., Dutta, B. N., *Indian J. Appl. Chem.* **24**, 186–92 (1961); *C.A.* **58**, 4377*f* (1963).

(13) Agrawal, K. C., Dutta, B. N., *J. Proc. Inst. Chemists* (*India*) **34**, 73–9 (1962); *C.A.* **57**, 12635*f* (1962).

(14) Akerstrom, S., Lindahl, P.E.B., *Acta Chem. Scand.*, **16** (5), 1206–121, (1962); *Anal. Abstr.* **10**, 335 (1963).

(15) Akita, E., Ikekawa, T., *J. Chromatog.* **12**, (2), 250–1 (1963); *C.A.* **60**, 368*d* (1964).

(16) Alessandro, G., Dal Brollo, F., Mecarelli, E., *Boll. Chim. Farm.* **101**, 34–40 (1962); *Anal. Abstr.* **10**, 1568 (1963).

(17) Alexander, T. G., *J. Assoc. Offic. Agr. Chemists* **46**, 634–6 (1963).

(18) Alexander, T. G., *Ibid.*, pp. 902–4.

(19) Alexander, T. G., *J. Pharm. Sci.* **51**, (7), 702–703 (1962).

(20) Alexander, T. G., *Ibid.*, **52** (9), 910–12 (1963).

(21) Algeri, E. J., Katsas, G. G., Luongo, M. A., *Amer. J. Clin. Path.* **38** (2), 125–130 (1962).

(22) Allen, J., Gartside, B., Johnson, C. A., *J. Pharm. Pharmacol. Suppl.* **14**, 73–6 (1962).

(23) Amer, M. M., Hebeka, E. K., Said, F., *Egypt. Pharm. Bull.* **42**, No. 4, 233–41 (1960); *C.A.* **58**, 8854*d* (1963).

(24) *J. Pharm. Sci.* **53** (1), 98–100 (1964).

(25) Anatanarayanan, V. G., Kudalkar, V. G., Madiwale, M. S., Desai, H. H., Walawalkar, M. B., *Indian J. Pharm.* **21**, 263–6 (1959); *C.A.* **58**, 7786*h* (1963).

(26) Anders, M. W., Mannering, G. J., *J. Chromatog.* **7** (2), 258–260 (1962).

(27) Andersen, H. E., *Dansk Tidsskr. Farm.* **38** (1), 1–17 (1964); *C.A.* **60**, 7871*b* (1964).

(28) Andreeva, L. G., Figurovskii, N. A., *Zh. Analit. Khim.* **17**, No. 1, 105–8 (1962); *C.A.* **57**, 6027*d* (1962).

(29) Angadji, E., Colleter, J. C., *Bull. Soc. Pharm. Bordeaux* **101**, No. 3, 147–58 (1962); *C.A.* **58**, 8851*g* (1963).

(30) Anger, V., Ofri, S., *Talanta* **10** (12), 1302–3 (1963).

(31) Anker, L., Sonanini, D., *Pharm. Acta Helv.* **37** (5–8), 360–378 (1962); *C.A.* **57**, 1131*d* (1962).

(32) Apreotesei, C., Teodosin, M., *Farmacia* **10**, 321–30 (1962); *C.A.* **58**, 4375*b* (1963).

(33) Ardry, R., *Ann. Pharm. Franc.* **21** (1), 27–29 (1963); *Anal. Abstr.* **10**, 5364 (1963).

(34) Ardry, R., *Ibid.*, pp. 31–35; *Anal. Abstr.* **10**, 5373 (1963).

(35) Ariesan, V., Danciu, F., Ban, C., *Farmacia* **11** (7), 385–92 (1963); *C.A.* **60**, 9101*h* (1964).

(36) Artamonov, B. P., Bugreeva, E. V., *Med. Prom. SSSR* **16**, No. 12, 30–4 (1962); *C.A.* **58**, 10039*a* (1963).

(37) Asahi, Y., *Chem. Pharm. Bull.* **11** (10), 1241–6 (1963); *C.A.* **60**, 5283*c* (1964).

(38) Asahi, Y., *Takeda Kenkyusho Nempo* **20**, 30–2 (1961); *C.A.* **55**, 412*e* (1961).

(39) Ashworth, M. R. F., "Titrimetric Organic Analysis," Part I. Direct Methods, Interscience, New York, 1964.

(40) Auber, L., Novotny, A., *Gyogyszereszet* **8**, 25–7 (1964); *C.A.* **60**, 9102*d* (1964).

(41) Audisio, G., Ruggieri, R., *Giorn. Med. Mil.* **113**, (1), 50–3 (1963); *C.A.* **59**, 2592*h* (1963).

(42) Auerbach, M. E., Hefferren, J. J., Koehler, H. M., Pratt, E. L., *J. Pharm. Sci.* **51** (5), 491–3 (1962).

(43) Auterhoff, H., Sachdev, R., *Apotheker-Ztg.* **102**, No. 30, 921–23 (1962); *C.A.* **57**, 15240*i* (1962).

(44) Avetisyan, F. O., Chekaida, S. G., *Sb. Nauchn. Tr. Inst. Avtomatiki Gosplana Ukr. SSR* **1961**, No. 2, 177–85; *C.A.* **58**, 8854*c* (1963).

(45) Awe, W., Kienert, J., *Arch. Pharm., Berlin* **295** (10); *Mitt. dtsch. Pharm. Ges.* **32** (10), 173–176 (1962); *C.A.* **58**, 4376*f* (1963).

(46) Awe, W., Kienert, H. J., *Pharm. Acta Helv.* **38** (11), 805–11 (1963); *C.A.* **60**, 5280*d* (1964).

(47) Awe, W., Schulze, W., *Pharm. Zentralhalle* **107**, 1333–9 (1962); *C.A.* **58**, 4376*a* (1963).

(48) Awe, W., Tracht, H. G., *Pharm. Ztg. Ver. Apotheker-Ztg.* **107**, 429–32 (1962); *C.A.* **57**, 16749*c* (1962).

(49) Awe, W., Weigrebe, W., El-Shibini, H. A. M., Su, M. E., *Naturwissenschaften* **49** (23), 540 (1962); *Anal. Abstr.* **10**, 4352 (1963).

(50) Babini, B., Paolucci, G., Salvioli, G. P., Manfredi, G., Corsini, F., *Boll. Soc. Ital. Biol. Sper.* **38** (15), 728–729 (1962); *C.A.* **58**, 6070*g* (1963).

(51) Bachmann, C., *Pharm. Praxis, Beilage Pharmazie* **1961**, No. 10, 178–80; *C.A.* **57**, 6025*h* (1962).

(52) Bachmann, C., *Ibid.*, **1962**, No. 8, 131–3; *C.A.* **57**, 15241*i* (1962).

(53) Backe-Hansen, K., *Medd. Norsk Farm. Selskap* **24**, 9–30 (1962); *C.A.* **57**, 13885*g* (1962).

(54) Badoz-Lambling, J., Stojkovic, D., *Bull. Soc. Chim. France* **1963** (12), 2709–12; *C.A.* **60**, 9102*a* (1964).

(55) Svendsen, A. B., Kolboe, T., *Medd. Norsk Farm. Selskap.* **25** (8), 117–23 *C.A.* **60**, 3949*h* (1964).

(56) Bagdanova, V. N., *Tr. 1-go Mosk. Med. Inst.* **17**, 170–2 (1962); *C.A.* **59**, 3719*h* (1963).

(57) Bahjat, K. S., *J. Pharm. Sci.* **52** (10), 1006–7 (1963).

(58) Ballard, B. E., Nelson, E., *Ibid.*, **51** (10), 1009 (1962).

(59) Bami, H. L., *J. Proc. Inst. Chemists* **34**, 130–3 (1962); *C.A.* **58**, 4377*b* (1963).

(60) Bandyukova, V. A., *Uch. Zap., Pyatigorskii Gos. Farmatsevt. Inst.* **5**, 185–8 (1961); *C.A.* **59**, 2593*c* (1963).

(61) Banshchikova, T. M., Tabatarovich, A. K., *Materialy po Primeneniyu Lyumenestsentn. Analiza v. Med., Chita* **1960**, 17–21; *C.A.* **57**, 2334*f* (1962).

(62) Barakat, M. Z., Shaker, M., *Analyst* **89** (1056), 216–19 (1964).

(63) Barcza, Lajos, *Acta Pharm. Hung.* **33** (6), 266–70 (1963); *C.A.* **60**, 6703*e* (1964).

(64) Barilari, E. M., Katz, M., *Arch. Bioquim., Quim. Farm., Tucuman* **9**, 75–84 (1961); *C.A.* **57**, 12814*c* (1962).

(65) Barker, H. E. R., *Australian J. Pharm.* **43**, 790–2 (1962); *C.A.* **58**, 11170*h* (1963).

(66) Baroncini, O., *Attualita Lab.* **7**, 92–9 (1961); *C.A.* **57**, 15244*f* (1962).

(67) Barrett, C. B., Dallas, M. S., Padley, F. B., *Chem. Ind.* **1962**, 1050–1.

(68) Bartkowicz, S., *Dissertationes Pharm.* **14**, 189–96 (1962); *C.A.* **50**, 411*e* (1963).

(69) Bartlett, M. F., Sklar, R., Taylor, W. I., Schlittler, E., Amai, R. L. S., Beak, Peter, Bringi, N. V., Wenkert, E., *J. Am. Chem. Soc.* **84**, 622–30 (1962).

(70) Bartos, J., *Ann. Pharm. Franc.* **20**, 478–9 (192); *C.A.* **57**, 15243*i* (1962).

(71) Basu, K., Dutta, B. N., *Indian J. Pharm.* **24**, 176–80 (1962); *C.A.* **58**, 4379*b* (1963).

(72) Basu, K., Dutta, B. N., *J. Instn. Chem., India* **34** (3), 142–159 (1962); *Anal. Abstr.* **10**, 2452 (1963).

(73) Basu, N. K., Nasipuri, R. N., *Indian J. Pharm.* **25** (1), 6–7 (1963); *Anal. Abstr.* **10**, 5358 (1963).

(74) Bauer, W. P., Fondi, M., *Sci. Pharm.* **30**, 173–82 (1962); *C.A.* **58**, 4381*d* (1963).

(75) Baumgarten, G., Nover, L., *Arch. Pharm., Berlin* **295** (4), 311–313 (1962); *Anal. Abstr.* **9**, 4455 (1962).

(76) Baumler, J., Rippstein, S., *Helv. Chim. Acta* **44** (7), 2208–2210 (1961). *Anal. Abstr.* **9**, 4905 (1962).

(77) Bayer, E., *Gyogyszereszet* **8**, 21–4 (1964); *C.A.* **60**, 9101*b* (1964).

(78) Bayer, I., Katona, K., *Acta Pharm. Hung.* **28**, 216–18 (1958); *C.A.* **57**, 6024*i* (1962).

(79) Bayer, I., *Ibid.*, **29**, 53–63 (1959); *C.A.* **57**, 9955*c* (1962).

(80) Bayer, J., *Chimia* **15**, 555 (1961); *C.A.* **57**, 11316*a* (1962).

(81) Bayer, J., *J. Chromatog.* **8** (1), 123–125 (1962).

(82) Bayer, J., Liptay, G., *Z. Anal. Chem.* **191**, 335–8 (1962); *C.A.* **58**, 5457*b* (1963).

(83) Bebesel, P., Sirbu, I., *Acad. R.P.R., Std. Cercet. Chim.* **9** (2), 351–355 (1961); *Anal. Abstr.* **9**, 2886 (1962).

(84) Beckett, A. H., Choulis, N. H., *J. Pharm. Pharmacol. Suppl.* **15**, 236–45 (1963); *C.A.* **60**, 3950*a* (1964).

(85) Becker, A., Ehinger, F., *Z. Anal. Chem.* **187**, 110–14 (1962); *C.A.* **57**, 12637*b* (1962).

(86) Becker, A., Ehinger, F., *Ibid.*, **198** (2), 162–170 (1963); *C.A.* **60**, 10478*d* (1964).

(87) Beckett, A. H., Stenlake, J. B., "Practical Pharmaceutical Chemistry, Quantitative Analysis.," The Athlone Press, London, 1962.

(88) Bednar, J., *Pharmazie* **17**, 76–80 (1962); *Anal. Abstr.* **10**, 2878 (1963).

(89) Beekman, S. M., *J. Pharm. Sci.* **51**, 675–9 (1962).

(90) Beerthuis, R. K., Recourt, J. H., *Colloq. Intern. Centre Natl. Rech. Sci.* (*Paris*) **99**, 205–19 (1961); *C.A.* **58**, 5751*c* (1963).

(91) Beggs, B. H., Spencer, R. D., ANAL. CHEM. **34**, 1590–1594 (1962).

(92) Beijleveld, W. M., *Pharm. Weekbl.* **97** (6), 190–193 (1962); *Anal. Abstr.* **9**, 5377 (1962).

(93) Belen'kaya, S. L., Tikhomirova, G. P., *Khlebopekar. i Konditer. Prom.* **6**, No. 4, 3–4 (1962); *C.A.* **57**, 6028 (1962).

(94) Belikov, V. G., *Med. Prom. SSSR* **17** (9), 32–5 (1963); *C.A.* **60**, 5281*c* (1964).

(95) Belikov, V. G., *Uch. Zap., Pyatigorskii Gos. Farmatsevt. Inst.* **5**, 189–92 (1961); *C.A.* **59**, 1441*c* (1963).

(96) Bellomonte, G., *R. C. Ist. Sup. Sanit.* **25** (7, 8, 9), 581–590 (1962); *C.A.* **58**, 12370*h* (1963).

(97) Benes, I., Burianek, J., Cifka, J., *Ceskosl. Farm.* **11** (6), 281–286 (1962); *Anal. Abstr.* **10**, 329 (1963).

(98) Benes, I., Burianek, J., Cifka, J., *Congr. Sci. Farm., Conf. Comun.* **21**, Pisa 1961, 183–92; *C.A.* **59**, 383*b* (1963).

(99) Benesova, B., *Czech.* **103,556**, May 15, 1962; *C.A.* **58**, 8859*f* (1963).

(100) Benesova, B., *Ceskosl. Farm.* **11** (4), 199–205 (1962); *Anal. Abstr.* **9**, 4880 (1962).

(101) Bennett, R. D., Heftmann, E., *J. Chromatog.* **9**, 348–352 (1962).

(102) *Ibid.*, pp. 353–358.

(103) *Ibid.*, pp. 359–362.

(104) Beral, H., Demetrescu, E., Grintescu, P., Calafeteanu, I., *Farmaca Ed. Prat.* **18** (10), 544–8 (1963); *C.A.* **60**, 369*h* (1964).

(105) Beral, H., Demetrescu, E., Grintescu, P., Calafeteanu, I., Stoicescu, V., *Pharm. Zentralhalle* **101**, 754–7 (1962); *C.A.* **59**, 3720*a* (1963).

(106) Beral, H., Popescu, D., Constantinescu, T., *Ibid.*, **101** 631–635 (1962); *Anal. Abstr.* **10**, 3423 (1963).

(107) Beral, H., Demetrescu, E., Stoicescu, V., Grintescu, P., Calafeteanu, I., *Ibid.*, **101**, 260–5 (1962); *C.A.* **58**, 3269*g* (1963).

(108) Beral, N., Murea, L., Wermescher, B., *Rev. Chim.* (*Bucharest*) **13**, 239–40 (1962); *C.A.* **57**, 16751*h* (1962).

(109) Beran, M., Semonsky, M., *Ceskosl. Farm.* **11** (9), 440–451 (1962); *Anal. Abstr.* **10**, 3413 (1963).

(110) Bergel'son, L. D., Dyatlovitskaya, E. V., Voronkova, V. V., *Dokl. Akad. Nauk SSSR*, **141** (1), 84–86 (1961); *Anal. Abstr.* **9**, 5243 (1962).

(111) Bergy, M. E., Eble, T. E., Herr, R. R., *Antibiot. Chemotherapy* **11**, 661–1 (1961); *C.A.* **57**, 4766*f* (1962).

(112) Bermudez, D. N., Meier, G. N., Brieva, A. J., *Anales Real Acad. Farm.* **24** (1–2), 35–49 (1963); *C.A.* **60**, 3949*g* (1964).

(113) Bernshtein, V. N., Chuiko, I. V., *Uch. Zap. Pyatigorskii Gos. Farmatsevt. Inst.* **5**, 193–8 (1961); *C.A.* **59**, 1440*g* (1963).

(114) Bernshtein, V. N., Godyatskii, V. E., *Ibid.*, **5**, 153–6 (1961); *C.A.* **59**, 1439*b* (1963).

(115) Bernshtein, V. N., Luk'yanchikova, G. I., *Ibid.*, **5**, 165–9 (1961); *C.A.* **59**, 2592*f* (1963).

(116) *Ibid.*, pp. 157–64; *C.A.* **59**, 3720*b* (1963).

(117) Bertucat, M., Mesnard, P., Sylla, O., *Bull. Soc. Pharm. Bordeaux* **101** (1), 37–40 (1962); *Anal. Abstr.* **10**, 1985 (1963).

(118) Bestova, J., *Chem. Zvesti* **17**, 672–5 (1963); *C.A.* **60**, 1540*a* (1964).

(119) Betina, V., *Ibid.*, **15**, 848–52 (1961); *C.A.* **58**, 5453*c* (1963).

(120) *Ibid.*, pp. 859–64.

(121) Betina, V., *Some Gen. Probl. Paper Chromatog., Symp. Liblice, Czech.* **1961**, 153–6; *C.A.* **59**, 11188*c* (1963).

(122) Betina, V., Nemec., P., *Chem. Zvesti* **15**, 853–8 (1961); *C.A.* **58**, 5453*c* (1963).

(123) Betts, T. J., *Pharm. J.* **189**, 479–81 (1962); *C.A.* **58**, 4372*h* (1963).

(124) Beyrich, T., *J. Chromatog.* **13** (1), 181–7 (1964).

(125) Beyrich, T., *Pharmazie* **17**, 280–2 (1962); *C.A.* **57**, 8658*e* (1962).

(126) Bican-Fister, T., *Acta Pharm. Jug.* **12** (2), 73–78 (1962); *Anal. Abstr.* **10**, 2450 (1963).

(127) Bican-Fister, T., *J. Pharm. Pharmacol.* **14**, 280–3 (1962).

(128) Bican-Fister, T., Andrec, A., *J. Pharm. Sci.* **52** (2), 219–20 (1964).

(129) Bican-Fister, T., Kajganovic, V., *J. Chromatog.* **11** (4), 492–5 (1963).

(130) Bresemeyer, M. E., *J. Assoc. Offic. Agr. Chemists* **45**, 593–4 (1962).

(131) Bilinska-Kozicka, U., Terpilowski, J., *Dissert. Pharm., Krakow* **14** (4), 467–472 (1962); *Anal. Abstr.* **10**, 3852 (1963).

(132) Bird, R. F., Stevens, S. G. E., *Analyst* **87**, 362–365 (1962).

(133) Birinboim, S. M., *Farmatsevt. Zh.* (*Kiev*) **16**, No. 5, 34–8 (1961); *C.A.* **59**, 383*a* (1963).

(134) Birinboim, S. M., *Ibid.*, **18** (5), 41–4 (1963); *C.A.* **60**, 6704*e* (1964).

(135) Birkinshaw, V. J., Smith, K. L., *J. Pharm. Pharmacol.* **14** 95T-96T (1962).

(136) Birner, J., *Australian J. Sci.* **25**, 416 (1963); *C.A.* **59**, 3718*d* (1963).

(137) Biryukova, I. N., *Aptechn. Delo* **10**, No. 5, 35–42 (1961); *C.A.* **57**, 2335*g* (1962).

(138) Bjorling, C. O., Berggren, A., Nygard, B., *Acta Chem. Scand.* **16**, 1481–92 (1962); *Anal. Abstr.* **10**, 786 (1963).

(139) Bjorling, C. O., Willman-Johnson, B., *Ibid.*, **17** (10), 2638–43 (1963); *C.A.* **60**, 10475*g* (1964).

(140) Blagojevic, Z., Bunovic, M., Bradic, P., *Acta Pharm. Jugoslav.* **9**, 93–6 (1959); *C.A.* **58**, 4378*f* (1963).

(141) Blake, M. I., Nona, D. A., *J. Pharm. Sci.* **52** (10), 945–8 (1963).

(142) Blake, M. I., Siegel, F. P., *Ibid.*, **51** (10), 944–946 (1962).

(143) Blattna, J. Deyl, Rosmus, J., *Mikro-chim. Acta* **1962** (5), 868–873; *Anal. Abstr.* **10**, 824 (1963).

(144) Blaug, S. M., Ebersman, D. S., *J. Pharm. Sci.* **53** (1), 35–9 (1964).

(145) Blazek, J., *Farmatsevt. Zh.* **17**, 9–13 (1962); *C.A.* **59**, 9740*f* (1963).

(146) Blazek, J., Kracmar, J., *Ceskosl. Farm.* **11** (4), 210–212 (1962); *Anal Abstr.* **9**, 4914 (1962).

(147) Blazek, J. Spinkova, V., Stejskal, Z., *Pharmazie* **17**, 497–514 (1962); *C.A.* **58**, 5454*g* (1963).

(148) Blinov, N. O., Yakubov, G. Z., Khokhlova, Yu. M., *J. Chromatog.* **8**, 522–5 (1962).

(149) Bobranski, B., Syper, L., *Microchem. J. Symp. Ser.* **2**, 749–56 (1962); *C.A.* **58**, 6647*a* (1963).

(150) Bobtelsky, M., Barzily, I., *Anal. Chim. Acta* **28** (1), 82–90 (1963); *Anal. Abstr.* **10**, 5236 (1963).

(151) Boeckel, C. R. C., *Sem. Med., Buenos Aires*, **120**, 344–345 (1962); *Anal. Abstr.* **10**, 4420 (1963).

(152) Boehme, H., Kreutzig, L., *Deut. Apotheker-Ztg.* **103** (17), 505–8 (1963); *C.A.* **59**, 2590*g* (1963).

(153) Bogdanski, K., Jeszka, J., Kenner, K., *Chem. Anal.* (*Warsaw*) **5**, 781–9 (1960); *C.A.* **57**, 4042*d* (1962).

(154) Bogs, U., *Pharm. Praxis, Beilage Pharmazie* **1962** (3), 43–44; *Anal. Abstr.* **10**, 3844 (1963).

(155) Bohley, P., *Naturwissenschaften* **49** (14), 326 (1962); *Anal. Abstr.* **10**, 1127 (1963).

(156) Boitsov, E. N., Finkel'shtein, A. I., *Zhur. Anal. Khim.* **17** (6), 748–750 (1962); *C.A.* **58**, 3889*b* (1963).

(157) Bolton, S., *J. Pharm. Sci.* **52** (11), 1071–4 (1963).

(158) D'Alessio de Carnevale Bonino, R. C., *Publ. Inst. Invest. Microquim., Univ. Nacl. Litoral* (*Rosario, Arg.*) **24**, No. 22, 83–93 (1958); *C.A.* **58**, 3873*f* (1963).

(159) *Ibid.*, pp. 94–8 (1958); *C.A.* **57**, 15243*g* (1962).

(160) Bonner, W. A., DeGraw, J. I., *J. Chem. Educ.* **39** (12), 639–640 (1962).

(161) Bonnischsen, R., Maehly, A. C., Frank, A., *J. Forensic Sci.* **6**, 411–443 (1961).

(162) Boon, P. F. G., *J. Pharm. Pharmacol. Suppl.* **14**, 116–18 (1962).

(163) Borchert, O., *Pharmazie* **16**, 181–96 (1961); *C.A.* **57**, 2334*d* (1962).

(164) Borg, D. C., Cotzias, G. C., *Proc. Natl. Acad. Sci. U. S.* **48**, 617–23 (1962).

(165) *Ibid.*, pp. 623–42.

(166) *Ibid.*, pp. 643–52.

(167) Bors, L., *Acad. Rep. Populare Romine, Filiala Iasi, Studii Cercetari Stiint Chim.* **13** (2), 277–82 (1962); *C.A.* **59**, 11188*g* (1963).

(168) Bose, B. C., Vijayvargiya, R., *J. Pharm. Pharmacol.* **14** (1), 58–60 (1962).

(169) Bose, P. C., Singh, R. K., Ray, G. K., Mukerji, B., *J. Sci. Ind. Res.* (*India*) **210**, 386–7 (1962); *C.A.* **58**, 3270*g* (1963).

(170) Bouw, P. S., *Suara Pharm. Madjalah* **4**, No. 2, 41–4 (1959); *C.A.* **58**, 12369*d* (1963).

(171) Boyd, G. S., Hutton, H. R. B., *Biochim. Biophys. Act.* **69** (2), 419–420 (1963); *Anal. Abstr.* **10**, 2527 (1963).

(172) Bozsai, G., Dupcza, K., Kelemen-Kuettel, I., Mosonyi, M., Zabrak, D., *Gyogyszereszet* **7**, 199–203 (1963); *C.A.* **60**, 3947*f* (1964).

(173) Bozsai, I., Dupcza, K., Kelemen-Kuettel, I., Mozsonyi-Sata, L., *Ibid.*, **3**, 139–43 (1959); *C.A.* **58**, 6647*d* (1963).

(174) Bozsai, Mrs. Imre, Mosonyi, Mrs. Mihaly, *Acta Pharm. Hung.* **32**, 255–9 (1962); *C.A.* **58**, 6649*b* (1963).

(175) Bradshaw, W. H., Douglas, J. F., ANAL. CHEM. **34** 1172 (1962).

(176) Braeuniger, H., Moede, F., *Pharm. Zentralhalle* **101**, 383–9 (1962); *C.A.* **58**, 8855*b* (1963).

(177) Braeckman, P., van Severen, R., De Jaeger-Van Moeseke, L., *Pharm. Tijdschr. Belg.* **40** (7), 113–28 (1963); *C.A.* **60,** 1541*f* (1964).
(178) Brandstaetter-Kuhnert, M., Hoffmann, R., Senn, M., *Microchem. J.* **7,** 357–74 (1963); *C.A.* **60,** 3948*c* (1964).
(179) Brandt, R., Olsen, M. J., Cheronis, N. D., *Science* **139** 1063–1064 (1963).
(180) Brazhnikova, M. G., Kudinova, M. K., *Nature* **200** (4902), 167–8 (1963).
(181) Breyer, B., Biegler, T., *Collection Czech. Chem. Commun.* **25,** 3348–55 (1960); *C.A.* **57,** 10926*d* (1962).
(182) Brierley, A., Langbridge, D. M., *Analyst* **86,** 709–13 (1961).
(183) Brink, H., *Pharm. Weekbl.* **97** (14), 505–507 (1962); *Anal. Abstr.* **10,** 327 (1963).
(184) Brochmann-Hanssen, E., *J. Pharm. Sci.* **51,** 1017–31 (1962).
(185) Brochmann-Hanssen, E., Baerheim Svendsen, A., *Ibid.*, **51,** 318–21 (1962).
(186) *Ibid.*, p. 393.
(187) *Ibid.*, pp. 938–941.
(188) *Ibid.*, pp. 1095–8.
(189) Brochmann-Hanssen, E., Baerheim Svendsen, A., *Ibid.*, **52** (12), 1134–6 (1963).
(190) Brodasky, T. F., Anal. Chem. **35,** 343–5 (1963).
(191) Brodasky, T. F., *J. Pharm. Sci.* **52,** 233–5 (1963).
(192) Brodasky, T. F., Lummis, W. L., *Ibid.*, **52,** 230–2 (1963).
(193) Brown, E. R., *J. Pharm. Pharmacol.* **15** (6), 379–85 (1963).
(194) Brown, J. K., Malone, M. H., Stuntz, D. E., Tyler, V. E., *J. Pharm. Sci.* **51,** 853–6 (1962).
(195) Brown, K. S., Kupchan, S. M., *J. Chromatog.* **9** (1) 71–80 (1962).
(196) Brown, T. L., *J. Pharm. Sci.* **52,** 274–5 (1963).
(197) Brownell, W. B., Chadde, F. E., Theivagt, J. G., Wimer, D. C., Anal. Chem. **35,** 143R–160R (1963).
(198) Bruinvels, J., *Experientia* **19** (10), 551–2 (1963); *C.A.* **60,** 1540*e* (1964).
(199) Brunjes, S., Wybenga, D., Johns, V. J., Jr., *Clin. Chem.* **10** (1), 1–12 (1964); *C.A.* **60,** 7872*h* (1964).
(200) Bruno, S., *Boll. Chim. Farm.* **102,** 478–80 (1963); *C.A.* **59,** 15123*a* (1963).
(201) Buda, L., *Gyogyszereszet* **4,** 217–18 (1960); *C.A.* **60,** 5281*d* (1964).
(202) Budimlic, B., Dumanovic, D., *Glasnik Hem. Drustva, Beograd* **27** (5–6), 293–9 (1962); *C.A.* **59,** 12594*d* (1963).
(203) Bukowska, H., Bielowska, M., *Acta Polon. Pharm.* **19** (5), 417–420 (1962); *Anal. Abstr.* **10,** 3881 (1963).
(204) Bulant, V., Parizkova, H., *Sb. Praci 10 (Desetin) Vyroci Zalozeni Ustavu* **1962,** 76–80; *C.A.* **59,** 11189*e* (1963).
(205) Bulenkov, T. I., *Med. Prom. SSSR* **17** (9), 26–9 (1963); *C.A.* **60,** 5280*e* (1964).
(206) Bullerwell, R. A. F., *J. Polarographic Soc.* **8** (1), 2–9 (1962).
(207) Burger, K., *Acta Pharm. Hung.* **32,** 180–7 (1962); *C.A.* **57,** 13892*f* (1962).
(208) Burger, K., *Magy. Kem. Folyoirat* **68,** 474–9 (1962); *C.A.* **58,** 7788*c* (1963).
(209) Bussmann, G., *Pharm. Acta Helv.* **39** (9), 690–701 (1963); *C.A.* **59,** 15121*d* (1963).
(210) Busson, F., Carbiener, R., Lanza, J., *Cahiers Tech. Centre Natl. Coord. Etudes Rech. Nutr. Aliment.* **5,** 36 pp. (1960); *C.A.* **57,** 12812*a* (1962).
(211) Butler, W. P., *J. Assoc. Offic. Agr. Chemists* **45,** 597–9 (1962).
(212) Byrom, P., Turnbull, J. H., *Talanta* **10** (12), 1217–21 (1963).
(213) Callahan, J. J., Litterio, F., Britt, E., Rosen, B. D., Owens, J., *J. Pharm. Sci.* **51,** 333–5 (1962).
(214) Calo, A., Cardini, C., Mariani, A., *Farm. Aikakauslehti* **72,** 286–92 (1963); *C.A.* **60,** 375*b* (1964).
(215) Campbell, J. A., *J. Pharm. Sci.* **51,** 270–2 (1962).
(216) Cann, F. L., *J. Forensic Sci. Soc.* **3,** 33–6 (1962); *C.A.* **58,** 3271*b* (1963).
(217) Canterelli, G., *Farmaco, Ed. Prat.* **17** (12), 728–731 (1962); *Anal. Abstr.* **10,** 4358 (1963).
(218) Cardini, C., Cavina, G., Cingolani, E., Mariani, A., Vicari, C., *Ibid.*, **17** (10), 583–592 (1962); *Anal. Abstr.* **10,** 4423 (1963).
(219) Carni-Catsadima, I., *Arch. Inst. Pasteur Hellenique* **7,** 123–4 (1961); *C.A.* **57,** 2857*g* (1962).
(220) Carro, C. R., Suarez, M. L., *Galenica Acta (Madrid)* **14,** 517–21 (1961); *C.A.* **57,** 6028 (1962).
(221) Casey, P. J., Wagner, W., *Proc. Penn. Acad. Sci.* **36,** 281–5 (1962).
(222) Casinelli, J. L., Sinsheimer, J. E., *J. Pharm. Sci.* **51,** 336–8 (1962); *C.A.* **57,** 960*f* (1962).
(223) Castillo, P. M., Leon, Benjamin, *Anales. Real Acad. Farm.* **24** (1–2), 31–3 (1963); *C.A.* **60,** 1541*a* (1964).
(224) Castren, E., *Farm. Aikakauslehti* **71,** 351–7 (1962); *C.A.* **58,** 8858*f* (1963).
(225) Gavina, G., Cingolani, E., Amormino, V., Giraldez, A., *Farmaco (Pavia) Ed. Prat.* **17,** 393–403 (1962); *C.A.* **57,** 16757*e* (1962).
(226) Cavina, G., Cingolani, E., Giraldez, A., *Ibid.*, **17** (3), 149–159 (1962); *Anal. Abstr.* **9,** 4423 (1962).
(227) Caws, A. C., Lawrence, B. E., *J. Pharm. Pharmacol. Suppl.* **14,** 59–62 (1962); *C.A.* **58,** 7785*d* (1963).
(228) Ceausescu, D., *Farmacia (Bucharest)* **10,** 543–9 (1962); *C.A.* **58,** 7787*g* (1963).
(229) Cecy, C., *Tribuna Farm. (Brazil)* **31,** 2–12 (1963); *C.A.* **60,** 1542*d* (1964).
(230) Ceglarski, R., Roman, J., *Farm. Polska* **18** (14), 327–329 (1962); *Anal. Abstr.* **10,** 1172 (1963).
(231) Celap, M. B., Janjic, T. J., Jevtic, V. D., *Mikrochim. Ichnoanal. Acta* **1963** (5–6), 1043–5; *C.A.* **60,** 6702*a* (1964).
(232) Celeste, A., Turczan, J., *J. Assoc. Offic. Agr. Chemists* **46,** 1055–9 (1963).
(233) Cerri, O., Maffi, G., *Boll. Chim. Farm.* **100,** 940–50 (1961); *C.A.* **57,** 11467*i* (1962).
(234) *Ibid.*, pp. 951–4.
(235) *Ibid.*, pp. 954–5.
(236) Cerutti, G., Vezzini, W., *Chim. Ind. (Milan)* (**43**), 784–5 (1961); *C.A.* **60,** 13890*f* (1964).
(237) Chafetz, L., *J. Pharm. Sci.* **52** (12), 1193–5 (1963).
(238) Chakrabarti, J. K., *J. Chromatog.* **8** (3), 414–416 (1962).
(239) Chang, J. C., Honig, A. B., Warren, A. T., Levine, S., *J. Pharm. Sci.* **52,** 603 (1963).
(240) Chatten, L. G., "Titrimetric Methods," *Proc. Symp. Cornwall, Ont., Can.* **1961,** 129–70; *C.A.* **57,** 7385*e* (1962).
(241) Chatten, L. G., Anderson, J. H., *Can. Pharm. J., Sci. Sect.* **96,** No. 1, 73–5 (1963).
(242) Chatten, L. G., Harris, L. E., Anal. Chem. **34** (11), 1495–1501 (1962).
(243) Chatten, L. G., Mainville, C. A., *J. Pharm. Sci.* **52,** 146–9 (1963).
(244) Cheeseman, G. W. H., Poller, R. C., *Analyst* **87,** 366–368 (1962).
(245) Ch'en, Chiung-Hsueh, *J. Taiwan Pharm. Assoc.* **13,** 2–9 (1961); *C.A.* **58,** 3274*b* (1963).
(246) Cheneau, M., Guiochon, G., *Bull. Soc. Chim. France* **1963** (11), 2601–6; *C.A.* **60,** 5276*g* (1964).
(247) Chiang, H. C., Chen, W. F., *J. Pharm. Sci.* **52,** 808–9 (1963).
(248) Chin, L., Picchioni, A. L., Childs, R. F., *J. Pharm. Sci.* **52** (9), 907–9 (1963).
(249) Chulski, T., Lamb, D., *Ibid.*, **52,** 169–71 (1963).
(250) Cieplinski, E. W., Anal. Chem. **35,** 256–7 (1963).
(251) Cieszynski, T., *Farm. Polska* **18,** No. 3, 55–7 (1962); *C.A.* **57,** 12637*h* (1962).
(252) Cima, L., Mantovan, R., *Farmaco (Pavia) Ed. Prat.* **17,** 473–81 (1962); *C.A.* **57,** 16986*b* (1962).
(253) Ciogolea, Gh., Creanga, S., Mihai, T., Petroniu, L., *Ann. Pharm. Franc.* **21** (9–10), 681–4 (1963); *C.A.* **60,** 5277*b* (1964).
(254) Ciogolea, Gh., Morait, Gh., Baloescu, C., Teodorescu, N., Creanga, Silvia, Tudor, M., Petroniu, Laurentia, Ionescu, Georgeta, *Farmacia (Bucharest)* **10,** 257–61 (1962); *C.A.* **58,** 4379*g* (1963).
(255) Ciurlo, R., *Rass. Chim.* **14,** No. 3, 103–4 (1962); *C.A.* **57,** 16749*i* (1962).
(256) Claeys, P. B. C., Delcambre, J. P., Traisnel, M., *Bull. Soc. Chim. France* **1962,** 1654–7; *C.A.* **58,** 3269*h* (1963).
(257) Clair, E. G., *Analyst* **87,** 499–500 (1962).
(258) Clarke, E. G. C., Kalayci, S., *Nature* **198,** 783 (1963).
(259) Cochin, J., Daly, J. W., *Experientia* **18** (6), 294–295 (1962); *Anal. Abstr.* **10,** 275 (1963).
(260) Coeur, A., Alary, J., Bauduin, N., *Boll. Chim. Farm.* **101,** 131–5 (1962); *C.A.* **57,** 4746*c* (1962).
(261) Coeur, A., Alary, J., Bauduin, N. *Congr. Sci. Farm., Conf. Comun., 21°, Pisa* **1961,** 342–6; *C.A.* **59,** 1440*a* (1963).
(262) Coeur, A., Bouvet, R., Boucherle, A., *Lyon Pharm.* **13,** 287–305 (1962); *C.A.* **58,** 4375*a* (1963).
(263) Cohen, A. J., Anal. Chem. **35** 128–131 (1963).
(264) Cohen, E. N., *J. Lab. Clin. Med.* **61** (2), 338–345 (1963).
(265) Cohen, J., Lach, J. L., *J. Pharm. Sci.* **52,** 132–6 (1963).
(266) Cohn, G. L., Pancake, E., *Nature* **201** (4914), 75–6 (1964).
(267) Colombatti, V., *Farmaco, Ed. Prat.*, **18** (4), 214–217 (1963); *Anal. Abstr.* **11,** 1463 (1964).
(268) Comer, J. P., Ribley, A. M., *J. Pharm. Sci.* **52** (4), 358–361 (1963).
(269) Conine, J. W., *Proc. Am. Assoc. Coll. Pharm. Teachers' Seminar* **13,** 77–91 (1961).
(270) Constantinescu, A., Stan, T., *Farmacia (Bucharest)* **11** (6), 379–381 (1963); *C.A.* **60,** 6704*h* (1964).
(271) Constantinescu, A., Stan, T., Popovici, N., *Ibid.*, **10,** 477–81 (1962); *C.A.* **58,** 12370*h* (1963).
(272) Cordier, P., Dupontrene, J., *Boll. Chim. Farm.* **101,** 462–8 (1962); *C.A.* **57,** 16750*b* (1962).
(273) Cordier, P., Dupontreue, J., *Congr. Sci. Farm., Conf. Commun.,* **21°**, *Pisa* **1961,** 347–53; *C.A.* **59,** 8545*a* (1963).
(274) Costa, H. A., *Rev. Farm. (Buenos Aires)* **102,** 57–65 (1960); *C.A.* **75,** 4762*a* (1962).
(275) Couling, T. E., Goodey, R., *J. Pharm. Pharmacol. Suppl.* **15,** 295–300 (1963).
(276) Covello, M., Schettino, O., *Ann. Chim. (Rome)* **52,** 1135–46 (1962); *C.A.* **58,** 12369*b* (1963).
(277) Cox, P. L., Heotis, J. P., *J. Agr. Food Chem.*, **10** (5), 402–3 (1962).
(278) Craig, L. C., *J. Am. Med. Assoc.* **186** (5), 473–80 (1963).

(279) Crippen, R. C., Freimuth, H. C., ANAL. CHEM. **36,** 273–5 (1964).

(280) Crisler, J. P., *Univ. Microfilms* (*Ann Arbor, Mich.*), Order No. **62-6267,** 114 pp.; *Dissertation Abstr.* **23,** 2303–4 (1963).

(281) Crisler, J. P., Witt, N. F., Hecht-Crisler, M., *Mikrochim. Ichnoanal. Acta,* **1963,** (1), 104–110; *Anal. Abstr.* **10,** 5333 (1963).

(282) Cromp, C. A., *J. Forensic Sci.* **8,** 477–80 (1963).

(283) Cummings, A. J., Martin, B. K., Wiggins, L. F., *J. Pharm. Pharmacol.* **15,** 56–62 (1963).

(284) Czerepko, K., *Microchim. Acta* **1958,** 638–45; *C.A.* **57,** 4039*d* (1962).

(285) Czerepko, K., Wolosowicz, N., *Talanta* **10,** 813 (1963).

(286) Dalev, D., Gagauzov, I., *Nauchni. Tr. Visshiya Med. Inst., Sofiya* **7,** No. 5, 119–24 (1960); *C.A.* **58,** 3270*d* (1963).

(287) Danek, A., Skora, M., *Dissert. Pharm., Krakow,* **14** (4), 461–465 (1962); *Anal. Abstr.* **10,** 3871 (1963).

(288) Danielsson, B., Arvidsson, I., *Svensk Farm. Tidskr.* **67** (7), 173–181 (1963); *Anal. Abstr.* **11,** 1466 (1964).

(289) Danilovic, M., Ristic, N., *Arhiv Farm.* (*Belgrade*) **12,** No. 2, 112–14 (1962); *C.A.* **58,** 10038*e* (1963).

(290) Das Gupta, V., *Indian J. Pharm.* **25,** 161–3 (1963); *C.A.* **59,** 1439*d* (1963).

(291) Datta, D. D., Bami, H. L., Ghosh, D., *India J. Pharm.* **24,** 215–17 (1962); *C.A.* **58,** 10039*c* (1963).

(292) Dave, J. B., Patel, J. L., *Indian J. Pharm.* **21,** 226–9 (1959); *C.A.* **58,** 4375*h* (1963).

(293) Davidek, J., Blattna, J., *J. Chromatog.* **7,** 204–10 (1962).

(294) Davidek, Jiri, *Sb. Vysoke Skoly Chem. Technol. Praze Oddil Fak. Potravinarske Technol.* **4,** Pt. 2, 87–114 (1960); *C.A.* **60,** 9101*e* (1964).

(295) Davies, R. O., Kalow, W., *Can. J. Biochem. Physiol.* **41** (9), 1991–5 (1963); *C.A.* **59,** 13769*g* (1963).

(296) Davis, J. E., *J. Crim. Law, Criminol. Police Sci.* **52,** 459 (1961).

(297) Davis, T. W. M., Farmilo, C. G., Osadchuk, M., ANAL. CHEM. **35,** 751–5 (1963).

(298) Davis, T. W., Genest, K., Lane, R., Farmilo, C. G., *Proc. Can. Soc. Forensic Sci.* **1,** Paper 3, 29 pp. (1962).

(299) de Abreu, M. M. V., *Rev. Port. Farm.* **13** (2), 141–4 (1963); *C.A.* **60,** 370*e* (1964).

(300) Debska, W., *Bull. Soc. Sci. Lettres Pozan Ser. C* **11,** 97–102 (1962); *C.A.* **57,** 13886*g* (1962).

(301) Degtyareva, A. P., *Tr. Gos. Nikitsk. Botan. Sada* **36,** 173–86 (1962); *C.A.* **59,** 12595*b* (1963).

(302) Delaville, J., *Ann. Biol. Clin.* (*Paris*) **20,** 479–81 (1962); *C.A.* **57,** 15240*d* (1962).

(303) DeMarco, J. D., Marcus, A. D., *J. Pharm. Sci.* **51,** 1010–11 (1962).

(304) Denton, S., *Analyst* **87,** 234–236 (1962).

(305) Deppeler, H., Becker, A., *Z. Anal. Chem.* **198** (4), 344–7 (1963); *C.A.* **60,** 3947*g* (1964).

(306) Despiller, O. D., Konakhovs'ka, S. M., Lifshifts, Ya. I., *Farmatsevt. Zh.* (*Kiem*) **18** (2), 42–3 (1963); *C.A.* **60,** 5277*d* (1964).

(307) Devyatnin, V. A., Fedorova, G. A., *Tr. Vses. Nauchn.-Issled. Vitamin. Inst.* **7,** 95–104 (1961); *C.A.* **58,** 8858*h* (1963).

(308) *Ibid.,* **8,** 97–102 (1961); *C.A.* **57,** 2338*d* (1962).

(309) Devyatnin, V. A., Moizhes, M. Ya., *Med. Prom. SSSR* **17** (11), 50–1 (1963); *C.A.* **60,** 7873*f* (1964).

(310) Devyatnin, V. A., Solunina, I. A., *Tr. Ves. Nauchn.-Issled. Vitamin. Inst.* **7,** 107–12 (1961); *C.A.* **58,** 5455*c* (1963).

(311) Deys, H. P., *Pharm. Weekblad* **97,** 805–15 (1962); *C.A.* **58,** 4372*h* (1963).

(312) Diamantstein, T., Lorcher, K., *Z. Anal. Chem.* **191** 429–431 (1962); *Anal. Abstr.* **10,** 3831 (1963).

(313) Dibbern, H. W., Rochelmeyer, H., *Arzneimittel-Forsch.* **13** (1), 7–16 (1963); *Anal. Abstr.* **10,** 4874 (1963).

(314) Dick, J., *Rev. Chim., Acad. Rep. Populaire Roumaine* **7,** No. 1, 145–50 (1962); *C.A.* **59,** 1438*d* (1963).

(315) Dick, J., Drugarin, C., *Acad. R.P.R. Baza Cercet. Stiint. Timisoara, Stud. Cercet. Stiint. Chim.* **8** (3–4), 205–217 (1961); *Anal. Abstr.* **9,** 5406 (1962).

(316) Dick, J., Ristici, J., *Acad. Rep. Populare Romine, Baza Cercetari Stiint. Timisoara, Studii Cercetari Stiinte Chim.* **9,** No. 3–4, 269–72 (1962); *C.A.* **59,** 1095*e* (1963).

(317) Dimitrijevic, B., Petrovic, B., Marcetic, A., Orlic, M., *Arhiv Farm.* (*Belgrade*) **12** (6), 539–40 (1962); *C.A.* **59,** 15123*e* (1963).

(318) Doepke, W., *Arch. Pharm.* **295,** 605–6 (1962); *C.A.* **58,** 1896*a* (1963).

(319) Dolezal, J., Novozamsky, I., Zyka, J., *Coll. Czech. Chem. Commun.* **27** 1830–1834 (1962); *Anal. Abstr.* **10,** 1705 (1963).

(320) Domagalina, E., Pyzyborowski, L., *Chem. Anal. Warsaw* **7** (6), 1153–1165 (1962); *Anal. Abstr.* **10,** 2882 (1963).

(321) Domange, L., Longuevalle, S., *Arch. Pharm. Chemi* **69** (3), 118–124 (1961); *Anal. Abstr.* **9,** 4870 (1962).

(322) Domokos, T., *Magy. Kem. Folyoirat* **67,** 538–41 (1961); *C.A.* **57,** 6023*f* (1962).

(323) Dooley, M. D., Branagan, C. A., Jr., *Am. J. Hosp. Pharm.* **19,** 608–9 (1962).

(324) Dragulescu, C., Policec, S., *Acad. R.P.R., Baza Cercet. Stiint. Timisoara, Stud. Cercet. Stiint. Chim.* **9** (1–2), 33–40 (1962); *Anal. Abstr.* **10,** 1978 (1963).

(325) Dressler, A., *Arch. Toxikol.* **17,** 293–4 (1959); *C.A.* **60,** 7874*c* (1964).

(326) Drey, R. E. A., *J. Pharm. Pharmacol. Suppl.* **15,** 210–15 (1963); *C.A.* **60,** 3949*c* (1964).

(327) Dudzik, Z., Ludwicki, H., Szewko S., *Farm. Polska,* **18** (6), 132–134 (1962); *Anal. Abstr.* **10,** 1144 1963).

(328) Dultz, G., *Dtsch. ApothZtg.* **101** (42), 1339–1346 (1961); *Anal. Abstr.* **9,** 2901 (1962).

(329) Dupcza, K., Kelemen-Kuttel, I., *Acta Pharm. Hung.* **33** (5), 202–5 (1963); *C.A.* **60,** 6705*c* (1964).

(330) Dupacza, K., Kelemenz-Kuettel, I., Vastagh, G., *Ibid.,* **29,** 6–10 (1959); *C.A.* **57,** 8660*d* (1962).

(331) Dusinsky, G., *Chem. Zvesti* **17,** 265–7 (1963); *C.A.* **59,** 12596*c* (1963).

(332) Dusinsky, G., Tyllova, M., *Ceskasl. Farm.* **9,** No. 2, 60–2 (1960); *C.A.* **58,** 4376*d* (1963).

(333) Dusinsky, G., Tyllova, M., *Ibid.,* **11** (7), 359–360 (1962); *Anal. Abstr.* **10,** 1174 (1963).

(334) Dutkiewicz, T., Lisikowa, I., *Acta Polon. Pharm.* **19** (2), 149–155 (1962); *Anal. Abstr.* **10,** 1147 (1963).

(335) Dutton, W. L., Hirt, R. C., *J. Pharm. Sci.* **51** (9), 911 (1962).

(336) Dvoryantsev, G. G., Sheinker, Yu. N., *Zhur. Anal. Khim.* **17** (7), 883–889 (1962); *Anal. Abstr.* **10,** 2444 (1963).

(337) Dworschak, E., *Gyogyszereszet* **7,** 378–80 (1963); *C.A.* **60,** 2722*a* (1964).

(338) Dyer, W. G., Gould, J. P., Maistrellis, N. A., Peng, Tai-Chan, Ofner, P., *Steroids* **1,** 271–85 (1963).

(339) Dzotsoti, S. Kh., *Azerb. Khim. Zh.* **1962,** No. 1, 143–51; *C.A.* **58,** 10039*f* (1963).

(340) Dzyuba, N. P., Georgievs'kii, V. P., **16,** No. 11, 31–3 (1962); *C.A.* **58,** 10041*f* (1963).

(341) Eberhardt, H., Freundt, J., Langbein, J. W., *Arzneimittel-Forsch.* **12,** 1087–9 (1962); *C.A.* **58,** 6647*g* (1963).

(342) Ebersman, D. S., *Univ. Microfilms* (*Ann Arbor, Mich.*), *Order No.* **622386,** 83 pp.; *Dissertation Abstr.* **23,** 207 (1962).

(343) Eckschlager, K., *Ceskosl. Farm.* **11** (7), 360–362 (1962); *Anal. Abstr.* **10,** 1162 (1963).

(344) Egerts, V., Simanska, M., Hillers, S., *Latvijas PSR Zinau Akad. Vestis Kim. Ser.* **1961,** No. 2, 199–240; *C.A.* **57,** 16749*b* (1962).

(345) Ehrlich-Rogozinsky, S., Cheronis, N. D., *Microchem. J.* **7,** (3), 336–56 (1963); *C.A.* **60,** 371*g* (1964).

(346) Eiden, F., Stachel, H.-D., *Dtsch. ApothZtg.* **103** (5), 121–124 (1963); *Anal. Abstr.* **10,** 2467 (1963).

(347) Ellert, H., Jasinski, T., Galicka, N., *Acta Polon. Pharm.* **18** (6), 521–523 (1961); *Anal. Abstr.* **9,** 3887 (1962).

(348) Ellert, H., Jasinski, T., Weclawska, K., *Ibid.,* **19** (1), 75–78 (1962); *Anal. Abstr.* **9,** 3416 (1962).

(349) El-Mangouri, H. A., Nour-El-Dine, H. M., *Egypt. Pharm. Bull.* **42,** 319–24 (1960); *C.A.* **58,** 12369*g* (1963).

(350) Elvidge, D. A., Peutrell, B., *J. Pharm. Pharmacol.* **13,** (Suppl.), 111T-116T (1961).

(351) Endriz, J. D., *J. Assoc. Offic. Agr. Chemists* **46** (4), 637–41 (1963).

(352) Epstein, E., *Clin. Chim. Acta* **7** (5), 735–737 (1962); *Anal. Abstr.* **10,** 759 (1963).

(353) Ermakova, N. M., Korchagin, V. G., Vakulenko, N. A., Sidorova, A. I., *Med. Prom. SSSR* **15,** No. 11, 50–2 (1961); *C.A.* **57,** 2336*h* (1962).

(354) Etchegoyen, E., Raffo Arias, J., Amorin, J. L., *Rev. Farm.* (*Buenos Aires*) **103,** 199–204, 247–55, 258–60 (1961); *C.A.* **58,** 7785*c* (1963).

(355) Ettre, L. S., *Rev. Ital. Sostanze Grasse* **39,** 587–88 (1962); *Anal. Abstr.* **10,** 4329 (1963).

(356) Everhard, M. E., Goodhart, F. W., *J. Pharm. Sci.* **52,** 281–3 (1963).

(357) Evstratova, K. I., Perel'man, Ya. M., *Med. Prom. SSSR* **16,** No. 10, 49–50 (1962); *C.A.* **58,** 12369*a* (1963).

(358) Fahmy, I. R., El-Deeb, S. R., Wahba, S. K., *Egypt. Pharm. Bull.* **42,** No. 4, 23–9 (1960); *C.A.* **57,** 16752*i* (1962).

(359) Fairbairn, J. W., Shellard, E. J., Talalaj, S., *Planta Med.* **11,** 92–7 (1963); *C.A.* **60,** 5276*b* (1964).

(360) Fairbairn, J. W., Simic, S., *J. Pharm. Pharmacol.* **15** (5), 325–8 (1963).

(361) Fairbairn, J. W., Wallsel, Gamila, *J. Pharm. Pharmacol. Suppl.* **15,** 216–21 (1963).

(362) Fait, L., *Farm. Obzor* **32** (2), 53–7 (1963); *C.A.* **60,** 372*g* (1964).

(363) Fang, Hong-Ju, Zhou, Tong-Hui, *Hua Hsueh Hsueh Pao* **29** (1), 64–6 (1963); *C.A.* **59,** 2591*c* (1963).

(364) Farkas, E., Krasznai, I., *Gyogyszereszet* **6,** No. 6, 385–87 (1962); *C.A.* **58,** 11167*f* (1963).

(365) Farkas, S., Bayer, I., *Acta Pharm. Hung.* **32,** 263–9 (1962); *C.A.* **58,** 7784*d* (1963).

(366) Farmilo, C. G., *Proc. Can. Soc. Forensic Sci.* **1,** Paper 1, 3 pp. (1962); *C.A.* **58,** 4376*h* (1963).

(367) Farnsworth, N. R., Euler, K. L., *Lloydia* **25,** 186–95 (1962); *C.A.* **58,** 8850*f* (1963).

(368) Fassina, G., *Boll. Soc. Ital. Biol.*

Sper. **36** (24), 1417–18 (1960); *C.A.* **60,** 900*d* (1964).
(369) *Ibid.*, pp. 1419–21 (1960); *C.A.* **60,** 9100*f* (1964).
(370) Faure, F., Blanquet, P., *Bull. Soc. Pharm. Bordeaux* **101,** 7–28 (1962); *Anal. Abstr.* **10,** 3858 (1963).
(371) Faure, F., Blanquet, M. P., *Ibid.*, **102** (2), 79–88 (1963); *C.A.* **59,** 13484*h* (1963).
(372) Feck, J., *Acta Polon. Pharm.* **20** (3), 225–228 (1963); *Anal. Abstr.* **11** 1467 (1964).
(373) *Federal Register* **28,** 12269–72 (Nov. 20, 1963).
(374) *Ibid.*, **28,** 14494–6 (Dec. 31, 1963).
(375) Feigl, F., *Anal. Chim. Acta* **26,** 424–6 (1962).
(376) Felder, E., Pitre, D., Tiepolo, U., *Minerva Med.* **53,** 1699–1703 (1962); *C.A.* **57,** 13889*h* (1962).
(377) Feldmann, E. G., Koehler, H. M., *J. Dental Research* **39** (2), 313–19 (1960); *C.A.* **60,** 7872*c* (1964).
(378) Feltkamp, H., *Deut. Apotheker-Ztg.* **101** (8), 207–10 (1961); *Anal. Abstr.* **8,** 4811 (1961).
(379) Feltkamp, H., *Ibid.*, **102** (40), 1269–1273 (1962); *Anal. Abstr.* **10,** 3408 (1963).
(380) Fenn, G. D., *Univ. Microfilms (Ann Arbor, Mich.), Order No.* **62-3450,** 70 pp.; *Dissertation Abstr.* **23,** 453 (1962).
(381) Fil, U. G., Mukhtarova, E., Mutset, T. I., *Farmatsevt. Zh. (Kiev)* **18** (2), 20–7 (1963); *C.A.* **60,** 5278*b* (1964).
(382) Finholt, Per, Paulssen, R. B., Higuchi, T., *J. Pharm. Sci.* **52** (10), 948–54 (1963).
(383) Fischer, R., Benkendorf, F. G., Kochan, Ch., *Duet. Apotheker-Ztg.* **103** (11), 301–10 (1963); *C.A.* **60,** 368*e* (1964).
(384) Floderer, I., Horvathy, V., *Acta Pharm. Hung.* **32** (5), 193–198 (1962); *Anal. Abstr.* **10,** 2881 (1963).
(385) Fog, J., Jellum, E., *Nature* **195,** 490 (1962).
(386) Forist, A. A., Madden, S. T., *J. Pharm. Sci.* **50** (3), 269–70 (1961).
(387) Franciszek, A., Ewaryst, P., *Farm. Polska* **18,** No. 8, 180–3 (1962); *C.A.* **58,** 8858*f* (1963).
(388) Frauch, P., Lang, E., *Pharm. Acta Helv.* **38** (9), 676–89 (1963); *C.A.* **59,** 15121*h* (1963).
(389) Frerejacque, M., De Graeve, P., *Ann. Pharm. Franc.* **21** (6), 509–28 (1963); *C.A.* **59,** 12591*b* (1963).
(390) Freund, R. J., *Arzneimittel-Forsch.* **12** (6), 614–617 (1962); *Anal. Abstr.* **10,** 331 (1963).
(391) Frgala, J., *Czech.* **105,745,** Nov. 15, 1962, Appl. Nov. 3, 1960; *C.A.* **59,** 15217*d* (1963).
(392) Friebel, H., Patel, N., *Anaesthesist* **11,** 356–8 (1962); *C.A.* **58,** 6651*a* (1963).
(393) Fried, R., *Arch. Pharm., Berlin* **32** (11) (1962); *Mitt. dtsch. pharm. Ges.*, **32** (11), 189–191 (1962); *Anal. Abstr.* **10,** 3879 (1963).
(394) Fried, Rudolph, *Mitt. Deut. Pharm. Ges.* **34,** (11) 189–91; [Publ. in *Arch. Pharm.* **295,** (11) 1962]; *C.A.* **58,** 10041*h* (1963).
(395) Friedman, R. L., Raab, W. J., Anal. Chem. **35,** 67–9 (1963).
(396) Frimpter, G. W., Bass, A., *J. Chromatog.* **7,** 427–30 (1962).
(397) Froemming, K. H., Schenck, G., Simon, H., *Arch. Pharm.* **296,** 557–61 (1963); *C.A.* **60,** 3951*b* (1964).
(398) Fukuzawa, H., Shiizu, K., *Yakuzaigaku* **23,** 112–15 (1963); *C.A.* **60,** 2723*a* (1964).
(399) Fulop, L., Vereph, I., Mortonfi, L., *Farmacia* **11,** 301–4 (1963); *C.A.* **59,** 9735*e* (1963).
(400) Fulton, C. C., *Microchem. J.* **6** (1), 51–65 (1962).
(401) Fursov, A. F., *Aptechn. Delo* **11** (3), 63–6 (1962); *C.A.* **60,** 10480*b* (1964).
(402) Fuwa, T., Kido, T., Tanaka, H., *Yakuzaigaku* **23,** 102–5 (1963); *C.A.* **60,** 3951*g* (1964).
(403) Gaenshirt, H., *Arch. Pharm.* **296,** No. 2, 129–34 (1963); *C.A.* **58,** 11167*a* (1963).
(404) Gallo, G. G., Chiesa, L., *Farmaco (Pavia), Ed. Prat.* **18** (4), 206–13 (1963); *C.A.* **59,** 8545*e* (1963).
(405) Gallo, G. G., Chiesa, L., Sensi, P., *Ibid.*, **17,** 668–78 (1962); *C.A.* **57,** 16750*c* (1962).
(406) Gamp, A., Studer, P., Linde, H., Meyer, K., *Experientia* **18,** 292–3 (1962); *C.A.* **57,** 6582*h* (1962).
(407) Ganshirt, H., *Arch. Pharm., Berlin* **296** (2), 73–79 (1963); *Anal. Abstr.* **11,** 335 (1964).
(408) Garratt, D. C., Johnson, C. A., King, R. E., *J. Pharm. Pharmacol. Suppl.* **15,** 206–9 (1963); *C.A.* **60,** 3950*d* (1964).
(409) Garrett, E. R., *Indian J. Pharm.* **25,** 285–97 (1963); *C.A.* **59,** 15125*b* (1963).
(410) Garrett, E. R., *J. Pharm. Sci.* **51,** 1036–1039 (1962).
(411) Garrett, E. R., *Ibid.*, **52,** 797–9 (1963).
(412) Garrett, E. R., *Ibid.*, **53,** 42–51 (1964).
(413) Garrett, E. R., Herr, R. R., *Ibid.*, **51,** 1034–6 (1962).
(414) Garrett, E. R., Johnson, J. L., *Ibid.*, **51,** 767–70 (1962).
(415) Garrett, E. R., Johnson, J. L., Alway, C. D., Anal. Chem. **34,** 1472–7 (1962).
(416) Garrett, E. R., Olson, E. C., *J. Pharm. Sci.* **51,** 764–6 (1962).
(417) Garrett, E. R., Umbreit, G. R., *Ibid.*, **51,** 436–44 (1962).
(418) Garrett, E. R., Weber, D. J., *Ibid.*, **51,** 387–9 (1962).
(419) Gavrilin, G. F., Perel'man, Ya. M., *Med. Prom. SSSR* **17** (6), 48–9 (1963); *C.A.* **59,** 15122*d* (1963).
(420) Gay, S., *Biochim. Appl.* **9,** 65–70 (1962); *C.A.* **57,** 13889*b* (1962).
(421) Genest, K., Farmilo, C. G., Anal. Chem. **34** (11), 1464–1468 (1962).
(422) Genkina, G. L., Abubakirov, N. K., *Med. Prom. SSSR* **17** (11), 52–4 (1963); *C.A.* **60,** 6703*d* (1964).
(423) Gerritsma, K. W., van Rheede, M. C. B., *Pharm. Weekblad* **97,** 765–76 (1962); *C.A.* **58,** 6646*a* (1963).
(424) Ghafoor, M. A., Huyck, C. L., *J. Pharm. Sci.* **51** (9), 894–896 (1962).
(425) Ghelardoni, M., Fedi, M., *Boll. Chim. Farm.* **101,** 26–30 (1962); *C.A.* **57,** 958*g* (1962).
(426) Ghimicescu, Gh., Musteata-Ghimicescu, Camelia, Dumbrava, Elvira, *Acad. Rep. Populare Romine Studii Cercetari Chim.* **11** (1), 103–10 (1963); *C.A.* **59,** 15121*a* (1963).
(427) Giacomasso, P. P., *Arch. Sci. Med.* **115** (5), 316–34 (1963); *C.A.* **60,** 3948*f* (1964).
(428) Girard, M., Rousselet, F., *Ann. Pharm. Franc.* **20** (2), 109–115 (1962); *Anal. Abstr.* **9,** 3872 (1962).
(429) Giroux, J., Puech, A., *Ibid.*, **21** (6), 469–76 (1963); *C.A.* **59,** 12593*d* (1963).
(430) Giusti, J., Di Paco, G. F., *Boll. Chim. Farm.* **100,** 425–429 (1961); *Anal. Abstr.* **9,** 4907 (1962).
(431) Gjaldback, J. Chr., Sorensen, K. W., *Dansk Tidsskr. Farm.* **37** (9), 213–19 (1963); *C.A.* **60,** 1539*b* (1964).
(432) Glombitza, K. W., *Arzneimittel-Forsch.* **13,** 513 (1963); *C.A.* **59,** 8544*a* (1963).
(433) Godyatskii, V. E., *Izv. Vysshikh Uchebn. Zavedenii, Khim. i Khim. Tekhnol.* **6** (4), 562–6 (1963); *C.A.* **60,** 3471*d* (1964).
(434) Godyatskii, V. E., *Uch. Zp., Pyatigorskii Gos. Farmatsevt. Inst.* **5,** 175–80 (1961); *C.A.* **59,** 3718*f* (1963).
(435) Godyatskii, V. E., Starostenko, A., Starostenko, V., *Ibid.*, **5,** 181–3 (1961); *C.A.* **59,** 2591*a* (1963).
(436) Golab, T., Layne, D. S., *J. Chromatog.* **9,** 321–330 (1962).
(437) Golowinsky, E., *Compt. Rend. Acad. Bulg. Sci.* **15** (3), 277–279 (1962); *Anal. Abstr.* **10,** 319 (1963).
(438) Goodey, R., Couling, T. E., Hart, J. E., *J. Pharm. Pharmacol. Suppl.* **14,** 122–6 (1962).
(439) Goryacheva, N. S., Solov'eva, Z. I., *Med. Prom. SSSR* **16,** No. 4, 33–6 (1962); *C.A.* **57,** 8658*d* (1962).
(440) Gosselinckx, F., Parmentier, G., *Chromatog. Symp., 2nd, Brussels* **1962,** 181–90; *C.A.* **60,** 1543*b* (1964).
(441) Gosztonyi, T., Marton, J., Kemeny, V., Vecsei, P., *J. Chromatog.* **10,** 29–34 (1963).
(442) Gottfried, N. S., *Am. J. Hosp. Pharm.* **19,** 310–14 (1962).
(443) Grabowicz, W., *Chem. Anal., Warsaw* **8** (1), 79–85 (1963); *Anal. Abstr.* **10,** 5371 (1963).
(444) Gracza, L., Szasz, K., *Arch. Pharm., Berlin,* **295** (11), 859–860 (1962); *Anal. Abstr.* **10,** 3850 (1963).
(445) Gracza, M., Szasz, G., Vegh, A., *Acta Pharm. Hung.* **33** (6), 252–6 (1963); *C.A.* **60,** 6704*d* (1964).
(446) Grade, K., Matthies, H., *Z. Chem.* **3** (6), 229 (1963); *C.A.* **59,** 8125*f* (1963).
(447) Graham, D. M., Spencer, Mary, Anal. Chem. **36** (1), 210–13 (1964).
(448) Graham, H. D., *J. Pharm. Sci.* **53** (1), 86–91 (1964).
(449) Green, J., Marcinkiewicz, S., McHale, D., *J. Chromatog.* **10** (2), 158–183 (1963); *Anal. Abstr.* **11,** 382 (1964).
(450) Grlic, Lj., *Farm. Glasnik* **19,** 163–72 (1963); *C.A.* **59,** 11190*g* (1963).
(451) Grochmalicka, J., *Arhiv Farm. (Belgrade)* **12,** No. 3, 157–62 (1962); *C.A.* **58,** 1117*b* (1963).
(452) Groeger, D., Erge, D., *Pharmazie* **18,** 346–9 (1963); *C.A.* **60,** 367*a* (1964).
(453) Guadagnini, G., *Boll. Chim. Farm.* **102** (6), 397–401 (1963); *C.A.* **59,** 8545*f* (1963).
(454) Gulubov, A., Kitova, M., *Sb. Trudy Vissh. Med. Inst. Plovdiv* **16,** 17–22 (1962); *Anal. Abstr.* **10,** 3412 (1963).
(455) Gupta, V. D., *J. Pharm. Sci.* **51,** 1195 (1962).
(456) Gusev, S. I., Ketova, L. A., *Sb. Nauchn. Tr. Permsk. Gos. Med. Inst.* **1962** (41), 148–51; *C.A.* **60,** 9100*g* (1964).
(457) Guttman, D. E., *Proc. Am. Assoc. Coll. Pharm. Teachers' Seminar* **13,** 63–76 (1961).
(458) Guttman, D. E., Brooke, D., *J. Pharm. Sci.* **52** (10), 941–5 (1963).
(459) Guven, K. C., *Eczacilik Bulteni* **4,** 137–40 (1962); *C.A.* **58,** 3270*c* (1963).
(460) Guven, K. C., Bayraktar, G., Mentes, O., *Ibid.*, **5** (9–11), 118–24 (1963); *C.A.* **60,** 5279*f* (1964).
(461) Guven, K. C., Eranil, M., *Ibid.*, **4,** 68–73 (1962); *Anal. Abstr.* **10,** 3845 (1963).
(462) Guven, K. C., Tarcan, S., *Bull. Fac. Med. Istanbul* **24,** 314–22 (1961); *C.A.* **58,** 1307*d* (1963).
(463) Guven, K. C., Tarcan, S., *Eczacilik Bulteni* **4,** 162–6 (1962); *C.A.* **58,** 5456*h* (1963).

(464) Gyenes, I., Bayer, J., *Pharmazie* **16**, 211–17 (1961); *C.A.* **57**, 4761*g* (1962).

(465) Halmekoski, Jaakko, *Suomen Kemistilehti* **B35**, 209–12 (1962); *C.A.* **58**, 7789*h* (1963).

(466) Halmekoski, Jaakko, *Ibid.*, **B36**, 40–2 (1963); *C.A.* **59**, 384*b* (1963).

(467) *Ibid.*, pp. 58–60; *C.A.* **59**, 2589*h* (1963).

(468) Halmekoski, Jaakko, Hannikainen, Helka, *Ibid.*, **35B**, 221–4 (1962); *C.A.* **58**, 7350*a* (1963).

(469) Halot, D., *Ann. Pharm. Franc.* **19** (7–8), 477–482 (1961); *Anal. Abstr.* **9**, 2900 (1962).

(470) Halot, D., *Ann. Pharm. Franc.* **19** (7–8), 483–493 (1961); *Anal. Abstr.* **9**, 2906 (1962).

(471) Hamilton-Miller, J. M. T., Smith, J. T., Knox, R., *J. Pharm. Pharmacol.* **25** (2), 81–91 (1963).

(472) Hara, Shoji, *Kagaku No Ryoiki* **17**, 196–206 (1963); *C.A.* **60**, 7*d* (1964).

(473) Harting, H. L., *Pharm. Ind.* **25**, 57–9 (1963); *C.A.* **58**, 13722*b* (1963).

(474) Hattori, Tetsuyasu, Nishiumi, Michinori, *Arch. Prac. Pharm., Japan*, **21** (1), 9–14 (1961); *Anal. Abstr.* **10**, 3876 (1963).

(475) Haug, U., Panescu, I., *Pharmazie* **18** (5), 340–1 (1963); *C.A.* **60**, 372*c* (1964).

(476) Hauton, J. C., *J. Chromatog.* **7**, 341–5 (1962).

(477) Hayden, A. L., *J. Pharm. Sci.* **51**, 617–22 (1962).

(478) Hayden, A. L., Sammul, O. R., Selzer, G. B., Carol, J., *J. Assoc. Offic. Agr. Chemists* **45**, 797–900 (1962).

(479) Hayes, J. R., Jr., DuBuy, H. G., *Anal. Biochem.* **7** (3), 322–7 (1964).

(480) Head, W. F., Jr., *J. Pharm. Sci.* **50** (12), 1041–1044 (1961).

(481) Healton, D. C., *J. Assoc. Offic. Agr. Chemists* **45**, 616–20 (1962).

(482) Healton, D. C., *Ibid.*, **46**, 652–4 (1963).

(483) Hedner, P., *Acta Pharm. Tox., Kbh.* **18** (4), 407–413 (1961); *Anal. Abstr.* **9**, 3399 (1962).

(484) Hefferren, J. J., Klessig, R. S., Dietz, C. L., *J. Dental Res.* **42**, 793–802 (1963).

(485) Hennart, C., *Chim. Anal.* **44** (1), 8–11 (1962); *Anal. Abstr.* **9**, 3423 (1962).

(486) Hentrich, K., *Pharmazie* **18**, 405–9 (1963); *C.A.* **60**, 373*d* (1964).

(487) Hernandez, R., Jr., Axelrod, L. R., ANAL. CHEM. **35** (1), 80–83 (1963).

(488) Herrmann, J., Grossmann, H. G., *Pharm. Zentralhalle* **101**, 743–8 (1962); *C.A.* **59**, 1442*f* (1963).

(489) Herting, D. C., Drury, E. J. E., Harris, P. L., *Anal. Biochem.* **4** (6), 459–475 (1962).

(490) Herzmann, H., Hennig, A., *Z. Naturforsch.* **17b**, 646–54 (1962); *C.A.* **58**, 1307*c* (1963).

(491) Hetman, J. S., *Lab. Pract.* **12** (8), 727–8, 740 (1963).

(492) Higuchi, Takeru, *Proc. Am. Assoc. Coll. Pharm. Teachers' Seminar* **13**, 119–24 (1961).

(493) Higuchi, W. I., Lau, P. K., Higuchi, T., Shell, J. W., *J. Pharm. Sci.* **52**, 150–3 (1963).

(494) Higuchi, W. I., Rowe, E. L., Hiestand, E. N., *Ibid.*, **52**, 162–4 (1963); *C.A.* **58**, 11168*e* (1963).

(495) Hiller, K., *Pharmazie* **16**, 600–603 (1961); *Anal. Abstr.* **10**, 1152 (163).

(496) Hillers, S., Egerts, V., Simanska, M., *Akad. Nauk. Latv. SSR Inst. Organ. Sinteza* **1962**, 17–27; *C.A.* **59**, 3719*b* (1963).

(497) Hishta, Charles, Reschke, R. F., *Microchem. J. Symp. Ser.* **2**, 679–86 (1962); *C.A.* **58**, 8851*h* (1963).

(498) Hoerhammer, L., Wagner, H., Koenig, H., *Deut. Apotheker-Ztg.* **103** (17), 502–4 (1963); *C.A.* **59**, 12593*f* (1963).

(499) Hoffman, A. J., Mitchell, H. I., *J. Pharm. Sci.* **52**, 305–6 (1963).

(500) Hofstetter, R., Muhlemann, H., *Pharm. Acta Helv.* **38**, 218–23 (1963); *C.A.* **59**, 2591*a* (1963).

(501) *Ibid.*, pp. 349–57 (1963); *C.A.* **59**, 11190*e* (1963).

(502) Hoft, E., Schultze, H., *J. Prakt. Chem.* **19** (5–6), 260–265 (1963); *Anal. Abstr.* **11**, 1319 (1964).

(503) Hoil, W. W., Michaels, T. P., Sinotte, L. P., *J. Pharm. Sci.* **52**, 817–18 (1963).

(504) Holbrook, A., Bailey, F., Bailey, G. M., *J. Pharm. Pharmacol. Suppl.* **15**, 274–7 (1963).

(505) Holbrook, A., Barlow, F. S., Bailey, F., *Ibid.*, **15**, 232–5 (1963).

(506) Hollis, D. P., ANAL. CHEM. **35** (11), 1682–4 (1963).

(507) Hollos, J., *Gyogyszereszet* **7** (5–6), 204–10 (1963); *C.A.* **60**, 1540*g* (1964).

(508) Homan, J. D. H., *Med. Prom. SSSR* **16**, No. 4, 20–7 (1962); *C.A.* **57**, 8659*h* (1962).

(509) Horak, F., *Chem. Zvest.* **17**, 795–7 (1963); *C.A.* **60**, 9099*d* (1964).

(510) Horak, P., Holoubek, J., Bumba, V. Cekan, Z., *Coll. Czech. Chem. Commun.* **27**, 1037–42 (1962); *C.A.* **58**, 10038*f* (1963).

(511) Horvath, D., *Magy. Kem. Folyoirat* **68**, No. 7, 324–5 (1962); *C.A.* **57**, 15243*a* (1962).

(512) Hossein, A. M., Kassim, Aly, Hamad, Hassan, A. A., *Egypt. Pharm. Bull.* **42**, No. 4, 395–409 (1960); *C.A.* **58**, 4380*f* (1963).

(513) Houk, A. E. H., *J. Pharm. Sci.* **52**, 738–43 (1963).

(514) *Ibid.*, pp. 743–7.

(515) Hoyem, T., *J. Assoc. Offic. Agr. Chemists* **45**, 902–905 (1962).

(516) Hrdy, O., *Ceskosl. Farm.* **11**, 192–6 (1962); *Anal. Abstr.* **9**, 4858 (1962).

(517) *Ibid.*, pp. 255–259; *Anal. Abstr.* **9**, 5402 (1962).

(518) Hrdy, O., Kolcova, J., *Ceskosl. Farm.* **11** (4), 185–187 (1962); *Anal. Abstr.* **9**, 4858 (1962).

(519) Huang, C. L., Sands, F. L., *J. Chromatog.* **13** (1), 246–9 (1964).

(520) Huang, C.-S., Wang, H., Wang, L.-L., *Yao Hsueh Hsueh Pao* **10** (6), 372–5 (1963); *C.A.* **59**, 13768*h* (1963).

(521) Hurst, L. S., "Titrimetric Methods," *Proc. Symp. Cornwall, Ont., Can.* **1961**, 179–85.

(522) Huttenrauch, R., *Pharm. Zentralh.* **102** (4), 203–208 (1963); *Anal. Abstr.* **11**, 1452 (1964).

(523) Huttenrauch, R., *Z. Physiol. Chem.* **326**, 166–167 (1961); *Anal. Abstr.* **9**, 5379 (1962).

(524) Huttenrauch, R., Keiner, J., *Pharm. Zentralhalle* **102** (10), 615–17 (1963); *C.A.* **60**, 7874*f* (1964).

(525) Huttenrauch, R., Klotz, L., *Experientia* **19** (2), 95–96 (1963); *Anal. Abstr.* **10**, 5408 (1963).

(526) Huttenrauch, R., Resch, U., *Arch. Pharm. Berlin* **295** (4), 256–260 (1962); *Anal. Abstr.* **9**, 4428 (1962).

(527) Ibadov, A. Yu., Gafurov, Sh., *Dokl. Akad. Nauk Uz. SSR* **1961**, No. 9, 31–3: *C.A.* **58**, 12368*h* (1963).

(528) Ichibangase, H., Kojima, S., *J. Pharm. Soc. Japan* **82** (12), 1616–1620 (1962); *Anal. Abstr.* **10**, 5391 (1963).

(529) Ichimura, Y., *Bunseki Kagaku* **8**, 557–61 (1959); *C.A.* **58**, 10041*b* (1963).

(530) Ichimura, Y., *Ibid.*, **10** (6), 623–625 (1961); *Anal. Abstr.* **10**, 3414 (1963).

(531) Ichimura, Y., Nishimoto, K., *Ibid.*, **11**, 1024–7 (1962); *C.A.* **57**, 16748*d* (1962).

(532) Iconomou, N., Buechi, J., Schumacher, H., *Pharm. Acta Helv.* **38**, 175–88 (1963); *C.A.* **59**, 2593*b* (1963).

(533) Iconomou, N., Buechi, J., Schumacher, H., *Ibid.*, **38**, 275–92 (1963); *C.A.* **59**, 9736*f* (1963).

(534) Iconomou-Petrovitch, N., Buechi, J., Schumacher, H., *Ibid.*, **37** (9–10), 622–638 (1962); *C.A.* **58**, 4378*g* (1963).

(535) *Ibid.*, **37**, pp. 638–710; *Anal. Abstr.* **10**, 3718 (1963).

(536) *Ibid.*, **37**, pp. 748–69; *C.A.* **58**, 7784*b* (1963).

(537) *Ibid.*, **38** (1), 26–33 (1962); *Anal. Abstr.* **10**, 4872 (1963).

(538) *Ibid.*, **38** (2), 102–118 (1963); *Anal. Abstr.* **11**, 319 (1964).

(539) Igloy, M., *Magy. Kem. Lapja* **18** (12), 622–4 (1963); *C.A.* **60**, 10476*d* (1964).

(540) Iguchi, S., Yamamoto, M., Aoyama, T., *Yakugaku Zasshi* **83**, 721–3 (1963); *C.A.* **59**, 12595*a* (1963).

(541) Ikeda, D., Yu, T., *Bitamin* **21**, 230–6 (1960); *C.A.* **60**, 3954*e* (1964).

(542) Ikekawa, T., Iwami, F., Akita, E., and Umezawa, H., *Penishirin Sono Ta Koseibusshitsu Ser. A* **16** (1), 56–7 (1963); *C.A.* **60**, 1539*c* (1964).

(543) Ikram, M., Bakhsh, M. K., ANAL. CHEM. **36** (1), 111–12 (1964).

(544) Ikramov, L. T., *Vopr. Ispol'z. Mineral'n i Rast. Syr'ya. Sredn. Azii* (*Tashkent. Akad. Nauk Uz. SSR*) *Sb.* **1961**, 160–3; *C.A.* **58**, 10040*h* (1963).

(545) Ikramov, L. T., Aliev, Kh., *Aptechn. Delo* **11** (5), 68–9 (1962); *C.A.* **60**, 10478*b* (1964).

(546) Iliev, M., *Farmatsiya* (*Sofia*) **12**, No. 1, 27–9 (1962); *C.A.* **58**, 8852*c* (1963).

(547) Illig, M., Buechner, M., *Acta Biol. Med. Ger.* **11**, 6–13 (1963); *C.A.* **60**, 1541*a* (1964).

(548) Ilver, K., Jackerott, Aa., *Dansk. Tidsskr. Farm.* **37** (6), 129–40 (1963); *C.A.* **59**, 11190*g* (1963).

(549) *Ibid.*, pp. 225–36; *C.A.* **60**, 3951*d* (1964).

(550) Inczedy, J., Gimesi, O., *Acta Chim. Acad. Sci. Hung.* **31** (4), 347–356 (1962); *C.A.* **58**, 933*f* (1963).

(551) Inoue, T., *Hoshi Yakka Daigaku Kiyo* **10**, 17–19 (1961); *C.A.* **57**, 959*d* (1962).

(552) Invernizzi, I., Sampietro, C., *Boll. Lab. Chim. Provinciali* **14** (2), 127–134 (1963); *Anal. Abstr.* **11**, 1923 (1964).

(553) Ioffe, I. S., Sukhomlinov, F. K., Leproskii, A. N., Bukhman, L. B., *Zh. Prikl. Khim.* **36**, 629–32 (1963); *C.A.* **59**, 2596*b* (1963).

(554) Iritani, N., Arizono, K., *Yakugaka Zasshi* **83**, 1083–6 (1963); *C.A.* **60**, 9102*b* (1964).

(555) Iritani, N., Suzuki, N., *Ibid.*, **82**, 1073–6 (1962); *C.A.* **57**, 15242*a* (1962).

(556) Iritani, N., Suzki, N., *J. Pharm. Soc. Japan* **83**, (1), 98–100 (1963); *Anal. Abstr.* **11**, 336 (1964).

(557) Irmscher, H., *Pharm. Praxis, Beilage Pharmazie* **1961**, 184; *C.A.* **57**, 13889*c* (1962).

(558) Irudayasamy, A., Natarajan, A. R., *Analyst* **87**, 831–832 (1962).

(559) Isarenko, N. Ya., Orlov, Yu. E., Shraiber, M. S., *Med. Prom. SSSR* **17** (9), 38–40 (1963); *C.A.* **60**, 2721*d* (1964).

(560) Iso, T., *J. Biochem.* (*Tokyo*) **51**, 345–49 (1962); *C.A.* **57**, 11471*a* (1962).

(561) Ivanov, N. P., Il'ich, G. K., *Lab. Delo* **8**, No. 2, 24–7 (1962); *C.A.* **57**, 4195*d* (1962).

(562) Ivashkiv, E., *J. Pharm. Sci.* **51**, 698–9 (1962).

(563) Izawa, Y., Kimura, W., *J. Japan Oil Chem. Soc.* **11** (12), 627–630 (1962); *Anal. Abstr.* **10,** 4256 (1963).

(564) Izmailov, N. A., Mushinskaya S. Kh., Danel'yants, V. A., *Med. Prom. SSSR* **17** (7), 47–50 (1963); *C.A.* **59,** 12594*c* (1963).

(565) Jackson, D. S., "Titrimetric Methods," *Proc. of 1961 Symposium on Titrimetric Methods, Cornwall, Ontario, May 8–9,* **1961,** Plenum Press, New York, 1961, 185 pp.

(566) Jackwerth, E., *Arch. Pharm., Berlin* **295** (10), 779–795 (1962); *C.A.* **58,** 4376*d* (1963).

(567) Jakovljevic, I. M., ANAL. CHEM. **35** (10), 1513–16 (1963).

(568) Jakovljevic, I. M., Fose, J. M., Kuzel, N. R., ANAL. CHEM. **34,** 410–413 (1962).

(569) Jancik, F., Korbl, J., *Ceskosl. Farm.* **11** (6), 305–308 (1962); *Anal. Abstr.* **10,** 336 (1963).

(570) Jancik, F., Koerbl, J., Sulovsky, J., *Czech.* **108,813,** Nov. 15, 1963, Appl. Nov. 29, 1962; *C.A.* **60,** 10482*c* (1964).

(571) Jayaraman, K. S., Ramanujam, S., Vijayaraghavan, P. K., *Curr. Sci.* **31** (7), 282–283 (1962); *Anal. Abstr.* **10,** 3455 (1963).

(572) Jindra, L., Hejlek, J., Janat, V., *Farm. Obzor* **32** (1), 6–11 (1963); *C.A.* **60,** 375*e* (1964).

(573) Johannesen, B., Sandal, A., *Medd. Norsk Farm. Selskap* **23,** 105–13 (1961); *C.A.* **57,** 15238*f* (1962).

(574) Johnson, C. A., *Mfg. Chemist* **34** (10), 480–2, 489 (1963).

(575) Johnson, C. A., King, R. E., *J. Pharm. Pharmacol. Suppl.* **14,** 77–82 (1962).

(576) Johnson, C. A., King, R. E., *Ibid.,* **15** (9), 584–8 (1963).

(577) Jonaitis, H., Rubikaite, B., *Lietuvos Fiz. Rinkinys, Lietuvos TSR Mokslu Akad., Leituvos TSR Aukstosios Moky-klos* **1,** No. 1–2, 207–16 (1961); *C.A.* **58,** 8857*e* (1963).

(578) Jones, R., Page, B. C., ANAL. CHEM. **36** (1), 35–8 (1964).

(579) Jones, W., *Pharm. J.* **191,** No. 5218, 459–62 (1963).

(580) *J. Pharm. Sci.* **53** (1), 101–3 (1964).

(581) Jue, J., Huyck, C. L., *J. Pharm. Sci.* **51,** 773–6 (1962); *C.A.* **57,** 1263*f* (1962).

(582) Jungalwala, F. B., Cama, H. R., *J. Chromatog.* **8,** 535–536 (1962).

(583) Jungnickel, H. E., Klinger, W., *Pharmazie* **18,** 353–5 (1963); *C.A.* **59,** 15120*e* (1963).

(584) Jusiak, L., Soczewinski, E., Waksmundzki, A., *Acta Polon. Pharm.* **19,** 193–8 (1962); *C.A.* **59,** 382*e* (1963).

(585) Kadar-Pauncz, J., *Acta Chim. Acad. Sci. Hung.* **35** (3), 297–299 (1963); *Anal. Abstr.* **10,** 2876 (1963).

(586) Kadis, B., Harris, C., Salhanick, H. A., *J. Chromatog.* **7,** 430–431 (1962).

(587) Kakac, B., *Ceskosl. Farm.* **12** (4), 177–182 (1963); *Anal. Abstr.* **11,** 1902 (1964).

(588) Kakac, B., Hudlicky, M., *Talanta* **9** (6), 530–533 (1962).

(589) Kakemi, K., Arita, T., Ohashi, S., *J. Pharm. Soc. Japan* **81** (5), 748–750 (1961); *Anal. Abstr.* **9,** 4896 (1962).

(590) Kakemi, K., Ohashi, S., *Yakugaku Zasshi* **82,** 338–42 (1962); *C.A.* **57,** 11310*f* (1962).

(591) Kakemi, K., Arita, T., Sezaki, H., Nadai, T., *Ibid.,* **82,** 297–9 (1962); *C.A.* **58,** 3270*b* (1963).

(592) Kakemi, K., Yokata, T., Nadai, T., *Ibid.,* **80** (2), 176–180 (1960); *Anal. Abstr.* **9,** 4459 (1962).

(593) *Ibid.,* pp. 180–183.

(594) Kala, H., *Pnarmazie* **16,** 297–305 (1961); *Anal. Abstr.* **10,** 1173 (1963).

(595) Kalinowska, Z. E., *Acta Polon. Pharm.* **20** (1), 69–75 (1963); *Anal. Abstr.* **10,** 4877 (1963).

(596) *Ibid.,* pp. 193–198; *Anal. Abstr.* **11,** 741 (1964).

(597) Kalinowska, Z., E., Bartnik-Kurzawinska, J., *Ibid.,* **19,** 45–53 (1962); *C.A.* **57,** 4765*i* (1962).

(598) Kalinowska, Z. E., Kochalska, J., *Ibid.,* **20,** 59–67 (1963); *Anal. Abstr.* **10,** 4879 (1963).

(599) Kalinowska, Z. E., Podkowska, L., Mieszczakowska, J., *Farm. Polska* **19** (15–16), 329–31 (1963); *C.A.* **60,** 10478*g* (1964).

(600) Kalinowski, K., Baran, H., *Acta Polon. Pharm.* **16,** 231–3 (1959); *C.A.* **58,** 8852*a* (1963).

(601) Kalinowski, K., Fecko, J., *Ibid.,* **20,** 53–58 (1963); *Anal. Abstr.* **10,** 4367 (1963).

(602) Kalinowski, K., Piotrowska, A., *Ibid.,* **20** (2), 199–204 (1963); *Anal. Abstr.* **11,** 746 (1964).

(603) Kalinowski, K., Zwierzchowski, Z., *Ibid.,* **20,** 303–308 (1963); *Anal. Abstr.* **11,** 1471 (1964).

(604) *Ibid.,* pp. 309–313; *Anal. Abstr.* **11,** 1470 (1964).

(605) Kamp, W., Klijsen, J. B., Ruward, R. H., *Pharm. Weekbl.* **97** (25), 889–894 (1962); *Anal. Abstr.* **10,** 3868 (1963).

(606) Kaneko, H., Fujimoto, R., Namba, K., Hayashi, K., *J. Pharm. Soc. Japan* **82,** 1493–1496 (1962); *Anal. Abstr.* **10,** 5357 (1963).

(607) *Ibid.,* pp. 1489–1492; *Anal. Abstr.* **10,** 5357 (1963).

(608) Kao, Tzu-I, T'ien, Ping-Shih, *Hua Hsueh Hsueh Pao* **27** (3), 167–72 (1961); *C.A.* **59,** 12594*g* (1963).

(609) Kapadia, G. J., Roa, G. S., *J. Pharm. Sci.* **53** (2), 223–4 (1964).

(610) Karawya, M. S., El-Deeb, S. R., *Egypt. Pharm. Bull.* **42,** No. 4, 107–16 (1960); *C.A.* **57,** 16752*f* (1962).

(611) Karpitschka, N., *Mikrochim. Ichnoanal. Acta* **1963,** 157–64; *C.A.* **58,** 10042*d* (1963).

(612) Kartnig, T., *Pharm. Zentralhalle* **98,** 495–9 (1959); *C.A.* **59,** 2591*b* (1963).

(613) Kas, V., *Prakt. Lekar* **43,** (3) 112–14 (1963); *C.A.* **59,** 1439*e* (1963).

(614) Kashima, T., Watari, T., Mikubo, H., *Eisei Shikensho Hokok* **79,** 59–63 (1961); *C.A.* **59,** 1438*c* (1963).

(615) Kassau, E., *Deut. Apotheker Ztg.* **100** (38), 1102–6 (1960); *C.A.* **60,** 1542*a* (1964).

(616) Kato, K., Sato, K., Tsuda, T., *Yakugaku Zasshi* **83,** 815–17 (1963); *C.A.* **59,** 15119*e* (1963).

(617) Katona, K., *Gyogyszereszet* **3,** 143–5 (1959); *C.A.* **58,** 3274*b* (1963).

(618) Katz, M., *Publ. Inst. Invest. Microquim. Univ. Nacl. Litoral* **24** (22) 144–65 (1958); *C.A.* **57,** 16752*h* (1962).

(619) Kaufmann, H. P., Das, B., *Fette Seif., Anstrichmitt.,* **64** (3), 214–217 (1962); *Anal. Abstr.* **9,** 4489 (1962).

(620) Kawai, S., Takeuchi, S., *Bunseki Kagaku* **10,** 473–8 (1961); *C.A.* **58,** 6646*g* (1963).

(621) Kawamoto, H., *Kumamoto Med. J.* **15** (2), 69–72 (1962); *Anal. Abstr.* **10,** 2888 (1963).

(622) Kazyak, L., Knoblock, E. C., ANAL. CHEM. **35,** 1448–52 (1963).

(623) Kediovski, M., *Bil. Farmats. Drusht. NRM* **9** (2), 1–5 (1962); *Anal. Abstr.* **10,** 3400 (1963).

(624) Kelly, C. A., *Talanta* **11** (2), 175–87 (1964).

(625) Kerenyi, I., *Gyogyszereszet* **4,** 86–95 (1960); *C.A.* **60,** 5276*k* (1964).

(626) Kerenyi, I., *Pharm. Zentralh.* **100** (12), 650–663 (1961); *Anal. Abstr.* **9,** 2893 (1962).

(627) Kertesz, P., *Acta Pharm. Hung.* **33** (4), 150–1 (1963); *C.A.* **60,** 1540b (1964).

(628) Kessler, W. V., Christian, J. E., *U. S. At. Energy Comm.* **TID-14330,** 7 pp. (1961); *C.A.* **59,** 9736*d* (1963).

(629) Kitazawa, T., Naito, M., Yoshida, F., *J. Pharm. Soc. Japan* **80** (1), 116–118 (1960); *Anal. Abstr.* **9,** 4464 (1962).

(630) Kho, B. T., Klein, S., *J. Pharm. Sci.* **52** (4), 404–406 (1963).

(631) Khorlin, A. Ya, Bochkov, A. F., *Izv. Akad. Nauk SSSR, Otd. Khim. Nauk* **1962,** 1120–1; *C.A.* **57,** 12812*b* (1962).

(632) Kimura, H., *Yakugaku Zasshi* **81,** 1699–702 (1961); *C.A.* **58,** 1301*h* (1963).

(633) Kimura, H., *Ibid.,* **81,** 1703–5 (1961); *C.A.* **58,** 1302*d* (1963).

(634) Kirschner, M. A., Fales, H. M., ANAL. CHEM. **34** (12), 1548–1551 (1962).

(635) Kiryakov, H., *Compt. Rend. Acad. Bulgare Sci.* **16** (1), 43–4 (1963); *C.A.* **60,** 2723*f* (1964).

(636) *Ibid.,* pp. 385–7 (1963); *C.A.* **60,** 5281*b* (1964).

(637) Kiss, L., Zajta, E., Rozsondai, B., *Gyogyszereszet* **6,** 129–35 (1962); *C.A.* **57,** 11315*f* (1962).

(638) Klavehn, M., Rochelmeyer, H., Seyfried, J., *Deut. Apotheker-Ztg.* **101** (4), 75–8 (1961); *C.A.* **60,** 10477*e* (1964).

(639) Kleflin, Z., *Acta Pharm. Jugoslav.* **11,** 23–31 (1961); *C.A.* **57,** 959*b* (1962).

(640) Klein, S., Kho, B. T., *J. Pharm. Sci.* **51** (10), 966–970 (1962).

(641) Kliman, B., Foster, D. W., *Anal. Biochem.* **3,** 403–7 (1962).

(642) Klimaviciene, E., *Lietuvos TSR Aukstuju Mokyklu Mokslo Darbai, Med.* **4,** 197–205 (1963); *C.A.* **60,** 2723*e* (1964).

(643) Kloecking, H. P., *Pharmazie* **16,** 604–11 (1961); *C.A.* **57,** 2338*a* (1962).

(644) Kloecking, H. P., *Ibid.,* **17,** 218–19 (1962); *C.A.* **58,** 8851*f* (1963).

(645) Kloecking, H. P., *Ibid.,* **18,** 342–5 (1963); *C.A.* **59,** 15120*c* (1963).

(646) Kloecking, H. P., *Z. Chem.* **2** (10), 310 (1962); *Anal. Abstr.* **10,** 3424 (1963).

(647) Kloecking, H. P., *Z. Med. Labortech.* **3,** 289–300 (1963); *C.A.* **59,** 2591*b* (1963).

(648) Kloecking, H. P., Joschko, E., *Z. Chem.* **2,** 350 (1962); *C.A.* **58,** 7784*g* (1963).

(649) *Ibid.,* p. 373 (1962); *C.A.* **59,** 382*d* (1963).

(650) Kloecking, H. P., Walter, H., *Pharmazie* **17,** 277–80 (1962); *C.A.* **58,** 10040*b* (1963).

(651) *Ibid.,* pp. 525–6; *C.A.* **58,** 5453*e* (1963).

(652) Klofutar, C., Pelan, B., *Acta Pharm. Jugosl.* **13** (1), 3–12 (1963); *Anal. Abstr.* **10,** 5355 (1963).

(653) Klotz, L., Huettenrauch, R., *Arch. Pharm.* **296** (11), 721–5 (1963); *C.A.* **60,** 5281*a* (1964).

(654) Knabe, J., *Pharm. Ztg. Ver. Apotheker-Ztg.* **107,** 69–71 (1962); *C.A.* **60,** 10476*b* (1964).

(655) Knights, B. A., Thomas, G. H., ANAL. CHEM. **34,** 1046–8 (1962).

(656) Knuppen, R., *Z. Vitamin.-Horm.-u.-Fermentforsch.* **12** (5), 355–401 (1963); *Anal. Abstr.* **10,** 5335 (1963).

(657) Kodama, Y., Hoshida, H., Tanabe, H., *Yakuzaigaku* **23,** 158–62 (1963); *C.A.* **60,** 2725*e* (1964).

(658) Koen, V., *Farmatsiya, Sofia,* **12** (3), 36–40 (1962); *Anal. Abstr.* **10,** 4351 (1963).

(659) Koen, V., *Ibid.*, **13** (4), 41–7 (1963); *C.A.* **60,** 2333*h* (1964).
(660) Koenig, K., Pohloudek-Fabina, R., *Pharm. Zentralhalle* **99** (11), 683–91 (1960); *C.A.* **60,** 9102*d* (1964).
(661) Kofler, M., Sommer, P. F., Bolliger, H. R., Schmidli, B., Vecchi, M., *Vitamins and Hormones* **20,** 407–439 (1962); *Anal. Abstr.* **10,** 3464 (1963).
(662) Kogan, O. M., *Farmatsevt. Zh. (Kiev)* **18** (1), 37–43 (1963); *C.A.* **59,** 11187*h* (1963).
(663) Kolesnikova, V. G., *Sb. Trudy Odessk. Med. Inst.* **1961** (15), 155–159; *Anal. Abstr.* **9,** 5161 (1962).
(664) Kolos-Pethes, E., *Gyogyszereszet* **4,** 339–41 (1960); *C.A.* **60,** 5280*h* (1964).
(665) Koluseva, A., Koen, V., *Farmaciya* **10,** 26–8 (1960); *C.A.* **59,** 2593*g* (1963).
(666) Kolusheva, A., Nin'o, N., *Farmatsiya (Sofia)* **9** (4), 36–40 (1959); *C.A.* **59,** 15125*e* (1963).
(667) Kolusheva, A., Nin'o, N., *Ibid.*, **13** (3), 21–5 (1963); *C.A.* **60,** 1542*b* (1964).
(668) Kolusheva, A., Toncheva, P., *Ibid.*, **11,** (6) 13–18 (1961); *C.A.* **58,** 8851*d* (1963).
(669) Konczal, W., Malek, B., *Biul. Inst. Roslin Leozniczych* **8** (4), 145–50 (1962); *C.A.* **59,** 8543*g* (1963).
(670) Konecny, J., Pohorsky, J., Vachek, J., *Cesk. Farm.* **12,** 204–9 (1963); *C.A.* **59,** 1442*g* (1963).
(671) Konii, S., *J. Pharm. Soc. Japan* **82** (11) 1571–1573 (1962); *Anal. Abstr.* **10,** 5376 (1963).
(672) Konkoly-Thege, I., Pungor, E., Schulek, E., *Ann. Univ. Sci. Budapest Rolando Eotvos Nominatae, Sect. Chim.* **2,** 497–502 (1960); *C.A.* **57,** 2334*b* (1962).
(673) Konstantinova, El., Boichinov, Khr., *Farmatsiya (Sofia)* **13** (4), 30–7 (1963); *C.A.* **60,** 2721*e* (1964).
(674) Kopacova, L., Vrba, C., *Farm. Obzor* **32** (1), 1–6 (1963); *C.A.* **60,** 368*c* (1964).
(675) Koppel, V. I., *Aptechn. Delo* **11** (1), 40–4 (1962); *C.A.* **57,** 1219*a* (1962).
(676) Korchagin, V. B., Ermakova, N. M., Druzhinina, E. N., *Antibiotiki* **7** (5), 449–453 (1962); *Anal. Abstr.* **9,** 5398 (1962).
(677) Korchagin, V. B., Korobitskaya, A. A., Chaikovskaya, S. M., *Ibid.*, **7** (6), 562–566 (1962); *Anal. Abstr.* **10,** 324 (1963).
(678) Kori, S., Kono, M., *J. Pharm. Soc. Japan* **81,** 776–8 (1961); *Anal. Abstr.* **9,** 4877 (1962).
(679) Kori, S., Kono, M., *Ibid.*, **82** (9), 1211–1213 (1962); *Anal. Abstr.* **10,** 5356 (1963).
(680) Kori, S., Tamaki, Y., Fujita, M., *Ibid.*, **81** (7), 980–984 (1961); *Anal. Abstr.* **10,** 3840 (1963).
(681) Korzun, B. P., Brody, S. M., *J. Chromatog.* **7,** 337–340 (1962).
(682) Korzun, B. P., Brody, S., *J. Pharm. Sci.* **52** (2), 206–207 (1963).
(683) Korzun, B. P., Dorfman, L., Brody, S. M., ANAL. CHEM. **35** (8), 950–2 (1963).
(684) Koshy, K. T., Mitchner, H., *J. Pharm. Sci.* **52,** 802–3 (1963).
(685) Kovach, A., Vastagh, G., *Acta Pharm. Hung.* **29,** 149–55 (1959); *C.A.* **57,** 12633*h* (1962).
(686) Kovach, A., Vastagh, G., *Gyogyszereszet* **7** (9), 344–7 (1963); *C.A.* **60,** 3948*a* (1964).
(687) Kovacs, L., *Ibid.*, **6,** 259–267 (1962); *C.A.* **57,** 12637*i* (1962).
(688) Kowalczyk-Kaniewska, T., *Acta Polon. Pharm.* **19** (4), 341–344 (1962); *Anal. Abstr.* **10,** 3431 (1963).
(689) Kowalewski, Z., *Biul. Inst. Roslin Leczniczych* **7** (3), 211–238 (1961); *Anal. Abstr.* **9,** 4888 (1962).
(690) Kozuka, H., Motoyoshi, M., *Kagaku Keisatsu Kenkyusho Hokoku* **16** (1), 39–40 (1963); *C.A.* **59,** 1512*h* (1963).
(691) Kracmar, J., *Pharmazie* **16,** 341–50 (1961); *C.A.* **57,** 12630*e* (1962).
(692) Kracmar, J., Sladkova, J., *Ibid.*, **18,** 115–21 (1963); *C.A.* **59,** 1438*f* (1963).
(693) *Ibid.*, pp. 350–3; *C.A.* **60,** 1539*f* (1964).
(694) Kracmar, J., Zyka, J., *Ceskosl. Farm.* **11** (9), 459–463 (1962); *Anal. Abstr.* **10,** 3426 (1963).
(695) Kracmarova, J., Jindra, A., *Ibid.*, **11** (9), 457–459 (1962); *Anal. Abstr.* **10,** 3427 (1963).
(696) Kraus, P., *Cesk. Farm.* **12** (5), 246–9 (1963); *C.A.* **59,** 8544*f* (1963).
(697) Kraus, P., Simane, Z., *Ibid.*, **10** (4), 195–7 (1961); *C.A.* **60,** 6703*b* (1964).
(698) Krepinsky, J., *Ibid.*, **11** (4), 205–210 (1962); *Anal. Abstr.* **9,** 4878 (1962).
(699) Kreutzkamp, R., Hoelscher, P., Krenek, D., Mueller, D., *Deut. Apotheker-Ztg.* **99,** 1273–5 (1959); *C.A.* **60,** 3948*d* (1964).
(700) Krikova, N. I., *Uchenye Zapiski, Pyatigorskii Farm. Inst.* **3,** 138–43 (1959); *C.A.* **58,** 12370*f* (1963).
(701) Kristensen, K., *Dansk Tidsskr. Farm.* **36,** 170–83 (1962); *C.A.* **58,** 4378*h* (1963).
(702) Krivis, A. F., Supp, G. R., ANAL. CHEM. **35** (10), 1411–12 (1963); *C.A.* **59,** 11598*d* (1963).
(703) Krogerus, V. E., *Medd. Norsk Farm. Selskap* **24,** 1–6 (1962); *C.A.* **57,** 13894*b* (1962).
(704) Kubo, F., Imaoka, K., Kaneko, A., *Kyoritsu Yakka Daigaku Kenkyu Nemp* **6/7,** 1–5 (1961/2); *C.A.* **60,** 375*d* (1964).
(705) *Ibid.*, pp. 5–8.
(706) Kudymov, G. I., Savel'eva, G. I., Rubtsov, V. K., *Med. Prom. SSSR* **17** (10), 41–4 (1963); *C.A.* **60,** 7873*d* (1964).
(707) Kuehmstedt, H., Wagner, G., *Pharm. Praxis, Beilage Pharmazie* **1963** (8), 152–60; *C.A.* **59,** 15119*c* (1963).
(708) Kulev, I., Dzhoneidi, M., Neshkova, M., *Farmatsiya (Sofia)* **13** (5), 16–23 (1963); *C.A.* **60,** 6702*e* (1964).
(709) Kunze, F. M., Espinoza, L. B., *J. Assoc. Offic. Agr. Chemists* **46** (5), 899–901 (1963).
(710) Kupcinskaite, R., Navickiene, B., *Lietuvos TSR Aukstuju Mokyklu Mokslo Darbai, Med.* **4,** 207–14 (1963); *C.A.* **60,** 2721*g* (1964).
(711) Kuroda, K., *Yakugaku Zasshi* **83,** 445–54 (1963); *C.A.* **59,** 11192*d* (1963).
(712) Lachman, L., Urbanyi, T., Weinstein, S., *J. Pharm. Sci.* **52,** 244–9 (1963).
(713) Lachman, L., Urbanyi, T., Weinstein, S., Cooper, J., Swartz, C. J., *Ibid.*, **51,** 321–6 (1962).
(714) *Ibid.*, pp. 326–9 (1962).
(715) Lachman, L., Weinstein, S., Urbanyi, T., Ebersold, E., Cooper, J., *Ibid.*, **52,** 241–3 (1963).
(716) Ladomery, L. G., *Australasian J. Pharm. Suppl. No.* **44,** 10–11 (1963); *C.A.* **58,** 10041*h* (1963).
(717) Lang, B., Horvath, G., *Gyogyszereszet* **6,** (5) 178–81 (1962); *C.A.* **57,** 9956*i* (1962).
(718) Langbein, G., Meyer, M., *J. Chromatog.* **8,** 442–448 (1962).
(719) Larbre, Mrs. S., Dorche, M. J., *Bull. Trav. Soc. Pharm. Lyon* **6,** (2) 61–2 (1962); *C.A.* **58,** 6648*g* (1963).
(720) Laszlovszky, J., Barcza, L., *Gyogyszereszet* **7** (5–6), 192–6 (1963); *C.A.* **60,** 3952*h* (1964).
(721) Laszlovszky, J., Schulek, E., *Acta Pharm. Hung.* **29,** 217–22 (1959); *C.A.* **57,** 12634*c* (1962).
(722) Laugel, P., *Compt. Rend.* **255** (4), 692–694 (1962); *Anal. Abstr.* **10,** 3409 (1963).
(723) Laugel, P., Hasselmann, M., *Chim. Anal.* **44** (10), 433–435 (1962); *Anal. Abstr.* **10,** 2874 (1963).
(724) Leal, A. M., Inacio, M. M. L., Leal, M. M. L., *Rev. Port. Farm.* **12,** 476–81 (1962); *C.A.* **59,** 1440*h* (1963).
(725) Leal, A. M., Lopes, M. B. S. R., *Ibid.*, **13,** 48–54 (1963); *C.A.* **59,** 11188*f* (1963).
(726) Lee, K. T., *Anal. Chim. Acta* **26** (3), 285–289 (1962).
(727) Lee, K. T., Ho, Y. H., *J. Pharm. Pharmacol.* **14** (2), 123–124 (1962).
(728) Lee, S., Putnam, N. A., *J. Appl. Chem. (London)* **13,** 69–71 (1963).
(729) Leeman, H. G., Stich, K., *Helv. Chim. Acta* **45,** 1275–83 (1962); *C.A.* **57,** 6246*e* (1962).
(730) Lehman, R. W., *J. Pharm. Sci.* **53** (2), 201–4 (1964).
(731) Lemmich, E., Gjaldbaek, J. Chr., *Dansk Tidsskr. Farm.* **37,** 1–8 (1963); *C.A.* **58,** 10039*e* (1963).
(732) Lenartowicz, K., *Acta Polon. Pharm.* **20** (3), 233–235 (1963); *Anal. Abstr.* **11,** 1453 (1964).
(733) Leonard, M. A., *J. Pharm. Pharmacol.* **14** (*Suppl.*), 63T–65T (1962).
(734) Lerner, M., Mills, A. L., *Bull. Narcotics, U. N. Dept. Social Affairs* **15** (1), 37–42 (1963); *C.A.* **59,** 3719*c* (1963).
(735) Lerner, M., Mills, A. L., Mount, S. F., *J. Forensic Sci.* **8,** 126–31 (1963).
(736) Lespagnol, A., Devergnies, M., *Bull. Soc. Pharm. Lille* **1961,** (2) 85–7; *C.A.* **57,** 11311*b* (1962).
(737) Levine, J., *J. Assoc. Offic. Agr. Chemists* **45,** 595–7 (1962).
(738) Levine, J., *Ibid.*, **46** (4), 641–2 (1963).
(739) Levine, J., *J. Pharm. Sci.* **52** (11), 1015–31 (1963).
(740) Levine, V. E., Nachman, M., *J. Forensic Med.* **10,** 65–86 (1963).
(741) Leyda, J. P., Harris, L. E., *J. Pharm. Sci.* **51,** 725–8 (1962).
(742) Liang, P., Chou, T.-H., *Yao Hsueh Pao* **8,** 181–5 (1960); *C.A.* **57,** 13888*a* (1962).
(743) Liang, P., Chou, T.-H., *Ibid.*, **10** (2), 87–90 (1963); *C.A.* **59,** 13768*h* (1963).
(744) Lidaks, M., Lejins, Z., Shimanskay, M. V., *TioTEFA, Akad. Nauk Latv. SSR, Inst. Organ. Sinteza* **1961,** 9–17; *C.A.* **58,** 1304*b* (1963).
(745) Linek, K., Peciar, C., *Chem. Zvesti* **16,** 692–700 (1962); *C.A.* **58,** 8409*h* (1963).
(746) Lipchinski, A., Kulev, I., Neshkova, M., *Pharmazie* **18,** 616–18 (1963); *C.A.* **60,** 1539*d* (1964).
(747) Lippmann, I., Mattocks, A. M., *Sci. Sect. Am. Pharm. Assoc., Preprints Papers, Las Vegas, Nev. 1962,* **D-11,** 11 pp.; *C.A.* **60,** 1544*e* (1964).
(748) Lisboa, B. P., *Act Endocr. Copenhagen* **43** (1), 47–66 (1963); *Anal. Abstr.* **10,** 5341 (1963).
(749) Lisboa, B. P., *J. Chromatog.* **13** (2), 391–407 (1964).
(750) Lisboa, B. P., Diczfalusy, E., *Acta Endocr., Copenhagen* **40** (1), 60–81 (1962); *Anal. Abstr.* **10,** 767 (1963).
(751) Lisboa, B. P., Diczfalusy, E., *Ibid.*, **43** (4), 545–560 (1963); *Anal. Abstr.* **11,** 1436 (1964).
(752) List, P. H., Stein, E., *Dtsch. ApothZtg.* **102** (46), 1497–1498 (1962); *Anal. Abstr.* **10,** 3846 (1963).
(753) Litvinenko, L. M., Arlozorov, D. G., Koroleva, V. I., *Uch. Zap. Khar'kovsk. Univ., No.* **110,** *Tr. Khim.*

Fak. i Nauchn.-Issled. Inst. Khim., Khar'kovsk. Gos. Univ. No. **17,** 39–41 (1961); *C.A.* **57,** 2334*e* (1962).

(754) Liukkonen, A., *Arch. Pharm. Chemi* **68** (10), 493–500 (1961); *Anal. Abstr.* **9,** 4876 (1962).

(755) Liukkonen, Anna, *Farm. Aikakauslehti* **71,** 329–40 (1962); *C.A.* **58,** 8850*h* (1963).

(756) Lobanov, V. I., *Aptechn. Delo* **11** (3), 66–7 (1962); *C.A.* **60,** 10477*h* (1964).

(757) Lobanov, V. I., *Farmatsevt. Zh. (Kiev)* **18** (1), 49–51 (1963); *C.A.* **59,** 12594*b* (1963).

(758) Lobanov, V. I., *Sudebno-Med. Ekspertiza, Min. Zdravookhr. SSSR* **6** (1), 39–44 (1963); *C.A.* **59,** 3718*b* (1963).

(759) Locock, R., Teare, F. W., *Can. Pharm. J.* **95,** 536–41 (1962).

(760) Longo, E., *Boll. Chim. Farm.* **101,** 31–33 (1962); *Anal. Abstr.* **10,** 1167 (1963).

(761) *Ibid.*, pp. 41–2; *C.A.* **57,** 2336*e* (1962).

(762) Lucas, G. H. W., Fabierkiewicz, C., *J. Forensic Sci.* **8** (3), 462–76 (1963).

(763) Luduena, F. P., Snyder, A. L., Lands, A. M., *J. Pharm. Pharmacol.* **15,** 538–43 (1963).

(764) Ludwicki, H., Sobiczewska, M., *Farm. Polska* **19** (11–12), 228–30 (1963); *C.A.* **60,** 370*g* (1964).

(765) Luedy-Tenger, F., *Pharm. Acta Helv.* **37,** 770–89 (1962); *C.A.* **58,** 7784*a* (1963).

(766) Luers, R. B., Jr., Stadler, L. B., *J. Pharm. Sci.* **51** (9), 910–911 (1962).

(767) Lukas, G., *Sci. Pharm.* **30** (1), 47–53 (1962); *Anal. Abstr.* **9,** 4889 (1962).

(768) Lukats, B., Nagy, G. T., Gyenes, I., *Gyogyszereszet* **4,** 264–9 (1960); *C.A.* **60,** 5278*e* (1964).

(769) Luk'yanchikova, G. I., *Med. Prom. SSSR* **16,** (9) 46–8 (1962); *C.A.* **58,** 5454*h* (1963).

(770) Luk'yanchikova, G. I., *Uch. Zap., Pyatigorskii Gos. Farmatsevt. Inst.* **5,** 199–202 (1961); *C.A.* **59,** 1441*b* (1963).

(771) *Ibid.*, pp. 213–17; *C.A.* **59,** 1441*e* (1963).

(772) Luk'yanchikova, G. I., *U.S.S.R.* **148,956,** July 20, 1962, (Appl. Aug. 1, 1961); *C.A.* **58,** 1311*h* (1963).

(773) Luk'yanchikova, G. I., Bernshtein, V. N., *Sb. Nauchn. Dokl., Stavropol'sk. Kraevoe Otd. Vses. Khim. Obshchestva im. D. I. Mendeleeva* **1960** (1), 28–30; *C.A.* **59,** 3719*e* (1963).

(774) *Ibid.*, pp. 31–4; *C.A.* **59,** 3719*f* (1963).

(775) Lutynski, E. W., *Dissertationes Pharm.* **15,** 205–13 (1963); *C.A.* **60,** 371*a* (1964).

(776) Luukkainen, T., Vanden Heuvel, W. J. A., Horning, E. C., *Biochim. Biophys. Acta* **62** (1), 153–159 (1962); *Anal. Abstr.* **9,** 5384 (1962).

(777) MacDonald, A., Jr., Pflaum, R. T., *J. Pharm. Sci.* **52,** 816 (1963).

(778) Macek, K., Vanecek, S., *Pharmazie* **17,** 442–4 (1962); *C.A.* **58,** 12368*g* (1963).

(779) Machek, G., Lorenz, F., *Sci. Pharm.* **30,** 25–37 (1962); *Anal. Abstr.* **9,** 4872 (1962).

(780) Machek, G., Lorenz, F., *Ibid.*, **31,** 17–26 (1963); *C.A.* **59,** 1440*d* (1963).

(781) Madgearu, M., Prodescu, M., Solomon-Ionescu, I., *Farmacia (Bucharest)* **10,** 555–7 (1962); *C.A.* **58,** 7784*h* (1963).

(782) Madiwale, M. S., Rao, S. S., Dutta, N. K., *Indian J. Pharm.* **24,** 286–9 (1962); *C.A.* **58,** 6647*b* (1963).

(783) Maehly, A. C., Linturi, M. K., *Acta Chem. Scand.* **16** (2), 283–292 (1962); *Anal. Abstr.* **9,** 3885 (1962).

(784) Magnusson, G., *Nature* **195,** 591–2 (1962).

(785) Majlat, P., Bayer, I., *Acta Pharm. Hung.* **32,** 116–18 (1962); *C.A.* **57,** 4766*d* (1962).

(786) Mainville, C. A., Chatten, L. G., *J. Pharm. Sci.* **53** (2), 154–7 (1964).

(787) Mandak, M., Struhar, M., Klucarova, H., *Farm. Obzor* **32** (3), 97–105 (1963); *C.A.* **60,** 367*g* (1964).

(788) Marciszewski, H., *Chem. Anal. (Warsaw)* **5,** 617–24 (1960); *C.A.* **57,** 9956*d* (1962).

(789) Marcus, A. D., DeMarco, J. D., *J. Pharm. Sci.* **52** (4), 402–403 (1963).

(790) Marcuse, R., *J. Chromatog.* **7,** 407–8 (1962).

(791) Marenzi, A. D., Braegger, E. S., *Rev. Asoc. Bioquim. Arg.* **26,** (133–4) 45–8 (1961); *C.A.* **57,** 10131*f* (1962).

(792) Margarida, M., Braga, F., Baltazar, J., *Rev. Port. Farm.* **13** (2), 145–50 (1963); *C.A.* **60,** 368*d* (1964).

(793) Mariani, A., Vicari, C., *Farm. Aikakauslehti* **72,** 277–85 (1963); *C.A.* **60,** 373*a* (1964).

(794) Marini-Bettolo, G. B., *J. Chromatog.* **7,** 329–336 (1962).

(795) Marjanovic, M., Blagojevic, Z., *Arhiv Farm. (Belgrade)* **12** (5), 330–40 (1962); *C.A.* **60,** 368*a* (1964).

(796) Martin, J. L., Shaw, W. H. C., *Analyst* **88,** 292–296 (1963).

(797) Martonfi, L., Szantho, C. St. N., Formanek, Gy., *Rev. Med. (Targu-Mures)* **8,** 308–12 (1962); *C.A.* **59,** 381*e* (1963).

(798) Maruyama, M., Hasegawa, K., *Japan Analyst* **10** (5), 518–522 (1961); *Anal. Abstr.* **10,** 3307 (1963).

(799) Mary, N. Y., Brochmann-Hanssen, E., *Lloydia* **26,** 223–8 (1963); *C.A.* **60,** 7871*h* (1964).

(800) Masuda, Y., Ikekawa, N., *J. Pharm. Soc. Japan* **82** (12), 1664–1666 (1962); *Anal. Abstr.* **10,** 5367 (1963).

(801) Matis, J., Adamec, O., Galvanek, M., *Nature* **194,** 477–478 (1962).

(802) Matrka, M., Podstata, J., Sagner, Z., *Chem. Prumysl* **12** (10), 549–51 (1962); *Anal. Abstr.* **10,** 3326 (1963).

(803) Matsukawa, T., Yurugi, S., Oka, Y., *Ann. N. Y. Acad. Sci.* **98,** Art. 2, 430–44 (1962); *C.A.* **60,** 6706*f* (1964).

(804) Matsuoka, M., *Nippon Kagaku Zasshi* **82,** 1193–7 (1961); *C.A.* **57,** 13187*d* (1962).

(805) *Ibid.*, pp. 1198–1202 (1961); *C.A.* **57,** 13187*e* (1962).

(806) Matthews, J. S., *Biochim. Biophys. Acta* **69,** 163–5 (1963); *C.A.* **58,** 14043*d* (1963).

(807) Matthews, J. S., de los Angeles, M., *J. Chromatog.* **9,** 195–8 (1962).

(808) Matthews, J. S., Pereda V. A. L., Aguilera P. A., *J. Chromatog.* **9,** 331–338 (1962).

(809) Maturana, M. H., Dannier, C. A., Brieva, A. J., *Rev. Real Acad. Cienc. Exact., Fis. Nat. Madrid* **56,** 365–82 (1962); *C.A.* **57,** 13888*c* (1962).

(810) Maturova, M., *Chem. Zvesti* **16,** 375–9 (1962); *C.A.* **58,** 6645*h* (1963).

(811) Mauryama, M., Hayashi, N., Kishi, M., *Ann. Rep. Takamine Lab.* **13,** 176–181 (1961); *Anal. Abstr.* **11,** 327 (1964).

(812) Maybury, R. B., Barrette, J. P., Payfer, R., *J. Assoc. Offic. Agr. Chemists* **46** (6), 1060–2 (1963).

(813) Mazon, M., Serwin-Krajewska, M., *Chem. Anal., Warsaw* **7** (3), 653–657 (1962); *Anal. Abstr.* **10,** 2468 (1963).

(814) Mazor, L., Papay, M. K., *Acta Pharm. Hung.* **32,** 59–62 (1962); *C.A.* **57,** 956*g* (1962).

(815) Medvesek, L., *Farm. Vestnik (Ljubljana)* **12** (5–8), 97–108 (1961); *C.A.* **57,** 7386*g* (1962).

(816) Medwick, T., Schiesswohl, F., *J. Pharm. Sci.* **52** (9), 843–6 (1963).

(817) Meier, G. N., Kuhn, O. S., *Anales Real Acad. Farm.* **28,** 195–204 (1962); *C.A.* **58,** 8853*d* (1963).

(818) Melichar, B., Horka, J., Necesana, E., Malta, D., *Cesk. Farm.* **8** (10), 423–6 (1959); *C.A.* **60,** 7873*b* (1964).

(819) Melichar, B., Jakubec, I., *Ibid.*, **9** (3), 72–5 (1960); *C.A.* **60,** 7873*b* (1964).

(820) Mellinger, T. J., Keeler, C. E., *J. Pharm. Sci.* **51,** 1169–73 (1962).

(821) Melson, F., Hofmann, H., *Pharm. Zentralh.* **102** (2), 59–69 (1963); *Anal. Abstr.* **11,** 337 (1964).

(822) Menyharth, P., *Arch. Pharm.* **295,** 412–15 (1962); *C.A.* **57,** 9958*b* (1962).

(823) Merlis, V. M., Bagreeva, M. R., Veselovskaya, G. G., *Med. Prom. SSSR* **16,** (11) 33–6 (1962); *C.A.* **58,** 12370*h* (1963).

(824) Merodio, J. C., *An. Asoc. Quim. Argentina* **49** (3), 225–236 (1961); *Anal. Abstr.* **9,** 4820 (1962).

(825) Mesnard, P., Crockett, R., *Congr. Sci. Farm., Conf. Comun., 21°, Pisa,* **1961,** 702–9; *C.A.* **59,** 1441*f* (1963).

(826) Mesnard, P., Devaux, G., *Chim. Anal. (Paris)* **44,** 287–91 (1962); *C.A.* **58,** 5454*f* (1963).

(827) Metcalfe, L. D., Anal. Chem. **34,** 1849 (1962).

(828) Michelon, L. E., Kelleher, W. J., *Lloydia* **26,** 192–201 (1963); *C.A.* **60,** 1538*f* (1964).

(829) Milch, G., *Acta Pharm. Hung.* **32,** 55–8 (1962); *C.A.* **58,** 962*f* (1962).

(830) Milch, G., *Acta Pharm. Hung.* **32** (5), 206–210 (1962); *Anal. Abstr.* **10,** 2887 (1963).

(831) Milch, G., *Acta Pharm. Hung.* **33** (6), 257–64 (1963); *C.A.* **60,** 6705*b* (1964).

(832) Milch, G., *Gyogyszereszet* **4,** 342–3 (1960); *C.A.* **60,** 5279*g* (1964).

(833) Miller, R. P., *Antibiot. & Chemother.* **12** (12), 689–693 (1962).

(834) Mills, A. L., Anal. Chem. **35,** 416 (1963).

(835) Mincsev - Ralcseva, M., *Gyogyszereszet* **4,** 344–7 (1960); *C.A.* **60,** 6702*f* (1964).

(836) Mincsev-Ralcseva, M., *Ibid.*, **6,** 270–2 (1962); *C.A.* **57,** 16748*a* (1962).

(837) Mincsev-Ralcseva, M., *Ibid.*, **7** (5–6), 197–8 (1963); *C.A.* **69,** 3954*b* (1964).

(838) Mirimanoff, A., *Rev. Real Acad. Farm. (Barcelona)* **8,** 15–20 (1961); *C.A.* **59,** 12593*c* (1963).

(839) Mitra, R. K., Ray, G. K., *Indian J. Pharm.* **25,** 262–4 (1963); *C.A.* **59,** 8543*h* (1963).

(840) Mitra, R. K., Bose, P. C., Ray, G. K., Mukerji, B., *Ibid.*, **24** (7), 152–154 (1962); *Anal. Abstr.* **10,** 782 (1963).

(841) Mizsei, A., Szabo, A., *Nature* **196,** 1199–1200 (1962).

(842) Modrzejewski, B., Zommer, S., *Chem. Anal., Warsaw* **7** (3), 659–666 (1962); *Anal. Abstr.* **10,** 2466 (1963).

(843) Mody, S. M., Naik, R. N., *J. Pharm. Sci.* **52,** 201–2 (1963).

(844) Mody, S. M., Naik, R. N., Agrawal, S. R., *Indian J. Pharm.* **24,** 259–61 (1962); *C.A.* **58,** 3270*e* (1963).

(845) Moffitt, R. A., Meckler, W. G., Sautier, P. M., Sanderson, D. W., *J. Assoc. Offic. Agr. Chemists* **45,** 787–8 (1962).

(846) Mohay, J., Kedvessy, Gy., *Gyogyszereszet* **7,** 257–9 (1963); *C.A.* **60,** 2724*b* (1964).

(847) Mohay-Farkas, J., Mohay, J., *Ibid.*, **5,** 61–4 (1961); *C.A.* **60,** 5284*e* (1960).

(848) Mohoric, J., *Farm. Vestik (Ljubl-*

jana) **14** (4–6), 74–6 (1963); *C.A.* **60,** 5280*f* (1964).

(849) Moira, C., Contaxi, C., *Chim. Chronika, Athens* **26** (4), 65–69 (1961); *Anal. Abstr.* **9,** 4425 (1962).

(850) Moll, F., *Arch. Pharm., Berlin* **296** (3), 205–207 (1963); *Anal. Abstr.* **11,** 740 (1964).

(851) Moll, F., *Naturwissenschaften* **49,** 450 (1962); *C.A.* **57,** 16746*c* (1962).

(852) Momose, T., Matsukuma, T., Ohkura, Y., Nakamura, Y., *J. Pharm. Soc. Japan* **83** (2), 143–147 (1963); *Anal. Abstr.* **11,** 1457 (1964).

(853) Momose, T., Ueda, Y., Mukai, Y., Watanabe, K., *J. Pharm. Soc. Japan* **80** (2), 225–228 (1960); *Anal. Abstr.* **9,** 4472 (1962).

(854) Monnier, D., Ghaliounghi, Y., *Chimia* **16** (10), 340 (1962); *C.A.* **58,** 7788*d* (1963).

(855) Monnier, D., Ghaliounghi, Y., Saba, R., *Anal. Chim. Acta* **28,** 30–40 (1963); *C.A.* **58,** 7788*e* (1963).

(856) Monnier, D., Keller, H., *Helv. Chim. Acta* **45** (1), 290–308 (1962); *Anal. Abstr.* **9,** 3384 (1962).

(857) Monnier, D., Saba, R., Galiounghi, Y., *Ibid.*, **46** (7), 2558–65 (1963); *C.A.* **60,** 5285*a* (1964).

(858) Montequi, R., Fernandez de Valderrama, E., *Anales Real Acad. Farm.* **28,** 133–42 (1962); *C.A.* **58,** 7787*b* (1963).

(859) Moody, J. E., Jr., *J. Pharm. Sci.* **52,** 791–3 (1963).

(860) de Moras, J., Parado, Cl., Drevon, B., *Bull. Trav. Soc. Pharm. Lyon* **7** (3), 87–94 (1963); *C.A.* **60,** 7874*b* (1964).

(861) Morotomi, Y., Sekine, A., *Tekamine Kenkyusho Nempo* **14,** 73–8 (1963); *C.A.* **58,** 5455*b* (1963).

(862) Morris, W. W., Jr., Wilkie, J. B., Jones, S. W., Friedman, L., ANAL. CHEM. **34,** 381–4 (1962).

(863) Morvai, J., Gaizer, F., *Gyogyszereszet* **4,** 300–1; *C.A.* **60,** 7872*e* (1964).

(864) Morvay, J., *Acta Pharm. Hung.* **28,** (5–6) 200–6 (1958); *C.A.* **57,** 12636*b* (1962).

(865) Moses, V., *J. Chromatog.* **9** (2), 241–243 (1962).

(866) Murai, K., *Arch. Prac. Pharm., Japan* **21** (1), 58–60 (1961); *Anal. Abstr.* **10,** 3870 (1963).

(867) *Ibid.*, (2) pp. 124–127; *Anal. Abstr.* **10,** 3863 (1963).

(868) Murai, K., *J. Pharm. Soc. Japan* **81** (7), 1013–1016 (1961); *Anal. Abstr.* **10,** 3866 (1963).

(869) Murea, L., Pintilie, S., Sterescu, M., *Rev. Chim. (Bucharest)* **13,** 171 (1962); *C.A.* **57,** 1388*i* (1962).

(870) Murea, L., Wermescher, B., Beral, H., *Ibid.*, 12723–6 (1961); *C.A.* **57,** 16750*g* (1962).

(871) Murray, T. K., ANAL. CHEM. **34,** 1241–4 (1962).

(872) Murray, T. K., Pelletier, O., Campbell, J. A., *J. Pharm. Pharmacol.* **15** (3), 192–196 (1963).

(873) Musil, F., *Pracovni Lekarstvi* **14** (9), 426–429 (1962); *Anal. Abstr.* **10,** 2863 (1962).

(874) Musons, J., *Galenica Acta (Madrid)* **15,** 441–86 (1962); *C.A.* **59,** 15121*c* (1963).

(875) Mustafaev, M. M., *Uch. Zap., Pyatigorskii Gos. Farmatsevt. Inst.* **5,** 207–8 (1961); *C.A.* **59;** 2592*b* (1963).

(876) Muth, C. W., Darlak, R. S., English, H., Hammer, A. T., ANAL. CHEM. **34,** 1163–4 (1962).

(877) Nagase, Y., Baba, S., *J. Pharm. Soc. Japan* **81** (5), 619–621 (1961). *Anal. Abstr.* **9,** 4885 (1962).

(878) Naik, R. N., *J. Proc. Inst. Chemists (India)* **35,** 200–1 (1963); *C.A.* **60,** 7872*a* (1964).

(879) Nakamura, G. R., *J. Assoc. Offic. Agr. Chemists* **46** (4), 769–70 (1963).

(880) Nakamura, G. R., Ukita, C., *J. Forensic Sci.* **7,** 465–73 (1962).

(881) Nakano, M., *Yakuzaigaku* **22,** 158–60 (1962); *C.A.* **58,** 8852*e* (1963).

(882) *Ibid.*, pp. 267–9; *C.A.* **59,** 6199*b* (1963).

(883) Nakata, T., *Bitamin* **26,** 213–16 (1962); *C.A.* **60,** 10478*e* (1964).

(884) Nakatani, H., *Yakuzaigaku* **23,** 75–7 (1963); *C.A.* **59,** 12596*h* (1963).

(885) Narasimhachari, N., Rao, R. G., *Hindustan Antibiot. Bull.* **4,** 163–7 (1962); *C.A.* **57,** 16758*b* (1962).

(886) Nedergaard, M., *Arch. Pharm. Chemi* **69** (22), 745–750 (1962); *Anal. Abstr.* **10,** 4342 (1963).

(887) Nehring, K., Brandhoff, Hildegard, *Pharmazie* **16,** 89–91 (1961); *C.A.* **57,** 15237*i* (1962).

(888) Nelan, D. R., Robeson, C. D., *Nature* **193,** 477 (1962).

(889) Nelson, D. F., Kirk, P. L., ANAL. CHEM. **34** (8), 899–903 (1962).

(890) Nelson, J. P., *J. Gas Chromatog.* **1,** 27–29 (1963).

(891) Netien, G., Mery, L., *Bull. Trav. Soc. Pharm. Lyon* **6** (2), 35–9 (1962); *C.A.* **58,** 7787*d* (1963).

(892) Neto, J. S. A., *Rev. Brasil. Farm.* **44** (3), 151–60 (1963); *C.A.* **60,** 5274*h* (1964).

(893) Neu, R., *J. Chromatog.* **11** (3), 364–8 (1963).

(894) Neubauer, D., Mothes, K., *Planta Med.* **9,** 466–70 (1961); *C.A.* **57,** 2332*c* (1962).

(895) Neuwald, F., Fetting, K. E., *Pharm. Ztg.-Nachr.* **108** (43), 1490–1 (1963); *C.A.* **60,** 5277*g* (1964).

(896) Nicolies, K., *Pharm. Zentralhalle* **102** (7), 432–41 (1963); *C.A.* **59,** 15121*g* (1963).

(897) Niebergall, P. J., *Univ. Microfilms (Ann Arbor, Mich.), Order No.* **62-2772,** 139 pp.; *C.A.* **57,** 16741*g* (1962).

(898) Niemann, A., *J. Chromatog.* **9** (1), 117–118 (1962).

(899) Niijima, K., Okuyama, T., *Seikagaku* **35,** 396–403 (1963); *C.A.* **60,** 6710*b* (1964).

(900) Nikolic, K., *Acta Pharm. Hung.* **33** (3), 125–41 (1963); *C.A.* **60,** 366*e* (1964).

(901) Nikolic, K., Panic, L., *Acta Pharm. Jugoslav.* **11,** 145–9 (1961); *C.A.* **57,** 963*a* (1962).

(902) Nin'o, N., *Farmatsiya (Sofia)* **12** (2), 27–32 (1962); *C.A.* **57,** 6026*f* (1962).

(903) Niwaguchi, T., *Kagaku Keisatsu Kenkyusho Hokoku* **16**; 41–2 (1963); *C.A.* **59,** 15122*a* (1963).

(904) Nogami, H., Awazu, S., *Chem. Pharm. Bull. (Tokyo)* **10,** 1158–60 (1962); *C.A.* **58,** 12372*f* (1963).

(905) Nogami, H., Awazu, S., Iwatsuru, M., *Chem. Pharm. Bull. (Tokyo)* **11** (10), 1251–5 (1963); *C.A.* **60,** 9104*f* (1964).

(906) Nogami, H., Awazu, S., Kanakubo, Y., *Ibid.*, **11,** 13–18 (1963); *C.A.* **58,** 12372*h* (1963).

(907) Nogami, H., Awazu, S., Nakajima, N., *Ibid.*, **10,** 503–11 (1962); *C.A.* **57,** 15245*e* (1962).

(908) Nogami, H., Hasegawa, J., Nagai, T., Miyamoto, M., *Ibid.*, **10,** 747–52 (1962); *C.A.* **59,** 2594*h* (1963).

(909) Nogami, H., Nagai, T., *Ibid.*, **10,** 728–40 (1962); *C.A.* **59,** 2594*f* (1963).

(910) *Ibid.*, pp. 741–6; *C.A.* **59,** 2594*h* (1963).

(911) Nogueira, A. L., Faria, M. J., Ruivo, M. L. S., *Rev. Port. Farm.* **13,** 16–43 (1963).

(912) Nordstrom, C. G., Hakalax, R., Karumaa, L. J., *Suomen Kemistilehti* **36B,** 102–5 (1963); *C.A.* **59,** 7470*g* (1963).

(913) Nour-El-Dine, H. M., El-Mangouri, H. A., *Egypt. Pharm. Bull.* **42,** 325–33 (1960); *C.A.* **58,** 12369*e* (1963).

(914) Nuernberg, E., *Deut. Apotheker-Ztg.* **101** (10), 268–9 (1961); *C.A.* **60,** 372*h* (1964).

(915) Nussbaumer, P. A., *Pharm. Acta Helv.* **37,** 65–72 (1962); *C.A.* **57,** 961*b* (1962).

(916) Nussbaumer, P. A., *Ibid.*, pp. 161–168; *Anal. Abstr.* **9,** 4456 (1962).

(917) Nussbaumer, P. A., *Ibid.*, **38** (10), 758–66 (1963); *C.A.* **60,** 1541 (1964).

(918) Nyiredy, S., *Gogyszereszet* **6,** 459–64 (1962); *C.A.* **58,** 10039*b* (1963).

(919) Nyitray, L., *Acta Pharm. Hung.* **32,** 27–29 (1962); *Anal. Abstr.* **9,** 4879 (1962).

(920) Oakey, R. E., *J. Chromatog.* **8,** 2–16 (1962).

(921) Oberzill, W., *Sci. Pharm.* **30,** 141–63 (1962); *C.A.* **58,** 7784*d* (1963).

(922) Ochab, S., Borowiecka, B., *Chem. Anal., Warsaw* **8** (1), 99–102 (1963); *Anal. Abstr.* **10,** 5363 (1963).

(923) Ochab, S., Malysz, D., Borowiecka, B., *Ibid.*, **8** (4), 597–600 (1963); *C.A.* **60,** 370*c* (1964).

(924) Oelschlager, H., *Arch. Pharm., Berlin* **296** (1), 7–13 (1963); *Anal. Abstr.* **10,** 4886 (1963).

(925) Okazaki, Y., *Bunseki Kagaku* **11,** 1142–6 (1962); *C.A.* **58,** 7786*d* (1963).

(926) *Ibid.*, pp. 1147–51.

(927) *Ibid.*, pp. 1239–42; *C.A.* **58,** 8854*f* (1963).

(928) Okazaki, Y., *J. Pharm. Soc. Japan* **81,** 1618–22 (1961); *Anal. Abstr.* **10,** 3880 (1963).

(929) Okuda, S., Yamaguchi, S., Kawazoe, Y., Tsuda, K., *Chem. Pharm. Bull. (Tokyo)* **12** (1), 104–12 (1964); *C.A.* **60,** 14030*c* (1964).

(930) Okuda, Y., Horino, S., Masaki, H., Furukawa, Y., Yamasaki, M., *Rev. Polarog. (Kyoto)* **9,** 174–6 (1961); *C.A.* **60,** 5279*d* (1964).

(931) Olechnowicz-Stepien, W., Rzadkowska, H., *Dissert. Pharm., Krakow* **14** (3), 331–338 (1962); *Anal. Abstr.* **10,** 3418 (1963).

(932) Omboly, C., Derzsi, E., *Acta Pharm. Hung.* **32** (6), 246–250 (1962); *Anal. Abstr.* **10,** 3886 (1963).

(933) Onesanu, D., *Acad. Rep. Populare Romine, Studii Cercetari Biochim.* **6** (2), 281–4 (1963); *C.A.* **59,** 11189*b* (1963).

(934) Opavsky, J., Moudry, R., *Tech. Publ. Stredisko Tech. Inform. Potravinar. Prumyslu No.* **139,** 106–10 (1963); *C.A.* **60,** 6706*g* (1964).

(935) Ordonez, C. R., Celsi, S. A., *Rev. Farm. (Buenos Aires)* **104** (3–4), 55–61 (1962); *C.A.* **57,** 9956*a* (1962).

936) Orlov, Yu. E., Dzyuba, N. P., Shostenko, Yu. V., *Farmatsevt. Zh. (Kiev)* **18** (5), 36–9 (1963); *C.A.* **60,** 5279*b* (1964).

(937) Oslet-Conter, J., *Arch. Pharm. Chemi* **68** (11), 529–556 (1961); *Anal. Abstr.* **9,** 4902 (1962).

(938) Otani, S., *Yakuzaigaku* **23,** 138–44 (1963); *C.A.* **60,** 5286*c* (1964).

(939) Outch, W. T., *Australasian J. Pharm.* **42,** 1277–84 (1961); *C.A.* **57,** 8661*a* (1962).

(940) Ovechkin, G. V., *Zh. Obshch. Khim.* **33** (6), 1923–7 (1963); *C.A.* **59,** 11190*b* (1963).

(941) Ozsoz, B., *Turk Ijiyen Tecrubi Biyol. Dergisi* **22,** 59–63 (1962); *C.A.* **57,** 9957*d* (1962).

(942) Ozsoz, B., *Ibid.*, **23** (2), 172–8 (1963); *C.A.* **60,** 1541*g* (1964).

(943) Palmer, J. F., *J. Pharm. Pharmacol.* **15** (11), 777–8 (1963).

(944) Pan, S. C., ANAL. CHEM. **34,** 766–70 (1962).

(945) Pan, S. C., *J. Chromatog.* **8,** 449–456 (1962).
(946) Pan, S. C., *Ibid.*, **9,** 871–91 (1962).
(947) Panalaks, T., *J. Pharm. Sci.* **52,** 205–6 (1963).
(948) Panalaks, T., Campbell, J. A., Anal. Chem. **34,** 64–66 (1962).
(949) Panier, R. G., Close, J. A., *J. Pharm. Sci.* **53** (1), 108–10 (1964).
(950) Pankiewicz, H., *Farm. Polska* **19** (11–12), 234–8 (1963); *C.A.* **60,** 371*b* (1964).
(951) Panwar, K. S., Gaur, J. N., *J. Electroanal. Chem.* **3,** 348–9 (1962).
(952) Papariello, G. J., Mader, W. J., *J. Pharm. Sci.* **52,** 209–17 (1963).
(953) Pappas, A. C., Alstad, J., Lunde, G., *Radiochim. Acta* **1** (3), 109–117 (1963); *Anal. Abstr.* **11,** 1455 (1964).
(954) Paraak, V., *Acta Chim. Acad. Sci. Hung.* **33,** 121–33 (1962); *C.A.* **58,** 4382*d* (1963).
(955) Parikh, P. M., Mukherji, S. P., *Indian J. Pharm.* **25,** 269–70 (1963); *C.A.* **59,** 8544*b* (1963).
(956) Paris, R. R., Rousselet, R., *Farm. Aikakauslehti* **72,** 261–9 (1963); *C.A.* **60,** 367*c* (1964).
(957) Parker, K. D., Fontan, C. R., Kirk, P. L., Anal. Chem. **34,** 757–760 (1962).
(958) *Ibid.*, pp. 1345–6.
(959) Parker, K. D., Fontan, C. R., Kirk, P. L., *Ibid.*, **35,** 356–359 (1963).
(960) *Ibid.*, pp. 418–19.
(961) Parrak, V., *Aptechn. Delo* **11** (2), 79–82 (1962); *C.A.* **60,** 10477*a* (1964).
(962) Pasich, J., Stasiewska, K., *Acta Polon. Pharm.* **19** (2), 181–182 (1962); *Anal. Abstr.* **10,** 1170 (1963).
(963) Pasich, J., Stasiewska, K., *Farm. Polska* **18,** 261–2 (1962); *C.A.* **58,** 1303*h* (1963); *Anal. Abstr.* **10,** 1169 (1963).
(964) Pasich, J., Stasiewska, K., Szczesniewska, B., *Ibid.*, **10,** 508–9 (1961); *C.A.* **57,** 4762*d* (1962).
(965) Patel, N. K., *Univ. Microfilms (Ann Arbor, Mich.), Order No.* **62-4589,** 154 pp.; *Dissertation Abstr.* **23,** 1522–3 (1962).
(966) Patel, N. K., Foss, N. E., *J. Pharm. Sci.* **53** (1), 94–7 (1964).
(967) Paulig, G., Gansau, H., Knorr, P., Erbe, L., *Z. Anal. Chem.* **193** (1), 28–33 (1963); *Anal. Abstr.* **10,** 4884 (1963).
(968) Paulssen, R. B., Waaler, T., *Pharm. Acta Helv.* **37** (3), 125–132 (1962); *Anal. Abstr.* **9,** 4473 (1962).
(969) Paulus, W., Hoch, W., Keymer, R., *Arzneimittel-Forsch.* **13** (7), 609–10 (1963); *C.A.* **59,** 11190*c* (1963).
(970) Paulus, W., Janitzki, U., Hoch, W., *Ibid.*, **12,** 950–2 (1962); *C.A.* **58,** 7785*g* (1963).
(971) *Ibid.*, pp. 1086–7 (1962); *C.A.* **58,** 6647*c* (1963).
(972) Pavlovic, V., Trpinac, P., *Arhiv. Farm. (Belgrade)* **12,** 221–3 1962); *C.A.* **58,** 8852*b* (1963).
(973) Pawelczyk, E., Dopierala, T., *Farm. Polska* **18,** 234–7 (1962); *C.A.* **58,** 1303*g* (1963).
(974) Pawlaczyk, J., *Bull. Soc. Sci. Lettres Poznan Ser. C* **11,** 91–5 (1962); *C.A.* **57,** 15241*g* (1962).
(975) Peciar, C., Linek, K., *Chem. Zvesti* **15,** 895–901 (1961); *C.A.* **58,** 3271*g* (1963).
(976) Peciar, C., Linek, K., *Czech.* **105,-405,** Oct. 15, 1962, Appl. Apr. 10, 1961; *C.A.* **59,** 13771*f* (1963).
(977) Pellerin, F., Gautier, J. A., Barat, O., Demay, D., *Chim. Anal. (Paris)* **45,** 395–8 (1963); *C.A.* **59,** 11189*h* (1963).
(978) Pellerin, F., Gautier, J. A., Demay, D., *Ann. Pharm. Franc.* **20,** No. 2, 97–104 (1962); *C.A.* **57,** 15243*c* (1962).
(979) Pelletier, O., Campbell, J. A., *J. Pharm. Sci.* **51** (6), 594–595 (1962).
(980) Pelloni, V., Simionovici, R., *Rev. Chim. (Bucharest)* **13,** No. 3, 171–2 (1962); *C.A.* **57,** 9957*i* (1962).
(981) Pereira, J. N., *J. Biochem. Microbiol. Technol. Engng.* **3,** 79–85 (1961).
(982) Perelman, Ya. M., Evstratova, K. I., *Tr. Leningr. Khim. Farmatsevt. Inst.* **14,** 38–40 (1962); *C.A.* **60,** 10478*b* (1964).
(983) Pernarowski, M., Knevel, A. M., Christian, J. E., *J. Pharm. Sci.* **51** (7), 688–693 (1962).
(984) Pernarowski, M., Padval, V. A., *Ibid.*, **52** (3), 218–223 (1963).
(985) Pesakhovich, L. V., *Aptechn. Delo* **11** (3), 67–9 (1962); *C.A.* **60,** 10480*c* (1964).
(986) Pesez, M., *Lyon Pharm.* **14** (8), 363–4, 367–70, 373–4, 377–80, 383–4, 387–90, 393–4, 397–8 (1963); *C.A.* **60,** 5273*h* (1960).
(987) Pesez, M., Bartos, J., *Talanta* **10** (1), 69–73 (1963).
(988) Pesez, M., Robin, J., *Bull. Soc. Chim. France* **1962,** 1930–2; *C.A.* **58,** 11425*g* (1963).
(989) Petering, H. G., Van Giesen, G. J., *J. Pharm. Sci.* **52** (12), 1159–62 (1963).
(990) Petokovic, M., *Arhiv. Farm.* **13** (3), 161–3 (1963); *C.A.* **60,** 368*h* (1964).
(991) Petricic, J., Benas, L., *Acta Pharm. Jugoslav.* **13,** 63–8 (1963); *C.A.* **59,** 11187*e* (1963).
(992) Petrova, V. V., *Lekarstv. Syr'evye Resursy Irkutskoi Oblasti, Irkutsk, Sb.* (**3**), 174–84 (1961); *C.A.* **60,** 370*h* (1964).
(993) Pfandl, Kurt, *Deut. Apotheker Ztg.* **103,** 365–71 (1963); *C.A.* **59,** 382*e* (1963).
(994) Pfeifer, S., *Pharmazie* **17,** 128–133 (1962); *Anal. Abstr.* **10,** 3842 (1963).
(995) Pfeifer, S., *Ibid.*, **18,** 409–10 (1963); *C.A.* **60,** 371*e* (1964).
(996) Pico, F., Bastus, J., *Galencia Acta (Madrid)* **15** (5), 383–91 (1962); *C.A.* **59,** 6996*a* (1963).
(997) Pierzchalski, T., *Chem. Anal., Warsaw* **6** (5), 845–854 (1961); *Anal. Abstr.* **9,** 2890 (1963).
(998) Piette, L. H., Forrest, I. S., *Biochem. Biophys. Acta* **57,** 419–20 (1962).
(999) Pinella, S. J., *J. Assoc. Offic. Agr. Chemists* **46,** 649–51 (1963).
(1000) Pinto, O., Carrilho, O. M., *Rev. Portug. Farm.* **12** (1), 1–3 (1962); *Anal. Abstr.* **10,** 3878 (1963).
(1001) van Pinxteren, J. A. C., Verloop, M. E., *Pharm. Weekbl.* **97** (1), 1–10 (1962); *Anal. Abstr.* **9,** 5389 (1962).
(1002) Pinyazhko, I. R. M., *Farmatsevt. Zh. (Kiev)* **14,** 45–51 (1959); *C.A.* **57,** 2335*c* (1962).
(1003) Pinyazhko, I. R. M., *Ibid.*, **17,** (3) 26–8 (1962); *C.A.* **58,** 7783*g* (1963).
(1004) Pla-Delfina, J. M., *J. Soc. Cosmet. Chem.* **13** (5), 214–244 (1962); *Anal. Abstr.* **10,** 1873 (1963).
(1005) Pleticha, R., *Pharmazie* **15,** 478–85 (1960); *C.A.* **57,** 2333*h* (1962).
(1006) Pligin, S. G., *Farmatsevt. Zh. (Kiev)* **18** (1), 62–7 (1963); *C.A.* **59,** 12591*h* (1963).
(1007) *Ibid.* (3), pp. 38–43; *C.A.* **60,** 3950*b* (1964).
(1008) Plumel, M., *Ann. Pharm. Franc.* **20** (1), 34–41 (1962); *Anal. Abstr.* **9,** 3411 (1962).
(1009) Podmore, D. A., *Clin. Chim. Acta* **7,** 176–9 (1962).
(1010) Poethke, W., Berendt, H., Matschke, E., *Pharm. Zentralhalle* **102** (8), 492–5 (1963); *C.A.* **60,** 6702*c* (1964).
(1011) Poethke, W., Jaekel, Chr., *Arch. Pharm.* **296** (9), 627–34 (1963); *C.A.* **59,** 15122*g* (1963).
(1012) Poethke, W., Kinze, W., *Pharm. Zentralh.* **101** (11), 685–688 (1962); *Anal. Abstr.* **10,** 3841 (1963).
(1013) Poethke, W., Rao, D. A., *Sci. Pharm.* **31** (2), 116–24 (1963); *C.A.* **59,** 11188*b* (1963).
(1014) Poethke, W., Wigert, R., *Pharm. Zentralh.* **100** (12), 646–650 (1961); *Anal. Abstr.* **10,** 2888 (1963).
(1015) Poetter, H., *Pharmazie* **18,** 554–61 (1963); *C.A.* **60,** 1540*b* (1964).
(1016) Polaczek, L., Spychala, S., Gierlowska, J., *Acta Polon. Pharm.* **18** (4), 275–284 (1961); *Anal. Abstr.* **9,** 2908 (1962).
(1017) Ponomarev, V. D., *Uch. Zap., Pyatigorski Gos. Farmatsevt. Inst.* **5,** 221–4 (1961); *C.A.* **59,** 2591*h* (1963).
(1018) Poole, J. W., Monte, A. A., *J. Pharm. Sci.* **53** (2), 158–61 (1964).
(1019) Popa, I., *Farmacia (Bucharest)* **11** (2), 71–80 (1963); *C.A.* **59,** 3720*a* (1963).
(1020) Popa, M., Kain, N., *Rev. Chim. (Bucharest)* **13,** (2) 113 (1962); *C.A.* **57,** 11313*b* (1962).
(1021) Popa, M., Sterescu, M., *Ibid.*, **11,** 420 (1960); *C.A.* **57,** 16752*e* (1962).
(1022) Posgay, E., *Gyogyszereszet* **6,** 336–8 (1962); *C.A.* **58,** 3269*h* (1963).
(1023) Posgay, E., *Pharm. Zentralhalle* **101,** 471–6 (1962); *C.A.* **58,** 6649*a* (1963).
(1024) *Ibid.*, pp. 534–43; *C.A.* **58,** 7784*c* (1963).
(1025) Posgay-Kovacs, E., *Gyogyszereszet* **4,** 225–8 (1960); *C.A.* **60,** 6701*h* (1964).
(1026) Pouchol - Haumonte, J., Mlle Leroy, *Ann. Pharm. Franc.* **20** (12), 885–893 (1962); *Anal. Abstr.* **10,** 4370 (1963).
(1027) Pozdnyakova, V. T., Bukanova, N. V., *Farmatsevt. Zh. (Kiev)* **18** (3), 65–8 (1963); *C.A.* **60,** 3951*h* (1964).
(1028) Preuss, F. R., Knecht, A., *Deut. Apotheker-Ztg.* **102,** 386–92 (1962); *C.A.* **57,** 7389*d* (1962).
(1029) Prozorovskii, A. S., *Tr. 1-go Mosk. Med. Inst.* **18,** 135–45 (1962); *C.A.* **59,** 3717*g* (1963).
(1030) Przyborowski, L., *Ann. Univ. Mariae Curie-Sklodowska, Lublin-Polonia Sect. D* **17,** 25–32 (1962); *C.A.* **59,** 13769*c* (1963).
(1031) Puccini, A. V., Spiegel, A. J., *J. Pharm. Sci.* **51,** 496–7 (1962).
(1032) Raber, H., Ott, R., *Sci. Pharm.* **31,** 10–17 (1963); *C.A.* **59,** 1441*g* (1963).
(1033) Racz, I., *Acta Pharm. Hung.* **32,** 109–15 (1962); *C.A.* **57,** 4772*c* (1962).
(1034) Racz, I., Varsanyi, D., *Pharm. Zentralh.* **101** (1), 18–25 (1962); *Anal. Abstr.* **9,** 3404 (1962).
(1035) Radecke, H. R., *Pharmazie* **18,** 402–5 (1963); *C.A.* **60,** 366*f* (1964).
(1036) Ragettli, J., *Gyogyszereszet* **5,** 127–30 (1961); *C.A.* **60,** 5277*c* (1964).
(1037) Rahandraha, T., Chanez, M., Boiteau, P., Jaquard, S., *Ann. Pharm. Franc.* **21** (7–8), 561–7 (1963); *C.A.* **60,** 368*f* (1964).
(1038) Rahman, A. A., Fouli, A. M., *Egypt. Pharm. Bull.* **42,** (4) 373–84 (1960); *C.A.* **58,** 1303*e* (1963).
(1039) Ramanathan, V. S., *J. Sci. Ind. Res. (India)* **21*D*,** 449–60 (1962); *C.A.* **58,** 6648*d* (1963).
(1040) Ramanathan, V. S., *Indian J. Technol.* **1** (10), 388–90 (1963); *C.A.* **60,** 372*a* (1964).
(1041) Rapaport, L. I., Berzina, A. E., *Farmatsevt. Zh. (Kiev)* **17** (6), 25–32 (1962); *C.A.* **59,** 12592*c* (1963).
(1042) Rapaport, L. I., Solyanik, G. K., *Ibid.*, **18** (1), 31–7 (1963); *C.A.* **59,** 11187*f* (1963).
(1043) Rapson, H. D. C., Austin, K. W., Cutmore, E. A., *J. Pharm. Pharmacol. Suppl.* **14,** 66–72 (1962).

(1044) Rapson, H. D. C., Bird, A. E., *Ibid.*, **15,** 222–31 (1963).

(1045) Ray, S. C., *Ann. Biochem. Exptl. Med.* (*Calcutta*) **23** (10), 411–14 (1963); *C.A.* **60,** 6705*a* (1964).

(1046) Reisch, J., Bornfleth, H., Rheinbay, J., *Pharm. Ztg. Ver. Apotheker-Ztg.* **107** (28), 920–1 (1962); *C.A.* **60,** 372*e* (1964).

(1047) Reisch, J., Bornfleth, H., Rheinbay, J., *Pharm. Ztg.-Nach.* **108** (36), 1182–3 (1963); *C.A.* **60,** 3947*d* (1964).

(1048) *Ibid.*, pp. 1183–4 (1963); *C.A.* **60,** 3947*e* (1964).

(1049) Resplandy, A., *Mem. Inst. Sci. Madagascar Ser. B* **10** (1), 1–36 (1961); *C.A.* **60,** 13885*c* (1964).

(1050) Richter, J., *Pharmazie* **17,** 213–18 (1962); *C.A.* **57,** 8657*h* (1962).

(1051) Richter, J., Roethig, J., *Ibid.*, **17,** 686–9 (1962); *C.A.* **58,** 8853*c* (1963).

(1052) Riemschneider, R., Wierer, J., *Z. Anal. Chem.* **193** (3), 186–189 (1963); *C.A.* **59,** 1062*e* (1963).

(1053) Riffkin, C., *Am. J. Hosp. Pharm.* **20,** 19–22 (1963).

(1054) Rink, M., Riemhofer, M., *Deut. Apotheker-Ztg.* **102,** 1567–71 (1962); *C.A.* **58,** 6646*c* (1963).

(1055) Rink, M., Riemhofer, M., Romer, H., *Ibid.*, **103** (23), 719–23 (1963); *C.A.* **59,** 8545*h* (1963).

(1056) Robert, T. O., *Rev. Fac. Farm. Bioquim., Univ. Nacl. Mayor San Marcos* (*Lima*) **23,** 7–53 (961); *C.A.* **58,** 7787*h* (1963).

(1057) Roberts, H. R., Florey, K., *J. Pharm. Sci.* **51,** 794–9 (1962).

(1058) Roberts, H. R., Sino, M. R., *Ibid.*, **52,** 370–375 (1963).

(1059) Rosen, Earl, *Ibid.*, **52,** 98–100 (1963).

(1060) Rosenfeld, R. S., Lebeau, M. C., Shulman, S., Seltzer, J., *J. Chromatog.* **7,** 293–296 (1962).

(1061) Rothenberg, S. P., Garcia, R., *Nature* **200** (4909), 922 (1963).

(1062) Roushdi, I. M., Abdine, H., Hassanein, A. A., *Egypt. Pharm. Bull.* **42,** 293–8 (1960); *C.A.* **57,** 15239*h* (1962).

(1063) Roushdi, I. M., Abdine, H., Sadek, W., *Ibid.*, **42,** 299–307 (1960); *C.A.* **57,** 15239*g* (1962).

(1064) Roushdi, I. M., Abdine, H., Sadek, W., *J. Pharm. Sci. U. Arab. Rep.* **2,** (1) 139–45 (1962); *C.A.* **58,** 7785*f* (1963).

(1065) Rousselet, R., Paris, R., *Ann. Pharm. Franc.* **20,** 523–6 (1962); *C.A.* **58,** 10041*e* (1963).

(1066) Roux, A., Roux-Matignon, J., *Ann. Pharm. Franc.* **21,** 255–60 (1963); *C.A.* **59,** 1439*c* (1963).

(1067) *Ibid.*, pp. 345–348; *Anal. Abstr.* **11,** 1899 (1964).

(1068) Rozanov, A., Ya., *Vopr. Med. Khim.* **9** (3), 294–7 (1963); *C.A.* **59,** 13769*h* (1963).

(1069) Rozenberg, A. I., *Med. Prom. SSSR* **17** (7), 43–6 (1963); *C.A.* **11,** 11193*a* (1963).

(1070) Rozsa, P., *Acta Pharm. Hung.* **32** (3), 97–101 (1962); *Anal. Abstr.* **10,** 1992 (1963).

(1071) Rozynska, M., Wojcik, Z., *Acta Polon. Pharm.* **18** (5), 377–381 (1961); *Anal. Abstr.* **9,** 2918 (1962).

(1072) Ruell, T., *Bull. Soc. Chim. France* **1963** (8–9), 1897–8; *C.A.* **60,** 370*a* (1964).

(1073) Ruettiman, O., Flueck, H., *Sci. Pharm.* **31** (2), 161–9 (1963); *C.A.* **59,** 11190*d* (1963).

(1074) Rusiecki, W., Henneberg, M., *Farm. Polska* **18,** 203–10 (1962); *C.A.* **57,** 9953*f* (1962).

(1075) Russu, C., Cruceanu, I., Barcaru, V., *Pharmazie* **18** (12), 799–800 (1963); *C.A.* **60,** 9100*h* (1964).

(1076) Russu, C., Pelloni, V., Sterescu, M., *Rev. Chim.* (*Bucharest*) **14,** 48 (1963); *C.A.* **59,** 2595*g* (1963).

(1077) Ruzhentseva, A. K., Chivireva, A. M., Leiferova, N. G., *Med. Prom. SSSR* **16** (1), 46–7 (1962); *C.A.* **60,** 6702*h* (1964).

(1078) Ruzhentseva, A. K., Prokof'eva, E. E., *Ibid.*, **17** (9), 29–32 (1963); *C.A.* **60,** 5279*c* (1964).

(1079) Ryabukhina, O. O., *Farmatsevt. Zh.* **17,** 48–54 (1962); *C.A.* **59,** 9738*f* (1963).

(1080) Sahli, M., *Arzneimittel-Forsch.* **12,** 55–61, 155–61 (1962); *C.A.* **57,** 956*e* (1962).

(1081) Said, El. F., Amer, M. M., Said, A. A., *Congr. Sci. Farm., Conf. Comun., 21°, Pisa* **1961,** 475–9; *C.A.* **59,** 3719*d* (1963).

(1082) Said, F., Girgis, P., *Egypt. Pharm. Bull.* **42** (4), 217–24 (1960); *C.A.* **58,** 8855*d* (1963).

(1083) Sakurai, H., Sato, K., Tsuda, T., *J. Pharm. Soc. Japan* **82** (12), 1684–1687 (1962); *Anal. Abstr.* **10,** 5354 (1963).

(1084) Sakurai, H., Sato, K., Tsuda, T., Takada, M., *Ann. Rep. Takamine Lab.* **14,** 83–86 (1962); *Anal. Abstr.* **11,** 323 (1964).

(1085) Sakurai, H., Kato, K., Umeda, M., Tsubota, S., *Yakagaku Zasshi* **83,** 811–14 (1963); *C.A.* **59,** 15119*e* (1963).

(1086) Sakurai, H., Umeda, M., *J. Pharm. Soc. Japan* **81,** 736–740 (1961); *Anal. Abstr.* **9,** 4875 (1962).

(1087) Salas, G., Gardet, M., Colleter, J. C., *Bull. Soc. Pharm. Bordeaux* **102,** 33–44 (1963); *C.A.* **59,** 9735*h* (1963).

(1088) Salazar, L. E. G., Otalora, A., *Rev. Fac. Med. Univ. Nacl.* (*Bogota*) **31** (1), 35–9 (1963); *C.A.* **60,** 3949*d* (1964).

(1089) Salazar, R., del Pozo, A., *Galenica Acta* (*Madrid*) **16** (1), 45–54 (1963); *C.A.* **60,** 9100*c* (1964).

(1090) Salo, T., *Suomen Kem., B* **36** (1), 1–5 (1963); *Anal. Abstr.* **10,** 5396 (1963).

(1091) Salvesen, B., *Medd. Norsk Farm. Selsk.* **24** (12), 185–197 (1962); *Anal. Abstr.* **10,** 4369 (1963).

(1092) Selezneva, A. A., Wang, I-Kuang, *Tr. Leningr. Khim.-Farmatsevt. Inst.* **1962** (15), 101–4; *C.A.* **59,** 2594*b* (1963).

(1093) Santavy, F., *Chem. Anal.* (*Warsaw*) **3,** 357–68 (1958); *C.A.* **57,** 2820*g* (1962).

(1094) Santavy, F., *Pharmazie* **15,** 676–83 (1960); *C.A.* **57,** 6022*i* (1962).

(1095) Santoro, R. S., Matz, A. R., *J. Pharm. Sci.* **51** (10), 984–987 (1962).

(1096) Sarin, J. P. S., Chakravarty, R. B., Ray, G. K., Kukerji, B., *Indian J. Pharm.* **25,** 375–6 (1963); *C.A.* **60,** 7873*c* (1964).

(1097) Sarsunova, M., *Pharmazie* **18** (11), 748–50 (1963); *C.A.* **60,** 9100*b* (1964).

(1098) Sarsunova, M., Menkynova, J., Fellegi, J., *Pharm. Zentralhalle* **101,** 325–8 (1962); *C.A.* **58,** 4375*d* (1963).

(1099) Sarsunova, M., Schwarz, V., *Pharmazie* **17,** 527–9 (1962); *C.A.* **58,** 4378*g* (1963).

(1100) Sarsunova, M., Schwarz, V., *Ibid.*, **18,** 207–9 (1963); *C.A.* **59,** 1441*a* (1963).

(1101) Sasongko and Sasongko-Ponamon, J. A. S., *Suara Pharm. Madjalah* **5,** No. 4, 101–25 (1960); *C.A.* **58,** 12370*e* (1963).

(1102) Sato, K., Takada, M., *Arch. Pract. Pharm., Japan* **22** (1), 57–59 (1962); *Anal. Abstr.* **10,** 5372 (1963).

(1103) Savel'eva, G. N., Kudymov, G. N., *Materialy 2-oi* (*Vitoroi*) *Vses. Konf. Farmatsevtov* (*Moscow Gos. Izd. Med. Lit.*) *Sb.* **1959,** 296–8; *C.A.* **59,** 6996*b* (1963).

(1104) Sax, S. M., Migliore, P. J., *Anal. Biochem.* **3,** 150–7 (1962).

(1105) Scaringelli, F. P., *J. Assoc. Offic. Agr. Chemists* **46** (4), 643–5 (1963).

(1106) Schaub, K., *Pharm. Acta Helv.* **37** (11), 669–682 (1962); *Anal. Abstr.* **10,** 3888 (1963).

(1107) Scheibl, F., *Arch. Toxikol.* **17,** 357–64 (1959); *C.A.* **59,** 2594*a* (1964).

(1108) Schill, G., Marsh, M., *Svensk Farm. Tidskr.* **67** (14), 385–400 (1963); *Anal. Abstr.* **11,** 1458 (1964).

(1109) Schiller, P., *Acta Facultatis Pharm. Bohemoslov.* **7,** 7–57 (1962); *C.A.* **59,** 15119*i* (1963).

(1110) Schiller, P., Jombik, J., *Chem. Zvesti* **17,** 685–8 (1963); *C.A.* **60,** 1539*h* (1964).

(1111) Schilling, E. D., Burchill, P. I., Clayton, R. A., *Anal. Biochem.* **5** (1), 1–6 (1963).

(1112) Schlemmer, W., Bosse, J., *Arch. Pharm.* **296** (11), 785–91 (1963); *C.A.* **60,** 3948*g* (1964).

(1113) Schmidt-Kastner, G., *Naturwissenschaften* **51** (2), 38–9 (1964); *C.A.* **60,** 10476*e* (1964).

(1114) Schneider, H., Hofstetter, J., *Deut. Apotheker-Ztg.* **103** (43), 1423–4 (1963); *C.A.* **60,** 3950*g* (1964).

(1115) Schou, S. A., *Pharm. Acta Helv.* **38** (10), 741–5 (1963); *C.A.* **60,** 2724*c* (1960).

(1116) Schulek, E., Burger, K., *Ann. Univ. Sci. Budapest Rolando Eotvos Nominatae, Sect. Chim.* **2,** 531–6 (1960); *C.A.* **57,** 2337*f* (1962).

(1117) Schultz, O. E., Kratzer, R., *Arch. Pharm.* **295,** 444–54 (1962); *C.A.* **57,** 12638*c* (1962).

(1118) Schwartz, M. A., Granatek, A. P., Buckwalter, F. H., *J. Pharm. Sci.* **51,** 523–6 (1962).

(1119) Schwarz, V., *Pharmazie* **18,** 122–4 (1963); *C.A.* **59,** 3719*g* (1963).

(1120) Schwarz, V., Syhora, K., *Collection Czech. Chem. Commun.* **28,** 101–6 (1963); *C.A.* **59,** 11593*h* (1963).

(1121) Schwietert, H. R., Uffelie, O. F., *Pharm. Weekblad* **97,** 397–401 (1962); *C.A.* **57,** 9958*c* (1962).

(1122) *Sci. Pharm.* **30** (4), 230–287 (1962); *Anal. Abstr.* **10,** 4022 (1963).

(1123) Scott, R. B., *Develop. Appl. Spectry.* **1,** 17–23 (1962).

(1124) Seeboth, H., *Chem. Tech., Leipzig* **15** (1), 34–35 (1963); *C.A.* **58,** 13661*d* (1963).

(1125) Seidlein, H. J., Poggendorf, A., *Pharm. Praxis Beilage Pharmazie* **1963** (9), 192–7; *C.A.* **60,** 368*b* (1964).

(1126) Sekera, Anne, Sekera, Ales, *Congr. Sci. Farm., Conf. Comun., 21°, Pisa* **1961,** 816–19; *C.A.* **59,** 1442*a* (1963).

(1127) Sekiguchi, K., Obi, N., *Japan Analyst* **10** (11), 1288–1290 (1961); *Anal. Abstr.* **10,** 4346 (1963).

(1128) Sekiguchi, K., Ueda, Y., Nakamori, Y., *Chem. Pharm. Bull.* (*Tokyo*) **11** (9), 1108–22 (1963); *C.A.* **59,** 15123*e* (1963).

(1129) Selleri, R., Caldini, O., ANAL. CHEM. **33,** 1944 (1961).

(1130) Sellers, D. E., Greenlee, K. L., Van Atta, R. E., *Ibid.*, **34,** 441–2 (1962).

(1131) Sellers, D. E., Tang, S. S. N., Van Atta, R. E., *J. Chem. Educ.* **39** (8), 408–409 (1962).

(1132) Semenycheva, A. A., Chepurnykh, N. S., Smirnova, G. D., *Sb. Nauchn. Tr., Tsentr. Nauchn.-Issled. Aptechn. Inst.* **2,** 114–17 (1961); *C.A.* **59,** 12592*g* (1963).

(1133) Sferruzza, G., Rangone, R., *Congr. Sci. Farm., Conf. Comun., 21°, Pisa* **1961,** 830–9; *C.A.* **59,** 2592*c* (1963).

(1134) Shah, R. A., Fatima, N., *Mikrochim. Ichnoanal. Acta* **1963** (5–6), 1059–65; *C.A.* **60,** 3947*h* (1964).

(1135) Shah, R. C., Raman, P. V., Shah, B. M., *J. Pharm. Sci.* **52,** 167–8 (1963).

(1136) Shakh, Ts. I., Kagan, F. Yu, *Farmatsevt. Zh.* (*Kiev*) **18** (1), 13–17 (1963); *C.A.* **59,** 11188*c* (1963).

(1137) Shaw, W. H. C., Fortune, I., *Analyst* **87,** 187–196 (1962).

(1138) Shefter, E., *Univ. Microfilms* (*Ann Arbor, Mich.*), *Order No.* **63-2911,** 81 pp.; *Dissertation Abstr.* **23,** 3325–6 (1963).

(1139) Shell, J. W., *J. Pharm. Sci.* **51,** 731–5 (1962).

(1140) Shell, J. W., *Ibid.*, **52** (1), 24–29 (1963).

(1141) Sheverdyaeva, V. M., Bystrov, S. P., *Aptechn. Delo* **11** (5), 71–4 (1962); *C.A.* **60,** 10477*g* (1964).

(1142) Schmidt, E. V., Velikanova, V. I., *Sb. Tr. Posvyashch. XX-letiyu* (*Davadtsatiletiyu*) *Farmatsvet. Fak.* (*Tomsk: Tomskii Med. Inst.*) **1962,** 132–3; *C.A.* **59,** 11188*h* (1963).

(1143) Shostenko, Yu. V., Simon, I. S., *Med. Prom. SSSR* **17** (8), 41–4 (1963); *C.A.* **59,** 15122*b* (1963).

(1144) Siblikova, O., Cerna, M., Hais, I. M., *Ceskosl. Farm.* **11** (4), 187–191 (1962); *Anal. Abstr.* **9,** 4859 (1962).

(1145) Sidky, M. M., Mahran, M. R., *Arch. Pharm.* **296** (9), 569–70 (1963); *C.A.* **60,** 371*e* (1964).

(1146) Siedlanowska, H., *Farm. Polska* **19** (10), 214–15 (1963); *C.A.* **60,** 1538*h* (1964).

(1147) Siest, G., *Bull. Soc. Pharm. Nancy No.* **57,** 11–18 (1963); *C.A.* **59,** 12595*a* (1963).

(1148) Siest, G., Roos, F., Gabou, J. J., *Bull. Soc. Pharm. Nancy No.* **58,** 29–38 (1963); *C.A.* **60,** 2722*e* (1964).

(1149) Silva, E., *Anal. Chem., Proc. Intern. Symp., Birmingham Univ., Birmingham, Engl.* **1962,** 78–9; *C.A.* **59,** 11189*e* (1963).

(1150) Simoes, R., *Anais Fac. Farm. Odontol Univ. Sao Paulo* **19** (1), 31–8 (1962); *C.A.* **59,** 13769*d* (1963).

(1151) Simon, I. S., Shostenko, Yu. V., *Farmatsevt. Zh.* (*Kiev*) **14** (6), 51–3 (1959); *C.A.* **60,** 371*c* (1964).

(1152) Simon, I. S., Shostenko, Yu. V., *Zh. Obshch. Khim.* **32,** 1002–4 (1962); *C.A.* **58,** 1301*g* (1963).

(1153) Simoons, J. R. A., *Pharm. Weekblad* **98,** 225–42 (1963); *C.A.* **59,** 1437*b* (1963).

(1154) Sinsheimer, J. E., Smith, Edward, *J. Pharm. Sci.* **52** (11), 1080–5 (1963).

(1155) Sirbu, I., Bebesel, P., *Rev. Chim.* (*Bucharest*) **10,** 641–4 (1959); *C.A.* **57,** 15239*h* (1962).

(1156) Sivitskaya, O. K., *Aptechn. Delo* **11** (3), 38–41 (1962); *C.A.* **60,** 10478*e* (1964).

(1157) Sjoholm, I., *Svensk Farm. Tdskr.* **66,** 321–30 (1962); *C.A.* **57,** 13888*i* (1962).

(1158) Skibina, E. M., *Tr. Khar'kovsk. Farmatsevt. Inst. No.* **2,** 129–33 (1962); *C.A.* **60,** 10476*g* (1964).

(1159) Smith, B., *Trans. Chalmers Univ. Technol., Gothenburg* **1963,** No. 263, 51 pp.; *C.A.* **59,** 10*g* (1963).

(1160) Smith, D. L., Elving, P. J., Anal. Chem. **34** (8), 930–936 (1962).

(1161) Smith, E., Worrell, L. F., Sinsheimer, J. E., *Ibid.*, **35,** 58–61 (1963).

(1162) Smith, L. L., Foell, T., *J. Chromatog.* **9,** 339–344 (1962).

(1163) Smoczkiewiczowa, A., Grochmalicka, J., *Acta Polon. Pharm.* **18** (6), 515–519 (1961); *Anal. Abstr.* **9,** 3924 (1962).

(1164) Smolyanskaya, P. G., *Tr. Leningr. Khim. Farmatsevt. Inst.* **1961,** No. 13, 268–78.

(1165) Snell, J. F., *Advan. Tracer Methodol.* **1,** 234–46 (1963).

(1166) Soemantri, R. M., *Suara Pharm. Madjalah* **4,** No. 5, 139–46, No. 6, 157–61 (1959); *C.A.* **58,** 12369*d* (1963).

(1167) Soliman, S. A., Harris, L. E., *J. Pharm. Sci.* **52** (1), 43–46 (1963).

(1168) Solomon-Ionescu, I., Enache, S., Gamentzy, V., *Rev. Chim., Bucharest* **13** (7), 433 (1962); *Anal. Abstr.* **10,** 1171 (1963).

(1169) Solomon-Ionescu, I., St. Enache, Wermescher, B., *Rev. Chim.* (*Bucharest*) **13,** 433 (1962); *C.A.* **58,** 412*g* (1963).

(1170) Solomon-Ionescu, I., Popescu, D., Constantinescu, T., *Farmacia* (*Bucharest*) **10,** 491–6 (1962); *C.A.* **58,** 12370*c* (1963).

(1171) Solomon-Ionescu, I., Popescu, D., St. Enache, *Rev. Chim.* (*Bucharest*) **14** (9), 532 (1963); *C.A.* **60,** 3950*c* (1964).

(1172) Solomon-Ionescu, I., Popescu, D., Nicola, M., *Farmacia* (*Bucharest*) **10,** 627–9 (1962); *C.A.* **58,** 6648*b* (1963).

(1173) Solomon-Ionescu, I., Prodescu, M., Cuciureanu, E., *Ibid.*, **10,** 551–3 (1962); *C.A.* **58,** 7785*h* (1963).

(1174) Soltesz, J., Zsupan, K., Otvos, G., *Acta Pharm. Hung.* **28** (5–6), 229–36 (1958); *C.A.* **57,** 1263*g* (1962).

(1175) Sommer, L., *Z. Anal. Chem.* **187,** 7–16 (1962); *C.A.* **57,** 11831*b* (1962).

(1176) Sophoulis, E., *J. Pharm. Sci. U. Arab. Rep.* **2,** (1) 29–34 (1961); *C.A.* **58,** 8855*a* (1963).

(1177) Spaeth, E., Baptist, V. H., Roberts, M., Anal. Chem. **34,** 1342 (1962).

(1178) Spahr, J. L., Pernarowski, M., Knevel, A. M., *J. Pharm. Sci.* **51,** 749–52 (1962).

(1179) Spiteller, G., Kaschnitz, R., *Monatsh.* **95** (5), 964–80 (1963); *C.A.* **60,** 372*d* (1964).

(1180) Springer, V., Majer, J., *Ceskosl. Farm.* **12** (1), 6–10 (1963); *Anal. Abstr.* **10,** 5375 (1963).

(1181) Springer, V., Majer, J., Karlicek, R., *Ibid.*, **12,** 4–6 (1963).

(1182) Spross, B., *Congr. Sci. Pharm.* **1959,** 425–38; *C.A.* **58,** 12372*a* (1963).

(1183) Sprung, M., *Pharmazie* **16,** 515–19 (1961); *C.A.* **57,** 2344*b* (1962).

(1184) Srepel, B., *Farm. Glasnik* **18,** 64–7 (1962); *C.A.* **58,** 3269*f* (1963).

(1185) Srivastava, K. L., Tayal, J. N., Nanda, J. N., *LABDEV* (*Kanpur, India*) **1** (1–2), 49–50 (1963); *C.A.* **60,** 5282*f* (1964).

(1186) Srivastava, S. K., Srivastava, B. B. P., Ray, G. K., and Mukerji, B., *Indian J. Pharm.* **25,** 304–5 (1963); *C.A.* **59,** 15119*h* (1963).

(1187) Stachel, H. D., Eiden, F., *Deut. Apotheker Ztg.* **103** (24), 753–5 (1963); *C.A.* **59,** 12594*g* (1963).

(1188) Stahl, E., Kaltenbach, U., *J. Chromatog.* **5,** 458–60 (1961).

(1189) Stahl, E., Schorn, P. J., *Z. Physiol. Chem.* **325,** 263–74 (1961); *C.A.* **57,** 4761a (1962).

(1190) Stainier, C., Bosly, J., Dutreiux, Fr., Stainier, R., *Pharm. Acta Helv.* **38** (7–8), 587–95 (1963); *C.A.* **59,** 13771*b* (1963).

(1191) Stark, R., *J. Pharm. Sci.* **52** (12), 1180–2 (1963).

(1192) Starka, L., *Chem. Zvest.* **17** (8), 550–5 (1963); *C.A.* **60,** 13890*c* (1964).

(1193) Staroscik, R., *Farm. Polska* **18,** 112–13 (1962); **57,** 9953*e* (1962).

(1194) Steidle, W., *Planta Med.* **9,** 435–41 (1961); *C.A.* **57,** 11311*g* (1962).

(1195) Steimetz, E. P., *Bull. Soc. Pharm. Nancy No.* **55,** 9–14 (1962); *C.A.* **58,** 6645*g* (1963).

(1196) Steinbach, H., *Tuberkulosearzt* **17** (12), 761–5 (1963); *C.A.* **60,** 9100*a* (1964).

(1197) Steinegger, E., Gebistorf, J., *Pharm. Acta Helv.* **38** (12), 840–7 (1963); *C.A.* **60,** 7872*f* (1964).

(1198) Steinetz, B. G., Beach, V. L., Dubnick, B., Meli, A., Fujimoto, G., *Steroids* 1, 395–408 (1963).

(1199) Sterescu, M., Kain, N., *Rev. Chim.* (*Bucharest*) **13,** (3) 172 (1962); *C.A.* **57,** 12812*h* (1962).

(1200) Sterescu, M., Kain, N., Aftalion, H., *Ibid.*, **10,** 535–6 (1959); *C.A.* **57,** 9955*f* (1962).

(1201) Sterescu, M., Kain, N., Stoicescu, V., *Ibid.*, **11,** 534 (1960); *C.A.* **57,** 8659*f* (1962).

(1202) Sterescu, M., Keim, N., Dobrovici, M., *Ibid.*, **10,** No. 2, 108–9 (1959); *C.A.* **57,** 960*i* (1962).

(1203) Sterescu, M., Pelloni, V., *Ibid.*, **11,** 533–4 (1960); *C.A.* **57,** 11313*d* (1962).

(1204) Sterescu, M., Popa, M., St. Pola, *Ibid.*, **10,** 712 (1959); *C.A.* **57,** 11312*i* (1962).

(1205) Sterescu, M., Simionovici, R., Mihalca, G., Pelloni, V., Popa, M., Arizan, S. *Pharmazie* **18,** 471–4 (1963); *C.A.* **60,** 1541*f* (1964).

(1206) Stevens, H. M., *Med. Sci. & Law* **2** (4), 268–273 (1962).

(1207) Stockmann, C. C., *Chem. Zvesti* **16,** 365–7 (1962); *C.A.* **58,** 5454*c* (1963).

(1208) Stojanoff, N. S., Awramowa, B., *Pharm. Praxis, Beilage Pharmazie* **1962,** (11), 193–5; *C.A.* **58,** 7791*b* (1963).

(1209) Street, H. V., *Acta Pharm. Tox., Kbh.* **19** (4), 312–324 (1962); *Anal. Abstr.* **10,** 3873 (1963).

(1210) *Ibid.*, pp. 325–329; *Anal. Abstr.* **10,** 3839 (1963).

(1211) Street, H. V., *Chem. & Ind.* **1962,** (33), 1501–1502.

(1212) Street, H. V., *Forensic Sci. Soc. J.* **2,** 118–124 (1962).

(1213) Street, H. V., Niyogi, S. K., *J. Pharm. Sci.* **51,** 666–8 (1962).

(1214) Strel'nikova, N. D., Tronov, B. V., Shcheglova, L. T., *Sb. Tr. Posvyashch. XX-letiyu* (*Dvadtsatiletiyu*) *Farmatsvet. Fak.* **1962,** **42–7**; *C.A.* **59,** 12596*a* (1963).

(1215) Strohecker, R., Pies, H., *Z. Lebensmitt Untersuch.*, **118** (5), 394–397 (1962); *Anal. Abstr.* **10,** 4424 (1963).

(1216) Strohecker, R., Pies, H., Jansky, R., Ludwig, M., Menhorn, H. W., *Arch. Pharm., Berlin* **294** (12), 800–811 (1961); *Anal. Abstr.* **9,** 2933 (1962).

(1217) Studeny, J., Uhrova, D., *Chem. Prumysl* **12,** 553–4 (1962); *C.A.* **58,** 3889*f* (1963).

(1218) Stupnicki, R., Stupnicka, E., *J. Chromatog.* **9,** 235–237 (1962).

(1219) Subirana, A. E., *Rev. Real Acad. Farm.* (*Barcelona*) **8,** 9–13 (1961); *C.A.* **59,** 12592*a* (1963).

(1220) Sucker, H., *Deut. Apotheker-Ztg.* **101** (16), 441–3 (1961); *C.A.* **60,** 366*h* (1964).

(1221) Summa, A. F., *J. Pharm. Sci.*, **51,** 474–7 (1962).

(1222) Sund, R. B., *Medd. Norsk Farm. Selskap* **25** (7), 101–14 (1963); *C.A.* **60,** 5279*h* (1964).

(1223) Sune, J. M., Fauli, C., *Anales Real Acad. Farm.* **28,** 53–9 (1962); *C.A.* **57,** 16755*e* (1962).

(1224) Sunshine, I., Gerber, S. R., "Spectrophotometric Analysis of Drugs," C. C Thomas, Springfield, Ill., 1963, 253 pp.

(1225) Sunshine, I., Rose, E., LeBeau, J., *Clin. Chem.* 9 (3), 312–316 (1963).

(1226) Suprun, P. P., *Farmatsevt. Zh.* (*Kiev*) **17,** (2) 31–5 (1962); *C.A.* **58,** 7785*e* (1963).

(1227) Suprun, P. P., *Ibid.*, **18** (2), 43–51 (1963); *C.A.* **60,** 5278*f* (1964).

(1228) Suprun, P. P., *Med. Prom. SSSR*

16, (1) 47–9 (1962); *C.A.* **57,** 12635*h* (1962).

(1229) Suszko-Purzycka, A., *Prace Zakresu Towaroznawst. Chem. Wyssza Szkola Ekon. Poznan. Zeszyty Nauk. Ser. I No.* **10,** 105–11 (1963); *C.A.* **60,** 2724*a* (1964).

(1230) Suvorova, T. M., *Uch. Zap., Pyatigorskii Gos. Farmatsevt. Inst.* **5,** 147–51 (1961); *C.A.* **59,** 2950*c* (1963).

(1231) Suzuki, T., *Chem. Pharm. Bull.* (*Tokyo*) **10,** 912–21 (1962); *C.A.* **58,** 11169*h* (1963).

(1232) *Ibid.,* pp. 1017–23 (1962); *C.A.* **58,** 11169*a* (1963).

(1233) Svatek, E., Vachek, J., *Ceskoslov. Farm.* **11,** 69–73 (1962); *C.A.* **57,** 2335*b* (1962).

(1234) Svoboda, G. R., *Anal. Chem.* **34,** 1559–62 (1962).

(1235) Szabolcs, E., *Acta Pharm. Hung.* **32,** (4), 145–155 (1962); *Anal. Abstr.* **11,** 1468 (1964).

(1236) Szasz, G., Khin, L., Budvari, Mrs. R., *Acta Pharm. Hung.* **33** (6), 245–51 (1963); *C.A.* **60,** 6704*g* (1964).

(1237) Szasz, G. Khin, L., Takacs, M., Szasz, M., *Ibid.,* **28** (5–6), 219–28 (1958); *C.A.* **57,** 12638*h* (1962).

(1238) Szendey, G. L., *Arch. Pharm.* **296** (2), 126–9 (1963); *C.A.* **58,** 10040*c* (1963).

(1239) Szendey, G. L., *J. Pharm. Sci.* **51** (9), 906–908 (1962).

(1240) Szepesy, A., *Gyogyszereszet* **4,** 98–100 (1960); *C.A.* **60,** 5284*d* (1964).

(1241) Szepesy, A., *Ibid.,* **6,** 216–20, 267–70 (1962); *C.A.* **60,** 5284*f* (1964).

(1242) Szepesy, A., Bloch, E., *Ibid.,* **6,** 421–2 (1962); *C.A.* **59,** 1439*g* (1963).

(1243) Szigetvary, Mrs. F., Kuttel, D., *Ibid.,* **7** (1), 19–23 (1963); *C.A.* **60,** 5275*g* (1964).

(1244) *Ibid.,* (8), 297–305 (1963); *C.A.* **60,** 372*d* (1964).

(1245) Szilagyi, I., Valyi-Nagy, T., Szabo, I., Keresztes, T., Keresztes, T., *Nature* **201** (4914), 81–2 (1964).

(1246) Sztaricskai, F., *Acta Pharm. Hung.* **32** (2), 63–70 (1962); *Anal. Abstr.* **10,** 1982 (1963).

(1247) Sztark, W., *Dissert. Pharm., Krakow* **14** (1), 41–45 (1962); *Anal. Abstr.* **9,** 5411 (1962).

(1248) Sztark, W., *Dissertationes Pharm.* **14,** 197–9 (1962); *C.A.* **57,** 16750*f* (1962).

(1249) Sztark, W., Eckstein, M., *Ibid.,* **14,** 321–325 (1962); *Anal. Abstr.,* **10,** 4368 (1963).

(1250) Tabata, T., *Bitamin* **24,** 164–7 (1961); *C.A.* **60,** 6705*d* (1964).

(1251) Takacs, M., Szabo, B., Szasz, G., Khin, L., Szas-Zacsko, M., *Gyogyszereszet* **7** (4), 144–6 (1963); *C.A.* **59,** 8456*b* (1963).

(1252) Takahashi, R., Tachi, I., *Agric. Biol. Chem., Japan* **26** (11), 771–776 (1962); *Anal. Abstr.* **10,** 3928 (1963).

(1253) Takahashi, T., *Yakugaku Zasshi* **82,** 1349–54 (1962); *C.A.* 4379*h* (1963).

(1254) Takahashi, T., *Bunseki Kagaku* **8,** 536–42 (1959); *C.A.* **57,** 2338*g* (1962).

(1255) Takeuchi, Y., *J. Pharm. Soc. Japan* **82,** (2), 289–296 (1962); *Anal. Abstr.* **10,** 3887 (1963).

(1256) Takubo, T., Tadaoka, T., Sawai, T., *Yakuzaigaku* **22,** 67–9 (1962); *C.A.* **57,** 9960*i* (1962).

(1257) Talmage, J. M., Penner, M. H., Geller, M. J., *J. Pharm. Sci.,* **53** (1), 76–9 (1964).

(1258) Tanaka, I., Yoshizawa, H., Otani, S., Nakayashiki, J., Hara, E., *Yakuzaigaku* **22,** 147–50 (1962); *C.A.* **59,** 12596*f* (1963).

(1259) Tanker, M., *Bull. Fac. Med. Istanbul* **24,** 542–548 (1961); *Anal. Abstr.,* **10,** 2871 (1963).

(1260) Tapfer, D., *Gyogyszereszet* **6** (8), 295–300 (1962); *C.A.* **58,** 8851*c* (1963).

(1261) Tapia, H. V., *Anales Fac. Quim. Farm. Univ. Chile* **14,** 88–97 (1962); *C.A.* **60,** 3951*c* (1964).

(1262) Tarasenko, M. I., *Sb. Nauchn. Tr. Tsentr. Aptechn. Nauchn-Issled. Inst.* **2,** 118–29 (1961); *C.A.* **55,** 412*d* (1961).

(1263) Tarasenko, M. I., Ionin, S. M., *Tr. 1-go Mosk. Med. Inst.* **17,** 225–37 (1962); *C.A.* **59,** 3718*g* (1963).

(1264) Tarasenko, M. I., Timofeeva, N. G., *Ibid.,* **17,** 220–4 (1962); *C.A.* **59,** 3718*e* (1963).

(1265) Tarpo, E., Gheorghiv, M., *Farmacia* **11,** 235–42 (1963); *C.A.* **59,** 9736*b* (1963).

(1266) Tatsuzawa, M., *Bunseki Kagaku* **11,** 1055–9 (1962); *C.A.* **57,** 16748*f* (1962).

(1267) Tatsuzawa, M., *Japan Analyst* **10** (2), 129–133 (1962); *Anal. Abstr.* **10,** 2465 (1963).

(1268) Teijgeler, C. A., *Pharm. Weekbl.* (1962) **97** (14), 507–511; *Anal. Abstr.* **10,** 19 (1963).

(1269) Teixeira, M., Baltazer, J., *Rev. Port. Farm.* **13,** 44–7 (1963); *C.A.* **59,** 11188*h* (1963).

(1270) Terai, K., *Osaka Shiritsu Daigaku Igaku Zasshi* **8,** 487–9 (1959); *C.A.* **57,** 14102*h* (1962).

(1271) Thies, H., Ozbilici, Z., *Arch. Pharm., Berlin* **295** (9), 715–718 (1962); *Anal. Abstr.* **10,** 2884 (1963).

(1272) Thies, H., Ozbilici, Z., *Arch. Pharm.* **295** (3), 194–6 (1962); *C.A.* **58,** 6645*c* (1963).

(1273) Thilmans, Y., Smeyers, R., *Ann. Pharm. Franc.* **20,** 719–25 (1962); *C.A.* **58,** 6646*f* (1963).

(1274) Thoma, F., *Tuberkulosearzt* **16,** 362–8 (1962); *C.A.* **57,** 7391*h* (1962).

(1275) Thoma, K., Ullmann, E., Wolferseder, E., *Arch. Pharm.* **295,** 548–55 (1962); *C.A.* **57,** 16755*i* (1962).

(1276) Thompson, J. F., Arnold, W. N., Morris, C. J., *Nature* **197,** 380–381 (1963).

(1277) Tikhomirova, G. P., Belen'kaya, S. L., *Ukr. Khim. Zhur.* **29** (1), 97–99 (1963); *Anal. Abstr.* **10,** 4921 (1963).

(1278) Tishler, F., *Univ. Microfilms Order No.* **62-2796,** 124 pp. *Dissertation Abstr.* **23,** 83–4 (1962).

(1279) Tishler, F., Sheth, P. B., Giaimo, M. B., Mader, W. J., *J. Pharm. Sci.* **51** (12), 1175–1180 (1962).

(1280) Tishler, F., Worrell, L. F., Sinsheimer, J. E., *Ibid.,* **51,** 645–8 (1962).

(1281) Tokar, G., Simonyi, I., *Acta Chim. Acad. Sci. Hung.* **25,** 313–20 (1960); *C.A.* **60,** 7871*f* (1964).

(1282) Tolgyessy, J., Sarsunova, M., *Cesk. Farm.* **12** (5), 257–62 (1963); *C.A.* **59,** 8544*h* (1963).

(1283) Tomoda, M., Nishina, T., *Kyoritsu Yakka Daigaku Kenkyu Nempo* **6/7,** 31–4 (1961/2); *C.A.* **60,** 369*b* (1964).

(1284) Toncheva, P., Kolusheva, A., *Farmatsiya,* (*Sofia*) **12** (3), 27–30 (1962); *Anal. Abstr.* **10,** 4348 (1963).

(1285) *Ibid.,* (4), pp. 10–14; *C.A.* **58,** 4376*c* (1963).

(1286) Toncheva, P., Kolusheva, A., *Ibid.,* **13** (5), 10–16 (1963); *C.A.* **60,** 5276*c* (1964).

(1287) Torrado, J. M., *Farmacognosia* (*Madrid*) **22,** 93–5 (1962); *C.A.* **60,** 367*f* (1964).

(1288) Toth, Z., Szabo, D., *Acta Pharm. Hung.* **33** (5), 212–15 (1963); *C.A.* **60,** 6704*c* (1964).

(1289) Touchstone, J. C., Kasparow, M., Murawec, T., *Anal. Biochem.* **4** (2), 124–127 (1962).

(1290) Trachuk, S. V., Chekaida, S. T., *Med. Prom. SSSR* **16,** (7) 39–45 (1962); *C.A.* **55,** 413*b* (1961).

(1291) Trakman, Yu. G., *Sb. Nauchn. Tr. Tsentr. Aptechn. Nauchn.-Issled. Inst.* **2,** 85–92 (1961); *C.A.* **58,** 1306*h* (1963).

(1292) Travis, J., Robinson, A. D., *Can. J. Biochem. Physiol.* **40,** 1251–2 (1962); *C.A.* **57,** 15438*e* (1962).

(1293) Tsai, Chi-Kai, *Yao Hsueh Hsueh Pao* **8,** 193–6 (1960); *C.A.* **57,** 9955*d* (1962).

(1294) Tschudi-Steiner, I., Leupin, K., *Pharm. Acta Helv.* **37,** 274–82 (1962); *C.A.* **57,** 11311*e* (1962).

(1295) Tschudi-Steiner, I., Tranow, G., *Ibid.,* **38** (12), 848–53 (1963); *C.A.* **60,** 7874*f* (1964).

(1296) Tsuji, A., Hojo, M., *Bunseki Kagaku* **11,** 1255–62 (1962); *C.A.* **58,** 8852*h* (1963).

(1297) Tu, Chen-Yeh, Tsang, Feng-Ho, *Yao Hsueh Hsueh Pao* **10** (1), 10–14 (1963); *C.A.* **59,** 13768*f* (1963).

(1298) Tuchel, N., Antonescu, V., St. Moisescu, *Farmacia* (*Bucharest*) **10,** 213–18 (1962); *C.A.* **58,** 4380*h* (1963).

(1299) Tuchel, N., Lenhardt, E., *Ibid.,* **9,** 351–6 (1961); *C.A.* **57,** 970*h* (1962).

(1300) Tuckerman, M. M., *J. Pharm. Sci.,* **51,** 700–1 (1962).

(1301) Turi, P., Grossman, D., *Ibid.,* **52,** 809–10 (1963).

(1302) Turkevich, M. M., *Farmatsevt. Zh.* (*Kiev*) **18** (5), 30–1 (1963); *C.A.* **60,** 7873*h* (1964).

(1303) Tyihak, E., *Acta Pharm. Hung.* **32,** 122–8 (1962); *C.A.* **57,** 4762*c* (1962).

(1304) Ubatuba, F., Takab, S., *Anais Acad. Brasil. Cienc.* **32,** (1) 85–9 (1960); *C.A.* **57,** 11468*c* (1962).

(1305) Ueno, K., Tachikawa, T., *Japan Analyst* 11 (5), 554–558 (1962); *Anal. Abstr.* **11,** 340 (1964).

(1306) Ullmann, E., *Congr. Sci. Farm., Conf. Comun., 21°, Pisa* **1961,** 902–6; *C.A.* **59,** 1442*h* (1963).

(1307) Ullmann, E., Thoma, K., Zelfel, G., *Pharm. Acta Helv.* **38,** (7–8), 577–86 (1963); *C.A.* **60,** 1544*g* (1964).

(1308) Ulrich, W. F., "Developments in Applied Spectroscopy," **Vol. II,** Proc. of 13th Annual Symp. on Spectroscopy, Chicago, Ill., U.S.A. April–May 1962, pp. 130–141.

(1309) Umeda, M., Tsubota, S., Kajita, A., *Takamine Kenkyusho Nempo* **14,** 87–90 (1962); *C.A.* **58,** 4379*f* (1963).

(1310) Underhill, E. W., Youngken, H. W., Jr., Tsao, D. P. N., *Lloydia* **24,** 65–7 (1961).

(1311) Uno, T., Morishita, N., *J. Pharm. Soc. Japan,* **80** (12), 1679–1681 (1960); *Anal. Abstr.* **9,** 4458 (1962).

(1312) Uno, T., Yasuda, H., Kondo, T., *Ibid.,* **81** (4), 499–502 (1961); *Anal. Abstr.* **9,** 4897 (1962).

(1313) Unterman, W. H., *Rev. Chim.* (*Bucharest*) **13,** 618 (1962); *C.A.* **58,** 8853*f* (1963).

(1314) Unterman, W. H., *Ibid.,* **14** (5), 301–2 (1963); *C.A.* 59, 11189*c* (1963).

(1315) Uprety, M. C., Basu, D. K., Rao, V. K. M., *Indian J. Technol.* **1,** 397–8 (1963); *C.A.* **60,** 1544*a* (1964).

(1316) Uprety, M. C., Sen, R., Agrawal, D. K., Rao, V. K. M., *Ibid.,* **1,** 391–4 (1963); *C.A.* **60,** 375*b* (1964).

(1317) Urbanski, T., *Talanta,* **9** (9), 799–800 (1962).

(1318) Urx, M., Vondrackova, J., Kovarik, L., Horsky, O., Herold, M., *J. Chromatog.* **11,** 62–5 (1963).

(1319) Utsumi, I., Samejima, M., Kurita, H., *J. Vitaminol.* (*Kyoto*) **7,** 209–14 (1961); *C.A.* **57,** 4962*g* (1962).

(1320) Utsumi, I., Tanaka, N., Nagano, S., *Yakugaku Zasshi* **82,** 32–6 (1962); *C.A.* **57,** 3568*e* (1962).

(1321) Vachek, J., *Cesk. Farm.* **9** (10), 505–6 (1960); *C.A.* **60,** 2722*h* (1964).

(1322) Vachek, J., *Ibid.,* **10** (4), 187–8 (1961); *C.A.* **59,** 15120*d* (1963).

(1323) *Ibid.*, **10** (10), 515–517; *Anal. Abstr.* **9,** 3428 (1962).

(1324) *Ibid.*, **11** (9), 464–466 (1962); *Anal. Abstr.* **10,** 3434 (1963).

(1325) *Ibid.*, pp. 469–471; *Anal. Abstr.* **10,** 3432 (1963).

(1326) Vadodaria, D. J., Parikh, P. M., Mukherji, S. P., *Indian J. Pharm.* **24,** 180–3 (1962); *C.A.* **58,** 4376*g* (1963).

(1327) Vaedtke, J., Gajewska, A., *J. Chromatog.* **9,** 345–347 (1962).

(1328) Vagnina, L. L., *J. Ass. Off. Agric. Chem.* **45** (3), 600–608 (1962).

(1329) Vaidya, E. R., Huyck, C. L., *Am. J. Pharm.* **134,** 404–13 (1962).

(1330) Vaisman, G. A., Bushkova, M. M., Kogan, O. M., *Farmatsevt. Zh.* (*Kiev*) **17** (1), 15–20 (1962); *C.A.* **57,** 2333*c* (1962).

(1331) Vaisman, G. A., Chaikovs'ka, M. A., *Ibid.*, **17** (3), 20–5 (1962); *C.A* **58,** 7788*b* (1963).

(1332) Vaisman, G. A., Yampol'skaya, M. M., *Aptechn. Delo* **11** (5), 38–41 (1962); *C.A.* **60,** 10476*b* (1964).

(1333) Vaisman, G. A., Yashchenko, D. V., *Farmatsevt. Zh.* (*Kiev*) **18** (2), 33–7 (1963); *C.A.* **60,** 3955*a* (1964).

(1334) Vanden Heuvel, W. J. A., Horning, E. C., *Biochim. Biophys. Acta* **74** (3), 560–563 (1963); *Anal. Abstr.* **11,** 1439 (1964).

(1335) Vanden Heuvel, W. J. A., Haahti, E. O. A., Horning, E. C., *Clin. Chem.* **8** (4), 351–359 (1962).

(1336) Vasavada, S. A., Malik, P. A., Kulkarni, V. P., Anantanarayanan, K. G., Deliwala, C. V., *J. Sci. Ind. Res.* (*India*) **21C,** 96–9 (1962); *C.A.* **57,** 14097*h* (1962).

(1337) Vasbinder, H., van der Sijde, H. R., *Pharm. Weekbl.* **97** (17), 610–611 (1962); *Anal. Abstr.* **10,** 788 (1963).

(1338) Vasilenko, S. K., Kamzolova, S. G., Knorre, D. G., *Biokhimiya* **27,** 142–48 (1962); *C.A.* **57,** 1215*i* (1962).

(1339) Vasiliev, R., Burnea, I., Cosmin, A., Mangu, M., *Rev. Chim.* (*Bucharest*) **11,** 422 (1960); *C.A.* **57,** 16752*b* (1962).

(1340) Vasiliev, R., Chialda, I., Anastasescu, Gr., *Ibid.*, **14** (6), 351 (1963); *C.A.* **59,** 12594*h* (1963).

(1341) Vasiliev, R., Cosmin, A., Mangu, M., Burnea, I., *Ibid.*, **13,** 239 (1962); *C.A.* **57,** 16752*a* (1962).

(1342) Vasiliev, R., Mangu, M., *Ibid.*, **14** (1), 49 (1963); *C.A.* **59,** 2592*a* (1963).

(1343) Vasiliev, R., Scintee, V., *Pharmazie* **18** (11), 746–7 (1963); *C.A.* **60,** 9101*b* (1964).

(1344) Vasiliev, R., Scintee, V., Chialda, I., Sisman, E., Fruchter, J., Jecu, M., *Rev. Chim.* (*Bucharest*) **13,** 759–60 (1962); *C.A.* **59,** 1438*h* (1963).

(1345) Vasiliev, R., Scintee, V., Fruchter, J., *Farmacia* (*Bucharest*) **10,** 283–7 (1962); *C.A.* **58,** 4375*f* (1963).

(1346) Vasiliev, R., Scintee, V., Fruchter, J., *Rev. Chim.* (*Bucharest*) **13,** 170–1 (1962); *C.A.* **57,** 13890*b* (1962).

(1347) Vasiliev, R., Scintee, V., Sisman, E., *Ibid.*, **13** (1), 56 (1962); *Anal. Abstr.* **9,** 5405 (1962).

(1348) Vasiliev, R., Scintee-Pazarina, V., Sisman, E., *Ibid.*, **14** (6), 352–3 (1963); *C.A.* **59,** 12594*f* (1963).

(1349) Vasiliev, R., Sisman, E., *Ibid.*, **14** (9), 533–4 (1963); *C.A.* **60,** 3950*h* (1964).

(1350) Vasiliev, R., Sisman, E., Burnea, I., *Pharmazie* **17,** 606–8 (1962); *C.A.* **58,** 6649*c* (1963).

(1351) Vasiliev, R., Sisman, E., Burnea, I., *Rev. Chim.* (*Bucharest*) **13,** 557 (1962); *C.A.* **58,** 5454*g* (1963).

(1352) Vasilov, S. I., Nikolaev, V. I., *Metody Lyuminests, Analiza, Materialy VIII* (*Vos'mogo*) *Soveshch. po Lyuminests., Akad. Nauk Belorussk. SSR, Inst. Fiz.* **1959,** 126–31; *C.A.* **57,** 2336*i* (1962).

(1353) Vasilov, S. I., Nikolaev, V. I., Khamin, N. S., *Aptechn. Delo* **11,** (1) 34–9 (1962); *C.A.* **57,** 960*b* (1962).

(1354) Vassileva, B., *Compt. Rend. Acad. Bulgare Sci.* **16** (4), 369–72 (1963); *C.A.* **60,** 5276*f* (1964).

(1355) Vecerkova, J., Kacl, K., *Acta Univ. Carolinae, Med Suppl. No.* **14,** 295–302 (1961); *C.A.* **57,** 15241*h* (1962).

(1356) Vecerkova, J., Kacl, K., *Ceskosl. Farm.* **11** (3), 129–134 (1962); *Anal. Abstr.* **9,** 5410 (1962).

(1357) Vecerkova, J., Kohlicek, J., Kacl, K., *Pharmazie* **17** (7), 394–399 (1962); *Anal. Abstr.* **10,** 787 (1963).

(1358) Vecerkova, J., Solc, J., Kacl, K., *J. Chromatog.* **10,** 479–492 (1963).

(1359) Vecerkova, J., Sulcova, M., Kacl, K., *Pharmazie* **17** (1), 22–29 (1962); *C.A.* **57,** 4761*h* (1962).

(1360) Vecerkova, J., Sulcova, M., Kacl, K., "Some Gen. Probl. Paper Chromatog.," Symp., Liblice, Czech. **1961,** 95–104; *C.A.* **59,** 11188*e* (1963).

(1361) Vegh, A., Gracza, M., Szasz, G., Brantner, A., Budvari, Z., *Acta Pharm. Hung.* **33** (3), 105–11 (1963); *C.A.* **60,** 1541*d* (1964).

(1362) Vegh, A., Szasz, G., Gracza, P., *Gyogyszereszet* **3,** 184–5 (1959); *C.A.* **58,** 3271*e* (1963).

(1363) Vegh, A., Szasz, G., Brantner, A., Gracza, Maria, *Ibid.*, **7,** 421–3 (1963); *C.A.* **60,** 6703*a* (1964).

(1364) Vegh, A., Szasz, G., Kertesz, Piroska, *Pharmazie* **16,** 512–14 (1961); *C.A.* **55,** 412*d* (1961).

(1365) Vegh, A., Szasz, G., Papp, O. B., *Acta Pharm. Hung.* **29,** 212–16 (1959); *C.A.* **57,** 12635*a* (1962).

(1366) Vejdelek, Z. J., Kakac, B., *Cesk. Farm.* **9** (7), 359–64 (1960); *C.A.* **60,** 9100*a* (1964).

(1367) Velescu, G., *Farmacia* (*Bucharest*) **10,** 297–8 (1962); *C.A.* **58,** 4379*e* (1963).

(1368) Vertes, A., Ezer, E., Szejtli, J., Zala, Gy., *Gyogyszereszet* **7,** 418–20 (1963); *C.A.* **60,** 5275*h* (1964).

(1369) Vessman, J., Schill, G., *Svensk. Farm. Tidskr.* **66** (25), 601–618 (1962); *Anal. Abstr.* **10,** 791 (1963).

(1370) Vestergaard, Per, *Acta Endocrinol. Suppl.* **64,** 39, 1–35 (1962); *C.A.* **57,** 10128*d* (1962).

(1371) Vida, I., Vastagh, Gabor, *Acta Pharm. Hung.* **33** (5), 197–201 (1963); *C.A.* **60,** 6704*a* (1964).

(1372) Vida, L., *Ibid.*, **33,** 3–8 (1963); *C.A.* **59,** 5213*f* (1963).

(1373) Vieillefosse, R., Hanegraf, C., *Ann. Pharm. Franc.* **20,** 480–1 (1962); *C.A.* **57,** 1675*d* (1962).

(1374) Vieillefosse, R., Khalili-Varasteh, H., Hanegraaff, C., *Ibid.*, **20,** 855–63 (1962); *C.A.* **59,** 3720*f* (1963).

(1375) Vignoli, L., Cristau, B., Defretin, J. P., *Ibid.*, **21** (6), 477–85 (1963); *C.A.* **59,** 11190*h* (1963).

(1376) Villanova, R. G., Thomas, J., Bosch, F., Sune, J. M., *Ars Pharm.* **3,** 127–37 (1962); *C.A.* **59,** 13768*e* (1963).

(1377) Vincent, D., Segonzac, G., Sesque, G., *Ann. Pharm. Franc.* **20,** 455–62 (1962); *C.A.* **58,** 411*b* (1963).

(1378) V'lkova, A., Kolusheva, A., *Farmatsiya* (*Sofia*) **12** (2), 16–20 (1962); *C.A.* **57,** 8658*c* (1962).

(1379) Vogelenzang, E. H., *Farmaco* (*Pavia*), *Ed. Prat.* **18** (4), 189–98 (1963); *C.A.* **59,** 2591*e* (1963).

(1380) Voigt, R., *Pharmazie* **17,** 101–6 (1962); *C.A.* **57,** 3564*i* (1962).

(1381) Voight, R., Kaehler, A., *Pharm. Zentralhalle* **101,** 95–8 (1962); *C.A.* **57,** 9956*g* (1962).

(1382) Volkova, E. A., *Tr. Khar'kovsk. Farmatsevt. Inst. No.* **2,** 113–15 (1962); *C.A.* **60,** 6704*a* (1964).

(1383) Vondrackova, J., Urx, M., *Sb. Praci* **10**—i.e., *Desetin—Vyroci Zalozeni Ustavu* (*Vyzkum. Ustav Antibiot. Roztokach Prahy*) **1962,** 70–5; *C.A.* **59,** 11191*e* (1963).

(1384) Vorob'eva, V. Ya., Samsonov, G. V., *Antibiotiki* **8** (1), 73–76 (1963); *Anal. Abstr.* **10,** 5361 (1963).

(1385) Wagner, F. A., Vincent, M. C., *J. Pharm. Sci.* **51,** 365–6 (1962).

(1386) Wagner, G., Flotow, R., *Pharm. Zentralhalle* **101,** 179–87 (1962); *C.A.* **57,** 12636*a* (1962).

(1387) Wagner, H., *Congr. Sci. Farm., Conf. Comun., 21°, Pisa* **1961,** 911–16; *C.A.* **59,** 1440*b* (1963).

(1388) Wakazawa, T., Sugano, Y., Abe, M., Fukatsu, S., Kawaji, S., *J. Antibiotics* (*Tokyo*) *Ser. A* **14,** 180–6 (1961); *C.A.* **57,** 3566*d* (1962).

(1389) Waksmundzki, A., Kaczor, M., *Acta Polon. Pharm.* **19** (2), 143–147 (1962); *Anal Abstr.* **10,** 1161 (1963).

(1390) Waksmundzki, A., Ratajewicz, D., Soczewinski, E., *Ibid.*, **19,** 55–7 (1962); *C.A.* **57,** 3565*d* (1962).

(1391) Waksmundzki, A., Soczewinski, E., Ratajewicz, D., *Chem. Anal., Warsaw* **8** (1), 103–106 (1963); *Anal. Abstr.* **10,** 5377 (1963).

(1392) Waldi, D., *Arch. Pharm., Berlin,* 1962, **295** (7); *Mitt. dtsch. pharm. Ges.*, **32** (7), 125–128 (1962); *Anal. Abstr.* **10,** 1160 (1963).

(1393) Waldi, D., "Some Gen. Probl. Paper Chromatog.," Symp., Liblice, Czech. **1961,** 139–51; *C.A.* **59,** 15119*c* (1963).

(1394) Walter, W. G., *Univ. Microfilms* (*Ann Arbor, Mich.*), *Order No.* **62-4401,** 120 pp.; *Dissertation Abstr.* **23,** 1523 (1962).

(1395) Wasilewska, L., *Acta Polon. Pharm.* **20** (2), 211–212 (1963); *Anal. Abstr.* **11,** 737 (1964).

(1396) Watanabe, A., Yoshida, T., Ochi, H., *Bitamin* **20** (2), 205–12 (1960); *C.A.* **60,** 7875*a* (1964).

(1397) Watanabe, H., *Bunseki Kagaku* **9,** 360–3 (1960); *C.A.* **57,** 7387*c* (1962).

(1398) Watanabe, H., *Ibid.*, **11,** 233–7 (1962); *C.A.* **57,** 958*i* (1962).

(1399) Watanabe, H., *Ibid.*, **10** (3), 268–270 (1961); *Anal Abstr.* **10,** 2457 (1963).

(1400) *Ibid.*, pp. 271–274.

(1401) Watanabe, T., Endo, S., Iida, Y., *J. Antibiotics* (*Tokyo*) *Ser. A* **15,** 112 (1962); *C.A.* **57,** 1213*g* (1962).

(1402) Watson, J. R., Pernarowski, M., *J. Assoc. Offic. Agr. Chemists* **45,** 609–15 (1962).

(1403) Weaver, W. J., Reschke, R. F., *J. Pharm. Sci.* **52** (4), 362–364 (1963).

(1404) Webb, F. W., *J. Pharm. Pharmacol.* **13** (*Suppl.*), 136T–143T (1961).

(1405) Wegen, P. A. van der, *Pharm. Weekblad* **98** (17), 659–60 (1963); *C.A.* **59,** 15120*h* (1963).

(1406) Wegen, P. A. van der, *Ibid.*, **97,** 664–6 (1962); *C.A.* **58,** 6648*c* (1963).

(1407) Wegen, T. P. A. van der, *Ibid.*, (1963) **98** (4), 109–110; *Anal. Abstr.* **11,** 338 (1964).

(1408) Welsh, L. H., Summa, A. F., *J. Pharm. Sci.* **52,** 656–8 (1963).

(1409) Wermescher, B., Murea, L., Beral, H., *Rev. Chim., Bucharest* **13** (2), 105–107 (1962); *Anal. Abstr.* **10,** 1989 (1963).

(1410) Wesolowski, J. W., *Univ. Microfilms* (*Ann Arbor, Mich.*), *Order No.* **61-5621,** 59 pp.; *Dissertation Abstr.* **22,** 2762 (1962).

(1411) West G. B., Whittet, T. D., *J. Pharm. Pharmacol.* **14,** 93–4 (1962).

(1412) Westerink D., *Pharm. Weekbl.* **97** (24), 849–856 (1962); *Anal. Abstr.* **10,** 3869 (1963).

(1413) Weyers, J., Skora, M., *Dissertationes Pharm.* **14**, 201–5 (1962); *C.A.* **57**, 16746*i* (1962).
(1414) Wichlinski, L., Trzebinski, J., *Acta Polon. Pharm.* **20** (1), 31–34 (1963); *Anal Abstr.* **10,** 4875 (1963).
(1415) Wichtl, M. W., Fuchs, L., *Arch. Pharm. Berlin* **295** (5), 361–373 (1962); *Anal. Abstr.* **9,** 5395 (1962).
(1416) Wichtl, M., Peithner, G., Fuchs, L., *Planta Med.* **10,** 304–17 (1962); *C.A.* **58,** 10040*d* (1963).
(1417) Wilkie, J. B., *J. Assoc. Offic. Agr. Chemists* **46** (5), 920–5 (1963).
(1418) Wilkomirsky, F. T., Meier, G. H., Brieva, A. J., *Anales Real Acad. Farm.* **28,** (4) 311–17 (1962); *C.A.* **58,** 10041*d* (1963).
(1419) Windheuser, J. J., Higuchi, T., *J. Pharm. Sci.* **51,** 354–64 (1962).
(1420) Winek, C. L., Beal, J. L., Cava, M. P., *J. Chromatog.* **10** (2), 246–247 (1963).
(1421) Wisniewski, J. V., Spencer, S. F., *J. Gas Chromatog.* **2** (1), 34–5 (1964).
(1422) Wisniewski, W., Jablonski, S., Wojtyra, J., *Acta Polon. Pharm.* **19** (4), 325–32 (1962); *C.A.* **60,** 5277*c* (1964).
(1423) Wisniewski, W., Pietura, A., *Ibid.*, **20** (1), 43–51 (1963); *Anal. Abstr.* **10,** 4876 (1963).
(1424) Wojahn, H., Boll. E., *Deut. Apotheker-Ztg.* **100** (50), 1453–5 (1960); *C.A.* **60,** 369*f* (1964).
(1425) Wojciak, W., Wolska, E., *Med. Doswiadczalna Mickrobiol.* **15** (3), 255–62 (1963); *C.A.* **60,** 3952*a* (1964).
(1426) Wolff, J., *J. Pharm. Sci.* **52,** 93–5 (1963).
(1427) Wollenweber, P., *J. Chromatog.* **7** (4), 557–560 (1962); *Anal. Abstr.* **10,** 229 (1963).
(1428) Wollmann, Chr., Schulz, D., *Pharm. Praxis, Beilage Pharmazie* **1963** (7), 128–30; *C.A.* **59,** 15122*c* (1963).
(1429) Xu, Li-Xin, Zhou, Tong-Hui, *Acta Chim. Sinica* **28** (6), 391–393 (1962); *Anal. Abstr.* **10,** 4354 (1963).
(1430) Yalcindag, O. N., *Folia Pharm. (Istanbul)* **4** 536–40 (1962); *C.A.* **57,** 4762*i* (1962);
(1431) Yamaguchi, K., Ito, M., *J. Pharm. Soc. Japan* **81** (2), 179–181 (1961); *Anal. Abstr.* **9,** 4881 (1962).
(1432) Yamaguchi, S., Seki, I., Okuda, S., Tsuda, K., *Chem. Pharm. Bull. (Tokyo)* **10** (8), 755–7 (1962); *C.A.* **59,** 2590*a* (1963).
(1433) Yamamoto, R., Fujisawa, S., *Yakugaku Zasshi* **82,** 1396–9 (1962); *C.A.* **58,** 5456*c* (1963).
(1434) *Ibid.*, pp. 1400–4.
(1435) Yamamura, J., Kato, N., *Kagaku Keisatsu Kenkyusho Hokoku* **15,** 223–5 (1962); *C.A.* **57,** 12635*c* (1962).
(1436) Yamana, T., Mizukami, Y., Niikawa, Y., Murakami, Y., Yamamoto, Y., Setokawa, K., *Yakuzaigaku* **23,** 72–3 (1963); *C.A.* **69,** 11194*a* (1963).
(1437) Yamana, T., Sato T., *Ibid.*, **22,** 189–90 (1962); *C.A.* **58,** 8855*c* (1963).
(1438) Yamanaka, T., Asai, S., Aoki, J., *Arch. Pract. Pharm., Japan* **22** (1), 60–63 (1962); *Anal Abstr.* **10,** 5300 (1963).
(1439) Yamazaki, T., *J. Pharm. Soc. Japan* **83** (4), 402–405 (1963); *Anal. Abstr.* **11,** 321 (1964).
(1440) Yamazaki, T., *Yakugaku Zasshi* **83,** 402–5 (1963); *C.A.* **59,** 2590*d* (1963).
(1441) Yampol'ska, M. M., *Farmatsevt. Zh. (Kiev)* **18** (5), 8–12 (1963); *C.A.* **60,** 7871*d* (1964).
(1442) Yaskina, D. S., Filichkina, M. P., *Aptechn. Delo* **11** (4), 33–6 (1962); *C.A.* **60,** 10478*a* (1964).
(1443) Yates, S. G., *J. Chromatog.* **12** (3), 432–6 (1963).
(1444) Yavors'kii, M. P., *Farmatsevt. Zh. (Kiev)* **16** (5), 38–44 (1961); *C.A.* **59,** 382*g* (1963).
(1445) Yoshimura, M., Deki, M., *Bunseki Kagaku* **12** (10), 941–5 (1963); *C.A.* **60,** 1541*b* (1964).
(1446) Yoshimura, M., Deki, M., Tsukamoto, H., *Yakugaku Zasshi* **83,** 223–6 (1963); *C.A.* **59,** 383*a* (1963).
(1447) Yoshino, T., Seno, A., Sugihara, M., *J. Pharm. Soc. Japan* **80** (10), 1484–1486 (1960); *Anal. Abstr.* **9,** 4454 (1962).
(1448) Yoshino, T., Seno, A., Sugihara, M., *Kagaku To Kogyo (Osaka)* **36,** 404–9 (1962); *C.A.* **58,** 2475*f* (1963).
(1449) Yung, D. K., Pernarowski, M., *J. Pharm. Sci.* **52** (4), 365–370 (1963).
(1450) Zabrak, D., Zoltai, L., *Acta Pharm. Hung.* **33** (5), 206–11 (1963); *C.A.* **60,** 6702*g* (1964).
(1451) Zacharius, R. M., Talley, E. A., *J. Chromatog.* **7,** 51–55 (1962).
(1452) Zaitsev, V. A., *Sb. Nauchn. Tr. Tsentr. Aptechn. Nauchn.-Issled. Inst.* **2,** 101–13 (1961); *C.A.* **58,** 1304*a* (1963).
(1453) Zajta, E., *Acta Pharm. Hung.* **32,** 129–39 (1962); *C.A.* **57,** 4772*d* (1962).
(1454) Zak, B., Cohen, J. S., *J. Pharm. Sci.* **52** (9), 912–14 (1963); *C.A.* **59,** 13770*a* (1963).
(1455) Zalai, K., *Gyogyszereszet* **5,** 214–16 (1961); *C.A.* **60,** 5280*g* (1964).
(1456) Zaputryaev, B. A., Sil'vestrova, T. E., *Med. Prom. SSSR* **17** (8), 37–9 (1963); *C.A.* **60,** 369*e* (1964).
(1457) Zarnack, J., Pfeifer, S., *Pharmazie* **17,** 431–6 (1962); *C.A.* **58,** 3271*c* (1963).
(1458) Zarnack, J., Pfeifer, S., *Ibid.*, **19** (2), 111–23 (1964); *C.A.* **60,** 10477*c* (1964).
(1459) Zathurecky, L., Bauerova, O., Somoskeoy, G., Molnar, L., Suchy, S., *Ceskosl. Farm.* **12** (4), 171–177 (1963); *Anal. Abstr.* **11,** 1893 (1964).
(1460) Zinner, G., Deucker, W., *Naturwissenschaften* **49** (13), 300 (1962); *Anal. Abstr.* **10,** 1061 (1963).
(1461) Zommer, S., Lipiec, T., *Acta Polon. Pharm.* **20** (3), 229–232 (1963); *Anal. Abstr.* **11,** 1469 (1964).
(1462) Zuch, D. A., Conine, J. W., *J. Pharm. Sci.* **52,** 59–63 (1963).
(1463) *Ibid.*, pp. 63–6.
(1464) Zurkowska, J., Lukaszewski, M., Ozarowski, A., *Acta Polon. Pharm.* **20** (2), 115–120 (1963); *Anal. Abstr.* **11,** 744 (194)

Rubber

Coe W. Wadelin

Research Division, Goodyear Tire and Rubber Co., Akron, Ohio 44316

This is the eleventh in a series of review articles on rubber (*7–12, 95, 112, 147, 149*). The scope has been changed from previous reviews to include only chemical analysis. In general, rubber has been covered, but not plastics. For instance, ethylene-propylene rubber is included but not polyethylene or polypropylene. This requires arbitrary choices as the dividing line between rubber and plastics is not always clear. The previous policy of including methods for identification or determination of compounding ingredients in rubber but omitting methods dealing with the analysis of the compounding ingredients themselves has been continued.

The literature available to the author from October 1962, the end of the period covered by the last review (*112*), through September 1964 is covered. The labor of searching was taken over by the information retrieval system in the Technical Information Center of Goodyear's Research Division (*60*). The thoroughness of this system was checked by manually searching *Chemical Abstracts'* subject indexes for Volumes 57, 58, and 59. In no case was an article missed by the information retrieval system except where the original source was not available to the literature searchers. The system covers only the 350 periodicals received by the Goodyear Research Library but not secondary sources such as *Chemical Abstracts* and *Rubber Abstracts*.

The section on polymer characterization, while having the most references, is by no means comprehensive. The methods included are mostly chemical and infrared. It is hoped that subsequent reviews will more adequately cover this area.

The abbreviations recommended by ASTM Designation D1418-61T have been used (*2*). They are listed in Table I.

Table I. Abbreviations Recommended by ASTM (2)

BR	Butadiene rubber
IR	Isoprene rubber, synthetic
CR	Chloroprene rubber
NR	Isoprene rubber, natural
IIR	Isobutylene–isoprene rubber
NBR	Nitrile–butadiene rubber
SBR	Styrene–butadiene rubber
IM	Polyisobutylene
EPM	Ethylene–propylene copolymer

GENERAL INFORMATION

The format of ASTM Standards has been changed so that methods dealing with rubber are now in Part 28 (*1*). In looking for ASTM Methods, one should remember that there are five pertinent designations; D297 for rubber products, D1076 for NR latex, D1278 for crude NR, D1416 for SBR, and D1417 for SBR latex. There have been no changes in these methods since the last review. A new edition of Scott's "Standard Methods of Chemical Analysis" contains a section on rubber (*156*), which is essentially taken from ASTM Methods.

POLYMER IDENTIFICATION

Pyrolysis–Gas Chromatography. There seems to be no general agreement as to the best method for pyrolyzing a sample prior to gas chromatography of the pyrolyzate. It has been shown by several workers that time, temperature, and catalytic conditions must be carefully controlled for reproducible results. A tubular furnace packed with glass wool and operated at 700° C. gave relatively few pyrolysis products and therefore simple gas chromatograms (*34*). Pyrolysis in a quartz capillary for 26 seconds at 950° C. gave reproducible decomposition to small fragments (*62*). These conditions were applied to 150 polymers. Polyolefins including EPM were pyrolyzed at 550° C. (*154*). Only the uncondensable part of the pyrolyzate was swept onto the gas chromatography column to identify NR, NBR, BR, IIR, CR, or SBR (*77*, *142*). If a cold trap is used to retain heavy materials, styrene will be lost and SBR will be missed. The same materials were pyrolyzed at 500° C. and identified by their gas chromatograms on squalane, dinonyl phthalate, squalane–dimethyl sulfolane, or dinonyl phthalate–dimethyl sulfolane columns (*46*). In another procedure, the sample was pyrolyzed at 800° C. and only the condensed pyrolyzate was used for gas chromatography (*21*). Guiochon and Henniker concluded that pyrolysis followed by gas chromatography is rarely adequate for identification (*64*). When combined with an infrared spectrum much more can be done. They also pointed out that polyurethanes are not adequately identified by infrared or pyrolysis and gas chromatography. Fillers affect pyrolysis by their catalytic effects. Plasticizers which are not extractable, such as polyesters and polyacrylonitrile, cannot be distinguished from the main polymer by pyrolysis and gas chromatography. The identification of silicone rubber was also reported (*45*).

One of the seemingly simple techniques, namely, pyrolyzing the polymer directly on a platinum coil, suffers from changes in the temperature of the platinum with each successive pyrolysis. This is due to formation of platinum carbide with a resultant increase in resistance. However, for paint it is convenient to dip a platinum coil into the sample, turn on a small current to evaporate solvent, and then pyrolyze directly in the carrier gas stream (*124*). A tungsten filament can be substituted for platinum (*139*). This may be less prone to change in resistance. Perry has reviewed pyrolysis and gas chromatography and gives many references (*115*).

Infrared. Two reviews on the use of infrared spectra to identify rubber have appeared (*47*, *110*). For raw polymers, a film cast from a solution is recommended. Cured samples are pyrolyzed with both the liquid and gaseous pyrolyzates being examined. Carbon black-filled vulcanizates were swelled in methyl cyclohexane before cutting 2-micron sections (*32*). Their spectra can be used to identify both polymer and inorganic fillers. The technique is not universally applicable. For example, CR and NBR are not swollen by methyl cyclohexane. It is also inapplicable to oil-extended rubber. Others workers prepared microtome sections by first freezing the sample (*64*, *141*). A technique was described for making KBr pellets of polymers including NR and CR (*152*). Attenuated total reflectance was used to identify NR, NBR, IIR, and SBR filled with clay or $CaCO_3$ (*70*). The spectra of BR, poly(methylbutadiene), and poly(dimethylbutadiene) have been published (*30*). The spectra of a number of polymers and frequency assignments for CH_2, CH_3, aromatic CH, NH, C=O, OH, and OOH in the 1.0- to 2.7-micron region were published (*51*). They were all studied as films except IM which was in carbon tetrachloride solution. The spectra of polymer films on rock salt plates were recorded before and after exposure to ammonia, hydrogen chloride, and bromine (*20*). The changes can be used to identify functional groups. For example, carboxylic acids are converted to ammonium salts and carbon-carbon double bonds are brominated. In SBR, the phenyl ring is also brominated.

Mass Spectrometry. Phillips pyrolyzed rubber in an arc image furnace for 2 seconds at 3300° C. (*116*). Under these conditions there is a high yield of monomer. The relative amounts of butadiene, acrylonitrile, styrene, and isoprene shown by the mass spectrum of the pyrolysis gas can be used to identify SBR, BR, NBR, and NR. It is valuable to examine both the gaseous and liquid pyrolyzates by mass spectra (*69*). The liquid portion contains some high boiling pyrolysis products which would be missed if only the gaseous part were used. The mechanism of polymer pyrolysis has been studied (*3*).

POLYMER CHARACTERIZATION

General Information. The book edited by Ke covers infrared, optical methods, fluorescence, DTA (differential thermal analysis), NMR (nuclear magnetic resonance), x-ray and electron diffraction, column fractionation, and other techniques (*81*). The use of infrared spectroscopy in determining tacticity and orientation was discussed (*164*). Slonim concluded that NMR should not be used alone, but rather in conjunction with other physical and chemical methods (*135*). The use of NMR, infrared, and chemical reactivity in the characterization of stereoregular polymers (*6*) and the application of DTA to polymers were reviewed (*101*). Gas chromatograms of the products of pyrolysis for 15-second intervals at successively increasing temperatures result in a fingerprint of the polymer (*5*). In general, block copolymers can be differentiated from random copolymers.

NR and IR. Binder used polarization spectra, maleic anhydride adducts, and hydrogenation products to make band assignments in the infrared spectra of polyisoprene (*14*). The infrared spectrum of polyisoprene containing 75% *cis*-1,4 and 20% *trans*-1,4 structures was found to be different from that of a mixture of 80% hevea and 20% balata, which are 100% *cis*-1,4 and 100% *trans*-1,4, respectively (*85*). The bands at 1130 and 1150 cm.$^{-1}$ appear only in long sequences of *cis*-1,4 and *trans*-1,4, respectively. This phenomenon may be useful in distinguishing mixtures of the two polymers from a polymer in which the structures are randomly distributed. Consequently, the 572 cm.$^{-1}$ band for determination of *cis*-1,4, 980 cm.$^{-1}$ for *trans*-1,4, and 888 cm.$^{-1}$ for 3,4 are preferred for determination of overall composition. Schmalz and Geiseler, however, used the 840, 890, 910, 1130, and 1150 cm.$^{-1}$ bands to determine 1,2-, 3,4-, *cis*-1,4, and trans-1,4, structures (*127*). When the 2965 cm.$^{-1}$ band was used for *cis*-1,4, the *trans*-1,4 was found by subtracting 1,2- and *cis*-1,4 from the total unsaturation found chemically (*109*). The *cis*-1,4 is found to ±3%. After subtracting out the contribution of the 3,4- structure, found from the 890 cm.$^{-1}$ band, the 1415 and 1423 cm.$^{-1}$ bands were used to determine *trans*-1,4 and *cis*-1,4 (*31*). Kinetics of the reaction with perbenzoic acid have been used to establish infrared calibrations (*127*).

Chen used high resolution NMR to measure the shifts in the methyl protons and thereby to determine *cis*-1,4, *trans*-1,4, and 3,4- structures but not 1,2- (*27*). These measurements can be combined with NMR measurements of different types of protons (*26*) or infrared to get a complete structural analysis. The *cis*-1,4 and *trans*-1,4 measurements are accurate to 0.5% and the 1,2- and 3,4- to 2 to 3%. NMR was considered to be more accurate than infrared (*57*). As little as 0.3% 3,4- and 1% *trans*-1,4 in high *cis*-1,4 polymer and 1% *cis*-1,4 in high *trans*-1,4 polymer could be detected. Golub, Fuqua, and Bhacca detected no *cis*-1,4 in balata and no *trans*-1,4 in hevea. Therefore, they concluded that each contains more than 99% of its nominal structure (*57*). Chakravarty, Banerjee, and Sircar, on the other hand, isolated formic acid from the ozonolysis products of hevea (*25*), confirming the presence of 2.5% 3,4- structure reported by Binder and Ransaw (*16*).

The amount of methanol required to cause precipitation in a carbon tetrachloride solution of polyisoprene degraded by perbenzoic acid was used to determine the 3,4- (or 1,2-) content (*75*).

BR. Binder also assigned infrared absorption bands of BR based on polarization spectra, hydrogenated BR, maleic anhydride adducts, and isomerization experiments (*15*). One CS_2 solution was used to measure *cis*-1,4 and 1,2- structures and another of different concentration to measure *trans*-1,4 and 1,2- by infrared absorption (*13*). The 1,2- values were compared to see if the results were compatible. In this way both *cis*-1,4 and *trans*-1,4 could be measured in their optimum per cent transmission ranges regardless of the composition of the sample. The 724 cm.$^{-1}$ band was used for *cis*-1,4, 967 cm.$^{-1}$ for *trans*-1,4, and 911 cm.$^{-1}$ for 1,2-. Results from films cast from 4% solutions in benzene are less sensitive to gel content than measurements in solution (*79*). In high *cis*-1,4 polymers the *trans*-1,4 was measured by the absorbance ratio of the 966 cm.$^{-1}$ band to the 1440 cm.$^{-1}$ band and 1,2- was measured by the ratio of 912 cm.$^{-1}$ to 1440 cm.$^{-1}$ Both calibration curves were linear. *Cis*-1,4 was then determined by subtracting the sum of these from 100%. This avoids the errors in making a direct spectrophotometric measurement of a component at the 95% level but introduces the assumption that the sample is composed exclusively of the three structures mentioned above. A more reliable way to avoid the dependence on a spectrophotometric measurement of a component at the 95% level is to subtract the 1,2- and *trans*-1,4 content from the total unsaturation found by iodine chloride (*84*) or iodine bromide (*93*) addition. These measurements of unsaturation can be quite good if carried out with due regard to choice of solvent and corrections for splitting out and substitution (*92*).

Calibration for the 1,2- content can be made by kinetic studies with perbenzoic acid (*84*). *Cis*-1,4 and *trans*-1,4 are not differentiated as their rates are nearly the same. Calibration for both 1,2-, and *trans*-1,4 has been made from sodium polybutadiene which contains no *cis*-1,4 (*93*).

Aldehydes, ketones, 1,2-addition and *trans*-1,4 structure were found in butadiene popcorn polymer by its infrared spectrum in KBr pellets (*106*).

The method of determining 1,2-addition by measuring the amount of methanol required to precipitate a polymer solution after reaction with perbenzoic acid was also applied to BR (*75*). The sensitivity is less than in NR and IR.

SBR. The *cis*-1,4 butadiene content can be found from the absorption band at 740 cm.$^{-1}$ or by subtracting the 1,2- (910 cm.$^{-1}$) and *trans*-1,4 (965 cm.$^{-1}$) contents from total unsaturation found by iodine chloride addition (*84*). The styrene content found from the 700 cm.$^{-1}$ band can be used as a check on the total unsaturation determination.

NMR was used to determine styrene, 1,2- addition, and 1,4- addition (*130*). CCl_4 solutions containing $(CH_3)_4Si$ as internal standard were used.

EPM. Infrared studies have led to general agreement on the assignment of bands in the 815 to 720 cm.$^{-1}$ region. The 815 cm.$^{-1}$ band is assigned to an isolated methylene group, 752 cm.$^{-1}$ to groups of two, 733 cm.$^{-1}$ to groups of three, 726 cm.$^{-1}$ to four, and 722 cm.$^{-1}$ to five (*24, 67, 68, 107, 151*). Model compounds (*24*) and hydrogenated NR (*151*) have been used to establish this. These bands can be used to distinguish block copolymers from random copolymers (*67, 68*). Mixtures of homopolymers are like block copolymers and polyallomers. The 1154 cm.$^{-1}$ band is due to methyl groups on alternating carbon atoms (*40, 97, 151*). The absorbance ratio 752 cm.$^{-1}$/1156 cm.$^{-1}$ can lead to inferences about the catalyst system used. Trialkyl aluminum with VCl_3 or $TiCl_3$ gives all head to tail addition of propylene while alkyl aluminum sesquichloride with $VOCl_3$ or $VO(OR)_3$ gives some tail to tail addition (*151*).

EPM containing less than 75% ethylene leaves no residue on extraction with acetone, ether, hexane, or heptane while in mixtures of homopolymers, polyethylene is left undissolved (*107*).

Gas chromatography of pyrolysis products gives patterns which are similar for block copolymers and mixtures of homopolymers but different from random copolymers (*5, 64*).

IM. Infrared and NMR were used to show that polymer made with BF_3 catalyst has the same *gem*-dimethyl structure as that made by Ziegler catalysis (*4*). Ultraviolet and infrared spectra were used to show that irradiation by 4 m.e.v. electrons produced *t*-butyl, 1,1-dialkyl ethylene, 1,1,2-trialkyl ethylene, 1,1,2-trialkyl ethane, and conjugated diene structures (*73*).

NBR. The same bands used in BR, *cis*-1,4 at 724 cm.$^{-1}$, *trans*-1,4 at 967 cm.$^{-1}$, and 1,2- at 911 cm.$^{-1}$, were used to measure these structures in NBR (*76*). The need was pointed out for making the system as simple as possible extracting with ether or methanol before pyrolysis or infrared.

CR. The *cis*-1,4 and *trans*-1,4 content was determined by means of the 1652 and 1660 cm.$^{-1}$ bands (*43*). Spectra was measured in 10% solutions in $CHCl_3$ in 0.2-mm. cells. Reproducibility is ±5%.

Siloxane–Acetylene Polymers. An absorption band at 2100–2150 cm.$^{-1}$ was used to measure Si-H bands, 1595–1600 cm.$^{-1}$ for vinyl, 1625–1635 cm.$^{-1}$ for allyl, and 3050 cm.$^{-1}$ for unsymmetrical disubstituted ethylene (*117*).

Piperylene Rubber. Reduction of ozonolysis products to give acetaldehyde and acetic acid made possible the calculation of 1,2-addition (*161*). Treatment with benzoyl peroxide to form the epoxide followed by reaction with HI to get acetaldehyde confirmed the ozonolysis results.

Poly(1-Cyano-1,3-Butadiene). The nitrile stretching band in this polymer appears at 2241 or 2220 cm.$^{-1}$ depending on whether it is attached to a saturated or unsaturated carbon atom (*55*).

Derivatives of IR. Golub and Heller measured the degree of cyclization of polyisoprene by the absorbance ratio of the 833 to the 1380 cm.$^{-1}$ bands (*58*). Lee, Scanlan, and Watson compared the perbenzoic acid, phenyl iododichloride, ozone, and ICl methods for measuring unsaturation (*91*). All methods were consistent except ICl which gave higher results due to substitution. They preferred perbenzoic acid. The ability to correct for substitution in the ICl method by measuring the acid produced (*92*) is too often overlooked.

The residual unsaturation in hydrochlorinated *cis*-1,4 NR and IR and *trans*-1,4 NR and IR can be determined by high resolution NMR (*59*).

Infrared still is a widely used tool in characterization of polymer structure, although NMR is growing. The constant signal strength of NMR for a given structure gives it a decided advantage over infrared in which each band must be carefully calibrated.

DETERMINATION OF POLYMERS IN POLYMER MIXTURES AND CONSTITUENTS IN COPOLYMERS

SBR. The absorbance ratio of the 1639 cm.$^{-1}$ vinyl band and the 1601 cm.$^{-1}$ phenyl band was used to determine from 14 to 90% styrene in SBR

(*157*). The success of the method depends on the constant 20% 1,2- addition in emulsion polymerization of butadiene over a wide range of temperature and butadiene content. Analysis of polymers made by other methods will require other calibration data. Systems with low 1,2- content would require use of another band for the butadiene component.

EPM. The absorbance ratio of the 1150 and 720 cm.$^{-1}$ bands was used to determine the ethylene–propylene ratio (*40*). According to Lomonte, the 1154 cm.$^{-1}$ band is due to methyl groups on alternate backbone carbon atoms and is applicable to block copolymers and mixtures of polyethylene and polypropylene but not to random copolymers (*97*). The intensity of the 720 cm.$^{-1}$ band in a film of fixed thickness at 180° C. is proportional to ethylene content (*98*). The elevated temperature eliminates crystallinity effects. The absorbance ratio of the 720 cm.$^{-1}$ band to the 1041 cm.$^{-1}$ band was preferred by others (*68*). The overtone region permits the use of thicker films which are easier to handle. The absorbance ratio of the methyl band at 1.692 microns to the methylene band at 1.764 microns is proportional to ethylene/propylene ratio (*23*). Corish and Tunnicliffe made a thorough study of several absorbance ratios and found that the ratio of the 1380 cm.$^{-1}$ and 1460 cm.$^{-1}$ bands in a film at 120° C. is the best general purpose method for composition (*33*). The accuracy is ±1% in the region of 50% ethylene. The method is applicable to random copolymers, block copolymers, and mixtures of homopolymers. Samples which are soluble in CCl_4 can be analyzed by means of the 1380 cm.$^{-1}$ methyl band (*107*). The calibration was done with atactic polypropylene.

The liquid phase of the pyrolyzate has a vinyl band at 909 cm.$^{-1}$ and a vinylidene band at 889 cm.$^{-1}$ Their absorbance ratio was used to determine 10 to 50% propylene with a standard deviation of 1.35 mole % (*22*). The calibration was based on the nominal compositions of commercial samples, which is a risky foundation on which to base an analytical method. The pyrolyzate can also be subjected to gas chromatography. Using peak height ratios, blends of polyethylene and polypropylene were analyzed to ±3% (*62, 154*). Ethylene–propylene copolymers give chromatograms different from homopolymer blends of the same overall composition (*62*). EPM of known composition was made by decomposing known amounts of diazomethane and diazpropane together (*80*). This method of synthesis should be useful for making calibration samples.

NBR. The acrylonitrile content was determined by means of the nitrile band at 2237 cm.$^{-1}$ with precision of ±1% (*76*). It is important to extract the sample before making the determination. The nitrile content of NBR or the NBR content of polymer mixtures can be found from the nitrogen content determined by the Kjeldahl or Dumas method (*76*). In the latter application, the nitrile content of the NBR must be known. Of course, other nitrogen-containing materials must be absent or removed by extraction.

Polyurethanes. Polyester urethanes were broken down by saponification (*128*). The sodium salt was converted to free acid on an ion exchange column and the acid was titrated. The polyol was determined by the periodate or permanganate method. The amines were converted to diazonium salts and measured colorimetrically. Trimethylol propane was isolated by treatment of the polyurethane with phenethyl amine, converted to the triacetate, and measured by gas chromatography with precision of ±3% relative at the 1% level (*159*).

Butadiene-1,3-Pentadiene Copolymers. Infrared spectra of carbon tetrachloride solutions have bands at 1377 cm.$^{-1}$ due to 1,2-pentadiene addition, 1371 cm.$^{-1}$ due to 1,4-pentadiene addition, and at 1355 cm.$^{-1}$ due to 1,4-butadiene (*119*). Analysis by means of these bands agrees with radioactivity measurements on copolymers containing 0-40 mole % tagged 1,3-pentadiene.

1 - Pentene - 4 - Methyl - 1 - Pentene Copolymers. This polymer gives only a low yield of the starting monomers upon pyrolysis. However, mass spectra of the pyrolyzate can be used to determine the composition (*155*).

SBR/NR. Further study (*94*) of pyrolysis followed by infrared measurement (*148*) showed that the absorbance ratio of bands at 889 and 909 cm.$^{-1}$ was a function of the vulcanization recipe when pyrolysis was done at 550° C. However, when the temperature was increased to 950° C., six different curing systems gave variations of only ±1.3% (*94*). The liquid and gaseous portions of the pyrolyzate obtained at 600° C. were combined in cyclohexane and the absorbance was measured at 697 cm.$^{-1}$ to find the SBR content (*44*). An attempted calibration by mixing varied proportions of pyrolyzates of the two pure homopolymers gave results too high for SBR by 10%. When calibration was based on pyrolysis of known mixtures results were accurate to ±2% in the range from 5 to 95% SBR. The gaseous portion of the pyrolyzate obtained in an arc image furnace was analyzed by mass spectrometry (*116*). The ratio of the sum of the heights of the 53 and 54 mass number peaks to the sum of the heights of the peaks at 68 and 104 was correlated with composition. This method also was applied to BR/NR and SBR/BR mixtures.

NR/NBR. This mixture in a cured, filled covulcanizate was analyzed for total polymer by subtracting the sum of acetone, $CHCl_3$, and alcoholic KOH extracts, sulfur, ash, and carbon black from 100% according to ASTM D297-61T (*1, 53*). The NR was then found by chromic acid oxidation (*1*). The NBR remaining was accurate to 2%. From the nitrogen content the acrylonitrile/butadiene ratio of the NBR can be found.

DETERMINATION OF RUBBER

NR in hevea brasiliensis leaves was determined by measuring the unsaturation of the benzene extract (*102*). The extract was treated with bromine generated from KBr, $KBrO_3$, and HCl, then the excess was measured by adding KI and $Na_2S_2O_3$ and backtitrating with iodine. The amount of rubber was calculated with an empirical factor. The coefficient of variation was 1.53%. Interferences can be removed rapidly by extractions with ethanol and water (*104*).

Rubber in rubber–bitumen mixtures was determined from the iodine value of the acetone-insoluble portion (*144*). A sample of the unrubberized bitumen must be available to establish a correction factor for its iodine value.

IM in cured, filled stocks was determined by solubilizing the polymer with nitric acid (*19*). The polymer was then extracted with petroleum ether and $CHCl_3$, precipitated with ether, dried, and weighed. This is similar to the method of Kress (*86*).

UNSATURATION

The unsaturation in the side chains of 1,2- units in piperylene rubber was calculated from the amounts of acetic acid and acetaldehyde in the ozonolysis products (*161*). Ozonization was carried out in $CHCl_3$ at −40° C. and reduction was conducted catalytically with Pd on $CaCO_3$. In an alternative method, acetaldehyde was obtained by reacting an epoxide of piperylene rubber with HI. The epoxide was formed by the oxidation of the rubber with benzoyl peroxide. Good agreement of the results is claimed.

Ozonolysis was applied to BR to determine the amounts of 1,2- and 1,4- units (*160*), and it was also used to determine the unsaturation of NBR (*138*). Ozonolysis of IIR was carried out in the presence of dibutyl sulfide to prevent attack on carbon–carbon single bonds (*100*). Unsaturation in EPM containing cyclooctadiene was determined by ozonization of the double bonds and spectrophotometric measurement of the active oxygen (*56*). The ozonized solution was treated with the leuco base of malachite green to form the corresponding dye, or treated with AlI_3 for subsequent determination of free iodine.

Halogenation with ICl was used to determine unsaturation in NBR (*76*) and in high impact polystyrene (*35*). The samples were treated with $CHCl_3$ and ICl and the excess ICl was determined. Unsaturation below 0.5 mole % in IIR was investigated successfully by the use of radioactive chlorine (*99*). Two atoms of chlorine were incorporated in the polymer for each double bond originally present. CCl_4 was used as the solvent.

SULFUR AND SULFIDES

The free sulfur content of SBR latex was determined by reaction of sulfur with Na_2SO_3 and by iodine titration of the $Na_2S_2O_3$ formed (*83*). Accuracy is 0.02% on a 0.05-g. sample. A method developed for the determination of sulfides present in vulcanized rubber compounds consists of treatment of the rubber with HCl in a CO_2 atmosphere to liberate H_2S, absorption of the H_2S in cadmium acetate, treatment with iodine, and back-titration with $Na_2S_2O_3$ (*90*). The free sulfur content of CR was obtained by measuring the polarogram of the acetone extract in a solution of sodium acetate in 1:1 methanol and acetic acid (*146*). In another method, cathode ray polarography was applied to ethyl acetate and isopropyl alcohol extracts of rubber, as well as to rubber solutions prepared with $CHCl_3$ (*150*). Methanol was used as the base electrolyte. The mean value of 10 measurements was 0.30%, with a standard deviation of 0.01%.

Alterations in unsaturation during cure of *cis*-IR were followed by changes in the infrared absorption spectra (*38*). Decrease in the intensity of absorption at 840 $cm.^{-1}$ was interpreted as decrease in unsaturation during cure. The resulting sulfide bonds were investigated from data in the ultraviolet region. The mechanism of sulfur–rubber reaction during vulcanization of *cis*-IR and -BR was investigated by studying the infrared spectra of vulcanizates (*133*). The occurrence of absorption at 962 $cm.^{-1}$ was attributed to the formation of cyclic sulfides and new conjugated double bonds. The determination of the free sulfur content of natural rubber vulcanizates was carried out by measuring the luminescence intensity of vulcanizates as a function of the time of cure (*122*).

Replica electron microscopy was used for the examination of the fracture surface of compounded synthetic rubbers to identify sulfur and other compounding ingredients (*120*).

RESIDUAL MONOMERS

A general method for residual monomers in latex involves dissolving the latex in acetic anhydride, 2,2-dimethoxypropane, or cyclohexanone and injecting the solution into a gas chromatograph (*158*). The first two reagents remove the water by reacting with it. Free styrene or α-methyl styrene in copolymers with butadiene was determined with accuracy of ±4% by measuring the ultraviolet spectrum of the isooctane, octane, heptane, or petroleum ether extract (*39*).

CURING AGENTS

Sulfenamide accelerators in extracts of vulcanizates were identified by extraction of the rubber with a mixture of CCl_4 and methanol, treatment of the extract with chlorine water to form amine hydrochlorides, paper chromatography of the amine hydrochlorides using acetic acid–water solution as the eluent, and identification of the various amine hydrochlorides (*72*). The method was applied to extracts of vulcanizates containing the sulfenamides of morpholine, diethyl amine, cyclohexyl amine, *t*-octyl amine, and *t*-butyl amine. Rubber accelerators of the thiuram series were also identified by paper chromatography (*118*).

Tetramethyl and tetraethyl thiuram mono- and disulfides alone and in mixtures in amounts of 3 to 5 μg. were determined by paper strip and circular paper chromatography using acetic acid–water solution as the mobile phase. Spots of mercaptobenzothiazole in paper chromatograms of acetone extracts were developed with saturated $Bi(NO_3)_3$ solution, cut out, and weighed for quantitative measurements (*140*). Benzothiazyl disulfide was determined by reduction to mercaptobenzothiazole prior to the chromatographic process. The average errors were 15 and 20%, respectively.

Quantitative chromatographic separation of sulfur from dimorpholinyl disulfide and tetramethylthiuram disulfide accelerators in vulcanizates was carried out by toluene elution of tagged sulfur from a column of alumina (*114*). The radioactive fraction was measured by scintillation counting with accuracy of ±5%. Acetone extracts were also separated on silica gel columns (*37*). The fractions were examined under ultraviolet radiation or by chemical tests.

Ethanol extracts of NR, SBR, NBR, CR, and IIR containing diazoaminobenzene, benzothiazyl disulfide, tetramethylthiuram disulfide, and mercaptobenzothiazole were analyzed in the ultraviolet region (*132*). The content of the ingredients was determined by comparing spectra of extracts of uncured rubber mixes and their vulcanizates. Mercaptobenzothiazole and benzothiazolesulfenamides were detected by comparing the spectra of untreated rubber extracts with those treated with NH_3 and hydrogen, respectively (*96*). Sulfur was identified by comparing the spectra of extracts treated with $NaBH_4$ and NH_3.

Examination of the fracture surface of compounded, unvulcanized rubber stocks by electron microscopy provides information on distinctive crystal habits of major compounding materials. The method was used to identify benzothiazyl disulfide, cyclohexyl benzothiazyl sulfenamide, and sulfur in SBR (*120*).

Mercaptobenzothiazole was determined by titrating the extract of a vulcanizate or an uncured rubber mix with iodine or $AgNO_3$ to an amperometric end point (*61*). Titration with $AgNO_3$ was conducted by adding an excess to the extract and back-titrating the unreacted silver with a solution of NaCl. This prevented the adsorption of mercaptobenzothiazole on the precipitate.

STABILIZERS

Identification. The superiority of separating an extract before attempting to identify stabilizers was pointed out by Fiorenza, Bonomi, and Piacentini (*48*). The additional datum of an *R* value from a chromatographic separation plus the availability of a relatively pure sample makes the effort worthwhile. The high absorptivities in the ultraviolet region make possible the examination of small amounts of material. This is an advantage over infrared in stabilizer identification. *p* - Phenylenediamine and several of its alkyl and aryl derivatives were identified by paper chromatography of extracts (*71*).

Fractions eluted from alumina or silica gel columns were examined by fluorescence under ultraviolet irradiation or chemical tests for identification (*37*). Paper electrophoresis was used to separate the azo dyes formed by treating the acetone extract of a vulcanizate with diazotized sulfanilic acid (*125, 126*). Aldehyde–amine accelerators interfere. Some phenolic stabilizers are not separated by this method. The visible spectra of 23 stabilizers treated with 3-methyl-2-benzothiazolone hydrazone hydrochloride and $FeCl_3$ were published for use in identification (*87*). Paper chromatography is also helpful, although many of the derivatives have the same R_f value (*88*).

If the stabilizers expected can be restricted to a limited number as in raw SBR, rapid color tests can be used. *N*-Phenyl-2-naphthylamine, acetone–diphenylamine condensation product, styrenated phenol, alkylated diphenylamine, and tris(nonylphenyl) phosphite can be distinguished in 15 minutes (*113*). Color tests are not very selective and the results should not be considered infallible.

New stabilizers are appearing constantly and, for positive identification, the unknown must be isolated and compared with a known sample, preferably by more than one physical property. Color tests for *N*-phenyl-2-naphthylamine; poly(2,2,4-trimethyl-1,2-dihydroquinoline); *N*-cyclohexyl-*N′*-phenyl-*p*-phenylenediamine; mercaptobenzoimidazole; styrenated phenol; nonylated cresol; and acetone–diphenylamine condensation product were also reported (*134*).

Ultraviolet spectra of extracts were also used for identification (*132*). Again, the lack of preliminary separation can result in missing constituents of mixtures. Identification of stabilizers in oil-extended rubber without separation from the oil is out of the question (*32*).

Determination. Fluorescence and phosphorescence were used to determine 0.01% *N*-phenyl-2-naphthylamine; poly(2,2,4-trimethyl-1,2-dihydroquinoline); and 2,2′-dimethyl-5,5′-di-*t*-butyl-4,4′-dihydroxy-disulfide in cements containing 5% polymer (*41*). This method is also applicable to thin films. It is rapid, for no extraction is needed.

N,N′-Diphenyl-*p*-phenylenediamine and *N*-phenyl-2-naphthylamine were determined in the presence of each other by the visible spectra of their 3-methyl-2-benzothiazolone hydrazone hydrochlorides (*87*). The derivatives made by coupling stabilizers with diazotized *p*-nitroaniline were measured colorimetrically (*54*). This method is satisfactory for determination if the identity of the stabilizer is known, but the spectra are not specific enough for identification.

Coupling with the diazonium salt of sulfanilic acid was also used to form colored products (*162*). Tris(nonylphenyl) phosphite was determined by hydrolysis and measurement at 296 mμ in alkaline and neutral media (*131*). This is similar to the method of Nawakowski (*108*).

Free radicals generated from 2,4,6-tri-*t*-butyl phenol were used to titrate 2,6-di-*t*-butyl-*α*-dimethylamino-*p*-cresol and 2,3-dimethyl tetramethylene-4′,4′′-dipyrocatechol (*111*). This method should be generally applicable to determination of stabilizers.

FREE CARBON

A factor was determined to correct the error caused by the presence of residual ash, carbonization of the polymer, and dry distillation of organic matter in the combustion method, eliminating the need for concurrent use of a standard sample (*145*). Carbon black in vulcanized IIR was determined by decomposing the rubber, extracting the polymer, drying and igniting the residue, and weighing before and after ignition (*19*). Quantitative determination of carbon black in rubber was also done by measuring the luminescence intensity of a crude rubber mixture as a function of its carbon black content (*114*).

A review was presented on published information about quantitative determination of carbon black in vulcanized and unvulcanized rubber stocks which included a discussion of the most commonly used methods—i.e., pyrolysis, nitric acid, high-boiling solvents, and *p*-dichlorobenzene (*49*).

METALS

EDTA Titration. The convenience of EDTA [(ethylenedinitrilotraacetic acid)] titration combined with preliminary separation has continued to produce improved methods for metals. This is exemplified by the work of Blenkin, who devised a scheme for Ca, Mg, Zn present as ZnO, Zn present as ZnS, Fe, Al, and Ba, all by titration with EDTA (*17*). The sample was first ashed, at a temperature lower than 600° C. to avoid conversion of ZnS to ZnO.

The ash was treated with *N* acetic acid to dissolve out Ca and Mg not present as silicates, and Zn present as ZnO. One aliquot was titrated at pH 6.8 for Zn present as ZnO, then the pH was increased to titrate the sum of Ca and Mg. Another aliquot was titrated at pH 11.5 for Ca only. Mg was calculated by difference. The residue insoluble in *N* acetic acid was treated with H_2O_2 to dissolve ZnS and convert it to $ZnSO_4$. The Zn in this portion was then titrated at pH 10.

The remaining insoluble residue was fused in NaOH and the fusion mass was dissolved in HCl. In one aliquot, Fe was titrated at pH 4.5. Excess EDTA was then added and back-titrated with Al to measure Al. Other aliquots of this solution were then titrated as before to find the Ca and Mg which didn't dissolve in *N* acetic acid because they were present as silicates. Fe and Al were masked with triethanolamine in the Ca plus Mg and Ca titrations.

A fresh portion of ash was dissolved in concentrated H_2SO_4 and poured into water to precipitate Ba as $BaSO_4$. The precipitate was dissolved in excess ammonium salt of EDTA and the excess was back-titrated with Mg at pH 10. Care must be exercised in this method, for success is based on selective dissolution of various groups of materials from the ash. Occlusion could cause some materials to be missed or to appear in the wrong place.

In another approach the ash is treated with hot 1:1 HCl to dissolve Al, Fe, Ca, Mg, and Zn (*153*). Al and Fe are precipitated with NH_4OH. The filtrate is made 2*N* in HCl to put Zn into the tetrachlorozincate anion. The solution is then passed through a polyamine anion exchange column where Zn is retained while Ca and Mg pass through. Ca plus Mg are titrated at pH 10 and Ca at pH 12, as above. Zn is then eluted with water and titrated at pH 10. The NH_4OH precipitate is dissolved in HCl and Fe is titrated at pH 1.0.

Excess EDTA is added to complex Al, the pH is adjusted to 4.8 to 6, and the excess EDTA is back-titrated with Fe. If much Al and Fe are present the NH_4OH precipitation is avoided to prevent loss of Zn, Ca, and Mg by coprecipitation. Then, after Zn is removed on the anion exchange column, Al and Fe are masked with triethanolamine during titration of Ca plus Mg. Ca alone is titrated in sugar solution. Al and Fe are titrated in the presence of Ca and Mg by the procedure outlined above. Ca and Mg do not interfere at the low pH.

An alternative route, after removal of the NH_4OH precipitate, is to titrate an aliquot for Zn plus Ca plus Mg at pH 10. Then Zn is precipitated as the sulfide in another aliquot (*18*). After that, Ca plus Mg and Ca are titrated as above. The NH_4OH precipitate is dissolved in HCl and titrated in strongly acid solution for Fe. Excess EDTA is added to complex Al, the pH is increased to 4.8, and the excess is back-titrated with Fe to measure Al. This method gave good agreement with the oxalate method for Ca, the magnesium ammonium phosphate method for Mg, and the ferrocyanide method for Zn.

The titration of Zn in the presence of Fe was accomplished at pH 4.5 by masking Fe with fluoride (*105*). Fe alone, Ca alone, or Mg alone did not interfere. Films of carboxylated rubber were dissolved in oleic acid and the metal attached to the carboxyl group was extracted with boiling 3*N* HCl (*89*). Then Ca, Mg, Al, Cr(III), or Ba was titrated with EDTA.

Other Titrimetric Methods. The silica filler in silicone rubber can be selectively dissolved by HF, KF, and HCl at 50° C. (*163*). K_2SiF_6 is then precipitated by adding ethanol and the precipitate is titrated with 0.5*N* KOH. In another method, total silicon is precipitated as quinoline molybdosilicate, which is titrated with NaOH (*17*).

Colorimetric Methods. EPM was analyzed for vanadium by the 3,3′-diaminobenzidine method (*136*), which agreed with neutron activation analysis. Silicone polymers were treated with $HClO_4$ and HF to remove Si (*52*). Aliquots of the resulting solution were analyzed for Fe with 2,2′-bipyridine and for Al with 8-hydroxyquinoline.

The leaves of Hevea brasiliensis were digested with HNO_3, H_2SO_4, and $HClO_4$ (*103*). The pH was adjusted to 2.8 to 3.0 and the iron (III) 8-hydroxyquinolate was extracted into $CHCl_3$ leaving Al in the aqueous phase. Fe was measured at 470 mμ with accuracy of 1.0%. The pH of the aqueous solution was then increased to 5.5 and the Al 8-hydroxyquinolate was extracted into $CHCl_3$. Al was measured at 385 mμ with accuracy of 1.4%. Ti was determined by dissolving the ash in H_2SO_4 and measuring its peroxide complex colorimetrically (*17*).

Activation Analysis. This method avoids the laborious task of decomposing the organic part of the sample by ashing or wet digestion. In addition, very high sensitivity is obtained. For example, short-lived nuclides were used to determine Ti to 5 p.p.b., Al to 1 p.p.b., and Cl to 10 p.p.b. in rubber made with Ziegler catalyst (*29, 63*).

X-Ray Fluorescence. Ziegler catalyst residues were also determined by x-ray fluorescence (*137*). The precision at levels of less than 150 p.p.m. was 4 p.p.m. for Ti, 9 p.p.m. for Al, 40 p.p.m. for Cl, and 2 p.p.m. for Fe.

Emission Spectrometry. The ash of rubber samples was dissolved in HCl or HCl and HNO_3 and the acid was subjected to spark excitation (*65*). Fe was determined to ±0.01% in the range 0.01 to 0.25%, Mn to 0.008% in the range 0.008 to 0.1%, and Cu to 0.01% in the range 0.01 to 0.15%.

Polarography. The ash of the rubber sample was leached with water, and Na, K, or Li was determined polarographically using $(CH_3)_4NI$ as the supporting electrolyte (*143*).

Ultrasound Adsorption. TiO_2 was determined by exposing the sample to ultrasound and measuring the heat rise of the sample (*42*).

Gravimetry. Total silicon in silicone rubber was determined by acid digestion of the sample followed by precipitation and weighing of SiO_2 (*163*). Alternatively, the precipitate can be weighed in Pt, treated with HF, and the residue reweighed to correct for materials other than SiO_2 in the precipitate. If Ti is present, $(NH_4)_2SO_4$ must be added during digestion to prevent contamination of the precipitate by TiO_2.

HALOGENS

Two methods were developed for determining the fluorine content of polymers (*66*). In one, complexing of fluoride ion by a standard solution of aluminum was followed by potentiometric titration of the excess aluminum ion with NaF using aluminum–nichrome electrodes. In the other, HF was formed as a result of ion exchange with a strongly acid resin and titrated with alkali. The accuracy of determination by both methods is ±0.4% in the range 35–75%.

ORGANIC ACIDS

A procedure used to determine free stearic acid in sodium BR and in SBR comprises extraction of the rubber with acetone and polarographic analysis of the extract in LiCl (*121*). Relative accuracy of the method is ±8%. Determination of free methacrylic acid in butadiene–styrene–methacrylic acid terpolymer latex was conducted by subjecting the latex to polarographic analysis in an aqueous solution of $(CH_3)_4NI$ (*121*). Carboxylic acid and carboxylate groups in polymers were detected by comparing the infrared spectra of untreated films of the polymer with those exposed to vapors of NH_3 and HCl, respectively (*20*). Colorimetric determination of sodium dibutylnaphthalene sulfonate in SBR was done by extraction of the rubber with ethanol or $CHCl_3$ and formation of color with methylene blue (*78*).

WATER

Moisture content of polycarbonates, polyesters, and polyurethanes was determined in a vacuum system by condensing the moisture and volatiles in a liquid nitrogen trap, re-evaporating, and measuring the pressure. Water was removed by absorption in CaH_2, the pressure was remeasured, and water was calculated from the drop in pressure (*36*).

EXTRACTION

A variety of raw polymers and cured stocks were extracted with 18 solvents to select one satisfactory for use in the analysis of unknown elastomers (*74*). Ethanol was the most effective in preventing solubilization of the polymers and in simplifying analysis of the extract. Extraction of plasticizers, accelerators, free sulfur, and stearic acid from IIR was carried out with an azeotropic 3:1 mixture of methyl ethyl ketone and ethanol (*50*).

GEL

The gel content of BR, SBR, IR, and NR was determined by dissolving the polymer in benzene, separating the gel from the solution by centrifugation, and weighing the solids isolated from the supernatant liquid (*123*).

LATEX

Latex was treated with monofunctional carbonyl reagents, such as hydroxylamine or 5,5′-dimethyl cyclohexane-1,3-dione to determine the carbonyl content (*129*).

OTHERS

Determination of oxygen in natural and synthetic rubber was carried out with accuracy of 2.8% by nuclear activation (*28*).

Phenyl–aniline–formaldehyde resin in reinforced SBR was determined by extracting the free resin with acetone and weighing the dried residual rubber (*82*).

Esters were detected by obtaining spectra of a polymer film before and after treatment with KOH in methanol (*20*).

LITERATURE CITED

(1) Am. Soc. for Testing and Materials, "1964 Book of ASTM Standards, Part 28," Philadelphia, 1964.
(2) *Ibid.*, p. 717.
(3) Anderson, H. C., *J. Polymer Sci., Pt. B* **2**, 115 (1964).
(4) Bacskai, R., Lapporte, S. J., *Ibid., Pt. A* **1**, 2225 (1963).
(5) Barlow, A., Lehrle, R. S., Robb, J. C., "Tech. of Polymer Sci., No. 17," p. 267, Gordon and Breach Science Publishers, Inc., New York, 1963.
(6) Bawn, C. E. H., *Ibid.*, p. 251.
(7) Bekkedahl, N., ANAL. CHEM. **22**, 253 (1950).
(8) *Ibid.*, **23**, 243 (1951).
(9) *Ibid.*, **24**, 279 (1952).
(10) *Ibid.*, **25**, 54 (1953).
(11) Bekkedahl, N., Stiehler, R. D., *Ibid.*, **21**, 266 (1949).
(12) Bekkedahl, N., Tryon, M., *Ibid.*, **27**, 589 (1955).
(13) Berger, M., Buckley, D. J., *J. Polymer Sci., Pt. A* **1**, 2945 (1963); *Rubber Chem. Technol.* **37**, 169 (1964).
(14) Binder, J. L., *J. Polymer Sci., Pt. A* **1**, 37 (1963).
(15) *Ibid.*, p. 47.
(16) Binder, J. L., Ransaw, H. C., ANAL. CHEM. **29**, 503 (1957).
(17) Blenkin, J., *Trans. Inst. Rubber Ind.* **40**, T123 (1964).
(18) Bogina, L. L., Martyukhina, I. P., *Kauchuk i Rezina* **20** (10), 34 (1961); *Soviet Rubber Technol.* (*Eng. transl.*) **20** (10), 27 (1961).
(19) *Ibid.*, **22** (12), 50 (1963); *C.A.* **60**, 9447*f* (1964).
(20) Brako, F. D., Wexler, A. S., ANAL. CHEM. **35**, 1944 (1963).
(21) Braun, D., *Farbe Lack* **69**, 820 (1963); *C.A.* **60**, 6929*d* (1964).
(22) Brown, J. E., Tryon, M., Mandel, J., ANAL. CHEM. **35**, 2172 (1963).
(23) Bucci, G., Simonazzi, T., *Chim Ind.* (*Milan*) **44**, 262 (1962).
(24) Bucci, G., Simonazzi, T., *J. Polymer Sci., Pt. C* **1964** (7), p. 203.
(25) Chakravarty, S. M., Banerjee, D., Sircar, A. K., *Trans. Inst. Rubber Ind.* **38**, T226 (1962).
(26) Chen, H. Y., ANAL. CHEM. **34**, 1134 (1962).
(27) *Ibid.*, p. 1793.
(28) Chepel, L. V., Chapyzhnikov, B. A., Viting, B. I., *Zh. Analit. Khim.* **18**, 865 (1963); *CA* **59**, 9326*e* (1963).
(29) Chinaglia, B., Ciuffolotti, L., Fasolo, G. B., Malvano, R., *Energia Nucl.* (*Milan*) **9**, 503 (1962); *C.A.* **58**, 1901*a* (1962).
(30) Ciampelli, F., Manovicia, I., *Gazz. Chim. Ital.* **91**, 1045 (1961); *C.A.* **57**, 998*g* (1962).
(31) Ciampelli, F., Morero, D., Cambini, M., *Makromol. Chem.* **61**, 250 (1963).
(32) Corish, P. J., *J. Appl. Polymer Sci.* **7**, 727 (1963).
(33) Corish, P. J., Tunnicliffe, M. E., *J. Polymer Sci., Pt. C* **1964** (7), p. 187.
(34) Cox, B. C., Ellis, B., ANAL. CHEM. **36**, 90 (1964).
(35) Crompton, T., *J. Polymer Sci., Pt. A* **1**, 347 (1963).
(36) David, D. J., Baumann, G. F., Steingiser, S., *SPE* (*Soc. Plastics Engrs.*) *Trans.* **2**, 231 (1962).
(37) Dobrovol'skaya, N. M., Provorov, V. N., Taradai, E. P., *Tr. Komis. po*

Analit. Khim. Akad. Nauk SSSR, Inst. Geokhim. i Analit. Khim. **13,** 191 (1963); *CA* **59,** 7737*h* (1963).

(38) Dogadkin, B. A., Pavlov, N. N., *Rubber Chem. Technol.* **36,** 262 (1963).

(39) Drugov, Y. S., *Kauchuk i Rezina* **23,** (1), 51 (1964); *C.A.* **60,** 13417*e* (1964).

(40) Drushel, H. V., Iddings, F. A., Anal. Chem. **35,** 28 (1963).

(41) Drushel, H. V., Sommers, A. L., *Ibid.*, **36,** 836 (1964).

(42) Faerman, V. T., USSR Patent **160,-359** (Jan. 16, 1964); *C.A.* **61,** 5806*h* (1964).

(43) Ferguson, R. C., Anal. Chem. **36,** 2204 (1964).

(44) Feuerberg, H., Gross, D., Zimmer, H., *Kautschuk Gummi* **16,** 199 (1963).

(45) Feuerberg, H., Weigel, H., *Z. Anal. Chem.* **199,** 121 (1964); *C.A.* **60,** 5641*c* (1964).

(46) Fiorenza, A., Bonomi, G., *Rass. Chim.* **15** (5), 197 (1963); *C.A.* **60,** 9447*d* (1964).

(47) Fiorenza, A., Bonomi, G., *Rubber Chem. Technol.* **36,** 1129 (1963).

(48) Fiorenza, A., Bonomi, G., Piacentini, R., *Ibid.*, p. 1119.

(49) Fiorenza, A., Rizzardini, L., *Ind. Gomma* **7** (4), 23 (1963).

(50) *Ibid.*, (11), 35 (1963).

(51) Foster, G. N., Row, S. B., Griskey, R. G., *J. Appl. Polymer Sci.* **8,** 1357 (1964).

(52) Fujiwara, S., Narasaki, H., Anal. Chem. **36,** 206 (1964).

(53) Ganguli, K. K., Chakravarti, A. P., Balakrishna, K. J., *J. Sci. Ind. Res. (India)* **21 D** (2), 58 (1962); *C.A.* **57,** 2384*h* (1962).

(54) Ghosh, A. K., Sircar, A. K., *J. Indian Chem. Soc.* **39,** 60 (1962).

(55) Giannini, U., Cambini, M., Cassata, A., *Makromol. Chem.* **61,** 246 (1963).

(56) Giuffre, L., Cassani, F., *Chim. Ind. (Milan)* **45,** 806 (1963); *C.A.* **59,** 7733*b* (1963).

(57) Golub, M. A., Fuqua, S. A., Bhacca, N. S., *J. Am. Chem. Soc.* **84,** 4981 (1962).

(58) Golub, M. A., Heller, J., *Can. J. Chem.* **41,** 937 (1963).

(59) Golub, M. A., Heller, J., *J. Polymer Sci., Pt. B* **2,** 723 (1964).

(60) Goodyear Tire and Rubber Co., *Rubber World* **146** (4), 66 (1962).

(61) Gordon, B. E., Melamed, E. A., Belova, N. A., *Kauchuk i Rezina* **21** (8), 53 (1962); *Soviet Rubber Technol. (Eng. transl.)* **21** (8), 46 (1962).

(62) Groten, B., Anal. Chem. **36,** 1206 (1964).

(63) Guinn, V. P., *Prod. Use Short-Lived Radioisotopes Reactors, Proc. Seminar, Vienna, 1962* **2,** 3 (1963); *C.A.* **59,** 6972*g* (1963).

(64) Guiochon, G., Henniker, J., *Brit. Plastics* **37** (2), 74 (1964).

(65) Gulimov, V. N., Martyukhina, I. P., *Kauchuk i Rezina* **22** (10), 54, (1963); *C.A.* **60,** 5715*e* (1964).

(66) Gurvich, D. B., Balandina, V. A., *Soviet Plastics (Eng. transl.)* **1962** (6) p. 49.

(67) Hagemeyer, H. J., Jr., *Mod. Plastics* **39** (10), 157 (1962).

(68) Hagemeyer, H. J., Jr., Edwards, M. B., *J. Polymer Sci., Pt. C* **1963** (4) p. 731.

(69) Happ, G. P., Maier, D. P., Anal. Chem. **36,** 1678 (1964).

(70) Harris, R. L., *Bull. Parenteral Drug Assoc.* **17** (4), 1 (1963); *C.A.* **60,** 5719*d* (1964).

(71) Herr, I., *Plaste Kautschuk* **10,** 98 (1963).

(72) *Ibid.*, p. 737.

(73) Higgins, G. M. C., Turner, D. T., *J. Polymer Sci., Pt. A* **2,** 1713 (1964).

(74) Hilton, C. L., *Rubber Age (N.Y.)* **91,** 968 (1962).

(75) Hoffman, M., *Makromol. Chem.* **57,** 96 (1962).

(76) Hofmann, W., *Rubber Chem. Technol. Pt. 2* **37** (2) 1 (1964).

(77) Hulot, H., Lebel, P., *Rev. Gen. Caoutchouc* **40,** 969 (1963); *Rubber Chem. Technol. (Eng. transl.)* **37,** 297 (1964).

(78) Isakova, N. A., Rakhmanina, A. M., Orlova, Z. N., *Kauchuk i Rezina* **21** (4), 48 (1962); *Soviet Rubber Technol. (Eng. transl.)* **21** (4), 42 (1962).

(79) Kastorskii, L. P., Mednikova, I. N., *Ibid.*, **22** (3), 55 (1963); *Soviet Rubber Technol. (Eng. transl.)* **22** (3), 46 (1963).

(80) Ke, B., *J. Polymer Sci.* **61,** 47 (1962).

(81) Ke, B., "Newer Methods of Polymer Characterization," Interscience, New York, 1964.

(82) Khoroshaya, E. S., Kovrigina, G. I., Dinzburg, B. N., Safrai, B. A., *Soviet Plastics (Eng. transl.)* **1962** (2), p. 60.

(83) Khoroshaya, E. S., Kovrigina, G. I., Narinskaya, A. P., Pisarenko, A. P., *Kauchuk i Rezina* **20** (12), 40 (1961); *Soviet Rubber Technol. (Eng. transl.)* **20** (12), 39 (1961).

(84) Kimmer, W., Schmalz, E. O., *Kautschuk Gummi* **16,** 606 (1963).

(85) Kossler, I., Vodehnal, J., *J. Polymer Sci., Pt. B* **1,** 415 (1963).

(86) Kress, K. E., Anal. Chem. **30,** 287 (1958).

(87) Kubota, T., Kuribayashi, S., Furuhama, T., *Nippon Gomu Kyokaishi* **35,** 662 (1962); *C.A.* **58,** 14265*h* (1963).

(88) *Ibid.*, p. 669; *C.A.* **58,** 14266*a* (1963).

(89) Kuznetsov, A. R., Arbuzov, G. A., Ezhova, T. I., *Kauchuk i Rezina* **22** (5), 51 (1963); *Soviet Rubber Technol. (Engl. Transl.)* **22** (5), 36 (1963).

(90) Lazarescu, I., Jilavu, M., *Ind. Usoara (Bucharest)* **10** (3), 101 (1963); *C.A.* **59,** 12991*d* (1963).

(91) Lee, D. F., Scanlan, J., Watson, W. F., *Proc. Roy. Soc. (London) Ser. A* **273,** 345 (1963).

(92) Lee, T. S., Kolthoff, I. M., Mairs, M. A., *J. Polymer Sci.* **3,** 66 (1948).

(93) Leonova, N. I., Tikhomirov, B. I., Yakubchik, A. I., *Polymer Sci. USSR (Eng. transl.)* **4,** 953 (1963).

(94) Lerner, M., Gilbert, R. C., Anal. Chem. **36,** 1382 (1964).

(95) Linnig, F. J., Tryon, M., Parks, E. J., *Ibid.*, **33,** 127*R* (1961).

(96) Lloyd, D. G., *Photoelec. Spectrometry Group Bull.* **1962** (14), p. 395; *C.A.* **58,** 5863*b* (1962).

(97) Lomonte, J. N., *J. Polymer Sci., Pt. B* **1,** 645 (1963).

(98) Lomonte, J. N., Tirpak, G. A., *J. Polymer Sci., Pt. A* **2,** 705 (1964).

(99) McNeill, I. C., *Polymer* **4,** 15 (1963).

(100) Maillard, A., Deluzarche, A., Dole Robbe, J. P., *Bull. Soc. Chim. France* **1963,** p. 549; *C.A.* **59,** 4143*h* (1963).

(101) Manley, T. R., "Tech. of Polymer Sci., No. 17," p. 175, Gordon and Breach, Science Publishers, Inc., New York, 1963.

(102) Middleton, K. R., *Analyst* **88,** 368 (1963).

(103) *Ibid.*, **89,** 421 (1964).

(104) Middleton, K. R., Westgarth, D. R., *Ibid.*, **88,** 544 (1963).

(105) Miksch, R., Hinteneder, L., *Kautschuk Gummi* **15,** WT 358 (1962).

(106) Miller, G. H., Larson, V. R., Pritchard, G. O., *J. Polymer Sci.* **61,** 475 (1962).

(107) Natta, G., Crespi, G., Valvassori, A., Sartori, G., *Rubber Chem. Technol.* **36,** 1583 (1963).

(108) Nawakowski, A. C., Anal. Chem. **30,** 1868 (1958).

(109) Nel'son, K. V., Skripova, L. S., Kozlova, N. V., *Zavodsk. Lab.* **29,** 704 (1963); *C.A.* **59,** 8951*b* (1963).

(110) Nikitin, V. N., Volchek, B. Z., *Intern. Chem. Eng.* **2,** 486 (1962).

(111) Paris, J. P., Gorsuch, J. D., Hercules, D. M., Anal. Chem. **36,** 1332 (1964).

(112) Parks, E. J., Linnig, F. J., *Ibid.*, **35,** 160R (1963).

(113) Parks, E. J., Milliken, L. T., Linnig, F. J., *Rubber Age (N. Y.)* **92,** 257 (1962).

(114) Pedersen, H. L., *Acta Chem. Scand.* **16,** 870 (1962); *C.A.* **57,** 15305*g* (1962).

(115) Perry, S. G., *J. Gas Chromatog.* **2** (2), 54 (1964).

(116) Phillips, J. K., *Appl. Spectry.* **17,** 9 (1963).

(117) Polyakova, A. M., Korshak, V. V., Suchkova, M. D., Vdovin, V. M., Chumayevskii, N. A., *Polymer Sci. USSR (Eng. transl.)* **3,** 315 (1962).

(118) Popov, A., *Compt. Rend. Acad. Bulgare Sci.*, **15** (2), 139 (1962).

(119) Porri, L., Carbonaro, A., Ciampelli, F., *Makromol. Chem.* **61,** 90 (1963).

(120) Prestridge, E. B., *Rubber Age (N. Y.)* **94,** 916 (1964).

(121) Provorov, V. N., Panasova, N. I., *Kauchuk i Rezina* **22** (1), 56 (1963); *Soviet Rubber Technol. (Eng. transl.)* **22,** No. 1, 40 (1963).

(122) Provorov, V. N., Zailseva, V. D., *Vestn. Tekhn. i Ekon. Inform. Nauchn-Issled. Inst. Tekhn.-Ekon. Issled. Gos. Komiteta Sov. Min. SSSR po Khim.* **1961** (1), p. 23; *C.A.* **57,** 13941*e* (1962).

(123) Purdon, J. R., Mate, R. D., *J. Polymer Sci., Pt. B* **1,** 451 (1963).

(124) Rybicka, S. M., *J. Oil Colour Chemists' Assoc.* **47,** 475 (1964).

(125) Sawada, M., Yamaji, I., Yamashina, T., *Nippon Gomu Kyokaishi* **35,** 284 (1962); *C.A.* **58,** 4707*d* (1963).

(126) *Ibid.*, p. 291; *C.A.* **58,** 4707*f* (1963).

(127) Schmalz, E. O., Geiseler, G., *Z. Anal. Chem.* **191,** 1 (1962); *C.A.* **58,** 7007*a* (1963).

(128) Schroder, E., *Plaste Kautschuk* **10,** 25 (1963); *Rubber Chem. Technol. (Eng. transl.)* **37,** 146 (1964).

(129) Sekhar, B. C., *Rubber Chem. Technol.* **35,** 889 (1962).

(130) Senn, W. L., Jr., *Anal. Chim. Acta* **29,** 505 (1963).

(131) Serafin, D., Janik, A., Maciejowski, F., *Chem. Anal. (Warsaw)* **9** (1), 65 (1964); *C.A.* **60,** 16083*c* (1964).

(132) Setkina, O. N., Popova, A. M., Galanov, O. P., *Kauchuk i Rezina* **21** (11), 53 (1962); *Soviet Rubber Technol. (Eng. transl.)* **21** (11), 42 (1962).

(133) Shipman, J. J., Golub, M. A., *J. Polymer Sci.* **58,** 1063 (1962).

(134) Sircar, A. K., Varma, J. P., Suryanarayana, N. P., *J. Sci. Ind. Res. (India)* **21D,** 73 (1962); *C.A.* **57,** 12683*b* (1962).

(135) Slonim, I. Y., *Rus. Chem. Rev. (Eng. transl.)* **31,** 308 (1962).

(136) Smith, A. J., Anal. Chem. **36,** 944 (1964).

(137) Smith, G. D., Maute, R. L., *Ibid.*, **34,** 1733 (1962).

(138) Spasskova, A. I., *Vestn. Leningr. Univ.* **18** (22); *Ser. Fiz. i Khim.* (4), 146 (1963); *C.A.* **60,** 10893*c* (1964).

(139) Stanley, C. W., Peterson, W. R., *SPE (Soc. Plastics Engrs.) Trans.* **2,** 298 (1962).

(140) Stepien, M., Gaczynski, R., *Chem. Anal. (Warsaw)* **6,** 1045 (1961); *C.A.* **57,** 998*i* (1962).

(141) Suito, E., Arakawa, M., *Nippon Gomu Kyokaishi* **34,** 884 (1961); *C.A.* **57,** 2386*a* (1962).

(142) Sung, C., *J. Korean Chem. Soc.* **7** (2), 96 (1963); *C.A.* **61,** 5762*c* (1964).

(143) Supin, G. S., *Teoriya i Prakt. Polyarogr. Analiza, Akad. Nauk Moldavsk. SSR, Materialy Pervogo Vses. Soveshch.* **1962,** p. 348; *C.A.* **59,** 5340*f* (1963).

(144) Szatkowski, W., *J. Appl. Chem. (London)* **13**, 64 (1963).
(145) Tanaka, E., Torigoe, Y., Takemoto, T., Kinumaki, H., Oda, M., *Nippon Gomu Kyokaishi* **35**, 758 (1962); *C.A.* **59**, 4143*c* (1963).
(146) Taylor, A. F., *Talanta* **11**, 894 (1964).
(147) Tryon, M., Anal. Chem. **29**, 714 (1957).
(148) Tryon, M., Horowitz, E., Mandel, J., *J. Res. Nat. Bur. Std.* **55**, 219 (1955).
(149) Tryon, M., Linnig, F. J., Anal. Chem. **31**, 767 (1959).
(150) Ulbrechtova, V., Kresta, J., *Chem. Listy* **57**, 1282 (1963); *C.A.* **60**, 5715*g* (1964).
(151) VanSchooten, J., Mostert, S., *Polymer* **4**, 135 (1963).
(152) Visapaa, A., *Valtion Tek. Tutkimuslaitos, Tiedotus* **4** (42), (1962); *C.A.* **57**, 7447*e* (1962).
(153) Vitalskaya, N. M., Pantaeva, N. F., *Kauchuk i Rezina* **21** (6), 53 (1962); *Soviet Rubber Technol. (Eng. transl.)* **21** (6), 45 (1962).
(154) Voigt, J., *Kunststoffe* **54**, 2 (1964).
(155) Wanless, G. C., *J. Polymer Sci.* **62**, 263 (1962).
(156) Welcher, F. J., ed., "Standard Methods of Chem. Anal.," 6th ed., *Pt. B*, p. 2146, Van Nostrand, New York, 1963.
(157) Wexler, A. S., Anal. Chem. **36**, 1829 (1964).
(158) Wilkinson, L. B., Norman, C. W., Buettner, J. P., *Ibid.*, p. 1759.
(159) Wittendorfer, R. E., *Ibid.*, p. 930.
(160) Yakubchik, A. I., Shostatskaya, I. D., Shikheeva, L. V., Vlasova, V. H., *Zh. Prikl. Khim.* **35**, 876 (1962); *J. Appl. Chem. USSR (Eng. transl.)* **35**, 844 (1962).
(161) Yakubchik, A. I., Smirnova, V. K., *Ibid.*, p. 159; *J. Appl. Chem. USSR (Eng. transl.)* **35**, 141 (1962).
(162) Yamaji, I., Sawada, M. Yamashina, T., *Nippon Gomu Kyokaishi* **35**, 774 (1962); *C.A.* **59**, 4143*a* (1963).
(163) Zadorozhnaya, Z. S., Ka aseva, N. P., *Kauchuk i Rezina* **21** (6), 51 (1962); *Soviet Rubber Technol. (Eng. transl.)* **21** (6), 44 (1962).
(164) Zbinden, R., "Infrared Spectroscopy of High Polymers," Academic Press, New York, 1964.

Contribution No. 306 from the Research Division, Goodyear Tire and Rubber Co., Akron, Ohio 44316.

Solid and Gaseous Fuels

R. F. Abernethy and J. G. Walters

Bureau of Mines, U. S. Department of the Interior, Pittsburgh, Pa.

Eight previous reviews have been made in this series on testing methods used in the evaluation of solid and gaseous fuels. The period covered in this review is October 1962 to September 1964 inclusive, and follows a format similar to the last review.

SOLID FUELS

This section reports on studies on methods of sampling, analyzing, and testing coal, coke, and related materials. The greater part of the investigations reported are related to present standard methods of evaluation and are proposed to improve accuracy and reduce cost and time of analysis. Several references to investigations on nonstandard tests are given in the miscellaneous tests section. It is believed that some of these will develop into tests to measure properties of solid fuels not now covered by standard procedures.

Sampling. Visman (*9A*) reported terms and quantities easy to measure that are common to all sampling experiments.

Ghosal (*3A*) proposes a theory to show how the factors of sampling variance differ between individual and bulk sampling.

Chandra (*1A*) points out the importance of standard methods of sampling by geologists in appraising coal deposits. A procedure is specified in detail from the selection of the sampling site to the submission of the subsample to the laboratory.

Hall (*5A*) developed a method of determining the bias of samples taken mechanically from a belt conveyor. He also gave conditions necessary to take accurate samples from washed and raw coal.

Nikolaev *et al.* (*7A*) described a mechanism that positions the coal cars on the track, extracts increments from various locations of the car, and composites them in the hopper of the sample preparation unit. The preparation device crushes, mixes, and divides the sample. The entire unit can be operated partly or fully automatically.

Gorelov (*4A*) devised a multiple sample divider consisting of a square vertical tube containing several sets of riffles. Each riffle was positioned at a right angle to the one immediately above. The discarded half from each riffle was fed back into a chute leading to the storage bin. The laboratory sample was collected in a cup at the end of the tube. Test results for ash and volatile matter agreed with the values obtained by the national standard method of sampling.

Semisalov (*8A*) evaluated the arc-swinging-bucket, cut-off, and horizontally moving-bucket types of coke samplers. The first two types of samplers gave a distorted size consist increment, the first one required a structural modification, and the second one required a change of operation. The third type was judged to be of the best design, but it was concluded that the bucket should start the cut on the side of the stream containing the smaller material in order that it would act as a cushion for the larger pieces of coke.

In a preliminary study Das Gupta *et al.* (*2A*) found that in sampling run-of-oven coke a 50-pound sample was required for a proximate analysis. Ash percentages in the different size fractions showed small variations.

Later work on coke sampling by Menon, Das, and Das Gupta (*6A*) and Das Gupta *et al.* (*2A*) discuss the influence of size, location in ovens, and manner of discharge on the physical properties of the coke. An alternate bias-free method of sample collection for physical testing is proposed.

Proximate Analysis. The proximate analysis is sometimes erroneously referred to as "approximate" analysis, as if the determinations were only approximations. The proximate analysis consists of accurately determined groups consisting of moisture, ash, volatile matter, and fixed carbon. The latter item is calculated by subtracting the sum of the first 3 items from 100.

Moisture. Abernethy, Tarpley, and Drogowski (*1B*) reported the effect of a number of processing variables on the determined moisture in coal by three gravimetric methods. Small and consistently lower values for moisture were obtained when air is used in place of nitrogen in the moisture determination. Other variables such as temperature, air flow, time, and atmosphere have pronounced effects on the final result.

Bull (*3B*) and Burns and Swaine (*4B*) reported that the minimum free-space oven is acceptable under certain specified modifications for the determination of moisture in brown coals.

Goldinov, Lukhovitskii, and Srubinskaya (*14B*) modified the CaH_2 method by heating both the sample and CaH_2 to 550° to 600° C. Identical results were obtained with those by weight-loss method for hydrated salts.

Schmidt and Telling (*34B*) examined the Dörr-Wäge, xylol, and the Feuma rapid drier methods for moisture in coal. The xylol method is recommended if the required sample is available.

In the search for radiometric methods to measure moisture in coke, coal, brown coal, and peat, studies have been made using neutron activation of hydrogen by Crump (*6B*), Netz (*29B*), Cudars and Skvortsova (*7B*), and Tornau and Waechter (*35B*). Rieke and Semmler (*33B*) have proposed improvements in the density and moisture gauges based on the neutron activation.

Hoffmann (*17B*) reported on the continuous optical moisture measurement by infrared reflectance. He evaluated the several factors affecting the test. The design and operation of the infrared moisture meter Ultrax and the quick-ashing apparatus Pozetto was described by Köhling and Moser (*24B*).

The moisture content of a moving coal feed was measured by Ladner and Stacey (*27B*) by passing the coal through the sampling coil of a magnetic resonance spectrometer. A. Pande and C. S. Pande (*31B*) covered in detail moisture measurement by nuclear magnetic resonance and infrared spectrometry.

VOLATILE MATTER. The development of international test methods for coal by Technical Committee 27 of the International Organization for Standardization (ISO) has engendered much work in the evaluation of testing procedures.

Nitadori *et al.* (*30B*) made a study of a number of methods on representative samples of Japanese coals in search for the most satisfactory method. In addition to procedures, factors such as particle size, composition of crucible, type of muffle, and optimum temperature, were examined. The ISO single-crucible method with suggested modifications in muffle design, crucible stand, and technique of placing crucible in muffle is being considered for adoption.

Radmacher and Scholz (*32B*) made a very detailed comparison of the ISO and German methods for volatile matter in coal. The German standard method gave values 0.03 to 0.18% lower than the ISO method.

Grabowska (*15B*) showed graphically the effects of temperature, rate of heating, and type of coal on the composition of the volatile matter of a bituminous coal. A relationship between bituminous coal metamorphism and kinetics of volatile constituents are schematically illustrated.

Dockalova and Kessler (*11B*) used a thermogravimetric balance to make a continuous record of the weight loss of a coke in a nitrogen atmosphere. They claim a more accurate volatile matter determination with a correction for the adsorbed complexes to give a true volatile matter.

The combustible volatile matter of coal was computed by making corrections for water and carbon dioxide contents determined from the pyrolysis products by Gal and Szava (*13B*).

Kanuf and Gruson (*21B*) analyzed the gases evolved on pyrolysis at 200° C. intervals up to 1100° C. They found the maximum evolution of CO and CO_2 between 500° and 700° C. A correlation was found between the CO_2 and the total volatile components.

The aromaticity of bituminous coals was measured by the volatile matter content by Czuchajowski (*8B*).

ASH. A modification of the standard tube method for volatile matter was used to determine the ash in coal and coke by Das Gupta, Chatterjee, and and Bhowmic (*10B*). By using a 0.2-gram sample, the time of test was reduced to 20 minutes in air and 8 minutes in oxygen.

A density meter was developed by the National Coal Board, Cheltenham, England (*5B*), to measure the ash content of coal from a cleaning plant. Its operation is based on the relationship of the coal and mineral matter.

Khristov and Vorob'ev (*22B*) made a theoretical statistical analysis of data from a cleaning plant and formulated a method of computing the ash content of flotation residues from the specific gravity.

Dahn and Brunner (*9B*) used the low back scattering of electrons to measure the ash content of lignite to $\pm 2\%$. It was necessary to keep the moisture content constant and standardize with known source coal.

Jirkovsky (*20B*) preferred the reflection of β to γ rays in the rapid measurement of ash in coal. It was less dangerous and was not contingent on a uniform thickness of the sample. The test required dry coal and was completed in 5 to 6 minutes.

Dresia, Friedrich, and Vogel (*12B*) studied the application of β and γ rays to the determination of ash in coals. They found the γ-ray method better suited for coals with more than 15% ash and accurate within $\pm 1.0\%$; also the β-ray method was better for the coals with less than 15% ash and gave an accuracy of $\pm 0.5\%$.

The application of β irradiation to the rapid determination of ash in coals was discussed by Krigman (*25B*). He discusses the effect of variations in chemical composition on the determination.

Methods of evaluating the ash content of coals in motion using γ-ray absorption were examined by Hardt (*16B*), Ivanchenko and Kobyakov (*19B*), and Koch and Windelband (*23B*). The thickness of the sample layer and moisture content did not affect the test.

Buhrmann (*2B*) described an automatic x-ray analyzer that measured ash continuously as the coal moved from the washery to car-loading station.

The amount of mineral matter in coal was found by Kulishenko and Dyukanov (*26B*) to be 1.14 times the ash residue. This factor was obtained by x-ray radiations passing through a special beaker holding the sample.

Mori and Taira (*28B*) used x-rays to study the ash contents of various coals. Calibration curves were prepared for each variety of coal.

Hudec and Schiller (*18B*) constructed a device to measure the ash in coals based on the radioactive Sr^{90} and Y^{90} encased in a lead block. Geiger-Muller detectors are connected to an electric voltmeter calibrated directly in ash per cent. A precision of 2.0% is claimed.

Ultimate Analysis. CARBON AND HYDROGEN. Thürauf and Thiemann (*6C*) examined the existing methods of determining C, H, and N and proposed a method based on the work of Kozlowski and Ledochowski that has the advantage of no interfering residual gases. The CuO was reduced with CO instead of H which is dissolved by the Cu.

The N in coke was measured by Kozhevnikova, Pogrebinskaya, and Kleptsina (*5C*) by using a Kjeldahl digestion mixture of 4.5 grams of MnO_2 and 4.5 grams of a finely ground mixture of 96 grams of K_2SO_4, 15 grams of $CuSO_4 \cdot 5H_2O$, and 3 grams Se.

SULFUR. Two novel methods of determining the S in coal and coke were examined by the British Coke Research Association (*1C*). The oxygen flask and the dissolution of $BaSO_4$ with subsequent titration methods were compared with the national standard and found to be satisfactory. They are rapid and are amenable to small samples.

Elfferich and Kreulen (*3C*) recommend the combustion tube method for determining S in solid fuels. The combustion gases are passed into a 3% H_2O_2 solution. Then, 20 ml. of a Hg_2OCl_2 (20 g./l.) and a mixed indicator methylene blue and methyl red are added. The H_2SO_4 formed is titrated with NaOH or $Na_4B_2O_7$.

Gruzdeva (*4C*) improved the national standard combustion method (GOST 4339–48) after studying the effect of temperature (850° to 1200° C.) absorbing solutions and addition of catalysts consisting of SiO_2 and Fe_2O_3. The optimum temperature found was 1000° C., and the proposed catalysts singly and in mixture were ineffective.

Visapää (*7C*) measured the S in coke by x-ray emission spectrography. The standard error of the method depends solely upon the counting statistics. A determination consisting of 32,000 counts for the line and 8000 for the background gives sufficient accuracy for technical purposes.

Drekopf, Gebert, and Winzen (*2C*) point out errors due to mineral matter in the determination of O in coal pyrolized in pure N. They suggest the removal of ash by HCl and HF treatment of the coal before pyrolysis. (Authors Note: The retention of Cl and Fl in the de-ashed coal can cause considerable interference.)

Calorific Value. The heat of combustion of coal as determined by the calorimeter is affected by the ash content. Schlattner (*1D*) conducted calorimetric and differential thermal analysis investigations to determine the extent of the interference.

Inorganic Constituents in Coal and Coal Ash. ASH ANALYSIS. A rapid method for the analysis of brown coal ash was reported by Volkhova (*44E*). The Ca was determined by titrating the oxalate with $KMnO_4$, Mg with Trilon B, and Al and Ti with $Zn(C_2H_3O_2)_2$. The Si had previously been removed by precipitating with gelatin.

Mineva (*34E*) and Gardelka (*19E*) used the complexometric titration method for determining Ca and Mg in coal ashes and slags.

Silica was determined by Laske (*31E*) using gelatin to precipitate the Si from the acid solution of the alkali fusion of the ash.

Ahmed and Kahn (*2E*) describe a chromatographic method of analysis of coal ash. They recommend a new solvent mixture consisting of BuOH, EtOH, and HCl.

X-ray fluorescence spectrometry was used by Sweatman, Norrish, and Durie (*38E*) in the analysis of 12 brown coal ash samples. These samples were also analyzed by the wet method, and the results compared satisfactorily. Less than 10 minutes was required to make the line and background counts for the eight elements, Fe, Ti, Ca, K, Cl, S, Si, and Al. An improvement was made in the determination of Si by substituting a pentaerythritol crystal for the more usual ethylenediamine ditartrate crystal.

Durie, Schafer, and Swaine (*13E*) used the wet chemical, atomic absorption spectrometric, and x-ray emission spectrometric methods on a series of brown coals and coal ashes. They recommended that for a rapid and direct method of analysis for inorganic constituents a combination of the atomic absorption and x-ray emission spectrometric methods should be used.

Clark and Swaine (*9E*) gave details on estimating Si, Fe, Ca, and Mg in coal ashes from semiquantitative spectrographic data. Silica ratios calculated from these data agree well with those calculated from quantitative chemical analyses.

TRACE ELEMENTS. Abernethy and Gibson (*1E*) revised a literature review on rare or trace elements found in coal and coal ash. This review of 380 references includes some information on the distribution and mode of occurrence of 34 elements.

A review similar to the one above by Bethell (*4E*) includes nearly 200 references to more than 50 elements.

Clark and Swaine (*8E*) found that the coals of New South Wales contained some 30 trace elements in quantities similar to those of coals from other parts of the world.

Ershov (*17E*) reported a selective enrichment of rare earth elements in the Kizelovsky coals. His studies suggested the occurrence of Y, Yb, Ge, and Be in organic combination while Ga, Cu, Pb, Ni, V, Cr, Mn, and Ti appeared to be in the detrital minerals.

On the basis of semiquantitative spectral analyses Kovacs, Kralik, and Policky (*27E*) report the mode of occurrence of 18 trace elements in North Bohemian coals.

Kudelasek (*28E*) reported the concentration of trace elements depended on the stratigraphic sequence and that fusain did not contain many trace elements.

ARSENIC. Kunstmann and Bodenstein (*29E*) demonstrated the micro method for the determination of As in coal. In addition to a tabulation of As contained in representative coals, a report on the behavior of As in coal during combustion and carbonization is given.

The Gutzeit method for As was modified by Jackwerth and Kloppenburg (*22E*) by using an x-ray fluorescence spectrometer after reaction with $HgBr_2$ test paper. A statistical examination of the analytical results shows a greater accuracy and fewer sources of error than the Gutzeit method.

The oxygen combustion flask method was used by Wilson and Lewis (*45E*) to determine As. A favorable gravimetric factor is obtained with the precipitation of the As as $NH_4(UO_2)AsO_4$ and subsequent ignition to U_3O_8. Phosphorus and vanadium form similar salts and must be removed.

Tsynovnikov *et al.* (*40E*) used the British Standard and other methods for the determination of As in a series of coals varying in rank from anthracite to peat inclusive. In general, the As content of coal increases with increasing pyrite content of the coal.

BORON. A relationship between the B content and organic carbon of sediments from coal measures was shown by Eagar (*16E*).

Boron and Fl in phosphatic deposits were determined by Kunstmann *et al.* (*30E*). A description of the analysis of a deposit removed from an economizer is given.

CHLORINE. Heilpern (*21E*) conducted a study to determine the Cl balance in the products of carbonization. He reported losses in Cl in ashing coal at 700°–900° C., as well as errors in the Eschka method for Cl in coal.

GALLIUM. Gallium was found in all worked coal seams of Warwickshire and selected seams from Shopshire and Staffordshire by Dalton and Pringle (*11E*).

GERMANIUM. The number of studies on Ge in coal reported in the literature for the past 2 years exceeds by far the studies for any other element. Consequently, the references used in this review have been confined to representative reports on methods of analysis.

One of the most important operations in determining the Ge content of coal is that of ashing. Menkovskii and Aleksandrova (*32E*) made a study of the behavior of Ge during ashing. Some of the factors considered were rate of heating, mineral content, total S, and ashing temperature. The study showed that the GeS was not lost in the thermal decomposition of coal. The optimum ashing conditions were reported to be 700° to 800° C., rate of heating 3.5°/min., and time of 3.5 to 4 hours.

The Ge content of coal ashes and slags was determined spectrographically by Dvořák (*15E*), Mironyuk (*35E*), and Filchovska (*18E*) with only minor variations from each other.

Davidyuk (*12E*) described a simple and rapid polarographic method for Ge in lignite ash. The time of test was about 2 hours.

Menkovskii, Gordon, and Nurminskii (*33E*) decomposed the coal with H_2SO_4 to prevent losses incurred when ashed by combustion in a muffle. The Ge was extracted with CCl_4 and determined colorimetrically with phenylfluorone. Molybdenum was also determined by this method.

A correlation method for determining Ge in coal was developed by Kizil'shtein and Syunyakova (*25E*) from the correlation between the total Ge and total ash content of the coal.

A direct method of extracting Ge from coal was proposed by Skrivan (*37E*) using CCl_4 with free Cl. Chlorine alone at 100° C. was also successful. A detailed description of the extraction apparatus is given.

Zahradnik *et al.* (*46E*) have shown that Ge is concentrated in the vitrains and humic acids. Due to the physical properties of the vitrains it is possible to concentrate the Ge in certain fractions by preparation and leaching.

MERCURY. Vasilevskaya, Shcherbakov, and Klimenchuk (*43E*) used a chemical method of determining the Hg content of coal. The coal substance was oxidized with H_2SO_4 and solid $KMnO_4$. The Hg was then extracted from the aqueous solution with dithizone in $CHCl_3$.

In a later study Vasilevskaya and Shcherbakov (*42E*) determined the various forms of Hg in coal by selectively extracting with H_2O, H_2SO_4, saturated

Na_2S, and 0.2% solution of I_2 in 2% KI.

PHOSPHORUS. Gonzalez (*20E*) made a survey of methods used in the determination of phosphorus in coal. Included in the study were volumetric, gravimetric, alkali fusion, and H_2SO_4 extraction methods. Comparable and satisfactory results were obtained by the volumetric and gravimetric methods, but the other two were unsatisfactory. A special study indicated that Ti did not interfere with the determination of P.

A chelometric method for P in coal was studied by Bhaduri, Ghose, and Majumdar (*5E*). The P was extracted from the coal ash with $HClO_4$ and precipitated with a standard $Bi(NO_3)_3$ solution. The excess Bi was titrated with EDTA. A standard deviation of less than 0.0015% was obtained by this method.

Biswas *et al.* (*6E*) developed a rapid semimicro method for P in coal ash. A 50-mg. sample of ash was digested with HNO_3, H_2SO_4, and $HClO_4$, diluted with water, and filtered. An aliquot was prepared for the usual Mo blue color test. A standard deviation of 0.004% was obtained, and the time required for a test was approximately 90 minutes.

The oxygen combustion flask method was used by Kirk and Wilkinson (*24E*) to determine the P in coke. A 30-mg. sample of ash was mixed with an equal weight of Eschka mixture on the filter paper used to place the sample in the flask. The products of combustion were absorbed in a H_2SO_4 solution and quantitatively transferred to a volumetric flask. An aliquot was used to prepare the Mo blue color test. The method is appraised as being suitable for the routine testing of coke for P.

Cook (*10E*) proved the presence of $Ca_5F(PO_4)_3$ in coal by x-ray diffraction analysis.

SCANDIUM. A paper chromatographic separation was used by Kaplan and Ol'shevskaya (*23E*) to determine the concentration of Sc in coal ash. The Sc and Y were precipitated from solution on a Y carrier as tartrates. The Sc is separated by paper chromatography from the Y and determined colorimetrically with Alizarine S. Small quantities of Fe do not interfere, but if more than 20% Al is present it must be removed with NaOH before the separation of the Sc and Y.

SELENIUM. Kononenko (*26E*) reported the extraction of Se from coal pyrite slimes.

SODIUM. On the basis of ease and freedom from interference, the HCl extraction of Na from brown coal ashes is preferred by Durie (*14E*).

STRONTIUM. Strontium and rubidium in vitrain ashes were determined by Tupper and Loring (*41E*) by use of stabile isotope dilution and the emission spectrograph.

Belcher and Brooks (*3E*) proposed an atomic absorption spectrophotometric method for the determination of Sr in coal ashes. With the sample in solution, lanthanum was added to overcome interferences before the solution was atomized. The method is rapid and preliminary separations are not necessary.

URANIUM. A simplified and very accurate method for the recovery of U by a fluorometric method was developed by Purkayastha and Saha (*36E*). It consists of ashing the coal at 520° C., extracting the U with a concentrated solution of $Fe(NO_3)_3$ and ether. The organic matter was destroyed by HNO_3, then saturated with NH_4NO_3, followed with an ether extraction. The ether extract is evaporated to dryness and ignited with Na_2CO_3–K_2CO_3–NaF flux at 600° C. The fluorescence was measured in a fluorometer.

VANADIUM. Vanadium was estimated in coke by Buncak (*7E*) with a rotating disk electrode dipping into the V solution. The sample was prepared by dissolving in HNO_3–HCl, evaporated to dryness, and digested with HCl. The color is measured with a spectrophotometer.

ZINC. Thurauf and Assenmacher (*39E*) discussed a photometric method of determining Zn in coal. Zinc forms with dithizone a red complex dithizonate soluble in $CHCl_3$ and is measured with a photometer. Cu, Ni, Co, Mn, Bi, and Pb interfere if they are present in ratios of more than 10:1 Zn.

Laboratory Coking Tests. PLASTICITY TESTS. An automatic method for determining the interval of the plastic state of coals was proposed by Bogoslovskii, Kudryashov, and Makarov (*1F*) based on the measurement of the resistance of a heated layer of coal to the passage of an inert gas. The inlet presure of the N was measured by a manometer that transmitted changes in pressure to a recorder. Optimum operating conditions were: rate of heating 13° C./min., 0.5-gram sample, and particle size of sample, 0 to 1 mm.

Gryaznov and Komarovskaya (*2F*) reported a method of determining the plasticity and apparent viscosity of coals during pyrolysis. A rotating viscometer consisted of two vertical coaxial cylinders of radius 1.10 and 1.25 cm. Three grams of 0- by 1.5-mm. coal were used.

The thickness of the plastic layer of coal was determined in an enlarged oven by Pyzhov (*10F*). An oven 400 mm. wide, 310 mm. high, and 285 mm. long holding 16.7 kg. of coal was used. Through a tube in the side of the oven, a 4-mm. probe was used to determine the thickness of the plastic layer every 40 minutes. When the temperature of the center of the charge reached 950° C., the coke was removed and used for drop shatter and tumbler tests. Test results indicated the strongest cokes were made with coals having an average thickness of 14 to 17 mm.

Haque *et al.* (*3F*) used the standard Sapozhnikov apparatus to measure the effect of rank, oxidation, and inert additives on the maximum thickness of the plastic layer of coal.

The Sapozhnikov plastometer test method was modified by Shapiro and Alterman (*12F*) by increasing the depth of the cup to 220 mm. They recommend two additional parameters in the evaluation of the caking capacity of coal. The maximum temperature drop across the plastic layer and the thermal stability of the plastic mass should be considered. It is claimed that the two additional parameters provided a clearer demarcation between coking coals.

Shapiro, Al'terman, and Keitel'gisser (*13F*) correlated the particle size of the sample with the thickness of the plastic layer, temperature of intensive decomposition, and the temperature drop in the plastic mass.

The test results of Kreulen-van Selms (*6F*), Pichler and Lauer (*9F*), and Schuhknecht and Schinkel (*11F*) show that a decrease in particle size gives an increase in contraction measured with the dilatometer.

Sheikhet *et al.* (*14F*) developed an apparatus to provide data on additional carbonizing characteristics of coals. It consists of an electrically heated steel block surrounded with insulating material. The sample is placed in tubes in vertical holes in the block. Counterbalanced rods resting on the sample register on a drum movement during swelling. It is claimed that a more detailed differentiation between coals is made.

A new coal viscometer is reported by Stephens (*15F*). It consists of concentric cylinders so arranged to eliminate end effects, and the shearing velocity gradient is nearly constant. The measuring annulus is of sufficient height to accommodate highly swelling vitrains. Slip at the surface of either cylinder is eliminated by avoiding surfaces cooler than the coal. The torque required to provide the speed of rotation is measured on a torque meter head consisting of a calibrated spiral spring. The test uses an inert atmosphere.

The softening of coals of different rank was determined by Juranek, Bussmann, and Seifert (*5F*). The temperature at which softening begins is determined by microscopic examination of the coke, and the resolidification temperature is found by reflectance measurements. The relation between these characteristic temperatures to coal rank and coking temperature is discussed. The testing of mixtures of coals of wide variation in rank shows that during coking each coal keeps its individual properties and real fusion never is attained.

Wuerker, Singh, and Chakraverty (*17F*) discuss the Audibert-Arnu dilatometer test as applied to the international classification of coals. A section of the report deals with the use of the electric furnace in determining the free-swelling index of coal.

CARBONIZATION. Jones (*4F*) gives a 15-year review of work on the kinetics of carbonization. There is general agreement on the kinetics of the plasticity of coal, but not on the kinetics of primary gasification. Included in the review is recent work on the chemistry of carbonization as related to coal structure and secondary reactions of tar.

The heat of carbonization was determined by Loison and Foch (*7F*) and Voloshin, Virozub, and Kazmina (*16F*). The first group of workers used a Jenkner retort and a 400-kg. oven. Variations in the oven throughput were measured by determining moisture content of the charge. The latter group used an electrically heated adiabatic calorimeter.

Petrik, Sarymsakov, and Belekov (*8F*) developed an improved oven to test coals for coking properties. Scale drawings include a quenching chamber.

Standard Methods. NATIONAL STANDARDS. The nearest approach to national standards for coal and coke in this country is the compilation of specifications, methods of test, and definitions of terms relating to coal and coke by the American Society for Testing and Materials, "Gaseous Fuels, Coal and Coke" (*1G*). In the first edition (1964) of this publication, 6 of 34 standards and tentative standards were approved by the American Standards Association.

Previous ASTM publications of standards were issued at intervals of several years and were contained in a relatively few large volumes. The present system combines gaseous and solid fuels into Volume 19 which is issued annually. Another basic change in ASTM operation is the approval of standards. Prior to 1964, most standards were approved by the society at the time of the annual meeting. The Administrative Committee on Standards now receives recommendations four times each year.

ASTM Committee D-5 on Coal and Coke recommended to the society that revisions be made in the standard method of drop shatter test for coke (D 148) and standard specifications for classification of coals by rank (D 388). Tentative standards on volatile matter in coal (D 271), tumbler test for coke

Table I. Status of Work of ISO Technical Committee 27

1.	ISO RECOMMENDATIONS PUBLISHED
R157-1960	Determination of forms of sulfur in coal
R158-1960	Determination of ash in hard coal
R159-1960	Determination of total sulfur in coal by the Strambi method
2.	ISO RECOMMENDATIONS APPROVED BY ISO COUNCIL FOR PUBLICATION
R331-1963	Determination of moisture in the analysis sample of coal by the direct gravimetric method
R332-1963	Determination of nitrogen in coal by the Kjeldahl method
R333-1963	Determination of nitrogen in coal by the semimicro Kjeldahl method
R334-1963	Determination of total sulfur in coal by the Eschka method
R335-1963	Determination of caking power of coal by the Roga method
R348-1963	Determination of moisture in the analysis sample of coal by the direct volumetric method
R349-1963	Audibert-Arnu dilatometer test for coal
R350-1963	Determination of chlorine in coal by the bomb-combustion method
R351-1963	Determination of total sulfur in coal by the high temperature combustion method
R352-1963	Determination of chlorine in coal by the high temperature combustion method
3.	DRAFT ISO RECOMMENDATIONS BEING EDITED FOR SUBMISSION TO ISO COUNCIL
DR236	Determination of carbon and hydrogen in coal by the Liebig method
DR550	Determination of the volatile matter of hard coal and of coke
DR551	Determination of the crucible swelling of coal
DR552	Determination of the Gray-King coke type of coal
DR553	Determination of total moisture in hard coal
DR569	Determination of the gross calorific value of coal by the calorimetric bomb method
DR570	Determination of moisture-holding capacity of hard coal
DR571	Determination of micum indices of coke
DR572	Determination of the shatter indices of coke
DR573	Determination of the fusibility of fuel ash
DR604	Graphical symbols for coal preparation plant
DR605	Determination of carbon and hydrogen in coal by the Sheffield high-temperature method
DR606	Determination of phosphorus in ash from hard coal
4.	DRAFT ISO RECOMMENDATIONS CIRCULATED TO ALL ISO MEMBER BODIES BY ISO GENERAL SECRETARIAT FOR POSTAL BALLOT
DR677	Determination of chlorine in coal by the Eschka method
DR678	Determination of arsenic in coal and coke
DR679	Determination of mineral matter in coal
DR680	Determination of ash of coke
DR681	Determination of total moisture in coke
DR682	Determination of moisture in the general analysis sample of coke
DR683	Size analysis of coke
DR684	Determination of the bulk density of coke in a small container
5.	DRAFT PROPOSALS BEING EDITED FOR TRANSMISSION TO ISO GENERAL SECRETARIAT AFTER POSTAL BALLOT IN ISO/TC 27
N678	Glossary of coal sampling terms
N711	Determination of carbon dioxide in coal by the pressometric method
N713	Determination of the yields of tar, water, gas, and coke by low temperature distillation of brown coals and lignites
N730	Sampling of hard coal
6.	DRAFT PROPOSALS SUBMITTED FOR POSTAL BALLOT IN ISO/TC 27
N733	Terminology for coal preparation
N736	Expression and presentation of results of coal cleaning tests
N739	Principles and conventions for flowsheets for coal preparation plant
N773	Determination of the gross calorific value of coal by the adiabatic bomb calorimeter
N778	Determination of carbon dioxide in coal by the gravimetric method
N780	Determination of the benzene soluble extract in brown coals and lignites
N784	Modification of coal analysis methods for the analysis of coke
7.	DRAFT PROPOSALS BEING EDITED FOR POSTAL BALLOT IN ISO/TC 27
SC2	Determination of moisture by the direct volumetric method in brown coals and lignites
WG2	Determination of the moisture-holding capacity of hard coal—rapid methods
WG8	Sampling of coke
WG8	Determination of bulk density of coke in a large container
WG8	Determination of apparent specific gravity of coke
WG8	Determination of the true specific gravity of coke
8.	DOCUMENTS SUBMITTED TO ISO/TC 27 FOR COMMENT
N776	Determination of the Hardgrove grindability index of coal
N786	Size analysis of coal
9.	OTHER ITEMS UNDER INVESTIGATION
SC1	Float and sink tests on coal
SC2	Determination of ash of brown coals and lignites
SC2	Sampling of brown coal
WG8	Glossary of coke terms
WG9	Direct determination of oxygen in hard coal

(D 294), and expansion or contraction of coal in the sole-heated oven (D 2014) were recommended for advancement to standard tests.

INTERNATIONAL STANDARDS. Technical Committee 27 on Solid Mineral Fuels of ISO has been very active in the development of standards for the international trade in solid fuels. Since 1950, most of the test methods have been advanced toward standards and only a few remain for action. No recommendations have been approved as ISO standards, however, a number have reached the next level of advancement of ISO recommendation.

Table I gives the status of the work through June 1964.

Miscellaneous. The determination of mineral carbonates in coal is required in a number of applications of coal analysis. Burns, Durie, and Swaine (*2H*) have shown that the generally accepted method of test is not applicable to brown coals and lignite. A rather simple method has been proposed for these fuels. Leithe (*10H*) used the normal method of evolution with acid with a modified titration finish. Butyl alcohol was added to the alkali to cause foaming and accelerate the rate of absorption of the CO_2. The excess alkali was titrated with standard HCl. Pringle (*14H*) used benzylamine to absorb the CO_2. A nonaqueous titration using potassium methoxide in benzene–methanol with thymol blue indicator was made to determine the CO_2.

Sarkar and Manchanda (*15H*) used an all-glass apparatus to carry out float-and-sink tests on minus $^1/_8$-inch coal with organic liquids. The use of this equipment eliminates the health hazard due to the organic liquids.

Hardgrove (*5H*) states that the relationship between the Hardgrove grindability index and pulverizer capacity is ideally a straight line provided sufficient amount of fines are removed to prevent cushioning. Sinha *et al.* (*17H*), after making many Hardgrove grindability index determinations, developed a method of calculating the HGI from relationships with volatile matter, moisture, and ash content.

During a study of several methods used to determine the moisture-holding capacity of coals, Krumin (*9H*) used the results to determine the forms of moisture in coal.

At the present time there is no generally accepted method of test for the petrographic analysis of a coal sample. The great amount of work being reported in the literature of practical application of petrographic evaluation of coals indicates with small doubt that a standard method will result. The prediction of coke strength from rank and petrographic composition of coals is reported by Brown, Taylor, and Cook (*1H*). Test data reported by Gin, Dahl, and Wilson (*4H*) show that the application of petrographic evaluations of coal blends is useful in predicting the quality of coke produced. Harrison (*6H*) used petrographic analysis in the control of a preparation plant to produce coal of definite specifications. Reflectance measurements were used by Murchison (*11H*) and Ponomareva, Lifshits, and Vyrich (*12H*) in the differentiation between various ranks and classes of coal.

Wolfe (*20H*) reported that the standard ash pyramids used in the fusibility of ash determination failed for certain brown coal ashes. Satisfactory results were obtained by molding a small cylinder around a platinum wire which was used to suspend the cylinder in the heating zone of the furnace.

Kijewska (*8H*) reported true densities of coals, cokes, and semicokes as determined by pycnometer using EtOH, MeOH, C_6H_6, H_2O, gasoline, tetralin, and xylene. Jones (*7H*) reported a microscopic method of determining the density of coal masses of <10 mg. with an accuracy of $\pm 0.05\%$. The sample was suspended in a liquid of graded density within a hollow prism.

The apparent density of coke was determined by Seichter and Gruson (*16H*) by drying at 125° C., cooling, and immersing the weighed sample in paraffin before measuring its volume in a Seger meter.

The form in which S occurs in coal is of great interest to those engaged in coke making, gasification, preparation, and classification. Edwards, Jones, and Newcombe (*3H*) reported that the nitric acid penetration of the coal was dependent on rank and that many coals of higher rank were not cleanly extracted. These coals had to be pulverized to -300 mesh to get complete pyrite extraction. Tyman (*18H*) defined volatile S as that S that goes into gaseous form on combustion or gasification of coal. The coal is burned in oxygen bomb, the liquid is removed, and the volatile S content determined.

The types of organic S in coals were studied by Wnekowska (*19H*) and Prilezhaeva *et al.* (*13H*). Extractions were made with alkaline $HgCl_2$ and $AgNO_3$.

GASEOUS FUELS

The methods reported are those which have been or can be applied specifically to blast-furnace top gas, carbureted water gas, coal gas, coke-oven gas, liquified petroleum gas, sludge gas, manufactured gas, natural gas, producer gas, synthesis gas, and water gas.

The review by Hobbs (*6J*) should be consulted for fundamental developments in gas analysis. Kienitz (*7J*) reviewed chemical and instrumental methods for determining macro, micro, and trace levels of about 100 inorganic and 100 organic substances with boiling points less than 20° C. Problems of separation, type of sample, classification and choice of method, and analysis of mixtures are discussed. Fuhrmann (*5J*) reviewed the utilization and techniques of automatic analyzers for the determination of trace impurities in gases. Current methods used in analyzing natural gas and the various methods of determining heats of combustion have been reviewed by Baranenko (*2J*).

Duchemin (*4J*) describes the utilization of spectrography, polarography, and chromatography for quality control and for the solution of the problems of the coke-oven industry. Special methods and instruments used to determine dust, moisture, and conditioning agents are also discussed. Burgess and Wood (*3J*) presented a literature review dealing with analytical methods, detection apparatuses, and properties of sludge gas.

The 43rd volume of the gas handbook has been issued, and a review summarizes (*1J*) the technical data on design, construction, maintenance, operation, materials, corrosion, utilization, measurement, and control.

Standards. The 1964 book of ASTM Standards, part 19, for Gaseous Fuels, Coal, and Coke (*1K*) lists several methods of testing and analysis that have been approved as tentative standards and several that have been revised.

A tentative method for the analysis of liquified petroleum (LP) gas by gas chromatography is presented (D 2163–63T). This method provides for the determination of C_2H_6, C_3H_8, C_3H_6, *n*-butane, isobutane, butylenes, and isopentane. Five liquid substrates and the column preparation of each is described. Another tentative standard is the analysis of natural gas by gas chromatography (D 1945–62T), in which an adsorption-type column and a partition-type column are required for a complete analysis. The analysis of reformed gas by gas chromatography is covered in tentative standard (D 1946–62T), which provides for the determination of H, O, N, CO, CO_2, CH_4, C_2H_6, and C_2H_4.

Revisions have been made in the method for determining the specific gravity of gaseous fuels (D 1070–63), the determination of the water vapor content of gaseous fuels by measurement of dew-point temperature (D 1142–63), and the lamp method for determining sulfur in petroleum products and liquefied petroleum (LP) gases (D 1266–62T).

Physical Methods. The number of investigations concerning the applicability of gas chromatography to the analysis of gaseous fuels far surpasses that for the other physical methods. These studies have not been confined to an analysis of the major constituents but have been refined so that a high degree of precision has been obtained in the determination of trace components. Fundamental developments in the field of gas chromatography have been reviewed by Juvet and Dal Nogare (*11L*).

Most of the recent investigations concerning the analysis of gaseous fuels by gas chromatography have utilized two or more columns to resolve the many components that are generally present or to shorten analysis time. Manka (*18L*) used a short silica gel column to separate CO_2; the remainder of the sample then passes through a spacer column while the CO_2 is eluted from the silica gel. A molecular sieve column is then used to resolve the other components. Kavan and Vlckova (*12L*) describe a two column apparatus in which they analyzed for H, N, CO, CH_4, C_2H_6, C_3H_8, C_4H_{10}, and C_2H_4. The columns were packed with activated carbon and Alusil. Analysis time was ~60 minutes, and 14 components were said to be resolved from Fischer-Tropsch synthesis gas. Satisfactory resolution of H, O, N, CO, CO_2, and CH_4 is claimed by Kipping and Jeffery (*13L*), using a 3-foot molecular sieve (*5A*) and an 8-foot charcoal column in series. The detector was a thermal conductivity cell, and argon was the carrier gas. A three column system was utilized by Miquel and Benard (*20L*) to resolve the permanent gases and hydrocarbons through C_5. The first was a 2-meter silica gel column containing 1.5% by weight squalene and maintained at 65° C. An activated charcoal column, 1 meter in length and kept at 100° C., was second. The third column was 15 meters long, contained tritolyl phosphate on kieselguhr, and maintained at 20° C. Columns one and three were used to determine the C_4 and C_5 hydrocarbons, and column two was necessary to obtain precise H and CH_4 determinations. Three columns were also used by Tung and Yu (*25L*) for natural gas analysis. Carbon dioxide was the carrier gas, column operating temperature was 20° C., columns were packed with silica gel and activated C, and H, N, CH_4, C_2H_6, C_3H_8, and C_4H_{10} were resolved and determined quantitatively.

A fibrillar colloidal boehmite, having a surface area of 275 m./g, with an acetate modified surface, was studied as an adsorbent for gas solid chromatography by Kirkland (*14L*). He observed efficient separation of low-boiling and permanent gases in conventional packed columns. An aqueous suspension of the boehmite was also used to coat the walls of capillary columns. Bruk *et al.* (*4L*) modified a rottenstone by the addition of 2% KOH and 8% adiponitrile and used this to pack a 584 × 0.4-cm. column. The column was operated at 40° C. and completely resolved butane and butylenes from isobutane, butadiene, and C_1–C_3 hydrocarbons in ~8 minutes. Hydrogen, nitrogen, helium, carbon dioxide, and air were all tried as carriers and gave similar results.

A method for analyzing natural, cracked, and other petroleum gases using a Janak (integral) and a differential recording chromatograph is described by Aliev and Aminova (*1L*). Satisfactory separation was obtained for the C_1–C_4 gases, but the C_5–C_6 were not completely resolved and were therefore combined. The detector is easily overloaded and was difficult to keep in operation.

Carrier gas reverse flow technique has been adapted to the analysis of gaseous fuels by Miller (*19L*) to combine the numerous hexane isomers and heavier components into a measurable group of peaks. This method is in course of adoption as a tentative standard by ASTM.

A combination of absorption and gas chromatography is presented by Sechenov *et al.* (*24L*), wherein CO, CO_2, and O are absorbed in appropriate solutions, and H, N, and CH_4 are determined chromatographically.

Galwey (*7L*) utilized a radioactive ionization detector (Lovelock) for the analysis of gases containing hydrogen, methane, carbon dioxide, carbon monoxide, and nitrogen. An 80-cm. column, packed with activated charcoal, was used, and the carrier gas was argon with a flow rate of 30 ml./min. Oxygen, sulfur dioxide, and *n*-pentane were also determined by this procedure. Studies of chemical kinetics involving permanent gases are described and a method for the removal of corrosive gases from samples is discussed. Wulfhekel and Schaal (*27L*) describe a detector based on the gas dependent changes in the arc-drop voltage (anode-cathode voltage drop during arcing time) of glow discharge. Argon is the carrier gas and separation of illuminating gas is accomplished with a 1-meter column, 4-mm i.d., packed with 5A molecular sieve, followed by a 1-meter capillary column.

A column containing AR-3 activated carbon was used by Lityaeva, *et al.* (*16L*) to determine <0.005 volume per cent CO and <0.001 volume per cent methane and acetylene. The column was maintained at 60° C. for the separation of all the components except ethylene, the column temperature was then raised to 105° C. to elute ethylene.

Colson and Brown (*6L*) discuss the speed and accuracy of gas chromatographic analysis and describe columns and operating conditions for the analysis of town gas, hydrocarbons in refinery gas, and Lurgi gas. Other uses of chromatography in the gas industry, such as the determination of odorants, gum formers, trace components, and the detection of very small gas leaks, are also discussed.

Mixtures of H, O, N, CO, and CH_4 were separated on activated carbon and molecular sieves by Turkel'taub and Porshneva (*26L*). Also described is a procedure for the rapid analysis of H, CO, CH_4, CO_2, and C_2H_2 and a study of the dependence of the degree of separation characterized by the coefficient k of low-boiling gases on the nature of the adsorbent.

An 8-foot Chromosorb P column (30- to 60-mesh), containing 25 weight per cent glyceryl triacetate, followed by a 9-foot molecular sieve column, was utilized by Isbell (*10L*) to analyze mixtures containing CO_2, HCN, $(CN)_2$, O, N, NO, CH_4, and CO.

Trace quantities of CO and CH_4 were determined by Muraosa and Iwaya (*21L*) using three columns in series. Activated carbon, activated alumina, and dioctyl phthalate on a solid support were the column packings. Determination of concentrations as low as 70 p.p.m. of CH_4 were made with the activated carbon column and 20 to 30 p.p.m. of CO with the activated alumina column.

Averill and Ettre (*3L*) obtained separation of methane, ethane, and propane on a 150-ft. (0.01-inch i.d.) capillary column coated with high vacuum silicone grease. Ethane, propane, propylene, butanes, butylenes, and C_5 hydrocarbons were determined in the effluent of an ethylene process by means of a silica gel column containing 0.5% Na_2CO_3 and 2% glycerol by Lulova *et al.* (*17L*).

Orechkin and Dantsig (*22L*) patented a procedure for determining small quantities of CO that utilized a preliminary separation of hydrocarbons on an activated carbon column at low temperatures.

Andreen and Kniebes (*2L*) utilized three columns to separate the C_2–C_4 mercaptans, C_2–C_5 sulfides, and thiophene. Liquid substrates and column lengths were β,β'-oxydipropionitrile (5 feet), *n*-hepthyl sulfide (3 feet), and 550 silicone oil (3 feet).

Colson (*5L*) analyzed for small quantities of high-boiling compounds such as 0.45 mg./l. of napthalene or 0.03 mg./l. of thiophene by drawing a measured volume of gas through a short metal column packed with a suitable absorbent and then inserting the column before the separatory column on the chromatograph. The short metal column was then heated rapidly for about 5 seconds, and the carrier gas stream carried the evolved material to the separation column.

Recommended absorbent for thiophene is 30% silicone oil on 30- to 60-mesh Chromosorb P. The short metal trap column is 12.5-cm. long and has an inside diamter of 4.1 mm. A current of 90 amperes at 1.3 volts for 5 seconds heated the center of the sample tube to 150° C.

Knipschild and Pagnier (*15L*) describe a procedure to determine small quantities of benzene, toluene, xylenes, and the higher molecular weight aliphatics in gaseous fuels. A column containing picric acid and fluorene is operated at 100° C. with He as the carrier gas. The method is said to determine as little as 0.1 gram of aromatic hydrocarbons per cubic meter of gas.

Helium and hydrogen in natural gas have been determined by Graf and Toth (*8L*) by absorption on a 70 cm. long, dry ice-acetone cooled column, connected in series with a 170 cm. long uncooled column, and then eluting with CO_2. Either an interferometer or thermal conductivity cell is used for detection. Greater than 25% N or O in the sample interferes with the analysis and this is removed by absorption in a 50% KOH solution.

Henderson (*9L*) advocates the use of gas chromatography as the quickest and most reliable method of analyzing the gases produced in sludge digestion. Analysis time is ~5 minutes. Techniques, which include the identification of chromatographic peaks by mass spectrometry, are described by Pichler *et al.* (*23L*). It is claimed that identification can be made by two mass spectra, whereas a large number of chromatograms would be necessary.

Chemical Methods. Coleman and Elkins (*5M*) have patented a gas analyzer that utilizes meters and absorbers in series to remove moisture, carbon dioxide, and other components by absorption. The sample is heated to 105° C. before analysis. Provision is made to install a thermal conductivity detector in the effluent stream to analyze for constituents not determined by absorption.

Baranenko, Krivosheeva, and Maksimenko (*1M*) describe a method for the determination of CO_2 in gases by absorption under static conditions in a standard solution of $Ba(OH)_2$. The CO_2 concentration is determined from the quantity of $Ba(OH)_2$ which reacted.

Wronkowski (*16M*) determined the CO_2 content by measuring, with a 1.0-ml. micropipet, the difference in volume of the sample after passing through a 30% KOH solution. The pipet terminates in a 100-ml. container and is connected to a bottle containing a colored NaCl solution. The results are reproducible to within 0.03% and are comparable to those from titrimetric and gravimetric methods.

Leithe (*11M*) describes a method whereby he determined CO_2 concentration as low as 7 p.p.m. in synthesis gas. The method consists of bubbling the gas, by means of a dispersion frit, into an excess of 0.01*N* NaOH which contains BuOH. After saturation with NaCl, the excess NaOH is titrated with HCl. Either azo violet or thymolphthalein may be used as an indicator.

Scholl (*13M*) reported a simple device for the continuous determination of CO_2 in flue gas. The apparatus compares the flow rates of the gas before and after the absorption of CO_2 in a suitable liquid. The decrease in the flow rate is proportional to the CO_2 content. The apparatus, operation, and calibration are described.

Grosskopf (*9M*) states that gas velocity is the main factor affecting colorimetric indicator tubes, and this has prompted him to arrange the known types into six major groups. In this manner, NH_3, CO, CO_2, HCl, SO_2, H_2S, Cl_2, NO, NO_2, Hg(v), CS_2, benzene, toluene, HCN, styrene, and hydrocarbons can all be detected and quantitatively estimated.

Charpenet (*2M*) claims that the collection of the sample over mercury in the dry state improves the conventional Orsat procedure.

Kavan and Base proposed (*10M*) a mixture containing NaHS, NaOH, pyrogallol, and Na β-anthraquinone for the oxygen absorber in the orsat apparatus. It is claimed to be more effective and even when nearly exhausted does not liberate CO.

Sulfur and Sulfur Compounds. Takens (*15M*) described a method of obtaining the concentration of total sulfur by burning the gas and passing the combustion products through a 3% H_2O_2 solution. The H_2SO_4 and HNO_3 or HNO_2 are neutralized with 0.02*N* NaOH, and the Na_2SO_4 is then titrated 0.02*N* with $BaCl_2$ in a conductimeter.

Golyand and Lazhrev (*8M*) determined COS and CS_2 spectrophotometrically after they were absorbed in 1% alcoholic diethylamine. This procedure can also be used to determine aromatic and dienic hydrocarbons or other compounds that absorb in the ultra violet region.

Clayton and Jones (*4M*) developed a gas blending procedure whereby they could prepare samples containing 0.2 to 100 p.p.m. of H_2S. Samples were then analyzed by the methylene blue method and by mass spectroscopy, and the calibration curves and reproducibility data are presented. Flow stability and reproducibility of the sampling technique permit a test reproducibility of ±5%.

Morriss and Meiners (*12M*) patented a method of determining methyl and ethyl mercaptans. The gas is bubbled through aqueous I_2 solution (0.1 gram of I_2 and 0.25 gram of KI/l.), and the pH of the resultant solution is compared with the blank to determine the quantity of mercaptan. H_2S must be removed before analysis, and this can be accomplished with granular $Pb(OAc)_2$ and K_2CO_3.

Another method, described by Stout and Early (*14M*), also requires the removal of H_2S before the mercaptan determination. A known quantity of gas is passed through a scrubber containing an aqueous solution of $AgNO_3$, to which has been added a small amount of Ag^{110}. Since the S in H_2S requires as much Ag^{110} (molar basis) as mercaptan S, all the H_2S is removed before scrubbing. Other sulfides do not interfere if a dilute $AgNO_3$ solution is used in which an aqueous solution of $AgNO_3$ is mixed with Ag^{110} in dilute HNO_3 to give 50 μg. of $AgNO_3$/ml. containing 0.05 μg. of Ag^{110}/ml. The total $AgNO_3$ should be 10 to 100 μg. with a maximum of 0.05*N* in HNO_3.

Fredericks and Harlow (*7M*) utilized a chromatographic separation and two detectors, thermal conductivity and microcoulometric, in series to effect a mercaptan determination. A 25-foot by $^1/_4$-inch column, packed with chromosorb W, containing 30% by weight of tricresyl phosphate, is used for the separation. The mercaptans are determined in the microcoulometric cell where they are automatically titrated with Ag. Hydrogen sulfide can be determined in the same analysis if it is present in approximately the same concentration as the mercaptans (500 p.p.m.). Me, Et, iso-Pr, and *n*-Pr mercaptans are resolved by this column but *sec*-Bu and iso-Bu mercaptans are not completely resolved.

A two-step procedure to determine SO_2 and SO_3 in flue gases is described by Chory (*3M*) and is said to be reproducible within 7 to 50 × 10^{-4}% by volume. Test time is approximately 15 minutes.

Carbonyls. Engelbrecht *et al.* (*6M*) have analyzed Fischer-Tropsch synthesis gas for $Fe(CO)_5$ in concentrations ranging from 0.05 to 500 mg./m³. The determination is made by adsorbing the carbonyl in a solution composed of 100 ml. of AcOH and 15 ml. of 30% H_2O_2 and then titrating with 0.05*N* $K_2Cr_2O_7$ or colorimetrically with *o*-phenanthroline.

Sampling. Chekanov (*1N*) developed an automatic gas sampler and analyzer that determines the composition and heat of combustion of the gas. The apparatus can be programmed to take average daily, hourly, or shift samples. A review of the various types of apparatuses for separating liquid drops and dust from gas samples has been compiled and evaluated by Lutoshkin (*3N*). Particle size, concentration, surface tension, and the rate, temperature, and pressure of the gas are stated to be the most significant factors affecting separation, and the effects of these variables are discussed.

Maksimov and Bushmelev (*4N*) also reviewed the factors affecting the separation and determination of moisture and particulate matter in gas samples. They also describe an apparatus for taking gas samples and present formulas for calculating moisture and dust contents.

Fickinger (*2N*) has developed a sampling procedure for flue gases whereby the sample cylinder is placed in the hot gas duct and the sample is drawn through a filter. This eliminates the problem of condensation and also removes most of the dust from the sample.

Moisture. Mikhailov (*2P*) derived an analytical function based on an analysis of experimental and theoretical data on the moisture content of natural gases, and on the basis of this function, constructed a nomogram for determining the moisture content of natural gases. The nomograph covers the temperature range, −40° C. to 110° C., and the pressure range, 1 to 300 atms.

Davis (*1P*) also has presented a nomograph for the moisture content of natural gas when the temperature and pressure are known.

Calorific Value. A commercial apparatus is described by Rosset (*2Q*), which gives a continuous measurement of the heating value of a gas and also permits adjustment of the heating value of a gas mixture to a constant value.

Krizan (*1Q*) has constructed an apparatus containing a metal block, used in place of a Junkers calorimeter, to determine the heating value of gases. The heating values are determined from a calibrated diagram, which was derived from data for known fuels.

Miscellaneous. Lille, Murd, and Kundel (*5R*) utilized frontal displacement for collecting the higher hydrocarbons present in natural gas. Gas chromatographic analysis of the first and second frontal displacement fractions indicate a predominance of C_3 hydrocarbons in the first fraction and saturated and unsaturated C_4 hydrocarbons in the second fraction.

Iijima *et al.* (*2R*) describe a procedure for determining benzene and toluene in coke-oven gas by observing the absorption in the ultraviolet region, specifically at 2537 A. A comparison of results determined by this method and by gas chromatography is presented.

A gas analyzer patented by Martin and Shields (*6R*) utilizes a spectrophotometer with a nonselective detector and a mechanically modulated beam in the ultraviolet, visible, or infrared range.

Bothe and Adler discuss (*1R*) the physical principles and development possibilities of an apparatus for the analysis of gases and vapors by nuclear radiation. The apparatus is especially suitable for the determination of electromagnetic gases such as O_2, H_2O, and halogens.

Lestienne (*4R*) has applied the classical electrochemical methods such as amperometry, differential titration, and polarography to the continuous analysis of gases that contain volatile acids, bases, carbonyl compounds, and alcohols by using an apparatus in which liquid flows slowly in a continuous thin film over a metal electrode. Response is instantaneous, and the method is claimed to be highly sensitive.

Methods for determining the cyclopentadiene content of coal gas were investigated by Kavan and Vlckova (*3R*), and they concluded that the fulvene method was most applicable.

Infrared gas analyzers, without spectral resolution, and their industrial application were reviewed by Schultz (*8R*).

Helium, argon, and carbon contents of gases were determined by Zartman *et al.* (*9R*). Helium was determined by volumetric condensation, argon by isotope dilution, and carbon by conversion to CO_2.

A diffusion method for the determination of hydrogen in gases has been developed by Ryabov (*7R*). The sample is introduced into a constant temperature chamber, maintained at 450° C., which contains a coiled evacuated Pd tube (2 by 0.2 mm.). The pressure in both the tube and chamber are recorded by means of Hg manometers for high H concentrations and ionization gauges for 10^{-10} volume % range. An accuracy of ±5% was reported.

LITERATURE CITED

SOLID FUELS

Sampling

(1A) Chandra, T. K., *J. Mines Metals Fuels* **11**, (2), 8–12 (1963).
(2A) Das Gupta, N. N., Ghosal, A., Chatterjee, N. N., Menon, M. V. P., *Ibid.*, **11**, (1), 15–18 (1963).
(3A) Ghosal, A., *J. Sci. Ind. Res.* (*India*) **21D**, 362–5 (1962).
(4A) Gorelov, P. N., Stepanov, Yu. V., *Coke Chem. USSR* **1963**, (7), 14–16.
(5A) Hall, D. A., *Colliery Eng.* **40**, 55–9 (1963).
(6A) Menon, M. V. P., Das, P. K., Das Gupta, N. N., *J. Mines Metals Fuels* **11**, (9), 1–6, 18 (1963).
(7A) Nikolaev, D. D., Iskra, G. S., Melnichenko, A. F., Makarevich, Yu. S., Starikov, A. A., Fomovskii, V. A., *Coke Chem. USSR* **1963**, (2), 5–8.
(8A) Semisalov, L. P., *Ibid.*, (6), 31–33.
(9A) Visman, J., *Dept. Min., Ottawa, Min. Branch Res. Rept.* **R93**, 39 (July 1962).

Proximate Analysis

(1B) Abernethy, R. F., Tarpley, E. C., Drogowski, R. A., *U.S. Bur. Mines Rept Invest*, **6440**, 12 (1964).
(2B) Buhrmann, H., *Control Eng.* **9**, (7), 121 (1962).
(3B) Bull, F. A., *Fuel* **42**, 203–10 (1963).
(4B) Burns, M. S., Swaine, D. J., *Ibid.*, pp. 505–6.
(5B) *Colliery Guardian* **208**, No. 5363, 151 (1964).
(6B) Crump, J. G., *Blast Furnace Steel Plant* **52**, 325–7 (1964).
(7B) Cudars, J., Skvortsova, N., *Latvijas PSR Zinatnu Akad. Vestis* **1962**, (5), 75–83.
(8B) Czuchajowski, L., *Arch. Gornictwa* **5**, 241–69 (1960).
(9B) Dahn, E., Brunner, G., *Isotopen Tech.* **2**, 201–4 (1962).
(10B) Das Gupta, N. N., Chatterjee, N. N., Bhowmic, B. B., *Fuel Res. Inst. News* **12**, (3–4), 95–9 (1962).
(11B) Dockalova, L., Kessler, F. M., *Paliva* **42**, 368–71 (1962).
(12B) Dresia, H., Friedrich, W., Vogel, J., *Brennstoff-Chem.* **43**, 149–52 (1962).
(13B) Gal, E., Szava, J., *Bányász. Kut Int. Közlemén* **5**, 200–5 (1962).
(14B) Goldinov, A. L., Lukhovitskii, V. I., Srubinskaya, G. Z., *Zhur. Anal. Khim.* **17**, 532–4 (1962).
(15B) Grabowska, H., *Koks, Smola, Gaz* **8**, (2), 49–52 (1963).
(16B) Hardt, L., *Glückauf* **98**, 823–31 (1962).
(17B) Hoffmann, K., *Chem. Ing. Tech.* **35**, 55–62 (1963).
(18B) Hudec, P., Schiller, P., *Automatizace* **6**, (9), 219–22 (1963).
(19B) Ivanchenko, E. Ya., Kobyakov, B. S., Izv. *Vysshikh Uchebn. Zavedenii, Gorn. Zh.* **5**, (5), 123–33 (1962).
(20B) Jirkovsky, R., *Sb. Ved. Praci Vysokej Skoly Banske Ostrave* **6**, 713–22 (1960).
(21B) Kanuf, G., Gruson, G., *Paliva* **42**, 364–7 (1962).
(22B) Khristov, A. A., Vorob'ev, I. E., *Koks i Khim.* **1964**, (5), 9–10.
(23B) Koch, S., Windelband, R., *Kernenergie* **7** (3), 176–83 (1964).
(24B) Köhling, R., Moser, T., *Glückauf* **99**, 1176–81 (1963).
(25B) Krigman, F. E., *Zavodsk. Lab.* **30**, 453–4 (1964).
(26B) Kulishenko, A. Z., Dyukanov, A. G., *Sb. Nauchn. Tr. Ukr. Nauchn.-Issled. Uglekhim. Inst.* **12**, 84–94 (1961).
(27B) Ladner, W. R., Stacey, A. E., *Brit. J. Appl. Phys.* **13**, (3), 136 (1962).
(28B) Mori, H., Taira, S., *Japan Analyst* **11**, 764–7 (1962).
(29B) Netz, V., *Messen, Steuern, Reglen, Berlin* **6**, 427–9 (1963).
(30B) Nitadori, J., Aoki, T., Sato, M., Koseki, K., *Resources Res. Inst. Japan, Rept.* **52**, 58 (1962).
(31B) Pande, A., Pande, C. S., *Instr. Pract.*, **16**, 1104–10 (1962).
(32B) Radmacher, W., Scholz, A., *Erdöl Kohle*, **15**, 96–101 (1962).
(33B) Rieke, F. F., Semmler, R. A., *Combustion*, **35**, No. 9, 25–7 (1964).
(34B) Schmidt, J., Telling, H., *Mitt. Inst. Energetik, Lpz.*, **46**, 356–64 (1962).
(35B) Tornau, R., Waechter, K. H., *Ver. Deut. Ingr. Z.* **104**, 901–2 (1962).

Ultimate Analysis

(1C) British Coke Res. Assoc., *Coke Res. Rept.*, No. 13, 7 (1961).
(2C) Drekopf, K., Gebert, S., Winzen, W., *Mikrochim. Ichnoanal. Acta* **2**, 327–47 (1963).
(3C) Elfferich, N. W., Kreulen, D. J. W., *Ned. Chem. Ind.* **5**, 146–8 (1963).
(4C) Gruzdeva, N. A., *Podgotovka i Koksovanie Uglei, Vost. Nauchn.-Issled. Uglekhim. Inst., Sb. Statei* **1963**, (4) 256–63.
(5C) Kozhevnikova, O. P., Pogrebinsk-

aya, M. L., Kleptsina, Z. B., *Ibid.*, **1962**, 412–14.
(6C) Thürauf, W., Thiemann, W., *Brennstoff-Chem.* **44**, (9) 275–80 (1963).
(7C) Visapää, A., *Teknillisen Kemian Aikakauslehi* **19** (14), 565–72 (1962).

Calorific Value

(1D) Schlattner, J., *Acta Chim. Acad. Sci. Hung.* **35**, 301–8 (1963).

Inorganic Constituents in Coal and Coal Ash

(1E) Abernethy, R. F., Gibson, F. H., *U.S. Bur. Mines Inform. Circ.* **8163**, 69 (1963).
(2E) Ahmed, M. N., Khan, R. M., *Pakistan J. Sci. Ind. Res.* **5**, 166–72 (1962).
(3E) Belcher, C. B., Brooks, K. A., *Anal. Chim. Acta* **29**, 202–5 (1963).
(4E) Bethell, F. V., *Brit. Coal Util. Res. Assoc., Monthly Bull.* **26**, (12), 401–30 (1962).
(5E) Bhaduri, B. P., Ghose, S. K., Majumdar, S. K., *Indian J. Technol.* **2**, (2) 62–64 (1964).
(6E) Biswas, N., Mukherjee, S. N., Dutta, P. B., Lahiri, A., *J. Sci. Ind. Res., India* **21D**, (3) 101–2 (1962).
(7E) Buncak, P., *Ropa Uhlie* **5** (5), 144–9 (1963).
(8E) Clark, M. C., Swaine, D. J., *Australia, CSIRO, Coal Res. Div., T.C.* **45**, 1–20 (1962).
(9E) Clark, M. C., Swaine, D. J., *Fuel London* **42**, (4), 315–18 (1963).
(10E) Cook, A. C., *Australian J. Sci.* **25**, 94–5 (1962).
(11E) Dalton, I. M., Pringle, W. J. S., *Fuel* **41**, 41–48 (1962).
(12E) Davidyuk, L. A., *Dopovidi Akad. Nauk Ukr. RSR* **1962**, 401–5.
(13E) Durie, R. A., Schafer, H. N. S., Swaine, D. J., *CSIRO Divn. Coal Res. Misc. Rept.* **178**, 13 (1963).
(14E) Durie, R. A., *Fuel* **42**, 421–4 (1963).
(15E) Dvořák, J., *Coll. Czech. Chem. Commun.* **28** (1), 221–8 (1963).
(16E) Eagar, R. M. C., *Nature, London* **196**, 428–31 (1962).
(17E) Ershov, V. M., *Geokhimiya* 306–8 (1961).
(18E) Filchovska, P. D., *Khim. i Ind. (Sofia)* **34**, 204–5 (1962).
(19E) Gardelka, K., *Sb. Ved. Praci Vysoke Skoly Banske Ostrave* **6**, 117–19 (1960).
(20E) Gonzalez, L., *Inst. Nacl. Carbon (Oviedo, Spain), Bol Inform.* **11** (54), 58–71 (1962).
(21E) Heilpern, S., *Koks, Smola, Gaz* **8** (2), 57–59 (1963).
(22E) Jackwerth, E., Kloppenburg, H. G., *Z. Anal. Chem.* **186**, 428–36 (1962).
(23E) Kaplan, B. Ya., Ol'shevskaya, I. V., *Zavodsk. Lab.* **29**, 26–7 (1963).
(24E) Kirk, B. P., Wilkinson, H. C., *Fuel* **43**, 105–9 (1964).
(25E) Kizil'shtein, L. Ya., Syunyakova, N. N., *Razvedka i Okhrana Nedr* **29** (9), 20–4 (1963).
(26E) Kononenko, N. I., *Nauchn. Tr. Permsk. Nauchn.-Issled Ugol'n. Inst., Sb.* (4), 40–45 (1962).
(27E) Kovacs, F., Kralik, J., Policky, J., *Sb. Ved. Praci Vysoke Skoly Banske Ostrave* **6**, 93–115 (1960).
(28E) Kudelasek, V., *Ibid.*, **5**, 457–81 (1959).
(29E) Kunstmann, F. H., Bodenstein, L. B., *J. S. African Inst. Mining Met.* **62**, 234–44 (1961).
(30E) Kunstmann, F. H., Harris, J. F., Bodenstein, L. B., van der Berg, A. M., *Fuel Res. Inst., Pretoria, Bull.* **63**, 45 (1963).
(31E) Laske, H., *Wiss. Z. Martin-Luther Univ. Halle-Wittenberg. Math.-Nat. Reihe* **11**, 873–8 (1962).
(32E) Menkovskii, M. A., Aleksandrova, A. N., *Zavodsk. Lab.* **29**, 797–9 (1963).
(33E) Menkovskii, M. A., Gordon, S. A., Nurminskii, N. N., *Ibid.*, **28**, 1321 (1962).
(34E) Mineva, R., *Khim. Ind., Sofia* **33** (3), 90–1 (1961).
(35E) Mironyuk, L. M., *Izv. Dnepropetr. Gorn. Inst.* **41**, Pt. 1, 198–9 (1962).
(36E) Purkayastha, B. C., Saha, S. R., *Indian J. Appl. Chem.* **25**, No. 1, 20–3 (1962).
(37E) Skrivan, P., *Sb. Vysoke Skoly Chem. Technol.-Praze, Mineral.* **6**, 391–404 (1962).
(38E) Sweatman, T. R., Norrish, K., Durie, R. A., *Australia, Commonwealth Sci. Ind. Res. Organ., Div. Coal Res., Misc. Rept.* **177**, 30 (1963).
(39E) Thurauf, W., Assenmacher, H., *Brennstoff-Chem.* **44**, 21–2 (1963).
(40E) Tsynovnikov, A. S., Pogrebinskaya, M. L., Filippova, A. A., Kleptsina, Z. B., *Koks i Khim.* **1964** (4), 17–20.
(41E) Tupper, W. M., Loring, D. H., *Geochim. Cosmochim. Acta* **24**, 314–15 (1961).
(42E) Vasilevskaya, A. E., Shcherbakov, V. P., *Dopovidi Akad. Nauk Ukr. RSR* 1494–6 (1963).
(43E) Vasilevskaya, A. E., Shcherbakov, V. P., Klimenchuk, V. I., *Zavodsk. Lab.* **28**, 415 (1962).
(44E) Volkhova, V. I., *Zavodsk. Lab.* **29**, 804–5 (1963).
(45E) Wilson, A. D., Lewis, D. T., *Analyst* **88**, 510–15 (1963).
(46E) Zahradnik, L., Tyroler, J., Vondrakova, Z., Stovik, M., *Sb. Geol. Ved, Technol. Geochemie*, 189–201 (1962).

Laboratory Coking Tests

(1F) Bogoslovskii, Yu. N., Kudryashov, V. I., Makarov, G. N., *Zavod. Lab.* **29**, 198–9 (1963).
(2F) Gryaznov, N. S., Komarovskaya, G. M., "Preparation & Coking of Coals," *Metallurg.* No. 3, 277–88 (1962).
(3F) Haque, R., Sharma, S. K., Das Gupta, N. N., Lahiri, A., *Indian J. Technol.* **1**, 322–7 (1963).
(4F) Jones, W., *J. Inst. Fuel*, **37**, 3–11 (1964).
(5F) Juranek, G., Bussmann, B., Seifert, G., *Brennstoff-Chem.* **44**, 97–104 (1963).
(6F) Kreulen-van Selms, F. G., *Ibid.*, p. 310.
(7F) Loison, R., Foch, P., *Rev. Ind. Minerale* **43** (9), 19–22 (1961).
(8F) Petrik, G. K., Sarymasakov, Sh., Belekov, O., *Koks i Khim.*, **1962**, No. 12, 13–16.
(9F) Pichler, H., Lauer, K. H., *Brennstoff-Chem.* **44**, 149–54 (1963).
(10F) Pyzhov, Yu. V., *Coke Chem USSR*, (7), 17–19 (1963).
(11F) Schuhknecht, W., Schinkel, H., *Brennstoff-Chem.* **44**, 202–6 (1963).
(12F) Shapiro, M. D., Alterman, L. S., *Coke Chem. USSR*, (10), 5–10 (1962).
(13F) Shapiro, M. D., Al'terman, L. S., Keitel'gisser, S. R., *Koks i Khim.* **1963**, (9), 10–14.
(14F) Sheikhet, A. M., Pyzhov, Yu. V., Goldberg, P. Ya., Rabukhina, G. G., *Coke Chem. USSR*, (6), 15–17 (1963).
(15F) Stephens, J. N., *Fuel* **42**, 175–8 (1963).
(16F) Voloshin, A. I., Virozub, I. V., Kazmina, V. V., *Coke Chem. USSR*, (3), 17–20 (1962).
(17F) Wuerker, R. G., Singh, M. M., Chakraverty, N., *Illinois Univ. Eng. Expt. Sta. Tech. Rept.* No. 1, 43 (1962).

Standard Methods

(1G) Am. Soc. Testing and Materials, "ASTM Standards, Gaseous Fuels, Coal and Coke," Vol. **19**, p. 434 1964.

Miscellaneous

(1H) Brown, H. R., Taylor, G. H., Cook, A. C., *Fuel, London* **43**, 43–54 (1964).
(2H) Burns, M. S., Durie, R. A., Swaine, D. J., *Ibid.*, **41**, 373–83 (1962).
(3H) Edwards, A. H., Jones, J. M., Newcombe, W., *Ibid.*, **43**, 55–62 (1964).
(4H) Gin, T. T., Dahl, C. L., Wilson, D. G., *Blast Furnace Steel Plant* **52**, 237–43, 290 (1964).
(5H) Hardgrove, R. M., *Combustion* **33**, 46 (Apr. 1962).
(6H) Harrison, J. A., *Min. Engr*, **123**, 346–57 (1963).
(7H) Jones, J. M., *J. Sci. Instr.* **38**, 303–4 (1961).
(8H) Kijewska, A., *Koks, Smola, Gaz* **5**, 3–9 (1960).
(9H) Krumin, P. O., *Ohio State Univ., Eng. Expt. Sta. Bull. No.* **195**, 78 (1963).
(10H) Leithe, W., *Z. Anal. Chem.* **193**, 16–23 (1963).
(11H) Murchison, D. G., *Bull. Inst. Mining Met.* **689** (4), 479–502 (1964).
(12H) Ponomareva, M. N., Lifshits, M. M., Vyrich, G. P., *Issled. i Klassifik. Uglei, Donetskii Nauchin.-Issled. Ugol'n. Inst.* No. **25**, Moscow, Sb. 52–95 (1962).
(13H) Prilezhaeva, E. N., Fedorovskaya, N. P., Miesserova, L. V., Domanina, O. N., Khaskina, I. M., *Tr. Inst. Goryuch. Iskop., Akad. Nauk SSSR* **21**, 159–68 (1963).
(14H) Pringle, W. J. S., *Fuel, London* **42**, 63–8 (1963).
(15H) Sarkar, G. G., Manchanda, S., *J. Min. Metals Fuels* **10**, (5) 17–20 (1962).
(16H) Seichter, A., Gruson, G., *Freiberg. Forschungsh.* **A271**, 79–82 (1963).
(17H) Sinha, N. C., Moitra, A. K., Majumdar, S., *J. Min. Metals Fuels*, **10**, (11), 1–6 (1962).
(18H) Tyman, Vl., *Paliva* **43**, No. 2, 37–41 (1963).
(19H) Wnekowska, L., *Prace Glownego Inst. Gornictwa, Komun. No.* **325**, 12 (1963).
(20H) Wolfe, J. D., *J. Inst. Fuel* **35**, 448–51 (1962).

GASEOUS FUELS

(1J) *Am. Gas J.* **191**, 186 pp. (1964).
(2J) Baranenko, S. E., *Tr. Gos. Vsesp Inst. po Proektir i Nauchn.-Issled. Rabotam* **2**, 109–23 (1961).
(3J) Burgess, S. G., Wood, L. B., *Inst. Sewage Purif., J. Proc. Part* **1**, 26–46 (1964).
(4J) Duchemin, M., *Dechema Mon.* **48**, 835–58 (1963).
(5J) Fuhrmann, H., *Glas-Instr.-Tech.* **8**, 340–3, 345, 347 (1964).
(6J) Hobbs, A. P., ANAL. CHEM. **36**, 130R–139R (1964).
(7J) Kienitz, H., *Z. Anal. Chem.* **192**, 160–89 (1963).

Standards

(1K) ASTM Standards, 1964, Part **19**, ASTM, Philadelphia, Pa. (1963).

Physical Methods

(1L) Aliev, Y. Y., Aminova, R. I., *Issled. Mineral'n. i Rast. Syr'ya Uzbekistana, Akad. Nauk Uz. S. S. R., Inst. Khim.* 106–15 (1962).
(2L) Andreen, B. H., Kniebes, D. V., *Proc. Operating Sect., Am. Gas Assoc.* **CEP-62-13**, 7 pp. (1962).
(3L) Averill, W., Ettre, L. S., *Nature* **196**, 1198–9 (1962).
(4L) Bruk, A. I., Vinogradova, L. M., Vyakhirev, D. A., *Tr. po Khim. i Khim. Tekhnol.* No. 1, 211–12 (1962).
(5L) Colson, E. R., ANAL. CHEM. **35**, 1111–12 (1963).
(6L) Colson, E. R., Brown, G. M., *J. Inst. Fuel* **35**, 3399–404 (1962).
(7L) Galwey, A. K., *Talanta* **9**, 1043–52 (1962).
(8L) Graf, L., Toth, J., *Abhandl. Deut. Akad. Wiss. Berlin, Kl. Chem., Geol. Biol.* No. **1**, 145–61 (1962).
(9L) Henderson, W. L., *Water Sewage Works* **107**, 312–13 (1960).
(10L) Isbell, R. E., ANAL. CHEM. **35**, 255–6 (1963).
(11L) Juvet, R. S., Dal Nogare, S., *Ibid.*, **36**, 36R–51R (1964).
(12L) Kavan, I., Vlckova, Z., *Prace. Ustavu Vyzkum Paliv* **3**, 203–15 (1963).
(13L) Kipping, P. J., Jeffery, P. G., *Res. Develop. Ind.* No. 9, 46–9 (1962).
(14L) Kirkland, J. J., ANAL. CHEM. **35**, 1295–7 (1963).
(15L) Knipschild, J., Pagnier, G., *Brennstoff-Chem.* **43**, 197–9 (1962).
(16L) Lityaeva, Z. A., Markosov, P. I., Aichenko, V. N., *Tr. Vses Neftegaz. Nauchn.-Issled. Inst. Krasnodarsk. Filial* No. 8, 110–25 (1962).
(17L) Lulova, N. I., Tarasov, A. I., Kuz'mina, A. V., Koroleva, N. M., *Neftekhimiya* **2**, 885–91 (1962).
(18L) Manka, D. P., ANAL. CHEM. **36**, 480–2 (1964).
(19L) Miller, A. J., *Rev. Inst. Franc. Petrole Ann. Combust. Liquids* **17**, 1317–27 (1962).
(20L) Miquel, R., Benard, C., *Compt. Rend.* **256**, 940–1 (1963).
(21L) Muraosa, K., Iwaya, K., *Kogyo Kagahu-Zasshi* **65**, 1550–4 (1962).
(22L) Orechkin, D. B., Dantsig, G. N., USSR Pat. **150,297**.
(23L) Pichler, H., Herlan, A., Schulz, H., *Brennstoff-Chem.* **43**, 269–76 (1962).
(24L) Sechenov, G. P., Bunina, N. N., Al'tshuler, V. S. S., *Tr. Inst. Goryuch. Iskop., Akad. Nauk SSR,* **18**, 253–8 (1962).
(25L) Tung, K. N., Yu, W., *Shih'yu Lien Chih.* No. 11, 36–7 (1960).
(26L) Turkel'taub, N. M., Porshneva, N. V., *Novosti Neft. i Gaz. Tekhn. Gaz. Delo* No. 11, 32–5 (1961).
(27L) Wulfhekel, H., Schaal, G., *Exptl. Tech. Physik,* **10**, 231–43 (1962).

Chemical Methods

(1M) Baranenko, S. E., Krivosheeva, V. I., Maksimenko, E D., USSR Pat. **157,559**.
(2M) Charpenet, L., *Chim. Anal.* **45**, 367–72 (1963).
(3M) Chory, J. P., *Brennstoff-Waerme-Kraft* **14**, 6.1–3 (1962).
(4M) Clayton, E. E., Jr., Jones, J. H., *Oil Gas J.* **61**, 82, 85–6 (1963).
(5M) Coleman, E. H., Elkins, G. H. J., Brit. Pat. **963,197**.
(6M) Engelbrecht, D. J., Scholtz, H. v. Z., van den Berg, J. P., *J. S. African Chem. Inst.* **17**, 38–40 (1964).
(7M) Fredericks, E. M., Harlow, G. A., ANAL. CHEM. **36**, 263–6 (1964).
(8M) Golyand, S. M., Lazhrev, V. I., *Zh. Analit. Khim.* **17**, 734–8 (1962).
(9M) Grosskopf, K., *Chemiker-Ztg.* **87**, 270–5 (1963).
(10M) Kavan, I., Base, J., *Prace. Ustav. Vyzkum Paliv* **2**, 218–23 (1960).
(11M) Leithe, W., *Z. Anal. Chem.* **193**, 16–23, 24–27 (1963).
(12M) Morriss, F. V., Meiners, A. F., U. S. Pat. **3,061,414**.
(13M) Scholl, F., *Zeigelindustrie* **15**, 843–5 (1962).
(14M) Stout, J. W., Jr., Early, E., *Proc. Am. Gas Assoc.* **CEP-62-2**, 4 pp. (1962).
(15M) Takens, W., *Gas* **82**, 123–5 (1962).
(16M) Wronkowski, C., *Med. Weterynar.* **17**, 115 (1961).

Sampling

(1N) Chekanov, A. A., *Haughn. Tr. Vses. Nauchn.-Issled. Inst. Podzemn. Gazifik. Uglei.* **6**, 62–5 (1962).
(2N) Fickinger, W., *Gas-Wasserfach* **103**, 1354–6 (1963).
(3N) Lutoshkin, G. S., *Novosti Neft. i Gaz. Tekhn., Gaz Delo.* **2**, 18–27 (1962).
(4N) Maksimov, V. F., Bushmelev, V. A., *Nauchn.-Tekhn. Inform. Tsentr. Inst. Nauchn.-Tekhn. Inform. Bumazhn. i Derevoobrabat. Prom. Tsellyuzno-Bumazhn., Gidrolisn. i Lesokhim. Prom.* **10**, 35–9 (1961).

Moisture

(1P) Davis, D. S., *Ind. Chemist* **38**, 565 (1962).
(2P) Mikhailov, N. P., *Novosti. Neft. i. Gaz. Tekhn. Gaz. Delo.* **7**, 29–33 (1962).

Calorific Value

(1Q) Krizan, V., *Chem. Zvesti* **16**, 769–73 (1962).
(2Q) Rosset, R., *Nature (Paris)* No. **3342**, 435–6 (1963).

Miscellaneous

(1R) Bothe, H. K., Adler, P., *Chem. Tech. (Berlin)* **16**, 169–74 (1964).
(2R) Iijima, Y., Miyazu, T., Araragi, M., Hashimoto, Y., Takeuchi, T., *Koru Taru* **14**, 449–53 (1962).
(3R) Kavan, I., Vlckova, Z., *Prace. Ustav. Vyzkum Paliv* **4**, 381–91 (1962).
(4R) Lestienne, A., *Compt. Rend.* **255**, 912–13 (1962).
(5R) Lille, Y., Murd, A., Kundel, K., *Khim. i Tekhnol. Goryuch. Slantsev. i Produktou Ikh Pererabotki* **12**, 142–51 (1963).
(6R) Martin, A. E., Shields, J., Ger. Pat. **1,133,920**.
(7R) Ryabov, V. P., *Zavodsk. Lab.* **29**, 824–5 (1963).
(8R) Schultz, G., *Dechema. Mon.* **43**, 287–98 (1962).
(9R) Zartman, R. E., Wasserburg, G. J., Reynolds, J. H., *J. Geophys. Res.* **66**, 277–306 (1961).

Water Analysis

M. W. Skougstad and M. J. Fishman

U. S. Geological Survey, Denver, Colo.

THIS BIENNIAL REVIEW, the eleventh in a series which began in 1949, generally covers contributions to methods of water analysis published during the period from October 1962 through September 1964. It follows the plan of the previous reviews, the most recent of which appeared in ANALYTICAL CHEMISTRY for April 1963 (*20*).

A Research Committee of the Water Pollution Control Federation publishes an annual review of literature related to water pollution control which regularly includes a section on analytical methods. The review of the literature of 1961 (*9*) included 81 references to methods for determining anions, biochemical oxygen demand, chemical oxygen demand, cyanides, dissolved oxygen, oxidation–reduction potential, organics, and others. Sixty-one references were included in their review of 1962 literature (*10*) and covered such topics as biological methods for pollution evaluation; instrumental methods, including conductivity, polarography, galvanic cell polarography, luminescence, and chromatography; nitrogen compounds, including organic amines and cyanides; and organic compounds, such as phenols, chlorinated compounds, and synthetic detergents. The most recent review (*11*), covering the literature of 1963, contained references to 116 publications dealing with these general topics plus such things as biocides, carboxylic acids, sulfur compounds, and suspended solids.

A review listing 102 references to methods for the determination of trace and other elements in water, soils, and plants was published by Borovik-Romanova and Kutsenko (*2*). Ballinger (*1*) prepared a review of polarographic methods for analyzing water and wastes. Physicochemical methods for the analysis of brines and mineral substances were reviewed by Mazel and Akulovich (*17*).

Mackereth (*15*) prepared a manual of water analysis methods for limnologists. A manual of recommended methods for the determination of copper, nickel, lead, iron, manganese, zinc, and cadmium in oilfield brines was prepared by Collins *et al.* (*3*). Methods used for certain oceanographic investigations have been detailed by Goya *et al.* (*7*); and chemical methods, suitable for determining pH, alkalinity, calcium, magnesium, sulfate, and chloride in rainwater samples, were described by Matveev (*16*).

Dall'Aglio and Tonani (*5*) tested several physical and chemical methods for analyzing surface waters in connection with geochemical investigations, and a number of analytical methods potentially useful for hydrogeochemical exploration were evaluated by Sokolov *et al.* (*21*). Konovalov (*12*) gave details of a portable field laboratory for performing colorimetric and titrimetric analysis for pH, heavy metals, calcium, magnesium, hardness, alkalinity, chloride, sulfate, iron(II), iron(III), and boron. A system for the rapid determination of several dissolved constituents by conductometric titrations and the necessary instrumentation for field use were described by Constable (*4*).

Several methods and techniques for the analysis of effluent water were noted and evaluated by Humphrey (*8*). The methods used by a commercial laboratory to analyze up to 60,000 water samples per year and methods for regular instrument calibration checks and for routine methods checking were described in a special publication by Sussman, Portnoy, and Jacobson (*23*).

The results of a cooperative study wherein a standard water sample was simultaneously analyzed for calcium, magnesium, sodium, potassium, hardness, chloride, sulfate, alkalinity, nitrate, and nitrite by 136 different laboratories were reported by Lishka, Kelso, and Kramer (*14*). The cation-anion balance method of checking the accuracy of water analyses was discussed in some detail by Terhaag (*24*).

Methods and techniques for determining trace elements have been reported by several investigators. Goldberg (*6*) discussed analytical problems connected with trace analysis of sea water. A study of the completeness of coprecipitation of tin(II), zinc, niobium, gallium, and germanium was made by Sokolov, Polyakov, and Lushnikov (*22*) who used radioactive isotopes to evaluate various coprecipitation systems. A rapid, indirect method for the determination of p.p.b. concentrations of total metal ions in reactor cooling water was developed by Pohl (*18*). A small measured amount of EDTA is added to the sample to form the metal complexes, after which an exactly equivalent amount of cadmium is added and the uncomplexed cadmium determined polarographically. As little as 0.1 p.p.b. of metal ions can be determined.

Neutron activation analysis methods for water were discussed by Leddicotte and Moeller (*13*) and by Selz, Haerdi, and Monnier (*19*).

ALKALI METALS

Several investigators have reported flame photometric methods for determining the alkali metals in water. Gupta and Ramaswamy (*17A*) found sodium and potassium concentrations in sea water which differed slightly from previously reported values. The sodium values were higher, after correcting for potassium, calcium, magnesium, and sulfate; the potassium results were slightly lower, after correcting for sodium, calcium, magnesium, and sulfate. Frantsuzova (*13A*) designed a flame photometer with interference filters for determining sodium and potassium. Calcium is removed by the addition of ammonium oxalate and a few drops of ammonium hydroxide. The filtrate is diluted to a specified volume and the sodium determined. The standards are also made up with ammonium oxalate and ammonium hydroxide. Potassium is determined directly. Flame photometric methods for determining lithium, sodium, potassium, rubidium, and cesium in thermal waters were examined critically by Golding and Speer (*15A*).

Collins (*4A*, *5A*) determined potassium, lithium, strontium, barium, manganese, cesium, and rubidium in oilfield waters by flame photometry. Standard addition techniques in an ethanol medium were used for determining potassium, lithium, and strontium. After removal of interferences, barium and manganese were determined in ethanol and propanol, respectively. The tetraphenylboron complexes of cesium and rubidium were extracted with nitroethane prior to determining their emission intensities. Extraction efficiencies for cesium and rubidium were determined by radiotracer methods.

Uzumasa, Nasu, and Terae (*27A*) reported a procedure for determining rubidium and cesium in natural waters by first forming their tetraphenylborates in the presence of potassium. The recovered precipitate is mixed with acetone, the mixture evaporated to dryness, and the tetraphenylborates decomposed by boiling with a solution containing mercuric chloride and ammonium hydroxide. After removing the decomposition product and evaporating the filtrate to dryness, hydrochloric acid is added and the sample again evaporated. The residue is then dissolved in water and rubidium and cesium determined in an oxygen-hydrogen flame at 795 and 852 mμ, respectively. Rubidium concentrations from 0.5 to 10 mg. per liter and cesium concentrations from 0.2 to 5 mg. per liter can be determined within $\pm 5\%$ even in the presence of 1000 p.p.m. of potassium. Sodium tetraphenylboron was also used by Feldman and Rains (*11A*) to determine cesium. Ammonium 12-molybdophosphate (AMP) was first used to absorb cesium from a large volume of water and the AMP then flocculated with aluminum nitrate solution and the residue separated by filtration and centrifugation, and redissolved in 1*M* sodium hydroxide solution. The cesium was extracted with sodium tetraphenylboron in a hexone–cyclohexane mixture and determined by flame photometry. The sensitivity was 0.005 μg. per ml. in the 3:1 hexone–cyclohexane reagent, and 0.05 μg. per ml. in aqueous solution containing 200 μg. per ml. of potassium as the chloride.

Chow (*2A*) described a flame photometric procedure for determining potassium in both fresh water and sea water. Studies were made to determine the extent of radiation and spectral interference of other major cations on the 767-mμ potassium line. None of the ions emitted appreciably at this wavelength. Sodium ions caused high potassium recovery, and chloride ions and sulfuric acid suppressed the intensity of the potassium spectral line. A standard addition procedure was used to eliminate the effect of these interfering constituents.

Yofe and Stiller (*29A*) developed a direct flame photometric method for determining lithium in brines. Lithium emission is measured at 671 mμ and a background correction made from the emission readings of the sample at 665 and 675 mμ. Jonsson (*19A*) determined low (0.010 $\pm$0.001 p.p.m.) concentrations of lithium by directly aspirating the sample without previous chemical treatment. If the samples contain 20% of a mixture of acetone and amyl alcohol (4:1), the intensity of lithium emission is increased three-fold. Calcium and strontium interferences are suppressed by the addition of aluminum; interference caused by stray sodium light is eliminated by a special absorption filter. Concentration of the sample is necessary for the lowest lithium concentrations.

Dobremyslova, Dvorak, and Novobilsky (*8A*) estimated the calcium and sodium content of waste waters by flame photometry. Ferraris (*12A*) determined, potassium, cesium, calcium, and strontium by flame photometry, and described the conditions used and the sources of errors.

Li and Huang (*22A*) used cation exchange to separate sodium and potassium, and a modified mercurimetric method to titrate the chlorides of each. The acidified sample is first passed through an ion-exchange column and sodium eluted with 0.2*N* hydrochloric acid. Potassium is then eluted with 0.5*N* hydrochloric acid. The samples, after evaporation to remove excess hydrochloric acid, are dissolved in dilute

HNO_3 and titrated with mercuric nitrate, using diphenylthiourea in alcohol as the indicator. Lithium is eluted with the sodium but may be removed by extraction with amyl alcohol prior to titration.

Mamedov and Emirdzhanova (*23A*) determined sodium in mineral and oil well waters by an arsenate–iodometric method. Sodium is precipitated as the zinc uranyl acetate, washed with ethanol, dissolved in hydrochloric acid, and converted to an insoluble arsenate compound. The precipitate is then washed, dissolved in hydrochloric acid, reprecipitated with ammonium hydroxide, dissolved with sulfuric acid, and titrated, after addition of benzene–potassium iodide solution, with sodium thiosulfate to decoloration of the organic layer.

An indirect complexometric method for determining sodium in mineral waters was described by Dolezal, Novozamsky, and Zyka (*9A*). Sodium is precipitated with cobalt uranyl acetate and determined by chelatometric uranium titration after reduction with ascorbic acid.

Gravimetric methods for determining sodium and potassium in waters of low mineralization were reported by Efendi and Sokolovich (*10A*). Sodium is determined with magnesium uranyl acetate, and potassium with either sodium or magnesium dipicrylaminate.

Three different paper chromatographic techniques were developed by Djavad-Nia (*7A*) for determining sodium, potassium, calcium, and magnesium in mineral waters. The first chromatogram was prepared directly from the water sample, the second used the sulfated water residue, and the third the precipitate of calcium and magnesium carbonates. The chromatogram developing reagents were ammoniacal 8-quinolinol for magnesium and calcium, $Zn(H)(UO_2)_3(OAc)_9$ for sodium, and $Na_3Co(NO_2)_6$ for potassium. The calcium and magnesium results compared favorably with results obtained by EDTA titration. Literature values for the same water sources were used for comparing sodium and potassium results.

Potassium in natural waters was determined turbidimetrically by Kriventsov (*21A*). Samples and standards were prepared by adding dry sodium chloride and $Na_3Co(NO_2)_6$, dissolving the solids, and allowing 30 minutes before comparison. In comparison with the gravimetric method for well waters containing from 25.1 to 424 mg. of potassium per liter, the method gave an error of -7.9 to $+6.1\%$.

Goremykin (*16A*) used a sodium sensitive glass electrode to analyze natural waters having any composition in which the sum of ions amounts to 5 to 6 grams per liter. Equations and sample calculations are presented.

Spectrographic techniques for determining manganese and potassium in formation water were presented by Berezina (*1A*). The method is more rapid and requires only inexpensive reagents and a smaller aliquot of water than noninstrumental methods. Chow and Goldberg (*3A*) described a mass spectrometric method for determining lithium in sea water. Lithium-6 is used as a tracer. Lithium is separated by ion exchange, using Dowex 50-X16 and $1N$ hydrochloric acid to elute the lithium. The elution is terminated when sodium first appears in the eluate. About 60% of the lithium is recovered in a solution relatively free of other cations. The solution is evaporated almost to dryness and about 5 μg. of the residue is transferred to a tantalum filament and the lithium concentration determined spectrometrically at 850° C., at which temperature lithium is emitted. Since only lithium has naturally occurring isotopes of mass numbers 6 and 7, the method is specific for lithium.

Several methods have been reported for determining cesium-137 in sea water and other types of water. Golchert and Sedlet (*14A*) separated cesium using hexachlorostannate. Coprecipitation of cesium with ferrocyanides was used by Shvedov, Ivanova, and Zharikov (*26A*), Ivanova and Shvedov (*18A*), and Mohanrao and Folsom (*24A*). The use of ammonium molydophosphate as a scavenger was reported by Morgan and Arkell (*25A*), Yamagata (*28A*), and Derecki *et al.* (*6A*).

Kalhousova (*20A*) described a visual colorimetric method for determining sodium peroxide in water based on its reaction with titanium tetrachloride. None of the other substances present in natural water react with titanium tetrachloride; however, nitrites, organics, copper, cobalt, an increased temperature, or a high pH result in destruction of the peroxide. The sensitivity of the determination is 1 mg. of Na_2O_2 per liter.

HARDNESS, ALKALINE EARTH METALS

Rowe (*28B*) developed a rapid, direct flame photometric method for determining calcium concentrations below 1 p.p.m. in the presence of large amounts of sulfate, silica, and bicarbonate without chemical separations. The depressive effects of silica, sulfate, aluminum, and phosphate on calcium are eliminated by adding magnesium. Background corrections, as well as the use of magnesium, eliminate interferences from sodium and potassium; perchloric acid eliminates bicarbonate interference. The results obtained for thermal waters agreed to results obtained by the usual EDTA titration method. Dziedzianowicz (*10B*) compared two available commercial flame photometers for their stability, accuracy, and sensitivity for determining traces of calcium in the presence of magnesium in boiler feed waters.

Lukin, Smirnova, and Zavarikhina (*22B*) proposed a new indicator, Calcion IREA, for determining calcium photometrically and complexometrically, and described its preparation. A solution of the indicator reagent is stable and its color reaction with calcium is both sensitive and selective. The method was applied to the determination of calcium in distilled water.

Bozhevol'nov and Kreingol'd (*4B, 5B*) described fluorescence complexometric methods for determining calcium in water. In one, calcium is titrated with EDTA in $0.1N$ sodium hydroxide with fluorescein complexon indicator. In the other, it is titrated in a $0.1N$ potassium hydroxide solution with Fluorexone indicator. A fluorometric method for determining magnesium using disalicylideneethylenediamine was reported by Serebryakova, Bozhevol'nov, and Godlina (*29B*). The sample is evaporated to dryness, ignited, and the residue dissolved in a solution of isobutylamine in dimethylformamide. Finally, disalicylideneethylenediamine in dimethylformamide is added and the fluorescence compared with standards under ultraviolet light. The fluorescence properties of nine azo compounds known to form calcium and magnesium derivatives were studied by Olsen and Diehl (*25B*). They found that 1-(2′-hydroxy-1′-benzene azo)-2-hydroxybenzene showed the most promise for the determination of magnesium in the presence of calcium. Diehl *et al.* (*8B*) used the above azo compound to determine between 2 and 25 μg. of magnesium in water fluorometrically and spectrophotometrically in the presence of up to 5 mg. calcium. Provisions were made to eliminate interferences from iron, aluminum, copper, and zinc. Detailed procedures are given for water as well as other types of material. They preferred the fluorometric method because of its greater sensitivity and because it requires less critical adjustment of conditions.

Katz and Navone (*19B*) described a method for the simultaneous determination of calcium and magnesium using a new indicator, Eriochrome Blue SE. Magnesium is first precipitated by adding sodium hydroxide to a pH of 12 to 13. Calcium is then titrated with EDTA with Eriochrome Blue SE indicator. The solution is then acidified with hydrochloric acid to dissolve the magnesium, buffered to pH 10.1, and titrated with EDTA using Eriochrome Black T indicator. The results obtained generally agree with those obtained by the standard EDTA method. Lacy (*20B*), also, described a photometric method for determining calcium and magnesium in the same sample. Eriochrome is added to a solution buffered to pH 10 and the solution titrated with EDTA at a constant flow rate; the titration is followed on a simple, semi-automatic apparatus consisting of a phototube, light source assembly, a measuring circuit for measuring the phototube output, and a recorder. Two distinct end points are observed on the titration curve. Other ions do not seriously interfere.

Januszkiewicz (*18B*) determined calcium, magnesium, and the hardness of natural waters and sewage with EDTA using Murexide and Eriochrome Black T as indicators. Interferences from other cations are eliminated with potassium cyanide. Guerello (*14B*) and Herring (*16B*) reported complexometric methods for calcium and magnesium in water. A study was made by Rial and Molins (*27B*) of the concentrations of calcium and magnesium in sea water. Calcium and magnesium were titrated with EDTA using Calcein and Eriochrome Black T indicators.

An automatic recording colorimeter was used by Demmitt and Burt (*7B*) to continuously measure water hardness. Di-sodium, magnesium EDTA, and Eriochrome Black T are added to the sample buffered to pH 10.1. The absorbance of the resulting solution is proportional to the sum of calcium and magnesium in the sample, or to its hardness up to 0.125 p.p.m. as calcium carbonate. If the pH is raised to between 11 and 12 during the chelation step, interference from iron(II), iron(III), and copper is virtually eliminated. A continuous flow-through cell assembly and a double beam photometer were used by Zizin *et al.* (*39B*) to determine water hardness. The indicator, which is not specified, is first added to the water. Then the sample flows continuously through the first cell of the photometer. Trilon B is then added continuously and the water flows through the second cell. The color difference between the two cells is measured by the instrument.

Wuenscher and Hoffmann (*38B*) described an improved reagent for rapidly determining residual hardness in boiler feed waters. The reagent consists of Calmagite, borax, sodium hydroxide, and EDTA. A tetrakisazo compound formed from benzidine and chromotropic acid was prepared by Hemmeler and Bernardini (*15B*) for use as a specific reagent for certain cations, particularly magnesium, and as a complexing indicator in the determination of water hardness. The sample is buffered to a pH of 10 with ammonium hydroxide and titrated with Complexon III using the above indicator. As little as 0.9 μg. of magnesium per ml. can be detected with this reagent in the absence of other ions.

Melamed (*23B*) used cation exchange to separate calcium and magnesium for the titrimetric determination of these ions in hard waters. A water sample, neutralized with hydrochloric acid, was passed through a column of resin. The column was washed with water, the washings and elutrient were combined and titrated with sodium carbonate solution using methyl orange indicator. The results are comparable to other methods.

Potentiometric titration of total hardness in water using a silver electrode was reported by Fritz and Garralda (*12B*). If a trace of silver is added to the sample prior to titration, magnesium, calcium, strontium, and barium can be titrated with EDTA directly at pH 9.0 to 10.5. Chloride interferes sometimes, but can be removed by anion exchange.

X-ray fluorescence spectrometry was used by Stone (*31B*) to determine strontium in tap water. Calcium and strontium were first coprecipitated as oxalates, the precipitate ignited at 500° C., and the resulting carbonates dissolved in nitric acid. The solution was then evaporated to dryness, dissolved in a rubidium chloride solution, and the intensities of the background and of the $K\alpha$ radiations of strontium and rubidium are measured. The rubidium, which is present as the internal standard, compensates for the absorption effect of the varying concentration of calcium. The results indicated that the determination of 0.5 μg. of strontium is feasible by this method. A standard addition technique was chosen by Visapaa (*36B*) for determining strontium by x-ray fluorescence. Strontium was added to the sample and counted, and the background intensity determined under the same conditions but without the addition of strontium. Strontium in the sample is determined by difference either by a calibration curve or by equations. Gloyna, Bhagat, and Felsing (*13B*) reported on the use of x-ray fluorescence for determining strontium and calcium in various materials, including water. They discussed the theory of x-ray fluorescence, experimental equipment used, test procedures, advantages, and sources of error.

Ion exchange and photometric measurement were used by Uesugi *et al.* (*34B*) to determine strontium in sea water. A measured quantity of calcium was added to a sample of sea water and the strontium coprecipitated with the calcium by the addition of oxalic acid. The precipitate was dried, ignited, dissolved with a little hydrochloric acid, and, finally, diluted with 0.3*M* ammonium hydroxide solution. The sample was then passed through a Dowex 50W-X12 ion-exchange column, calcium eluted with 1.5*M* ammonium acetate, and strontium eluted with 6*N* hydrochloric acid. The strontium elutant was evaporated to dryness, heated to remove ammonium chloride, and the residue dissolved and made up to volume with water. An ammonium chloride–ammonium hydroxide buffer and *o*-cresolphthalein complexone were added, and the absorbance measured at 575 mμ. Between 96 and 97% of this strontium was recovered and this contained less than 0.005% calcium.

A flame photometric method for determining strontium between 0.2 and 1.0 μg. per ml. based on the separation of calcium and strontium with acetone was reported by Svishchev *et al.* (*32B*). Elfers, Hallbach, and Velten (*11B*) described a flame photometric method for determining strontium. Strontium and other alkaline earths are separated from interfering ions by oxalate and nitrate precipitations. Strontium is then isolated by ion exchange and preferential chelation with Versene. Strontium is then determined flame photometrically. The detection limit is between 0.1 and 0.2 mg. per liter.

Szmytowna and Latour (*33B*) reported a gravimetric method for determining strontium in mineral waters. The sample is evaporated to dryness and fused with sodium carbonate. The residue is dissolved in hydrochloric acid, and magnesium, aluminum, and iron precipitated with ammonium hydroxide. Complexon III and ammonium sulfate are added and strontium sulfate precipitated at pH 4.5. Double precipitation is recommended to avoid coprecipitation of calcium.

Several radiostrontium methods have been reported. Morgan, Galbraith, and Gilcreas (*24B*) added a small amount of strontium carrier and sodium carbonate to the water sample and digested the floc at 90° to 95° C. The sample was cooled and all but 175 ml. of the supernatant removed. The pH was then adjusted to 4.8, a solution of EDTA–ammonium hydroxide added, and the solution filtered through Dowex AG 50W-X8. Calcium, magnesium, ammonia, and the alkalies were eluted first. Strontium was then eluted, the solution evaporated, and the dry residue transferred to a planchet and counted. Strontium-90 recovery at the 25 μμc. per liter concentration level is greater than 90%; the recovery at lower concentrations is between 80 and 90%. Ion exchange and selective precipitation of strontium from large quantities of calcium with potassium rhodizonate were used by Boni (*3B*) for the determination of total radiostrontium. Detailed procedures for determining strontium-90 in rain water were described by Osmond, Healy and Marshall (*26B*) and by Herszberg, Dutailly, and Burg (*17B*). Bakacs-Polgar and Kurcz-Csiky (*1B*) described a simple procedure for separating radioactive barium and strontium occurring in samples of rain water. Calcium is complexed with Complexon III and strontium and barium precipitated with potassium carbonate solution; the strontium is subsequently separated by controlled chromate precipitations.

Calcium-45 in cooling pond and treatment plant effluents was determined (*35B*) by adding carriers for calcium and strontium, removing strontium by successive precipitations with fuming nitric acid, and finally precipitating calcium oxalate which was β-counted.

An emission spectrographic method utilizing a plasma arc assembly for determining beryllium in oilfield waters was reported by Collins and Pearson (*6B*). Less than 1 p.p.b. of beryllium can be detected. Beryllium is extracted from the water with chloroform and acetylacetone. The standards are made up in synthetic brines and treated in an identical manner. The chloroform extracts are aspirated directly in the plasma arc and the spectra recorded on photographic plates. Shigematsu, Tabushi, and Isojima (*30B*) used acetylacetone for determining beryllium spectrophotometrically. Iron was first removed by precipitation with ammonium hydroxide at pH 8. EDTA, NaCl, and acetylacetone were added, the beryllium–acetylacetonate extracted with chloroform, and the absorbance measured at 295 mμ. Dunaeva (*9B*) described a colorimetric method for determining beryllium in water using Beryllon II. Ascorbic acid was added to reduce interference from ferric iron. Nickel cobalt, copper, magnesium, and calcium interfere but were complexed with EDTA.

Baumgartel and Kolb (*2B*) determined beryllium-7 in rain water by electronically subtracting the single components of the γ spectrum from the complex spectrum. Reproducibility was ±5%.

Between 10 and 500 μg. per ml. of barium in water can be determined by x-ray fluorescence, according to a procedure described by Visapaa (*37B*). To avoid matrix interference, heavy elements must be absent and the lighter ones present in only moderate amounts.

Spectrographic methods for the simultaneous determination of strontium, calcium, barium, manganese, titanium, and copper in waters and other materials were described by Litovchenko and Shipitsyn (*21B*).

IRON, MANGANESE, CHROMIUM, ALUMINUM, AND RHENIUM

Concentrations of iron between 0.001 and 0.1 p.p.m. in industrial waters were determined spectrophotometrically by Klump and Busch (*8C*). The sample was heated for 30 minutes at 90° C. after the addition of 2 ml. of 80% thioglycolic acid and 4 ml. of ammonium hydroxide. The sample was then cooled, ammoniated, and the iron measured.

Wilson (*24C, 25C*) and Tetlow and Wilson (*22C*) developed methods for determining "reactive," "nonreactive," and total iron content in boiler feed waters. "Reactive" iron is extracted into isopentanol as the red iron bathophenanthroline complex and its absorbance measured at 534 mμ. The detection limit is about 0.4 μg. per liter using a 200-ml. sample. The standard deviation is from ±0.2 to ±0.5 in the range 0.0 to 50 μg. of iron per liter. Several methods were investigated for determining "nonreactive" iron. They preferred the method of Klump and Busch (*8C*), and measurement at 530 mμ. For total iron content the "nonreactive" iron is converted to "reactive" iron with thioglycolic acid, then determined spectrophotometrically by a modification of the iron bathophenanthroline method. The detection limit and standard deviation is slightly higher than that for "reactive" iron alone. Kostrikin, Kalinina, and Dzysyuk (*9C*) reported on the difficulties of the sulfosalicylate method for iron in boiler waters. As little as 1 p.p.b. copper interferes and organics must be removed by heating to 500° to 600° C. Samples should not be stored for long periods of time and care must be taken when pipeting a sample if coarse particles are present in the water. Filtering the sample is also recommended. The filter paper and residue are also analyzed for iron.

A spectrophotometric method for determining iron in water with α-pyridil dioxime was described by Wakimoto (*23C*). At a pH of 7.4 to 7.8 a stable red-purple complex is formed with maximum absorbance at 595 mμ. Calcium, zirconium, thallium, cobalt, and copper interfere with the determination. The calibration curve is linear from 0.0 to 4.0 p.p.m. Shidlovskaya-Ovchinnikova (*19C*) described a method for determining nonionic iron in natural waters. The method offers several changes which improve and simplify a wet combustion method previously reported by the same author (*18C*). Oxalic acid is added dropwise to the hot solution until decoloration occurs. A universal indicator replaces thymol blue indicator. Freshly prepared sodium sulfite is used to reduce ferric iron, and bipyridine is used to determine the iron after the wet combustion. A bipyridine method was also used by Mokievskaya (*13C*) to determine ferrous, ferric, and organic iron in sea and interstitial waters. A photometer, consisting of a double-beam optical system, an amplifier, and a continuous flow cell assembly, was described by Ito and Musha (*7C*) for determining iron in water using thiocyanate. The results agreed with those obtained by a conventional spectrophotometer. The range of iron concentration varied from 1.87 to 8.99 p.p.m.

A catalytic method for determining iron was proposed by Kreingol'd *et al.* (*10C*). The method is based on the fading of the color of Stilbexon (a new luminescent reagent) in an acid medium in the presence of hydrogen peroxide and ferric iron. Preparation of the luminescent reagent is given. The maximum absorbance is between 420 and 480 mμ. The rate of oxidation is effected by pH, temperature, and peroxide concentration.

Potentiometric titration with either platinum foil or platinized porcelain electrodes was used by Zavodnov (*26C*) to determine ferrous iron in water. Sulfuric acid and iodine are added to two identical samples. One is titrated directly with sodium sulfite and the other titrated after the addition of Trilon B. The iron content of the sample is determined from the difference of the two titrations. Concentrations of iron between 1 and 100 p.p.m. were determined by Hellwig and Steenderen (*6C*) by direct EDTA titration. Iron must first be oxidized. Zinc, 5 p.p.m., and copper, 0.2 p.p.m., interfere. A 100-ml. sample was adjusted to pH 2.5 and 1 ml. indicator (sulfosalicylic acid in water) added. The solution was then heated to 50° C. and titrated until the color changes from red to colorless.

A fluorometric and a spectrophotometric method for determining micro amounts of aluminum between 0.00 and 0.50 p.p.m. in industrial waters were reported by Noll and Stefanelli (*15C*). Aluminum is first complexed with sodium fluoride and interfering substances are removed by a weak cation-exchange resin. A solution of 8-quinolinol is added to the filtrate and the aluminum complex extracted with chloroform. Aluminum is then determined fluorometrically or spectrophotometrically at 380 mμ. The standard deviation is ±0.02. Albinati (*1C*) determined aluminum by measuring the fluorescence of the reaction product of aluminum and Pontachrome Blue-Black-R. Absorption maxima were observed at 520 and 560 mμ, but the optimum wavelength was 605 mμ. Beer's law is followed up to 0.2 μg. of aluminum per ml. and the sensitivity is 0.0001 μg. of aluminum per ml. The effects of pH, solvent, reagent concentrations, heating time, and fluorescence stability of some 70 ions were also studied.

Lisenko, Mustafin, and Molot (*12C*) used Chromoxane Violet R to determine iron and aluminum in natural waters. At pH 6 the complexes of ferric iron and aluminum are pink-violet and red, respectively; however, iron and aluminum absorptions are additive. Aluminum was determined separately after adding ascorbic acid to complex the iron; iron is calculated by difference. Solomin and Fesenko (*20C, 21C*) described a complexometric method for determining ferric and ferrous iron, and aluminum in acid waters. Heavy metals are first removed by extraction with sodium diethyldithiocarbamate and carbon tetrachloride. A portion of the aqueous filtrate is adjusted to pH 1.0 to 1.5, heated to between 60° and 70° C., and ferric iron determined by EDTA titration with sulfosalicylic acid indicator. Ferrous iron can be determined separately after oxidation with ammonium persulfate. Ammonium hydroxide is added to an aliquot of sample to precipitate aluminum and ferric hydroxides. The residue is washed, dissolved in dilute hydrochloric acid, and the solution buffered to pH 4. Excess EDTA is then added and the solution titrated with zinc chloride solution using dithizone indicator. The aluminum content is calculated from the difference between the latter determination and the ferric iron determination. Calcium and magnesium is also determined in the aqueous filtrate using EDTA.

Goto, Komatsu, and Furukawa (*4C*) reported a modified formaldoxime method for manganese which eliminates interference from iron. After the formation of the iron and manganese formaldoxime color, the iron formaldoxime is decomposed with EDTA and hydroxylamine. No colored complex is formed by either iron or manganese if EDTA is added first.

Gustin and Sweet (*5C*) proposed a spectrophotometric method for manganese involving the reaction of manganese with 8-aminoquinoline to form a colored oxidation product which is extracted into a mixture of benzyl alcohol and chloroform. The absorbance of the complex is measured at 550 mμ. Care must be taken since the oxidation and extraction are pH sensitive. The sensitivity of the method is 0.006 p.p.m. manganese. Other oxidizing metals and high concentrations of chloride interfere. A qualitative test for manganese was reported by Barker (*3C*). Potassium iodide is added to a water sample and three drops of $(4\text{-}Me_2NC_6H_4)_2CH_2$ solution are added after 1 minute. If manganese is present, a blue color develops to maximum intensity in 30 seconds and then fades. Chloride, iron, and chloramine, in moderate amounts, do not interfere. The sensitivity is somewhat influenced by pH.

Plsko (*16C*) and Plsko and Blazej (*17C*) pointed out that certain organic substances interfere with the usual polarographic or colorimetric methods for determining chromium in tannery wastes. They described a spectrographic procedure in which organics do not interfere. A rapid qualitative method for sexivalent chromium, using a powdered reagent of diphenylcarbazide and oxalic acid, was reported by Bahensky, Zika, and Lastovkova (*2C*). The reagent, added to a 25-ml. sample, develops a pink to red-violet color if 0.2 p.p.m. of chromium is present.

Nechaeva (*14C*) described an extraction–colorimetric method for determining rhenium in natural waters. Rhenium is first concentrated on activated charcoal, and then extracted with methyl ethyl ketone from 5.5*N* sodium hydroxide solution. The color of an extracted rhenium-rhodanide complex, formed after reduction with stannous chloride, is then compared with standards. As little as 2 μg. of rhenium per liter can be detected.

Krusenstjern and Harst (*11C*) reported on the accuracy of colorimetric methods for determining chromium, iron, zinc, nickel, and copper in galvanic process wastes.

COPPER, ZINC, LEAD, CADMIUM, AND NICKEL

Ballinger and Hartlage (*2D*) described polarographic procedures for copper, cadmium, and zinc. The results obtained are similar to those of colorimetric procedures but the polarographic techniques are simpler. Each sample is evaporated to dryness, dried, and ashed at 600° C. The residue is dissolved in hydrochloric acid and the elements are determined polarographically. Dobychin and Aleskovskii (*9D*) proposed using metallic magnesium to concentrate trace amounts of copper, lead, zinc, and cadmium from large volumes of water. The magnesium is removed from the water, dissolved in hydrochloric acid, and the above metals are determined polarographically. A technique is given for the determination of these ions in the presence of iron, and a procedure for determining nickel. The relative error is about 20%. Rozenfel'd (*22D*) described polarographic methods for copper, lead, and zinc. The ions are extracted from a 1-liter water sample with dithizone in either carbon tetrachloride or chloroform. Copper is extracted first after acidification of the sample. Lead and zinc are extracted together at pH 8. The ions are then back extracted into hydrochloric acid solution. The lead and zinc extract is divided into two portions for the polarographic determination of each. Concentrations as little as 0.05 mg. of copper or zinc per liter and 0.1 mg. of lead per liter can be determined. Ariel and Eisner (*1D*) applied anodic stripping voltammetry to the determination of trace metals in brines and were able to detect zinc and cadmium at concentrations of $5.7 \times 10^{-7}M$ and $4.4 \times 10^{-8}M$, respectively. The standard deviation for the zinc determination was 3%; copper and lead were detected qualitatively.

A plasma arc was used by Sambueva and Shipitsyn (*23D*) to determine manganese and copper spectrographically in natural waters. Serious errors arise if the samples are first filtered, since the insoluble materials retain considerable amounts of trace elements.

Rona *et al.* (*21D*) investigated neutron activation for determining zinc and manganese in sea water. Zinc and manganese were coprecipitated from sea water with iron hydroxide. Diethyldithiocarbamate was then used to separate divalent manganese and zinc, and each determined by neutron activation.

Natelson, Leighton, and Calas (*19D*) attempted to determine manganese, iron, cobalt, copper, nickel, and zinc in sea water by x-ray spectrometry, but were able to detect only iron, nickel, copper, and zinc at the observed detection limit of 2 μg. Fabricand *et al.* (*11D*) determined certain trace metals in sea water by atomic absorption flame photometry. The technique was capable of detecting concentrations of 1 p.p.b. or less of nickel, iron, manganese, zinc, and copper.

Boichinova and Aleskovskii (*4D*) determined nickel and copper in natural waters by ascending paper chromatography. The paper is soaked with either dimethylglyoxime or dithiooxamide for nickel, and with either diethyldithiocarbamate or dithiooxamide for the copper determination. As the cation moves upward with the solvent, a colored peak forms, the height of which is proportional to the concentration of nickel or copper. Complete details were given by Chistyakov and Blagoveshchenskaya (*7D*) for determining copper, cobalt, and nickel by paper chromatography, both quantitatively and semiquantitatively.

Comte *et al.* (*8D*) reported a new colorimetric reagent, monophenyloxaldihydrazide, for determining copper. Two methods were presented: the first, a direct determination, estimates copper concentrations from a calibration curve prepared from solutions of known copper concentrations; the second is based on the standard addition principle. The results of the two methods agreed closely. The colored complex formed between copper and oxaldihydrazide in ammonia solution was described by Billikova and Billik (*3D*), and was used as the basis for a sensitive and selective photometric method; only cobalt above 1 p.p.m. interferes. Bougis (*5D*) compared the diethyldithiocarbamate and the 2,2′-diquinolyl methods for the determination of copper in sea water. Comparable results were found in the range of 0.1 to 20.0 μg. of copper per liter, the deviation being of the order of ±0.2. For normal water, the diethyldithiocarbamate method is simpler and easier, whereas, the 2,2′-diquinolyl method is more specific and more applicable to interstitial and polluted waters. The dibenzyldithiocarbamate, diethyldithiocarbamate, and neocuproine methods were compared for sensitivity, reproducibility, interference, simplicity, and reagent cost by Hissel and Cadot-Deithier (*12D*) for their usefulness in determining copper in boiler waters. The cupric ion complexes directly with the first two reagents; however, with the latter reagent, copper must first be reduced. In all three methods, the complexes are extracted with either carbon tetrachloride or chloroform and the absorbances measured. The first-named reagent was found to be most suitable. Wilson (*25D*) used the dibenzyldithiocarbamate method to determine copper in boiler feed waters, and described details of the procedure. The limit of detection is 0.3 μg. of copper per liter, and the standard deviation is ±0.2.

A fluorometric method for determining between 0.05 and 0.50 μg. of copper in 5 to 50 grams of water was described by Kreingol'd and Bozhevol'nov (*14D*). A sample aliquot is evaporated, treated with hydrochloric acid, and buffered to pH 6, and is titrated with Fluorexon to an end point of persistent fluorescence. There is no interference from manganese, iron, cobalt, and nickel. Bozhevol'nov and Kreingol'd (*6D*) used lumocupferron to determine, fluorometrically, less than 0.2 μg. of copper in 5 to 50 ml. of water. The sample is evaporated to dryness, dissolved with 2 drops of 0.1*N* hydrochloric acid and water, lumocupferron reagent added, and the fluorescence measured. There is no interference from less than 0.1 μg. per ml. amounts of iron, cobalt, nickel, and manganese.

Snow (*24D*) described spot tests for identifying certain inorganic substances, and an electrographic test for copper.

Nehring (*20D*) reported a new method for detecting less than 0.4 mg. of zinc per liter in water. Ammonium thiocyanate and pyridine are mixed with the sample, shaken with chloroform, and filtered. The zinc complex, if present, remains on the filter paper and will give a green color when a drop of copper sulfate is added.

Loveridge and Owens (*16D*) described a method for determining zinc-65 in water. Lead is first coprecipitated with strontium sulfate. The solution is then adjusted to pH 4.5, saturated with carbon tetrachloride and chloroform, and passed through a column of dithizone on cellulose acetate, which retains the zinc. Zinc is eluted with *N* HCl, and scavenges with cobalt, ruthenium, and iron are carried out. Tartaric acid is added to the solution, the pH adjusted to 4.5, and zinc precipitated as the quinaldinate and γ-counted.

Markova (*18D*) described a photometric method for determining lead in fresh and mineralized waters. Lead is first extracted from basic solution with dithizone in carbon tetrachloride, and reextracted into an aqueous phase with hydrochloric acid. Potassium ferrous cyanide, sodum tetraborate, and sulfarsazene are added and, after 30 minutes, the absorbance is measured. Carbon dioxide interferes and must be removed by boiling with hydrochloric acid. Lukin *et al.* (*17D*) used arsazen to determine lead. An aliquot of water is made acid to Congo red with hydrochloric acid. Sulfosalicylic acid and thiourea are added to mask interferences, the sample buffered with ammonium hydroxide–ammonium chloride, and extracted with arsazen in butyl alcohol. The color is compared to standard solutions containing 0.5 to 10 μg. of lead.

Kyuregyan (*15D*) showed that Plumbon, a new colorimetric reagent, can be used to determine 0.01 mg. or more of lead in water. Eight micrograms of iron and molybdenum, and 20 μg. of copper per liter interfere.

A spectrophotometric method for determining between 2 and 30 μg. of cadmium in sea water was reported by Ishibashi *et al.* (*13D*). Ten to 20 liters of sea water are acidified with hydrochloric acid. Cupric ion is added and copper and cadmium are precipitated with hydrogen sulfide. The precipitate is dissolved in aqua regia, evaporated to dryness, treated with hydrochloric acid, and passed through a cation-exchange column of Dowex 50W-X8. Cadmium is eluted and separated from the copper and any iron present with 0.5*N* hydrochloric acid. Cadmium is then extracted with 1-(2-pyridylazo)-2-naphthol (PAN) in benzene and the absorbance of the complex measured at 555 mμ.

Evstifeev and Kovalenko (*10D*) described a procedure for determining traces of nickel between 0.001 and 0.01 p.p.m. in natural water. The nickel is concentrated by deposition with zinc powder and subsequently determined photometrically with dimethylglyoxime. Copper, lead, cobalt, cadmium, sodium chloride, and magnesium sulfate do not interfere.

SILVER, GOLD, AND MERCURY

Uman (*3E*) described a spectrographic procedure for determining between 0.01 and 0.50 μg. of silver in samples of potable waters. An aliquot of water is diluted with a lithium sulfate matrix solution and a palladium internal standard. The sample is evaporated to dryness and a spectrogram made from the residue. A working curve is obtained from standards prepared in the same manner. Other ions found in potable waters do not interfere. Markova and Astaf'eva (*1E*) used a dithizone procedure to determine 3 μg., or less, of silver per liter. If more than 0.5 μg. per liter of mercury is present, it must be reduced with silver nitrate and ascorbic acid, a correction made for the silver so added, and the mixture than extracted with dithizone in carbon tetrachloride. Sulfuric acid and Complexon solutions are added and the color of the extract is compared with standards.

Weiss and Lai (*5E*), using radiotracers, investigated the cocrystallization of ultramicroquantities of 25 diverse elements from solutions of different hydrogen-ion concentrations with 2-mercaptobenzimidazole. High yields were obtained for tin, mercury, silver, tantalum, and gold. They applied this technique to the determination of gold in sea water. The gold in the residue is determined spectrophotometrically at 470 mμ using *p*-dimethylaminobenzalrhodanine and radiometric corrections made to determine the chemical yield. The concentration of gold found in sea water was 0.068 ± 0.003 μg. per liter. Pakhomova and Vysotskaya (*2E*) used a dithizone extraction method to determine gold in natural waters. The dithizone was specially purified in a mixture of 3% ascorbic acid and dilute ammonium hydroxide. The sensitivity of the determination was 0.00002 to 0.00003% with a relative standard deviation of less than 20%.

Vasilevskaya, Shcherbakov, and Levchenko (*4E*) described procedures for determining cationic, anionic, and total mercury in water. To determine cationic mercury, the sample is first acidified to 1*N* with sulfuric acid, and then saturated with chloroform and masking agents added to prevent interference from other ions. Finally, the mercury is extracted with dithizone in chloroform and the absorbance of the dithizone layer is measured at 490 mμ. To determine anionic mercury, the sample is saturated to pH 13 to 14 with sodium hydroxide and sodium sulfide to form the HgS_2^{-2} complex. This complex is stable and the mercury can be determined with dithizone after evaporating to dryness. Total mercury is determined by first boiling the sample with ammonium persulfate or potassium nitrate, and then proceeding as for anionic mercury.

VANADIUM, MOLYBDENUM, TITANIUM, ZIRCONIUM, NIOBIUM, TIN, GERMANIUM, AND URANIUM

An indirect spectrophotometric method for determining vanadium in natural waters was reported by Fishman and Skougstad (*3F*). It is based on the catalytic effect of vanadium on the rate of oxidation of gallic acid by persulfate in acid solution. The absorbance of the oxidation product of gallic acid is measured at 415 mμ and compared with standards treated in an identical manner. Optimum conditions are given for concentration of reactants, temperature, and reaction time. Concentrations in the range of from 0.1 to 8.0 μg. of vanadium per liter may be determined with a standard deviation of 0.2 or less. Possible interferences by substances commonly found in natural waters were investigated. Brudz *et al.* (*2F*) described a new reagent, sulfonazo, which gives a highly sensitive reaction with vanadium. As little as 0.05 μg. of vanadium per 5 ml. can be detected in water. A blue color is formed between vanadium and sulfonazo at pH 4.4 to 4.6, which shows maximum absorbance between 620 and 640 mμ. Sodium fluoride and thiourea are added to mask interferences. Other organic reagents containing the same reactive group as sulfonazo can be used. To determine vanadium in sea water, Suzuki and Takeuchi (*15F*) coprecipitated vanadium with ferric and ammonium hydroxides. The residue was dissolved in hydrochloric acid, sulfuric acid added, and the solution evaporated to white fumes. The residue was redissolved in water, potassium permanganate added, the vanadium extracted with *N*-benzoyl-*N*-phenylhydroxylamine in chloroform, and the absorbance of the organic layer measured at 530 mμ.

Konovalov (*9F*) described a procedure for determining molybdenum in natural waters in which molybdenum is coprecipitated with $MnO(OH)_2$ from a 0.5- to 1-liter sample. Subsequently, the molybdenum is complexed, extracted with an isoamyl alcohol–carbon tetrachloride mixture, and the absorbance of the extract determined on a photocolorimeter or spectrophotometer.

A spectrophotometric method for determining traces of titanium was reported by Herrmann (*7F*). Titanium is extracted from brines at pH 3 with 8-quinolinol in the presence of hydrogen peroxide. Titanium thiocyanate is then formed, extracted into trioctylphosphine oxide, and the absorbance measured at 432 mμ.

A method for determining zirconium-95 and niobium-95 in seaweed and sea water was described by Hampson (*6F*). Both elements are complexed with oxalic acid in nitric acid solution. Phosphoric acid and potassium chlorate are added and the oxalic acid destroyed, forming a precipitate of zirconium phosphate and niobic acid. Rare earth activities are removed with a double lanthanum fluoride scavenge, and zirconium is separated as barium fluorozirconate. After decontamination from other active isotopes, the residue is ignited to zirconium oxide. Similarly, niobium oxide is formed and the activities of each isotope determined by gamma-ray spectrometry.

Hamaguchi *et al.* (*5F*) have developed a neutron activation method for tin of general applicability for a variety of samples, including sea water. The samples and standards are sealed in ampoules, irradiated intermittently for three days, followed by carrier radiochemical separations consisting, mainly, of solvent extraction steps. The chemical yield is about 50% and as little as 0.1 μg. of tin can be determined.

Ilica (*8F*) described a new method for determining germanium in mine waters. The sample is acidified with hydrochloric acid, and the germanium extracted first by carbon tetrachloride and then reextracted into distilled water The aqueous solution is treated with gelatin and phenylfluorone, and the concentration of germanium determined colorimetrically. The results obtained were comparable to those obtained by coprecipitation of the germanium with ferrous hydroxide.

Singer and Marecek (*14F*) used Arsenazo III for determining as little as 1 μg. of uranium per liter. Uranium (VI) is first separated by coprecipitation with aluminum hydroxide, and the precipitate redissolved in hydrochloric acid. A bismuth reductor and ascorbic acid reduce the uranium(VI) to uranium (IV), Arsenazo III reagent is added, and the resulting blue complex measured at 665 mμ. This same reagent was also used by Reznikov and Mulikovskaya (*12F*) to determine uranium in natural waters. They separated uranium(IV) from interfering ions (iron and aluminum) by absorption on activated charcoal and elution with sodium carbonate. The uranium was then complexed with the arsenazo and determined spectrophotometrically. A fluorescence method was also reported by Reznikov and Mulikovskaya (*13F*). The sample is pretreated identically as above to remove interferences. The uranium is then fused with sodium fluoride in a platinum-wire loop and comparison made with standard beads under ultraviolet light.

Rapid chromatographic methods for estimating uranium concentrations in natural waters were described by Alberti, Grassini, and Di Ferrante (*1F*), and by Grassini and Alberti (*4F*). Ascending paper chromatography with isobutyl-methyl ketone–nitric acid separates uranium from interfering ions. The paper is treated either with sodium phosphate and then zinc acetate in a sodium acetate–acetic acid buffer, or with phosphoric acid and then zinc acetate-acetic acid. The yellow-green fluorescence of the chromatogram is compared with standards. As little as 0.001 μg. can be detected in a dilution of 1 p.p.m.

A differential luminescence photometer was constructed by Nikolov and Kamburov (*11F*) to determine the uranium concentrations in water from various sources. The uranium is fused with sodium carbonate and sodium fluoride after purification to eliminate quenchers.

An automatic polarograph was installed by Koyama, Michelson, and Alkire (*10F*) for continuous measurement of uranium in process-waste streams. The solution is analyzed directly with a dropping mercury electrode without either dilution, addition of supporting electrolyte, removal of dissolved oxygen, or control of temperature.

BORON, SELENIUM, AND TELLURIUM

Greenhalgh and Riley (*7G*) modified the curcumin method for boron to improve the reproducibility and applied the method to the analysis of boron in sea water. A sample containing less than 1.0 μg. of boron is pipeted into a reaction tube. Oxalic acid and curcumin are added, and the mixture is heated in an oil bath at 120° C. for 90 minutes. The reaction tube is removed, the sample diluted with ethanol, and the absorbance of the boron oxalate–curcumin complex measured at 548 mμ. The major ions found in sea water do not interfere to any extent. Rowe (*12G*) studied the effect of fluoride on the determination of boron by the dianthrimide method of Rainwater and Thatcher (*10G*). He found that no interference occurs when the amount of fluoride present is 1 mg. or less, since the fluoride is removed by evaporation with sulfuric acid.

Vera (*13G*) determined between 5 and 60 mg. of boron per liter, by an indirect spectrophotometric method after preliminary distillation of boron as methyl borate. Boron, complexed with potassium fluoride in acid solution, will reduce sodium molybdate to molybdenum blue. The absorbance is measured at 741 mμ after the reaction has proceeded for 60 minutes. Glebovich (*6G*) used both a titrimetric and a photometric carmine method to determine boron in fresh, salt, and petroleum waters. If the boron concentration was greater than 2 mg. per liter the titrimetric method was used. The method involved the titration of boric acid with 0.1*N* alkali using phenolphthalein indicator, with an error of less than 5%. With the photometric method, 0.1 to 1.0 mg. of boron per liter was determined with an error of less than 25%.

A volumetric method was used by Murakami (*9G*) for determining boron in sea water, brines, and bittern. The sample was acidified with hydrochloric acid and then neutralized to methyl orange with sodium hydroxide. Mannitol, potassium iodide, potassium iodate, and sodium thiosulfate were then added, and the solution titrated with iodine, using starch indicator.

Korobov and Lipovskii (*8G*) recommended the use of copper electrodes for the spectrographic determination of boron since carbon electrodes may contain boron. Details were given for preparation of the samples and for excitation conditions. Standards were prepared in a matrix similar to the object studied. For natural waters, they used pure salts of sodium chloride, calcium hydroxide, magnesium oxide, calcium carbonate, and sodium bromide.

A detailed investigation of the use of ion-exchange resins in methods for determining boron in natural waters was described by Eristavi, Brouchek, and Cheishvili (*5G*), Eristavi and Brouchek (*3G*, *4G*), and Brouchek (*1G*). Boron was first passed through a strong cation-exchange resin to remove ammonium calcium, magnesium, iron(II), iron(III), aluminum, and other cations. The eluate was adjusted to pH 2 and passed through a weak anion-exchange resin to remove sulfate, chloride, nitrate, phosphate, and some silica, and the effluent analyzed for boron by conventional volumetric or colorimetric methods. When the boron concentration was low, a third strong anion-exchange resin was used to concentrate the boron. The methods were checked under both laboratory and field conditions. Ion exchange increased the sensitivity significantly.

Rouquebert and Vitte (*11G*) described a method for determining selenium in natural waters using 3,3′-diaminobenzidine-hydrochloride. One to 10 liters of water were evaporated, reduced with hydrobromic acid solution, and the sample distilled. Bromine was removed from the distillate with phenol, and the distillate neutralized to pH 7 with ammonia. Finally, the pH was adjusted to 2.5 with acetic acid, 3,3′-diaminobenzidine added, and after two hours the selenium complex extracted with methyl phenol and the absorbance measured at 420 mμ.

Collins, Waters, and Pearson (*2G*) described procedures for the determination of selenium and tellurium in oilfield waters. Selenium may be determined by one of two methods: (1) semiquantitatively by reduction to elemental selenium with sulfur dioxide and comparing the red amorphous form with standards, or (2) quantitatively by reduction to selenite with concentrated hydrochloric acid, and reaction with 3,3′-diaminobenzidine. Tellurium may be determined by reducing it to the elemental form with hypophosphorus acid, and measuring the absorbance of the resulting red hydrosol at 400 mμ. Unfortunately, a typographical error in the formula for calculating selenium and tellurium concentrations, and an incongruity in the tables of data result in an apparent sensititivity for the method greater than is actually the case.

CHLORIDE, BROMIDE, AND IODIDE

The decoloration by choride of the bluish-violet diphenylcarbazone–mercury complex was the basis of a photometric method developed by Zimmermann and Stocmeier (*20H*) to detect 0.05 to 0.6 mg. of chloride per liter in 10-ml. samples. Tomonari (*16H*) also described a diphenylcarbazone method for chloride and reported that the complex of diphenylcarbazone and mercury is unstable but may be extracted into benzene and the absorbance measured. Between 0.01 and 0.2 p.p.m. chloride can be determined, but a number of ions interfere, including bromide, iodide, cyanide, thiocyanate, sulfide, thiosulfate, copper, silver, and ferric ions. Tomonari (*17H*) improved the mercuric thiocyanate method for determining 0.1 to 15 p.p.m. chloride. By increasing the concentration of mercuric thiocyanate and ferric iron, a higher sensitivity and a nearly straight calibration curve is obtained. The absorbance is measured at 460 mμ using a 1-cm. cell.

Woelk (*18H*) described a method for determining 2 to 100 μg. of chloride in very pure water. The method consists of oxidizing chloride to chlorine with concentrated sulfuric acid and potassium periodate. With a stream of hydrogen, the chlorine is swept into an absorption cell containing potassium cyanide, barbituric acid, and pyridine. The chlorine reacts with the potassium cyanide to form the halogen cyanide, which forms a colored complex with pyridine and barbituric acid.

Panasyuk and Yaroshevich (*13H*) improved the sharpness of the end point of the mercurimetric titration method for chloride by the use of a combined indicator (diphenylcarbazone + malachite green). A nephelometric method was used by Palei and Udal'tsova (*12H*) to determine chloride concentrations between 2 and 100 μg. per liter. Acetone was used to decrease the solubility of the silver chloride precipitate formed by the addition of a silver nitrate–lead nitrate solution. The relative absorbance is measured with the use of a green filter after the solutions have stood for 20 minutes in the dark, and in chlorine-free atmosphere. Temperature also affects the accuracy.

The effects of variations in concentration, temperature, and pressure on the e.m.f. of the Ag/AgCl and other electrode systems suitable for the determination of chloride in sea water are discussed by Koske (*9H*). Kalvoda and Gladyshev (*7H*), and Gladyshev and Kalvoda (*3H*), described the use of oscillographic polarography for determining chloride, bromide, and iodide in waters.

Zitomer and Lambert (*21H*) determined bromide spectrophotometrically as a function of its inhibition on the chlorination of ammonia. Preliminary cation exchange was used if iron and ammonia (in excess of 0.01 p.p.m.) are present. The sample was then adjusted to pH 5.6 and ammonia and hypochlorite were added. After 3.5 minutes, sodium nitrite and cadmium iodide–starch solution were added, and the absorbance read at 615 mμ. The determination is rapid and applicable to concentrations of bromide in the range of 0.02 to 1.20 p.p.m.

Bystritskii, Aleskovskii, and Bardin (*1H*) developed a new potentiometric method for determining part-per-billion levels of bromide in water. The difference in the potentials of a Ag/AgBr electrode between the sample and a reference solution was used to calculate bromide concentrations. A description of the Br-Ag electrode used is given in detail. The reference electrode is Hg | Hg$_2$SO$_4$ | H$_2$SO$_4$, with 0.01N H$_2$SO$_4$ as the reference solution.

Proskuryakova, Shveikina, and Chernavina (*14H*) compared titrimetric, colorimetric, and kinetic methods for the determination of iodine. The thiocyanate–nitrite (kinetic) and Brilliant Green (colorimetric) methods were the most useful based on sensitivity, precision, and time for analysis. Yatsimirskii *et al.* (*19H*) also described a thiocyanate–nitrite catalytic method for determining as little as 0.001 μg. of iodide per ml. in mineral waters, brines, and river and supply waters. A modified ceric–arsenite method was used by Stolc (*15H*) to determine iodine in water and a variety of other materials. After the reaction had proceeded for 20 minutes at 40° C., the reaction tube was cooled to 4° C.; after 10 minutes brucine acetate solution was added, and the mixture warmed at 100° C. for 15 minutes. The orange color which developed was then measured at 430 mμ. The recovery of known amounts of iodide averaged 95%.

A simple, rapid, and automatic procedure based on the catalytic effect of iodine on the reduction of cerium(IV) by arsenite for determining 1 to 50 μg. of iodide per liter was described by Hadjiionnou (*4H*). An automatic spectrotitrator with dial readout was used to record the results. Known amounts of iodide added to 4 water samples gave recoveries ranging from 97 to 102%.

Other investigators, including Ceausescu (*2H*) and Milicevic, Knezevic, and Custovic (*10H*), reported modifications of the ceric sulfate–arsenite method, and Miller and Shneider (*11H*) described a catalytic titrimetric method sensitive to 2.5 μg. of iodide per liter.

Feigl's catalytic reaction, based on the oxidation-reduction between chloramine T and tetra base-acetate, was used by Jungreis and Gedalia (*6H*) for the rapid determination of 0.0025 to 0.02 μg. of iodide per ml. A 10-ml. aliquot was buffered to pH 4.8, and tetra base-acetate added. Chloramine T was then added, mixed, and the absorbance at 600 mμ measured after 30 seconds.

Kobyak and Bondareva (*8H*) developed a new titrimetric method for determining iodide in the presence of large amounts of chloride and bromide. Iodide is titrated directly in acid solution using calcium hypochlorite. Two reactions occur; first, iodide is oxidized to free iodine, and second, iodine is oxidized to iodine mono-chloride.

Iwasaki, Utsumi, and Kang (*5H*) described a solvent extraction method for determining perchlorate in sea water. A 1:1 ratio methylene blue: perchlorate complex is extracted from acid solution with 1,2-dichloroethane and the absorbance measured at 655 mμ. A single extraction yields about 95% of the complex. Most interferences can be minimized by masking with mercuric ion, or by a backwash with sulfuric acid.

FLUORIDE

Banerjee and Chowdhury (*1J*) described an ion-exchange titrimetric method for determining fluoride in water. The sample is passed through a cation-exchange resin and the eluate titrated with thorium nitrate using the sodium salt of 2-(*p*-sulfophenylazo)-1,8-dihydroxynaphthalene - 3,6 - disulfonic acid as indicator. Ion exchange was also used by Kelso, Matthews, and Kramer (*4J*) for determining fluoride. The fluoride was adsorbed on an anion-exchange resin, Dowex 2X-8, and eluted with an acidic beryllium solution. Sodium 2 - (*p* - sulfophenylazo) - 1,8-dihydroxynaphthalene - 3,6 - disulfonate was then added to the eluate and the absorbance measured at 570 mμ. The ion exchange eliminated the need to distill the sample to remove interfering ions. If aluminum was present, ammonium-EDTA and sodium hydroxide were added to the samples and standards prior to the ion-exchange step. Cation exchange and precipitation of sulfide, chloride, and sulfate was used by Li (*7J*) for treatment of water samples prior to determining fluoride using Xylenol Orange. Thorium–Xylenol Orange complex was added to the treated sample, the solution adjusted to pH 3, and the absorbance measured at 560 mμ. Between 2 and 12 p.p.m. fluoride can be determined.

A method for concentrating fluoride with a suspension of magnesium hydroxide was described by Shapiro and Kolesnikova (*13J*). The suspension is prepared by adding sodium hydroxide to a solution of magnesium sulfate, heating, decanting, and washing the magnesium hydroxide suspension with water to remove traces of sulfate. To concentrate fluoride, 20 ml. of suspension is added to 250 ml. of water, the solution is heated, and after the precipitate settles, the solution is decanted. The precipitate is then dissolved with nitric acid and the fluoride determined by the thorium–alizarin or other convenient titration method. The results obtained are comparable to those obtained by the classical distillation procedure.

Beyermann (*2J*) developed an electrochemical procedure, using an aluminum anode and a platinum or aluminum cathode, for determining fluoride in water and other materials. Fluoride must first be separated by ion exchange or distillation since many other ions react with the aluminum anode. A voltage (1000 volts) was applied between the electrodes in a solution containing 5% potassium nitrate and 1% glycine. The addition of fluoride causes an increase of current proportional to the concentration of fluoride. The detection limit is $1 \times 10^{-5}\%$ in a 1-gram sample. Sensitivity is dependent upon thickness of oxide layer on the electrodes, area of the anode, pH, concentration of electrolytes, and temperature.

A simple colorimetric method was reported by Kobayashi (*6J*) for determining fluoride in water. Iron 5 p.p.m., aluminum 0.2 p.p.m., sulfate 40 p.p.m., and phosphate 0.25 p.p.m. do not interfere. Details are given for preparing the zirconium–quinalizarin reagent which is added to samples and standards and comparison made after 20 minutes. Fluoride concentration below 3.5 mg. per liter can be determined to within 0.1 mg. per liter.

Meyling and Meyling (*8J*) modified the zirconium–alizarin method for determining fluoride in natural water. The free alizarin formed when fluoride is present was extracted with pentanol and the absorbance of the organic layer measured at 430 mμ. To compensate for interfering ions, a known amount of fluoride was added to one of two identical samples before adding the other reagents. The method is accurate to ±0.05 p.p.m. fluoride. To determine fluoride in waste waters, Khosht and Lyashcheva (*5J*) coagulated the turbidity and color of these waters with sodium hydroxide and ferric chloride. The samples were then filtered and fluoride determined using zirconium–alizarin reagent. Palin (*9J*, *10J*) suggested certain modifications in the zirconium–alizarin method which result in a more intense color and in elimination of interferences due to alkalinity, chloride, iron, sulfate, color, turbidity, aluminum, and residual chlorine. The reagents were prepared as stable pellets.

Quentin (*11J*) separated fluoride from interfering ions by steam distillation. He first evaporated up to 1 liter of water containing 10 to 50 μg. of fluoride to a volume of about 10 to 50 ml. The sample was then acidified and steam distilled. Fluoride was determined in the distillate by either the zirconium–alizarin or zirconium Eriochrome Cyanine methods. Bhakuni and Sharma (*3J*) reported that aluminum does not interfere in the zirconium Eriochrome Cyanine method for fluoride if the temperature is elevated slightly (35° C.). Using this information, they described a method for estimating fluoride in the presence of aluminum. Sarma (*12J*) investigated the Megregian method and found that the calibration curve is linear only up to 1 or 1.4 p.p.m. By modifying the relative proportions of reagents, up to 2 to 3 p.p.m. can be determined with confidence, and it is possible to achieve maximum sensitivity and reliability for any concentration range.

SULFATE AND SULFIDE

Bulusu and Sen (*3K*) compared turbidimetric, titrimetric, and ion-exchange methods for determining sulfate in water. They preferred the ion-exchange method since it is simple, inexpensive, and accurate to ±1.0 mg. of sulfate per liter in the absence of organic acid anions. The direct titration of sulfate with barium perchlorate in 80% isopropanol, using Thorin as the indicator, was preferred by Palaty (*20K*) for the analysis of boiler feed waters.

A simple fluorescence method for determining sulfate in distilled water was described by Yanysheva and Sazonova (*32K*). A sample is evaporated to about 3 to 4 ml. Two milliliters of 96% ethanol and 0.5 ml. of 2×10^{-4} *M* thorium nitrate are added. After 5 minutes, 1 ml. of salicylfluorone is added, the volume adjusted to 10 ml., and the fluorescence measured and compared with standards prepared from solutions containing 0.5 to 4 μg. sulfate.

Chaudhuri, Purohit, and Bhargava (*4K*) developed a new turbidimetric method to determine sulfate in brackish waters. Sulfate is precipitated with barium in a suspending medium containing 50% glycerin, 10% glucose, 2.5% hydrochloric acid, and 37.5% distilled water, and the absorbance measured at 450 mμ and compared with standards containing 0 to 160 p.p.m. sulfate. A similar method was reported by Supatashvili (*27K*) in which a reagent consisting of barium chloride ethanol, and ethyleneglycol is added to the sample and the turbidity measured against a blank.

Ugol'nikov and Galkin (*29K*) described a potentiometric method using a platinum–silver bimetallic electrode for determining sulfate in mineral water. The sample is acidified with hydrochloric acid, potassium ferricyanide and ferrocyanide and ethanol are added, and the sample titrated with lead nitrate solution. Results agree with those obtained gravimetrically.

Matveev and Nechiporenko (*12K*) and Nechiporenko (*15K–17K*) described direct titration methods for determining sulfate and chloride in various types of waters. Ion exchange is used to eliminate cation interferences. The sulfate is titrated with lead nitrate solution, using either diphenylcarbazone or dithizone as indicator. Chloride can be titrated in the same sample using mercuric nitrate and diphenylcarbazone indicator. A description of the various methods and techniques is given. Concentration of sulfate by ion exchange and titration with barium chloride was used by Kralova and Shibaeva (*11K*) to determine traces of sulfate in very pure water. Sulfate was adsorbed on an anion-exchange resin and eluted with ammonium carbonate solution. The sulfate is then titrated using 2-(2-carboxyphenylazo) - 7 - (2 - sulfophenylazo) - 1,8 - dihydroxynaphthalene - 3,6 - disulfonic acid indicator. The titration may also be carried out with $0.002M$ Arsenazo III. For 50 to 100 μg. of sulfate, the experimental error was 5%.

Bozhevol'nov and Kreingol'd (*2K*) determined sulfate indirectly by precipitating sulfate with an excess of barium and titrating the excess with EDTA, using Fluorexon as a fluorescence indicator. An indirect EDTA method for determining sulfate in soils and waters was described by Moghe, Talati, and Mathur (*14K*). Calcium is first determined with ammonium purpurate indicator. The indicator is destroyed with hydrochloric acid and magnesium determined, using Eriochrome Black T. The indicator is again destroyed, the sample acidified, a known excess of barium chloride added to precipitate the sulfate, and the remaining barium titrated with EDTA, using Eriochrome Black T indicator. Grabarov (*7K*) used a similar procedure for determining sulfate except that calcium and magnesium were determined in a separate sample and Trilon B was used to titrate the excess barium with Chrome Dark Blue indicator. A similar method was reported by Rapp.-Sik (*25K*), and Novak (*19K*) described an indirect method wherein a known amount of barium chloride is added and the excess is titrated with Complexon III using Fluorexon indicator. Calcium titrates with barium and must be corrected for.

Gupta and Ananthanarayanan (*8K*) described a volumetric benzidine hydrochloride method for determining sulfate in sea water. All cations are removed by anion exchange and chloride precipitated with silver nitrate. An accuracy of ±0.4 to 0.5% was obtained by this method. Dashevskii (*5K*) suggested certain improvements in techniques in the standard benzidine method.

Nogina and Kobyak (*18K*) determined sulfate in natural waters by buretless titration with barium nitrate, using carboxyarsenazo as indicator. The preparation of the hydrostatic tube used for the titration is described. Five to 500 mg. of sulfate per liter in a 5-ml. sample is titrated with $0.01N$ barium nitrate solution until the indicator changes from crimson to the blue color of the barium carboxyarsenazo complex. Samples containing less than 5 mg. of sulfate per liter must be concentrated prior to analysis.

An indirect volumetric method for determining sulfate was described by Bolberitz and Csanady (*1K*). The water is passed through a column of Amberlite IR-120, and the eluate titrated with sodium hydroxide solution. The milliequivalents of chloride and nitrate are subtracted from the total acid concentration found and the difference multiplied by a factor of 48, which gives the amount of sulfate in mg. per liter. A double indirect method for sulfate was reported by Tolnay and Rapp.-Sik (*28K*). A portion of a neutralized sample is passed through a cation-exchange resin and the eluate titrated with sodium hydroxide. This gives the total cations in the sample. A second portion of sample is mixed with a known amount of neutralized barium chloride, and filtered. The filtrate is passed through the ion-exchange column and titrated. Subtraction of this value from the first gives the excess barium present. The sulfate is then calculated from the difference between the total barium added and the excess barium.

A spot test was devised by Meditsch and Castiel (*13K*) for estimating 50 to 1000 p.p.m. sulfate. The sample is concentrated, chloride removed by electrolysis, and sulfate converted to sulfuric acid by a cation-exchange resin. Ten microliters of the eluate are spotted on filter paper and a drop of silver ethylenediamine chromate added. The diameter of the red spot that develops is logarithmically related to the sulfuric acid concentration. A simple and rapid method for the indirect photometric analysis of sulfate in soils and water with $BaCrO_4$–As_2O_3–EDTA was described by Hsieh and Jen (*9K*). A stable color is formed and there is no interference from sodium, potassium, calcium, magnesium, chloride, or nitrate. Phosphate interferes, and silver, iron, and aluminum will interfere if they are present in large amounts. A precision of 1 to 3% was obtained over the range of 5 to 120 mg. of sulfate per liter.

Sietz (*26K*) determined sulfate in drinking and industrial waters gravimetrically. The sample was added to a solution of barium chloride containing EDTA. The EDTA complexes any iron present. For samples containing approximately 100 mg. of sulfate the error was less than 0.5%.

A study was made by Pungor and Balazs (*21K*) of the detection limits for chloride and sulfate for high-frequency titrations. They found that chloride and sulfate could be determined in drinking water down to 2 μg. and 10 μg. per ml., respectively. Erdey, Gegus, and Vandorffy (*6K*) also developed high-frequency titration methods for determining alkalinity, chloride, total hardness, and sulfate in natural waters. The values obtained for these determinations agreed with values obtained by classical methods.

Johnson, McClelland, and Boster (*10K*) devised an inverse filtration technique to separate and preserve the zinc sulfide formed when zinc acetate fixative is added to water samples containing sulfides. The sulfide is subsequently titrated with iodine–thiosulfate, with greater assurance of minimal loss of the original sulfide. To determine sulfide in mineral springs, Quentin and Pachmayr (*23K*) added an aliquot of sample to an acid solution of zinc acetate. Potassium iodide crystals and potassium iodide solution are added and the solution diluted to 1 liter. The solution is then titrated with thiosulfate, using starch solution as indicator. Hydrogen sulfide can be determined by distillation into a zinc acetate solution and continuing as above. Quentin and Pachmayr (*22K*) also discussed applications of the methylene blue colorimetric procedure for sulfide. Zavodnov (*33K*, *34K*) described several indicators that can be used to determine hydrogen sulfide in mineral waters. These form a blue complex in the reaction between hydrogen sulfide, a ferric salt, and the indicator, such as *p*-phenylenediamine. The blue color is measured at 656 or 670 mμ, depending upon the instrument used. The sensitivity is 0.05 μg. of hydrogen sulfide per 25 ml. Copper and thiosulfate ions interfere.

Quentin and Pachmayr (*24K*) determined thiosulfate above 0.1 p.p.m. in mineral waters by the bleaching of methylene blue in acid solution.

Wayman (*30K*, *31K*) suggested the possibility of determining total sulfur in the absence of chloride and phosphate, and perhaps the simultaneous determination of sulfur and phosphorus in water, by neutron activation analysis.

SILICA, PHOSPHORUS, AND ARSENIC

Aunins and Tisheizers (*4L*) compared the picric acid, potassium chromate, and sodium silicate methods for determining silica in demineralized and colored waters. The sodium silicate method was considered the most accurate and convenient although the potassium chromate method was found satisfactory. The picric acid method is not recommended for high silica waters with a high color index.

Morrison and Wilson (*18L–20L*) made a detailed study of the molybdenum blue method for silica, including an investigation of conditions necessary to form α- and β-molybdosilicic acids, the stability of these acids during removal of the interfering molybdophosphoric acid, and the several reagents which may be used to reduce the acids to the blue complexes. They reported procedures for the determination of both "reactive" and total silica. "Reactive" silica are defined as those forms (mainly monomeric and dimeric silicic acid) which react with ammonium molybdate in less than 10 minutes under the conditions of the procedure. To determine "reactive" silica, β-molybdosilicic acid is reduced with 1-amino-2-naphthol-4-sulfonic acid. To determine total silica, α-molybdosilicic acid is reduced with stannous chloride solution, a necessary procedure because of the high salt concentrations resulting from the preliminary sodium carbonate fusion performed to obtain total silica. The molybdenum blue method was also investigated by Spillner (*24L*) who studied the effect of temperature on the formation of the first phase of the complex. The temperature is less critical when forming the reduction product. The concentration of the reducing solution is not critical.

To determine silica in colored waters by the molybdenum blue method, Umezaki (*26L*) first destroyed humic acids and other organic substances with permanganate in acid solution. The excess permanganate was then decolorized with sodium nitrite which has no effect on the reduction of the silicomolybdic acid. Herrig (*10L*) used citric rather than oxalic acid to suppress the phosphate interference, and found also that the blue complex is more stable in the presence of citric acid.

The silica method based on the reduction of silicomolybdic acid with 1-amino-2-naphthol-4-sulfonic acid was modified by Allen (*3L*) and automated for continuous operation. He described a 4-component instrument consisting of a voltage stabilizer, a one-lamp, twin-beam colorimeter, a strip-chart recorder, and a sample-proportioning pump. The system can monitor 0.0 to 7.0 p.p.m. silica in boiler water. A solvent extraction procedure was described by Sono, Watanabe, and Mitsukami (*23L*) for determining ultramicro amounts of silica in boiler water. Silica is complexed, reduced to molybdenum blue, and the colored product extracted with 5% polyoxyethylenated laurylamine and chloroform. The absorbance of the chloroform extract is measured at 750 mμ. Up to 0.31 p.p.m. silica can be determined with an error of ±5%.

Kemula and Rosolowski (*14L*) determined 0.05 μg. of silica per ml. polarographically with an error of ±8%. The method was based on the formation and reduction of γ-molybdosilicic acid.

Procedural details were given by Krapivina and Khachvankyan (*15L*) for determining ionic, monomolecular, and colloidal forms of silica in mineral waters. Hydrosilicate ions are determined by precipitation with calcium chloride and the monomolecular silica determined in the filtrate by the molybdenum blue procedure. Two methods were presented for determining colloidal silica: the first, the difference between the total silica content determined gravimetrically and the total content of nondissociated molecules and ions; and the second, the conversion to the ionic form and the difference obtained colorimetrically from total monomolecular and ionic silica.

Jones and Spencer (*13L*) compared five different methods for determining inorganic phosphate in sea water. From their study they concluded that the molybdenum blue method of Harvey (*8L*) was the most convenient.

A new photometric method for determining phosphate in water was reported by Breitling (*6L*). A 100-ml. sample is acidified with sulfuric acid and a 50-ml. portion of this treated with a vanadate-molybdate reagent. The absorbance of the treated portion is measured at 365 mμ. using the remainder of the acidified sample as the blank. To determine polymeric phosphates, the acidified sample is first refluxed and the phosphate determined as above. No reducing reagents are required in either procedure. Abbott and Emsden (*1L*) and Abbott, Emsden, and Harris (*2L*) also described the use of a vanadomolybdate solution for determining from 0.04 to 6.0 p.p.m. phosphate as P_2O_5 in water. Up to 500 p.p.m. chloride, 40 p.p.m. fluoride, 200 p.p.m. silica, and 2 p.p.m. iron did not interfere.

Semenov, Semenova, and Datsko (*22L*) described an extraction procedure for determining phosphate in water. A molybdic acid–sulfuric acid solution is added to an aliquot of water and the phosphomolybic acid extracted with butanol. Stannous chloride solution is then added to the extract and the blue color measured at either 620 or 720 mμ. A photocolorimeter with red filter may be used.

Heron (*9L*) reported that changes in phosphate concentrations during storage can be avoided by collecting the water sample in a polyethylene bottle which has been treated with a potassium iodide–iodine solution. Any decrease in phosphate concentration which may occur is attributed to bacterial action and not to absorption of phosphate by the polyethylene. To determine phosphate, a large volume of water was acidified, mixed with ammonium molybdate, and the phosphomolybdic acid extracted into hexanol and reduced with stannous chloride. It was possible to detect less than 1 μg. of phosphate per liter.

A method was described by Szekielda (*25L*) for determining phosphorus in particulate matter found in sea water. The solids from up to 1 liter of water are collected on filter paper which is then dried and burned between platinum spirals in an atmosphere of oxygen. The phosphate so recovered was then determined colorimetrically with ammonium molybdate and stannous chloride.

In determining organic phosphate in natural water, Datsko and Semenov (*7L*) used potassium perchlorate to speed up the rate of oxidation. Excess perchlorate was reduced with sodium sulfite and the excess sulfite removed by boiling. Between 5 and 50 μg. of phosphorus could be determined. No interference occurred from the other minerals found in water.

Molinski and Szporek-Dybkowska (*17L*) described a colorimetric procedure for determining sodium hexametaphosphate. The sample is first hydrolyzed to orthophosphate by heating with hydrochloric acid for 30 minutes and the phosphate determined colorimetrically by a molybdenum blue procedure.

A rapid method for determining arsenic-76 and phosphorus-32 in reactor effluent water was described by Johnson (*12L*). An extraction procedure is used to separate the two ions. The organic phase which contains the phosphorus-32 is dry mounted on a 1-inch stainless steel dish for β-counting. Arsenic-76 is directly γ-counted after separation from the phosphorus-32. Further purification is not necessary due to the discrete γ-energy of 0.56 m.e.v. for arsenic-76.

Ballinger, Lishka, and Gales (*5L*) applied the silver diethyldithiocarbamate (SDDC) method to the determination of arsenic in river water, domestic water, and sewage. The method is based on the reduction of arsenic to arsine by zinc in acid solution. The resulting arsine is passed into an absorbing solution containing SDDC, and the color formed is measured at 560 mμ. The method has a precision of ±0.01 mg. per liter for arsenic concentrations of from 0.01 to 0.3 mg. per liter; accuracy is about the same. The method is superior to both the Gutzeit and the heteropoly blue methods in precision, accuracy, time of analysis, and ease of manipuation.

An extraction of an arsenic–thionalide complex with ether and subsequent determination as molybdenum blue was used by Nakaya (*21L*) to determine arsenic in natural water. Arsenic was first reduced to As^{+3} and then complexed with thionalide. The complex was then extracted with ether, back-extracted with a hypobromite solution, and the arsenic finally determined by the molybdenum blue method. Most ions which are found in natural waters do not interfere.

A Gutzeit procedure was described by Mazo and Shafran (*16L*). The water sample is made neutral to methyl orange, the arsenic oxidized to AsO_4^{-3}, and then precipitated with ammonium aluminum sulfate and ammonium hydroxide solutions. The precipitate is filtered, washed, and dissolved in sulfuric acid solution. The solution is then transferred to an arsenic apparatus, stannous chloride and zinc are added, and the liberated arsine absorbed on mercuric bromide paper. The color is then compared with standards.

A polarographic procedure was used by Hofmann (*11L*) to determine arsenic in water. Arsenic must first be reduced to the trivalent state. The polarogram is made in an acid solution and, from a number of acids studied, tartaric acid was found to be the most suitable. Organic matter, however, must first be removed by a nitric and sulfuric acid treatment. A great number of other ions were investigated and only cadmium interferes. Concentrations of arsenic above 0.3 mg. per liter can be detected. The accuracy is ±3%.

NITRATE, NITRITE, AND NITROGEN COMPOUNDS

Direct ultraviolet absorption methods for determining nitrate in water have been described by several investigators. Armstrong (*4M*) found that adding an equal volume of sulfuric acid to samples containing nitrate and chloride shifts the absorption wavelength from 210 to 230 mμ. The absorption of other substances is less at the 230-mμ wavelength and, in some cases, diminishes in the presence of sulfuric acid. The sulfuric acid also increases the reactivity of the nitrate ion and it is easier to destroy it by a reducing agent to allow accurate measurement of the nonnitrate absorbance of the solution. Navone (*29M*) described a direct ultraviolet procedure measuring the absorbance of nitrate at 210 mμ against a blank in which the nitrate ion has been destroyed by the action of a zinc–copper couple in an acid medium. This cancels out all interfering substances except for nitrite. The method was applied to the determination of nitrate in potable water. Goldman (*17M*) compared the phenoldisulfonic acid and ultraviolet absorption methods for determining nitrate in water. If care is taken in removing chloride from the sample, the phenoldisulfonic acid method is satisfactory. However, the ultraviolet method is simpler and faster and equally reliable if corrections are made for high nitrite, chromium, and synthetic detergents.

In the past two years, several nitrate methods have been reported which are based on the reduction of nitrate to nitrite and subsequent diazotization and coupling. Williams and Coote (*41M*) found that hydrogen sulfide interferes when nitrate is reduced by hydrazine in the presence of a copper catalyst; it was presumed that the hydrogen sulfide poisoned the catalyst. Addition of more catalyst and boiling the sample did not completely overcome the effect in the analysis of sea water samples. Edwards *et al.* (*12M*) developed a method to determine nitrate in waste water effluents and in water by carefully controlled reduction of nitrate to nitrite with zinc in acid solution. The amount of zinc and the reduction time are very critical. The diazotization step with sulfanilic acid is carried on during the reduction period. The excess zinc is filtered and the diazo compound coupled with naphthylamine. Beer's law is followed up to 6.2 mg. of nitrate per liter. Chow and Johnstone (*7M*) determined nitrate in sea water by reduction of nitrate with zinc dust in ammoniacal solution in the presence of manganese(IV) catalyst. Diazotization with sulfanilamide occurs simultaneously with the formation of nitrite. They found that the reduction of nitrate was more efficient and reproducible at temperatures below 10° C. The diazo compound is coupled with *N*-1-naphthylethylenediamine in acid medium after filtration. The reduction yield was calculated to be 85 to 90%. Morris and Riley (*28M*) determined nitrate in sea water by converting the nitrate to nitrite with about a 91% efficiency by passing the sample through a column of amalgamated cadmium filings. The nitrite was diazotized and coupled with sulfanilamide and naphthylethylenediamine, respectively, in acid solution and the absorbance measured at 543 mμ. Normal conditions of temperature, salt concentration, sulfide, and pH found in sea water do not interfere. In the procedure described by Fishman, Skougstad, and Scarbro (*14M*) nitrate is reduced to nitrite by hydrazine using a copper catalyst in hot alkaline solution (90° C.). The solution is then cooled in a water bath and diazotized with sulfanilamide and coupled with 1-naphthylethylenediamine. The reduction is carried out at a pH between 10 and 11, and, to prevent precipitation of certain hydroxides, all metal ions are removed by cation exchange prior to reduction. Nitrite interferes and must first be removed by extraction with an isoamyl alcohol–carbon tetrachloride mixture. A procedure for determining nitrite is also presented.

Dal Pont, Newell, and Staniforth (*8M*) compared the hydrazine reduction and strychnidine methods for estimating nitrate in sea water. For low concentrations of nitrate (less than 10 μg.-atoms of nitrate per liter), they preferred the hydrazine method even though it is less convenient. At high concentrations of nitrate, both methods gave similar results.

Vaccaro, Thunberg, and Ketchum (*40M*) developed a procedure for determining nitrate in the presence of high nitrite concentrations. Nitrite is oxidized to nitrate by an ozonization technique, and the resulting nitrate determined by the catalytic hydrazine reduction procedure. Nitrite is determined separately and nitrate determined by difference.

Rosca and Vasilescu (*35M*) modified the brucine method for nitrate to avoid the interference of organic material, and to prolong the shelf life of the brucine reagent. Air bubbled through the alkalinized sample for 20 minutes decreases organic matter and oxidizes ferrous iron. For nitrate concentrations between 0.75 and 30 mg. per liter, the error is 2%, or less. A critical study of the brucine method was made by Fadrus and Maly (*13M*). Stability of the reagent, the effects of the temperature rise when sulfuric acid is added, oxidative decomposition during the period of color development, and adequate acid for color development are extremely critical factors; however, little or no interference occurs from most ions commonly found in water. A technique to determine less than 1 mg. of nitrate per liter in ocean, estuarine, and fresh waters by the brucine method was reported by Jenkins and Medsker (*21M*). Excess chloride is added to mask minor chloride variations and the reaction conditions are carefully controlled by even heating in a boiling water bath and rapid cooling afterwards in a cold bath. The method gave reproducible results for nitrate concentrations from 0.05 to 0.8 μg. per liter.

Yen (*42M*) found that sodium diphenylbenzidinesulfonate was a satisfactory reagent for determining 5 to 100 mg. of nitrate nitrogen in sea water. Chloride, nitrite, and other oxidizing agents at temperatures between 10° and 30° C. did not interfere. A 3-ml. sample was acidified with 2.5 ml. concentrated sulfuric acid. After 40 minutes, one drop of 0.2% sodium diphenylbenzidinesulfonate was added and the absorbance measured at 566 mμ. Konnov (*24M*) reported a similar procedure for the determination of nitrate in sea water. Ammonia was also determined by a method involving the use of cool, filtered Nessler reagent in an alkaline medium.

Makitie (*26M*) described a spectrophotometric method for determining less than 10 μg. of nitrate in aliquots of soil extracts, water, and other materials. Chloride is first removed from the sample by precipitation with silver sulfate. The solution is decolorized with charcoal and evaporated to dryness. Sodium 1-naphthol-4-sulfonate in sulfuric acid solution is added to the residue, the solution buffered at pH 8.5, diluted to final volume, and the absorbance measured.

A polarographic technique for determining nitrate, based on the reduction of nitrate at the dropping mercury electrode in slightly acid solution, and in the presence of uranyl ion, was described by Frazier (*16M*). Nitrite is the only ion which interferes seriously, giving readings 54% as high as those obtained from equivalent concentations of nitrate. Nitrite must be determined by an independent method and proper corrections made. With care, the nitrite can be destroyed by the addition of one drop saturated sulfamic acid before a reading is made. The procedure is satisfactory for nitrate as low as 0.2 mg. per liter. Henrioul (*19M*) determined nitrate in water and foods by direct polarographic determination with 2,4-phenoldisulfonate and obtained values which compared favorably with those obtained by colorimetric methods. Rapp.-Sik (*34M*) determined nitrate in water using indigo carmine.

Sawicki *et al.* (*38M*) compared 52 spectrophotometric methods for determining nitrite. The methods were compared with respect to molar absorptivity, selectivity, sensitivity, simplicity, color stability, and conformity to Beer's law.

A single, stable powdered reagent consisting of equal amounts of sulfanilic acid and 1-naphthylamine-hydrochloride was employed by Goodman (*18M*) for field or laboratory determination of nitrite in water. A 0.1-gram portion of the reagent is added to 50 ml. of sample, the sample shaken, and the absorbance measured at 520 mμ. Comparisons may also be made visually against permanent colored standards prepared from crystal violet and basic fushin dyes. The continuous determination of nitrite in waste water was described by O'Brien and Fiore (*31M*) based on a modified diazotization–coupling method.

Methods for determining nitrite in water and other materials, based on the formation of nitrosophenols which form colored complexes with ferrous iron, were evaluated by Peach (*32M*). The sensitivity, advantages, and limitations of seven different phenols were considered.

Methods for determining ammonia by the formation of the blue indophenol color have been described by several investigators. Acuna (*2M*) added chlorine water, a drop of manganese sulfate solution, and sodium phenate to a sample of water, and compared the resultant color intensity with Nessler ammonium chloride solution standards, or spectrophotometrically. A similar method, using hypochlorous reagent instead of chlorine water, was described by Rossum and Villarruz (*37M*). The absorbance of the indophenol was measured at 630 mμ. The sensitivity of the method was 0.01 mg. of ammonia per liter. Tetlow and Wilson (*39M*) based their method on the reaction of ammonia with sodium phenate and sodium hypochlorite in the presence of acetone. Small amounts of acetone increase the sensitivity ten times and also produce a faster rate of color development. Interference from copper is eliminated by using EDTA. The method was applied to the determination of ammonia in boiler feed water and similar high-purity waters. The detection limit was about 0.0013 p.p.m. The standard deviation varied from about ±0.001 to ±0.005 p.p.m. for ammonium concentrations of 0.05 to 0.5 p.p.m.

Roskam and Langen (*36M*) stated that thymol in acetone is more stable and sensitive than phenol for determining ammonia by the indophenol method. The method they described was applied to the determination of ammonia in sea water. CDTA (cyclohexyl-trans-1,2-diaminetetraacetic acid) was used to complex magnesium which would otherwise interfere. Datsko and Kaplin (*9M*) described an indophenol method using sodium hypobromite as the oxidant. The method is sensitive to 0.01 mg. of ammonia nitrogen per liter, with an accuracy of 10 to 15% in the range of concentration of from 0.01 to 0.10 mg. ammonium per liter. Kaplin and Fesenko (*23M*) used a similar method to analyze colored and turbid waters, but went one step further to remove interference by extracting the indophenol into chloroform and re-extracting the indophenol into an aqueous phase with 0.3% caustic soda and measuring the absorbance. There was no decrease in sensitivity. Newell and Dal Pont (*30M*) described an extraction procedure for determining ammonia in sea water. The ammonia is oxidized with phenol to monochloramine at pH 8 and the monochloramine coupled with phenol at pH 3.4, forming quinone chlorimide. The latter compound is extracted into hexanol. Sodium hydroxide is added to the hexanol which causes phenol to react further with quinone chlorimide to form indophenol. The absorbance of the hexanol layer is then measured. Demmitt (*10M*) described a procedure for the continuous measurement of trace concentrations of ammonia in reactor cooling water systems by the indophenol reaction. Up to 25 p.p.m. of ammonia can be determined.

Nessler reagent was used by Apostolache (*3M*) to determine ammonia in natural waters. Ferrous iron, calcium, magnesium, sulfides, and hydrogen sulfide interfere, and are removed by the addition of zinc acetate and distillation with sodium carbonate. Justatowa (*22M*) determined ammonia in water and aqueous salt solutions by calculating the partial pressure of ammonia above an aqueous solution containing less than 10% nonvolatile salts. Prochazkova (*33M*) discussed the effects of nitrates and nitrites on the determination of organic nitrogen and ammonia in water by Kjeldahl oxidation. Dudova (*11M*) converted organic nitrogen to ammonia by heating the sample to 500° C. with sodium hydroxide in hydrogen. The ammonia was then oxidized to free nitrogen with sodium bromite and the excess of the latter determined iodometrically.

Methods and instrumentation for determining cyanogen compounds were reviewed by Lancy and Zabban (*25M*). They discussed the applications and limitations of these methods and described the use of instrumentation for the determination of cyanides and the measurement and control of the extent of treatment in the oxidation of cyanides by either chlorine or hypochlorite. A modified pyridine–benzidine method for determining cyanide and thiocyanate between 0.05 and 2 mg. per liter in river and waste waters was described by Husmann, Malz, and Bortliss (*20M*). Improvements were achieved by standardizing the period between reaction and color measurement, and by keeping the pH at 4.2.

Bucksteeg, Kalweit, and Dietz (*6M*) developed a method to differentiate between certain complex cyanides. The complex cyanides of zinc, cadmium, nickel, and copper(II) yield hydrogen cyanide when distilled at pH 5.2 to 5.5. The cyanide complex of copper(I) can be oxidized to copper (II) with potassium dichromate. Zinc acetate may be added to precipitate the complexes and prevent the evolution of hydrogen cyanide from $Fe(CN)_6^{-3}$ and $Fe(CN)_6^{-4}$. Distilled hydrogen cyanide may be determined by either the colorimetric pyridine–benzidine or the silver nitrate titration method.

Bark and Higson (*5M*) proposed a *p*-phenylenediamine colorimetric method for determining 0.005 to 100 p.p.m. of cyanide in water. Abbott (*1M*) determined 0.05 to 1.0 p.p.m. of cyanide based on the stable color formed between cyanide and barbituric acids. Miller *et al.* (*27M*) used the direct and spontaneous response of a rapidly rotating gold electrode to cyanide ion in lithium hydroxide solution, to detect and measure extremely small concentrations of cyanide. Distillation from slightly alkaline solution was suggested by Fleps (*15M*) as a means of separation and determination of free and complexed cyanides.

ALKALINITY AND pH

Park, Oliphant, and Freund (*6N*) pointed out that an inductive conductivity meter, commonly used to determine the salinity of sea water, can also be used to determine its alkalinity. A sample, titrated with 1.00*N* hydrochloric acid shows little change in conductivity until the major components which contribute to total alkalinity have been neutralized. In analyzing 10 samples whose alkalinity ranged from about 2.25 to 2.45 meq. per liter, they found that values determined by the conductivity method differed from those obtained by the pH method by no more than 0.9%. Barnes (*1N*) described several special precautions that are necessary to obtain pH and alkalinity measurements in the field which are accurate to within ±0.02 pH and ±0.6 p.p.m. bicarbonate, respectively. Using carefully calibrated electrometric pH equipment to accurately determine the pH of the sample, the true pH of the end point of the alkalinity titration may be calculated as a function of temperature, ionic strength, and total alkalinity. Roberson *et al.* (*7N*) made a comparison of pH, alkalinity and specific conductance values determined in the field with values determined in the laboratory from 5 to 120 days later. The field determinations, although generally higher, better represent the actual condition of the water in its natural environment. Three titrimetric methods for determining the alkalinity of sea water were evaluated by Tsurikova (*8N*) who concluded that, for field use, the direct titration of a 10-ml. sample with 0.01*N* hydrochloric acid is the most convenient and accurate. A brief description of an apparatus for the continuous determination of alkalinity has been published (*9N*).

A glass-electrode system and cells for pH measurements in mineral waters at up to 6 atm. and 60° C. were described by Kryukov (*5N*). Hirsch (*3N*) discussed certain problems encountered in systems for continuous measurement of the pH of polluted water, including electrode fouling and corrosion, and means of minimizing or eliminating these difficulties.

A pH meter and procedures used in connection with oceanographic research and sea water analysis were described by Kabanov (*4N*). Disteche and Dubuisson (*2N*) reported on the design and operation of a glass electrode assembly which permitted precise pH measurements at depths as great as 2300 meters, with an accuracy of 0.05 pH unit or less.

OXYGEN DEMAND

A review of methods for the determination of chemical oxygen demand (C.O.D.) was published by Zdybiewska (*9P*). Twenty references were included.

Ballinger and Lishka (*1P*) studied the reliability and precision of the standard biological oxygen demand (B.O.D.) and C.O.D. determinations. A sample consisting of sterile distilled water, glucose, and glutamic acid, and seeded with domestic sewage was prepared and analyzed by several laboratories. A geometric standard deviation of approximately 20% for the B.O.D. and 8% for the C.O.D. was obtained. The study also revealed that the 2- and 3-day B.O.D. values were as precise as the 5-day values.

Drozdov, Vladimirova, and Kuptsova (*4P*) made a comparison of the dichromate and iodate methods for C.O.D. determinations and concluded that the dichromate method was the more rapid and convenient. Dobbs and Williams (*2P*) modified the standard dichromate method to minimize chloride interference. Mercuric sulfate is added to form a soluble mercuric chloride complex that completely resists oxidation under conditions of the C.O.D. test. Since the need for a chloride correction is eliminated and a separate chloride analysis is, therefore, not required, a more rapid, accurate, and useful method results.

Dissolved oxygen and B.O.D. measurements were made by Eye, Reuter, and Keshavan (*5P*) using a stationary, plastic-covered platinum electrode system. The results were as accurate as those obtained by the standard Winkler method. The electrode assembly consisted of a stationary platinum cathode, covered with a polyethylene membrane 2 to 4 mils thick and connected to a silver electrode by a supporting electrolyte of potassium chloride or hydroxide. The electrodes were assembled in a glass dip cell and the analyses made in a sealed flask to prevent reaeration during measurements. Voltages were measured at a constant temperature.

Reference tables of data which simplify the selection of dilutions and the calculations of B.O.D. determinations were described by Greenberg and Lennette (*6P*). A rapid, modified dichromate method for estimating B.O.D. in waste water was reported by Donlan (*3P*). The results are in close agreement with the 5-day B.O.D. of the waste water. An 8-hour total B.O.D. test using mass culture aeration and C.O.D. values was described by Hiser and Busch (*8P*). Hart, Hart, and Hess (*7P*) recommended the use of light-weight 16-oz. polyethylene bottles for collecting samples for B.O.D. determinations.

OXYGEN AND OTHER GASES

A review with 43 references of methods for determining dissolved oxygen in water was prepared by Burianek (*10Q*). Apte *et al.* (*4Q*) compared colorimetric and polarographic methods for determining dissolved oxygen and found satisfactory agreement with the conventional titrimetric method. The Winkler method, a polarographic method, and a commercial platinum–silver electrode method were studied by Hoak and Bramer (*28Q*). Neither the Winkler nor the polarographic method is suitable for field work because of the number of manipulations required and the limited number of samples that can be analyzed in one day. The platinum–silver electrode, on the other hand, is rapid and convenient.

Grasshoff (*22Q, 24Q*) and Solov'ev (*58Q*) described various polarographic devices for determining oxygen in sea water. The methods are based on the reduction of oxygen at a platinum electrode. Grasshoff (*23Q*) also described the use of a dropping mercury electrode for determining oxygen in sea water. A polarographic method for determining oxygen in fresh water, incorporating an electric motor-driven pump above gold–zinc electrodes so that the water is continuously renewed, was reported by Speranskaya and Khaidarov (*59Q*). The use of a wide-bore polarograph for continuous dissolved oxygen measurements in the field was described by Wood (*69Q*). Spot checks with the Alsterberg modification of the Winkler test gave comparable results. Morgan and Bewtra (*49Q*) described a polarographic apparatus incorporating a flow-through cell and photographic recording of oxygen values.

A galvanic-cell analyzer for the continuous determination of oxygen in natural waters and wastes was described by Mancy, Okun, and Reilley (*43Q*), and by Mancy and Westgarth (*44Q*). Waclawik and Waszak (*66Q*) used a continuous photocolorimetric and automatic galvanic analyzer for oxygen measurements. High concentrations of oxygen were removed from the sample with an inert gas and subsequently determined in the gaseous phase by the photocolorimeter. Trace amounts of oxygen were determined by means of the galvanic cell. Conditions for the amperometric determination of oxygen were investigated by Kutyurin (*40Q*) who developed a procedure based on a controlled system involving a noble metal anode, such as platinum, and an active metal cathode, such as iron, zinc, or cadmium.

Eckfeldt and Shaffer (*15Q*) applied constant potential derivative coulometry for measuring dissolved oxygen.

Jessop and Holy (*30Q*) described a method for determining between 0.001 and 0.02 p.p.m. of dissolved oxygen in boiler feed water, based on the transfer of dissolved oxygen by hydrogen into an isolated analyzing solution containing the measuring cell electrodes.

Chenouard, Dirian, and Gabilly (*14Q*) described an apparatus for the continuous determination of oxygen. The increase in conductance of water after passage over pure thallium is related to the amount of dissolved oxygen. As little as 0.002 p.p.m. oxygen can be determined. A similar procedure was described by Lindsay (*41Q*) for the determination of between 0.008 and 80 p.p.m. oxygen.

Gubeli (*25Q*) described a direct electrometric method for determining micro amounts of oxygen dissolved in waters of mineral springs. The method, based on a zinc-carbon-oxygen generator, requires only special preparation of the carbon electrode. A continuous electrometric microdetermination of dissolved oxygen in water was reported by Elliott (*16Q*).

The reduced yellow form of Safranine Red T is oxidized to a red color by oxygen; Aleskovskii *et al.* (*1Q*, *2Q*) and Stepanova and Aleskovskii (*61Q*) used this reaction to determine oxygen. The absorbance of the solution is measured at 520 mμ. Beer's law is obeyed from 0 to 30 μg. of oxygen per liter. Extraction of the Safranine Red T into organic alcohols increased the sensitivity. An automatic analyzer was also described for measuring oxygen by the above procedure. Meyling and Frank (*47Q*) determined oxygen spectrophotometrically using indigo carmine. Oxygen oxidized the leuco base of indigo carmine to give a blue color which was measured at 620 mμ. From 1 to 8 p.p.m. of oxygen can be determined in a 0.1-ml. sample. St. John, Winefordner, and Silver (*60Q*) also described a procedure for determining oxygen, based on the indigo carmine method. The results are in fair agreement with those obtained by a modified Winkler method. Oxidation of $Cu(NH_3)_2Cl$ by oxygen swept from a water sample by an inert gas was used by Rezayeva (*52Q*) to determine oxygen photometrically. Trotti and Sacks (*64Q*) modified the Winkler method to determine oxygen spectrophotometrically. Instead of titrating, the iodine color is measured at 450 mμ. The standard deviation is about the same as for the titration method. Elliott (*17Q*) used a similar procedure but extracted the iodine into chloroform. Carvalho, Calado, and Moura (*13Q*) determined oxygen in demineralized water spectrophotometrically, based on the oxidation by oxygen of the manganous–EDTA complex to the red manganic–EDTA complex. The absorbance was measured at 490 mμ.

Several investigators suggested modifications of the Winkler method. Montgomery, Thom, and Cockburn (*48Q*) emphasized the precautions necessary to prevent loss of iodine vapor. Kliffmueller (*38Q*) investigated the influence of different organic materials. Grasshoff (*21Q*) made a careful study of the errors in the Winkler method when analyzing sea water, and described the precautions necessary for accurate determinations by both dead-stop titration and by manual titration. Smit and Ehrenburg (*57Q*) also pointed out special precautions which must be observed when determining very low concentrations of oxygen.

Needleman (*51Q*) investigated the errors arising in the presence of hydrazine and showed that they are due to a noncompensated reaction, between hydrazine and the manganese oxidized during the Winkler reaction. Burke's (*11Q*) modified Alsterberg oxygen procedure is of value in field work where small samples must be taken. A 10-ml. syringe is used to draw up the water sample and reagents.

Automatic analyzer systems were described by Lo (*42Q*) and by Tanno (*63Q*) for determining oxygen by the Winkler method.

A volumetric method for oxygen was developed by Gusev and Ketova (*26Q*). This method is based on the oxidation of added ferrous iron and subsequent titration of the resulting ferric iron with vanadium chloride.

Berka and Hofmann (*6Q*) determined traces of dissolved oxygen by visual titration using a hydroquinone reference solution with diphenylamine as indicator in the presence of pyrophosphate. Roskam and Langen (*53Q*) developed a new complexometric method. Ferric iron, formed from ferrous ethylenediamine sulfate, is titrated with EDTA in the presence of salicylic acid indicator. Berka and Hofmann (*7Q*) proposed a new method for determining dissolved oxygen in waste water. The samples are titrated with hydroquinone, either potentiometrically using a platinum indicator electrode, or visually with ferroin indicator.

A miniaturized dissolved oxygen field test kit capable of determining dissolved oxygen within ±0.1 mg. per liter was developed by Scott (*56Q*). Samsoni (*55Q*) developed a new type of sampler, consisting of a flask with dispenser and ground glass stopcock, to collect samples for dissolved oxygen determinations. The sample and reagents can be introduced without contact with atmospheric oxygen.

A neutron activation method for determining dissolved oxygen in rocks, minerals, and water was described by Volborth and Banta (*65Q*), Roumy (*54Q*) reported a method for determining small quantities of dissolved oxygen in water.

Taniguchi (*62Q*) employed a modified "Shibata-Miller" method to determine dissolved oxygen in sea water in the presence of organic matter. Dimethylglyoxime with potassium–sodium tartrate is used as the chelating agent. To determine dissolved oxygen in waste water, Mori, Goto, and Ishihara (*50Q*) removed chromate ions with barium nitrate and the excess barium with sodium sulfate, and then applied the "Shibata-Miller" method.

Galster (*19Q*) reported that ozone in drinking water can be determined either iodometrically or spectrophotometrically by reaction with *N*,*N*,*N'*,*N'*-tetramethyl-*p*-phenylenediamine in citric acid solution. A coulometric ozone sensor for field and laboratory measurements was tested by Wartburg, Brewer, and Lodge (*67Q*) for reliability, accuracy, and specificity. Peroxy acids, hydroperoxides, and sulfur dioxide were found to interfere.

In a critical survey of methods for determining the total gas content of water, Kanellopoulos (*31Q*, *32Q*) found only direct extraction methods to be reliable, and described suitable methods of sampling. Cantone and Gurrieri (*12Q*) determined methane, oxygen, nitrogen, and argon in water by mass spectrometry. The samples were degassed under vacuum and freed of water vapor and carbon dioxide with dry ice and liquid air. To determine carbon dioxide, the liquid air trap was omitted, and results were corrected for its solubility in water. Gas chromatography was used by both Kilner and Ratcliff (*37Q*) and by Williams and Miller (*68Q*) to determine gases dissolved in water.

Hissel (*27Q*) described a simple apparatus for determining hydrogen in boiler waters. Argon is circulated through the sample in a closed system, to establish a gas-liquid biphase equilibrium, and the hydrogen determined by gas chromatography. An indirect method of determining the hydrogen content of water by measuring the oxygen consumption when hydrogen is made to react on a finely divided platinum catalyst in the presence of excess oxygen was reported by Faber and Brand (*18Q*). The oxygen is measured by the Winkler method if the hydrogen content is between 3 and 15 ml. per liter, and by the *o*-toluidine method for hydrogen concentrations down to 0.3 ml. per liter.

A rapid radiometric method was used by Andreev (*3Q*) to measure the radon content of solutions. Radon activity of the order of 1.8 × 10^{-11} curie per ml. can be determined.

An automatic photometric method for determining between 0 and 0.5 p.p.m. of chlorine was described by Bossy (*9Q*). The method is based on the oxidation of iodide to iodine. The blue color formed between iodine and starch is proportional to the concentration of chlorine. The apparatus is so designed that 200 ml. of water and 2 ml. of potassium iodide–starch solution are added to the cell of the recording photometer during each 10-minute interval. Katz and Neukelekian (*34Q*) evaluated the cyanogen chloride method for determining chlorine in waste waters and compared the results with other methods. The results differed widely from the amperometric method, but the superiority of either method was not demonstrated. However, for tap water, the results of these two methods agreed with the starch–iodine and *o*-tolidine methods. Residual chlorine in chlorinated sea water was determined spectrophotometrically by Athavale, Krishnan, and Subramanian (*5Q*). The bleaching effect of chlorine on methyl orange was measured at 510 mμ. However, hydrogen peroxide, potassium dichromate, hydrated manganese dioxide, bromine, and permanganate were found to interfere. Houghton (*29Q*) developed a technique for removing turbidity from river water samples prior to determining color and residual chlorine. The sample is filtered through a mat of kieselguhr flocculated with sodium chloride. When determining chlorine, however, the sodium chloride is omitted because of its effect on color development in the *o*-tolidine method.

Berndt (*8Q*) suggested iodometric titrations for determining chlorine, sodium chlorite, and chlorine dioxide. Three titrations with thiosulfate are required. Chlorine plus one fifth of the chlorine dioxide is titrated in a neutral solution. The same solution is then acidified with sulfuric acid and four fifths of the chlorine dioxide plus sodium chlorite is determined. In a separate sample, the chlorine dioxide is reduced to sodium chlorite and determined; chlorine is bound with potassium cyanide. The concentration of the individual substances may then be calculated from the titration values. Kerenyi (*35Q*) suggested a modification in the third step of the above procedure to the extent that the solution is buffered with phosphate to pH 7 and both chlorine and chlorine dioxide were removed by bubbling nitrogen or air into the sample. Sodium chlorite is then determined. Kerenyi and Kuba (*36Q*) discussed the use of tyrosine to determine chlorine dioxide in the presence of chlorine. Karge (*33Q*) pointed out certain limitations of the method of Berndt (*8Q*), primarily because of the reaction between chlorine dioxide and sodium hypochlorite. He proposed an alternate method based on three measurements. The first two are determined by titration of iodine by phenylarsenious oxide solution: (1) chlorine and one fifth of the chlorine dioxide, and (2) chlorine, chlorine dioxide, and sodium chlorite. Chlorine is then determined separately, colorimetrically, with potassium cyanide, pyridine, and barbituric acid. To determine chlorine, chloramine, chlorine dioxide, and sodium chlorite, Meier-Ewert and Bruenner (*46Q*) obtained the individual results from four titrations using arsenous oxide and sodium thiosulfate.

Gaunt and Shanks (*20Q*) described the design and functioning of an apparatus to determine up to 0.1 p.p.m. of carbon dioxide in boiler feed water by gas chromatography. The carbon dioxide and other gases are removed from an acidified sample by boiling under reduced pressure and entrainment by a carrier gas (hydrogen). Kryukov and Zavodnov (*39Q*) described a technique for collecting and analyzing carbon dioxide in mineral waters. The sample is collected in a partially evacuated round-bottomed flask containing barium oxide, and the carbon dioxide subsequently determined gravimetrically. A rapid method for determining total carbon dioxide in waters was reported by Maros *et al.* (*45Q*).

DETERGENTS

A cooperative study of three different, published methods for the determination of alkylbenzenesulfonates (ABS) in water was made by three different laboratories. The results, reported by Bolton, Webster, and Hilton (*2R*), showed no significant difference in accuracy between the methods. The standard deviation increases with increasing detergent concentration. Wayman (*11R*) also found that the error in the determination of ABS by the methylene blue method depends upon its concentration. He reported no significant temperature error between 10° and 35° C., and little or no fading of the complex up to 60 minutes after extraction; there were, however, noticeable differences among the dyes from several suppliers. The relative advantages and disadvantages of five different methods for determining small amounts of ABS in sewage were discussed by Heinerth (*5R*), who attempted to minimize interferences due to other organic substances by either pretreatment with trimethylcetyl ammonium bromide or by adsorption of the surfactant on Dowex 21K. The first method gave reproducible but high results, while the second did not lend itself to satisfactory recovery of the adsorbed ABS.

The determination of anionic surface-active materials in sludge plant effluents should be made either immediately after collecting the sample or on a sample which has been rendered sterile by the addition of 50 p.p.m. of mercuric chloride, according to Gameson and Lewis (*4R*), who analyzed the results submitted by 30 cooperating laboratories who participated in the analysis of standard samples of partially nitrified effluent containing known amounts of Manoxol OT.

A simple, rapid test to indicate the presence of anionic detergents in drinking water was reported by Abbott (*1R*). A dilute aqueous solution of methyl green and a small volume of chloroform are added to an acidified aliquot of the test sample. After thorough mixing, the appearance of a green color in the separated solvent layer indicates presence of detergent in the sample. Michelsen and Maerki (*8R*) made certain changes in the methylene blue-chloroform extraction method in order to simplify it for both laboratory and field use. A shaker rack, which holds 16 separatory funnels, was described by Niemitz and Fuss (*9R*), who recommended it as a convenience in carrying out the chloroform extractions of the methylene blue complex. Sztafrowski (*10R*) pointed out that although some free methylene blue is extracted along with the detergent-material complex, the free dye may be reextracted by treatment with distilled water acidified with sulfuric acid.

Three different techniques were investigated by Frankhouser (*3R*) in a search for a simple, rapid, and accurate method for trace amounts of alkylbenzenesulfonates. Turbidimetric titration with a solution of a high-molecular-weight quaternary amine permitted determination of ABS concentrations of the order of 7 p.p.m. in tap water with an error of no more than 5.6%. Attempts to follow the titration by differential refractometry were unsuccessful, as were attempts to titrate ABS amperometrically with 1,10-*o*-phenanthroline titrant. Absorbance measurements in a 5-cm. cell at 223 mμ permitted estimation of ABS in tap water at the 1.4 p.p.m. level. Levchenko, Khudyakova, and Gavrilova (*7R*), using a stalagmometer, determined the surface tension of solutions containing known concentrations of nonionic surface-active agents. From this empirical curve they were able to estimate, from the similarly-measured surface tension of a test solution, the amount of surface-active material present. From 0.005 to 0.03% of these materials may be determined. Methods for the determination of certain nonionic surface-active reagents were discussed by Karapaev and Malkina (*6R*) who also described suitable procedures for their determination in industrial wastes.

PESTICIDES AND HERBICIDES

One-tenth p.p.m. or more of such pesticides as DDT, chlordane, lindane, Perthane, Phygon SK, silvex, and others can be determined in polluted stream waters by a paper chromatographic method described by Ceresia (*3S*). Three distinct persumptive tests, including paper chromatography for chlorinated pesticides and for organophosphates, and an enzymic method for organophosphates and carbamates, were included in a screening program set up by Hindin and Dunstan (*7S*) to monitor river water for traces of pesticides. Positive results on the preliminary tests are followed by gas chromatographic separation and identification of the specific pesticide contaminant. A review of paper and gas chromatographic methods, most of which were developed for the analysis of foods, but which may be adapted for water analysis, was prepared by Skrinde, Caskey, and Gillespie (*9S*).

Ultraviolet spectrophotometric and colorimetric methods for the determination of 2,4-dichlorophenoxy acetic acid in surface waters were developed by Aly and Faust (*2S*). The methods are adaptations and modifications of methods previously reported by others for the analysis of milk, soils, and other materials. The possibility of identifying 2 p.p.b., or less, of certain chlorinated hydrocarbon pesticides in surface waters, by a combination of a carbon filter and paper chromatographic techniques, was pointed out by Goodenkauf and Erdei (*5S*). They gave details of a procedure which involved carbon filtration and concentration of 2000 to 4000 gallons of sample, chloroform extraction of the soluble organics, separation into water- and ether-soluble fractions, weak- and strong-acid fractions, and finally basic and neutral fractions. Final identification was made by established paper chromatographic techniques.

A procedure for determining silvex, an aquatic herbicide, was reported by Gutenmann and Lisk (*6S*). Details of a preliminary ether extraction, methylation of the silvex, and detection by electron-affinity gas chromatography are included in their report. The quantitative estimation of as little as 0.05 mg. of silvex per liter was based on a working curve prepared from solutions containing known amounts of the material.

Abbott *et al.* (*1S*) devised procedures for the extraction and paper chromatographic, quantitative determination of microgram amounts of 6 organo-chlorine herbicides in water or soil. By means of thin-layer chromatography they also achieved complete separation of the 6 herbicides 2,4-D; 2,4,5-T; MCPA; MCPB; 2,4-DB; and dalapon. A group of three chlorophenoxyacetic acid derivatives can be extracted, isolated, and identified by paper chromatography and, finally, determined quantitatively by absorbance measurements of the solution resulting from treatment of the separated organo-chlorine compounds with chromotropic acid, according to procedures described by Erne (*4S*).

Weiss and Gakstatter (*10S*) showed that by using a 30-day exposure of sensitive fish species, it is possible, by means of a biochemical assay method, to detect as little as 0.1×10^{-3} mg. per liter of the more toxic organo-phosphorus pesticides, materials which inhibit brain acetylcholinesterase, the measured factor in the method.

A continuous, multichamber liquid-liquid extractor, with internal solvent recycle, was described by Kahn and Wayman (*8S*). The apparatus was designed specifically for extraction of traces of chlorinated pesticides from relatively large volumes of water.

ORGANICS

There are a number of published reports dealing with the determination of phenol and related materials in water, effluents, wastes, etc. Morkowski (*43T*) Abbott (*1T*), Sharonova and Shul'man (*65T*), Scher (*56T*), Babkin and Voloskovets (*4T*), Kanibalodskaya, Sidorskaya, and Shevlyakov (*26T*), Babeshkina, Kaplin, and Fesenko (*3T*), and Kaplin and Fesenko (*27T*), all reported colorimetric methods. Optimum conditions for the separation of phenol and cresols on strong base, anion-exchange columns prior to their determination colorimetrically with *p*-nitroaniline, were discussed by Magda, Chwaszcza, and Chmielowski (*39T*); methods suitable for the chromatographic separation and ultraviolet spectrophotometric determination of phenols and cresols were reported by Barker and Hollingworth (*5T*).

Phenol, cresols, xylenols, and other industrial and biological phenols, in concentrations of the order of p.p.b. in potable waters were determined by Scholz (*57T*). Low pressure, flash evaporation followed by extraction with ethyl acetate permitted a 10,000-fold concentration of these materials from a 2.5-liter sample. The final solution was analyzed by either paper chromatography or gas chromatography. Kogan and Turaeva (*30T*) adapted an iodometric method to the determination of phenol and other bromine-accepting compounds in natural waters; Naucke *et al.* (*45T*) investigated the specificity of certain spectrophotometric methods for phenol, substituted phenols, cresols, catechol, and similar compounds in waste water. A new, colorimetric method for determining small amounts of phenols in oil refinery waste waters was reported by Mitkalev, Avferenok, and Kozorezov (*41T*).

Several methods have been reported for the determination of formaldehyde and other aldehydes in waste water. Kuchumova, Verigo, and Mamontova (*32T*) and Bodyu and Fel'dman (*7T*) determined aldehydes in the range of from 4 to 50 mg. per liter polarographically, and Stankovic (*69T*) determined 0.02 to 1.0 mg. of formaldehyde per liter by a colorimetric method. Kavan (*28T*) was able to separate and identify the C_2 to C_5 alcohols by gas chromatography and he described conditions for their determination in waste waters. Colorimetric and polarographic methods for the determination of furfural were described by Dyatlovitskaya and Berezovskii (*14T*).

Lur'e and Antipova (*36T*) described a method to determine hexogen in industrial waste waters. Hexogen is first converted into formaldehyde by the action of sulfuric acid, and the formaldehyde determined colorimetrically with chromotropic acid.

The occurrence of humic acids in water has been investigated by several researchers. Semenov, Ivleva, and Datsko (*59T*, *64T*) described a procedure for the separation and determination of these acids by ultraviolet and visible spectrophotometry. The characterization and determination of humic acids by titration with potassium permanganate solution was described by Obenaus (*47T*). Rice, Simon, and Rice (*55T*) developed a relatively simple method for determining a group of naturally occurring organic acids which characteristically foul strongly basic anion-exchange resins. Infrared and ultraviolet examination of a butanol-ether extract provides information which assists in the identification and quantitative estimation of some of these acids. Using gas chromatography and infrared identification, Lamar and Goerlitz (*34T*) were able to establish the presence of 20 carboxylic acids at concentrations which ranged from 4 to 592 p.p.b. for the individual acids in certain surface water samples. The organic material in the sample was first concentrated by continuous liquid-liquid extraction, using butanol as the solvent. Valeric, butyric, pyruvic, lactic, succinic, and maleic acids were among those identified. The method used by Slowey, Jeffrey, and Hood (*67T*), based on extraction of a 6-liter sample of sea water with ethylacetate, preparation of the methyl ester of the extracted fatty acids of 10 to 20 carbon atoms length, and final separation and identification by gas liquid chromatography, enabled estimation of several saturated and unsaturated fatty acids including capric, lauric, myristic, palmitic, stearic, myristoleic, oleic, linoleic, and linolenic. A procedure for the determination of certain lower fatty acids, including butyric and iso- and *n*-valeric acids, was developed by Otsuki (*48T*). The free acids, obtained by steam distillation of the acidified residue from an alkaline evaporation of the original sample, were analyzed by gas chromatography with hydrogen flame detection.

Methods for concentrating and determining amino acids have been reported by Semenov, Ivleva, and Datsko (*58T*, *60T*, *62T*) and by Palmork (*49T*).

Several standard methods of measuring the color of water due to organic solutes were investigated by Ungar and

Thomas (*73T*). They obtained consistent results by measuring the absorbance at 455 mμ of samples which had first been filtered and then passed through an ion-exchange column.

Trichloroethylene in industrial wastes at concentrations of from 3 to 50 p.p.m. was determined nephelometrically by Effenberger and Deyl (*17T*). They first formed trichloroepoxyethane, converted this compound to dichloroacetylchloride which, in turn, is readily hydrolyzed to dichloroacetic acid and hydrochloric acid, the latter of which can be determined as silver chloride.

Bouquiaux and Mertens (*8T*) reported a method for the determination of oil. Benzene in water can be determined with a maximum sensitivity of 0.01 mg. per liter and with a reproducibility of about 2 to 5%, according to a procedure proposed by Devlaminck (*12T*). The benzene, separated by entrainment distillation, is first nitrated and the nitro-compound then extracted with methyl-ethyl ketone whose absorbance is measured at 560 mμ.

A conductometric method for the determination of organic carbon in waste waters was proposed by Effenberger (*16T*). The method, substantially shorter and more precise than gravimetric or volumetric methods, involves oxidation of organic carbon to carbon dioxide, which is then swept into a barium hydroxide solution, the change in conductance of which is proportional to the total amount of carbon compounds originally present. The measurement and control of hydrazine in boiler feed water was discussed by Massart and Missa (*40T*), who investigated the use of *p*-dimethylaminobenzaldehyde as a colorimetric reagent. A highly specific and sensitive method for determining 1,1-dimethylhydrazine was described by Pinkerton *et al.* (*52T*); optimum conditions for its reaction with sodium pentacyanoaminoferrate were established. Sztafrowski (*72T*) devised a procedure for the colorimetric determination of several aromatic hydrocarbons such as styrene, ethylbenzene, and phenylmethyl, which may be present in styrene production wastes. The determination of volatile hydrocarbons in cooling tower water by mass spectrometry was described by Hoggan (*24T*).

The reactions of a number of organic materials, including carboxylic and amino acids, toward several different oxidizing agents were studied by Gorizontova (*21T*). Zdenek and Skorepa (*79T*) compared several methods for determining sugar in water, and an automatic method for the determination of sugar in refinery wastes was developed by Congdon and Zaiatz (*10T*). The method, based on the resorcinol reaction in acid solution, minimizes interference effects of certain divalent ions. The detection of 10 mg. of caprolactam per liter by means of picryl chloride was described by Vaitsekhovskii, Zasimchuk, and Fainerman (*74T*). A method for determining caprolactam was proposed by Wierzbicki (*78T*).

A number of compounds, including several fatty acids, present in industrial wastes were removed by carbon extraction and separated and identified by gas chromatography, according to a procedure developed by Sproul, Caskey, and Ryckman (*68T*).

Permanganate oxidation and catalytic dichromate or persulfate oxidation of organic material were investigated by Leibnitz *et al.* (*35T*) who indicated a preference for the simpler persulfate oxidation method. Low concentrations of organic carbon in fresh or saline waters, including sewage effluent and river water, were accurately determined by Montgomery and Thom (*42T*), who first evaporated an aliquot of sample to dryness, then burned the residual solids in a stream of oxygen. The final measurement of the resulting carbon dioxide was made with an infrared gas analyzer. Hoak (*23T*) pointed out some of the disadvantages of carbon extraction and liquid-liquid extraction for removing and concentrating traces of organics. Although solvent extraction is preferable to carbon extraction, separation and identification by paper chromatography is frequently superior to both. He successfully identified a number of organics by such techniques, including certain phenols, acids, and alcohols.

Methods based on the analysis of phenol extracts of sea water obtained in connection with studies on the metabolites of sea plants and animals were described by Khailov (*29T*). A number of methods for determining hydrocarbons and oils were compared by Ladendorf (*33T*) who concluded that the most suitable are infrared analyses and pycnometer-extraction methods using tetrabromoethane as solvent. Methods for the determination of lignosulfonic acid in sea water were investigated by Komaki (*31T*).

Goncharova and Datsko (*19T*) studied the elementary composition and the oxidizability of the isobutanol extract of both fresh and sea water samples. The sensitivity of the method for the determination of reducing sugars by reduction of copper(II) can be increased substantially by spectrophotometrically determining copper after reaction with diethyldithiocarbamate, according to a publication by Semenov, Ivleva, and Datsko (*61T*, *63T*). The chromatographic adsorption and separation of several colored organic substances was investigated by Shevchenko, Goronovskii, and Zabarilo (*66T*). After separation on a calcium carbonate column, the several fractions were studied spectrophotometrically. A simplified method for determining protein material in fresh water was developed by Povoledo and Gerletti (*53T*).

A high temperature, wet oxidation procedure for the determination of organic carbon in water and wastes was investigated by Weber and Morris (*77T*). Methods used to determine organic carbon in certain coastal waters were described by Camps and Arias (*9T*). The ultraviolet absorption at 220, 230, and 250 mμ of polluted water samples was studied by Hanya and Ogura (*22T*) who were unable to correlate the observed variations in absorbances with specific organic substances.

Total carbon determinations by means of infrared analysis were also described by Van Hall, Safranko, and Stenger (*75T*), and Van Hall and Stenger (*76T*). Stankovic (*70T*) determined traces of hexamethylenetetramine in waste waters colorimetrically after hydrolysis in hot, concentrated sulfuric acid solution. Colorimetric and polarographic methods for the determination of nitrobenzene were described by Dyatlovitskaya and Potemkina (*15T*); and procedures for the determination of traces of aromatic hydrocarbons, including benzene, toluene, and styrene, were described by Lur'e and Panova (*38T*). A method for determining as little as 0.5×10^{-4} mg. of pyridine in samples of surface or waste waters was described by Mrkva (*44T*). Amine nitrogen, of the order of 3 to 4 μg. in a 250-ml. sample, was determined by Nemtseva, Semenov, and Datsko (*46T*) in natural waters. An extraction and colorimetric method for determining acetophenone was reported by Drugov (*13T*). A colorimetric method for the determination of nitrobenzene in waste waters of oil refineries was proposed by Golubeva (*18T*); as little as 0.1 mg. per liter can be determined with an error, in the absence of interferences, of ±5%. The application of gas chromatography to the determination of chlorinated cresols was described by Hrivnak and Schiessl (*25T*).

The direct bioassay of biotin in sea water by means of an unidentified marine bacterium was described by Antia (*2T*). The successful application of an isothermal distillation technique to the determination of the molecular weight of organic substances in water was reported by Goncharova, Stradomskaya, and Datsko (*20T*).

A relatively simple method for the determination of petroleum and turpentine in certain industrial waste waters was described by Przybylski (*54T*), and a detailed discussion of a molybdophosphoric acid method for turpentine was presented by Lur'e and Panova (*37T*). Analytical methods useful for the determination of a number of organic contaminants in oilfield waters were described by Bars, Kogan, and Fikhman (*6T*).

A highly specific method for determining chlorophyll *c* in sea water, based on acid conversion of extracted chlorophyll *c* to pheophytin *c*, was described by Parsons (*50T*). Additional discussions regarding the spectrophotometric determination of marine plant pigments, including the chlorophylls and total carotenoids, were presented by Parsons and Strickland (*51T*). A method for the extraction and determination of vitamin B_{12} in sea water was reported by Suprunov and Muravskaya (*71T*).

Crummett and Hummel (*11T*) determined optimum conditions for the determination of polyacrimides by both distillation–Nesslerization and by nephelometric methods.

RADIOACTIVITY AND ISOTOPIC ANALYSIS

Reviews of methods and apparatus for the measurement of radioactivity in water and wastes were recently published by Bosch (*7U*) and by Schoenberg (*38U*). Reynolds (*34U*) discussed criteria for methods and standards used for water analysis. Methods for the continuous direct measurement of radioactive substances in water were described by Gebauer and Mueller (*19U*) who also discussed the various practical detector systems and their advantages and limitations. Frantz (*18U*) presented details of a method used to measure the radioactivity of surface waters. Plans to study improved methods for specific isotope identifications and for the development of techniques for the separate determination of filterable and nonfilterable radioactivity, and the detection of gross α-, β-, and γ-activities of the order of 10^{-8} μc. per ml. were outlined by Grune, Hughes, and Gibson (*21U*).

Stradomskii (*40U*) developed a method for the determination of total activities of the order of 10^{-12} curie per liter on samples with an inorganic solids content of 3.5 to 4 grams per liter. An end-window counter measured the β-activity of the evaporated solid residue of a suitable sample aliquot, and the activity of the sample was then compared with similarly prepared standards containing known Sr^{90} activities. The advantages of the vibrating condenser electrometer over the commonly used proportional counters, for measuring low-level radioactivity in water, were discussed by Ledbetter and Gloyna (*26U*); and a new device for monitoring radioactivity levels in a liquid stream has been described (*33U*). In connection with a study of the accuracy of methods for measuring radioactivity in water, Eichholz, Hughes, and Nagel (*14U*) investigated the adsorption of traces of certain ions from dilute solutions onto glass and plastic surfaces.

A method for the detection and determination of radioactive contaminants was described by Anokhin, Bylinkina, and Drachev (*2U*), and methods for routine analysis of radioactive materials, including fission products, in rainwater samples were reported by Marques, Grade, and Viana (*28U*), by Lesigang (*27U*), and by Buchtela and Lesigang (*8U*). Nelepo (*29U*) described a scintillation counter used to measure the total radioactivity of sea water. A survey of methods and apparatus for measuring radioactivity in waste water, including continuous monitoring and portable equipment for control measurements, was recently presented by Schanze (*35U*). Haughey and Manganelli (*22U*) described a method used to measure the radioactivity of waste waters.

The several factors affecting the accuracy of gross β-activity determinations were discussed by Donlan (*11U*) who used Tl^{204} standards to permit calibration and evaluation of counting efficiency for samples of different dissolved solids content. Further discussions of errors and precautions necessary in β-activity measurements were included by Barker and Robinson (*4U*) who described a method based on standardization with Sr^{90}–Y^{90}. An ion-exchange technique for removal of trace amounts of Y^{90} and, thus, the determination of Sr^{90}, and the measurement of the gross β-activity of water was described by Hinzpeter (*23U*). A summary of methods used to monitor fallout Sr^{90} in water and other materials was published by Butler (*9U*). Kautsky and Schmitt (*24U*) described the procedure they developed for the determination of low α- and β-activities in sea water. After adding cesium carrier and chemical separation, the filtered solids obtained from a 5-liter sample were transferred to a planchet and counted in a flow counter. Self-absorption was determined to be 30% and a correction was made for the potassium content of the sample. An ion-exchange and coprecipitation technique for the determination of β-activity was proposed by Tolkendorf (*43U*).

Radium concentrations of the order of 10^{-16} to 10^{-17} curie per ml. were determined by α-counting according to a procedure described by Duquesne *et al.* (*12U*). Radium was chemically separated by coprecipitation and purification, the residue mixed with a zinc–silver sulfide phosphor and measured with a photomultiplier tube. Sugimura and Tsubota (*41U*) reported details of a method by which they were able to measure from 0.4 to 1.0 $\times$ 10^{-13} gram of Ra^{226} per liter. Carbonates are first precipitated and redissolved, then, after adding barium carrier, a sodium rhodizonate solution is added to separate the barium and radium which are then redissolved in dilute nitric acid and separated on a strong cation exchanger. The radium is determined by α-spectrometry. A method for the determination, by α-counting, of less than 4 $\times$ 10^{-9} μc. per ml. of Ra^{226} and of traces of Th^{230} were described by Ebersole *et al.* (*13U*). Interferences due to Th^{230} and Ra^{223}, in the case of the radium determination, and of iron, in the Th^{230} determination, are eliminated or allowed for. Foti and Freiling (*17U*) described a technique for determining trace quantities of uranium, neptunium, and plutonium in both sea and fresh water, and for determining the relative amounts of each in its various oxidation states. To measure Ra^{228} at concentration levels of the order of 0.21 to 0.60 $\times$ 10^{-4} gram per liter, Syromyatnikov, Mukashev, and Kapatsinkaya (*42U*) made a double precipitation of barium and radium sulfates, converted these to the corresponding carbonates by a sodium carbonate fusion, and finally measured the β-activity of Ac^{228} in the moist residue obtained from solution and gentle evaporation of the carbonates. The details of an apparatus, utilizing both sodium iodide and plastic scintillation counters, for determining trace amounts of γ-emitting isotopes, were described by Bizollon and Moret (*5U*). They determined the activity of contaminated river water, as well as specific isotopes such as Zr^{95}, Ru^{103}, Ce^{141} or Ce^{144}, and Ru^{106}.

Submicrogram amounts of thorium were determined by a fluorometric procedure developed by Sill and Willis (*39U*). The method, utilizing the reaction between thorium and morin in an alkaline solution of diethylenetriaminepentaacetic acid, will detect as little as 0.01 μg. with good precision.

A rapid method for the determination of radon in water was described by Kerr, Coomber, and Lewis (*25U*) who coprecipitated Bi^{214} with inactive bismuth carrier and were able to detect 20 $\mu\mu$c. per liter by simple β-counting. They pointed out the possible loss of radon from samples stored in polyethylene bottles due to the absorption of this gas by polyethylene. Details of a procedure for the determination of actinium in uranium processing effluents were described by Petrow and Allen (*31U*); purified actinium is obtained from the sample by a series of chemical separations and the in-growth α-activity is observed over a period of several days. Scheidhauer, Messainguiral, and Meiraneisio (*37U*) determined plutonium activity of the order of 7 $\times$ 10^{-11} μc. per cc. by coprecipitation with calcium fluoride, double extraction of plutonium after solution of the fluoride precipitate in nitric acid, first with a xylene solution of thenoyltrifluoroacetone and then with nitric acid, and finally either evaporating to dryness for direct measurement, or radiocounting after electrodeposition on gold. Scheidhauer and Messainguiral (*36U*) also developed a procedure for the selective separation and determination of Ru^{103} and Ru^{106} in river and waste waters.

A study of analytical methods and techniques used by two different laboratories for the determination of Cs^{137} was made by Folsom and Saruhashi (*16U*). They also investigated the relative accuracy of ship and shore laboratory operations. Proctor, Papadopulos, and Firminhac (*32U*) constructed a probe and portable seagoing counting equipment for depth measurements of γ-activity.

A bibliography of methods for the determination of tritium was prepared by Feldman (*15U*). Abdullaev *et al.* (*1U*) described the equipment used to measure tritium activity with a maximum sensitivity of 10^{-11} curie per ml., in which hydrogen is produced by reaction with an aluminum alloy. A method based on liquid scintillation counting after at least a 1000-fold concentration by electrolysis was described by Nishiwaki and Kawai (*30U*).

A rapid, convenient method for the routine determination of deuterium was described by Arnett and Duggleby (*3U*). Reaction of the sample with calcium hydride produces H_2 and HD in identical ratio to the relative amounts of hydrogen and deuterium in the sample. The HD fraction is then determined by standard gas chromatographic techniques using thermal conductivity detection. Tschang (*44U*) discussed several standard methods for isotopic analysis of water, including falling drop, pycnometric, and densitometric methods, and Chou (*10U*) described a procedure which he used to determine the deuterium content of certain bitterns. A differential pycnometric method was developed by Blaga, Blaga, and Chifu (*6U*) to determine small variations within the normal range of deuterium and O^{18} concentrations. A spectrophotometric method for determining the hydrogen content, from 0 to 100%, in heavy water was described by Gordon and Yamatera (*20U*). The absorbance of HDO is measured at 16,680 A. and corrections calculated and applied for the slight absorbances of H_2O and D_2O at this wavelength.

MISCELLANEOUS

Salinity determinations based on inductivity-conductance measurements have been described by Pritchard (*24V*) and by German (*11V*). Williams (*28V*) described a small portable salinometer which operates on flashlight batteries and provides an accuracy of at least ±0.3% salinity.

Several different methods used to determine suspended solids in waste water were evaluated by Smith and Greenberg (*27V*) who recommended the use of a glass fiber filter material to provide convenient and consistently reliable determinations. The use of glass fiber filters has also been described by Wyckoff (*29V*). Jenkins (*17V*) found that the use of preheated glass fiber filters enabled the determination of volatile suspended solids with an accuracy of ±3%, representing an improvement over the standard Gooch crucible method. The superior qualities of glass fiber filters for determining suspended matter and for certain gravimetric determinations were discussed by Giebler and Kempf (*12V*).

A newly designed instrument for taking water samples from streams, rapidly and at varying depths, and which is unaffected by the rate of streamflow, was described by Makinenko (*22V*); Holden (*15V*) described a simple, battery operated, automatic sampler for use in stream pollution studies. Instrumentation for the automatic and continuous measurement and recording of several parameters of importance in pollution studies, including turbidity, conductivity, pH, dissolved oxygen, and both air and water temperature, is described in a report by Keyser (*18V*). Continuous automatic monitoring of silica, hardness, phosphate, chloride, copper, iron, nickel, and chromate in the high-pressure boiler water of steam power plants was reported by Goldberg (*13V*); Britt (*7V*) described instrumentation and methods for stream or batch sampling and spectrophotometric determinations in the low parts-per-billion range of chloride, nitrate, nitrite, iron(II), iron (III), and ammonia in heavy water.

Atomic absorption spectrophotometric methods are finding extensive application in the field of water analysis. Butler and Brink (*8V*) developed procedures for the determination of magnesium, calcium, potassium, sodium, copper, and iron, and designed special burners, operating on acetylene or mixed propane-butane, to improve flame stability and steadiness of the absorbance readings. They found that the addition of 1500 p.p.m. of strontium chloride eliminates interference of magnesium and calcium when determining sodium, and also eliminated the interference of magnesium, sodium, potassium, aluminum, and phosphate when determining calcium. On the other hand, the addition of strontium chloride is unnecessary when determining copper and iron. Herrin (*14V*) discussed the design and construction of a relatively simple atomic absorption photometer for water analysis.

Alkali and alkaline earth metals were determined spectrographically, after concentration by evaporation, according to a procedure reported by Azcona (*3V*). Dickinson and Wheeler (*9V*) pointed out the specific advantages of using a "silver spark" rather than the more familiar "copper spark" method for determining trace metal concentrations in high purity water. They found that silver electrodes provide less general background and fewer interfering lines. For samples containing low total solids, the method is fast, sensitive, and is of the same order of precision as other commonly used spectrographic methods. A spectrographic procedure for determining lithium, barium, strontium, and boron in mineral waters was reported by Jedlewska (*16V*). The acidified samples were repeatedly evaporated in quartz dishes at 1000° C. and ther esidues analyzed spectrographically by comparison with similarly prepared standards.

The extraction and concentration of certain trace elements from dilute solutions by means of diethyldithiocarbamate was studied by Libina *et al.* (*20V*). Copper, zinc, lead, nickel, cobalt, and silver were found to be completely extracted over a pH range of from 3 to 9, whereas vanadium, molybdenum, tungsten, and niobium were best extracted at a lower and relatively narrow pH range, generally at a pH less than 3.0 and in the case of tungsten, at a pH of less than 1.7. The final determination was made by either sensitive colorimetric or emission spectrographic methods.

Concentration and extraction techniques, used in connection with sensitive colorimetric determinations, make possible the convenient field determinations of certain elements of particular interest. Conditions for performing separate chloroform extractions of water samples for the recovery of micro amounts of a number of heavy metals, including nickel, cobalt, silver copper, vanadium, tin, molybdenum, iron, lead, manganese, zirconium, titanium, aluminum, and bismuth were described in considerable detail by Klimov (*19V*). The extractants included sodium diethyldithiocarbamate, ammonium nitrosophenylhydroxylamine, and 8-hydroxyquinoline. The extracts were evaporated and the resulting residues examined spectrographically. Standards were prepared by adding appropriate amounts of solutions of the metals to a solution containing potassium sulfate and carefully measured amounts of strontium and cerium which provided reference lines. Branche and Ropert (*6V*) developed spectrographic techniques for determining lithium, beryllium, tin, vanadium, molybdenum, lead, rubidium, cesium, and thallium in soils, water, and rocks. Conditions for preparing the samples and for determining lithium on one sample portion, beryllium, tin, vanadium, molybdenum, and lead on another portion, beryllium by a direct power-injection method, and rubidium, cesium, and thallium by line-width measurements, were presented. The spectrographic determination of certain micro elements after concentration by coprecipitation was described by Onufrienok, Glazunova, and Solodovnikova (*23V*). Belyaev *et al.* (*4V*) determined trace amounts of iron, lead, tin, nickel, silver, copper, and antimony which were extracted from 250-ml. aliquots of high-purity water, using sodium diethyldithiocarbamate and chloroform. The extract was subsequently evaporated on pure graphite powder and examined spectrographically.

Methods for determining trace amounts of gallium, aluminum, zinc and cadmium, and calcium using fluorescence and chelating agents were reported by Bozhevol'nov *et al.* (*5V*). They used 2,2′,4-trihydroxy-5-chloro-1, 1′ - azobenzene - 3 - sulfonic acid and extraction of the complex with isoamyl alcohol to determine as little as 1 p.p.b. of gallium. As little as 0.5 p.p.b. aluminum was detected using *o*-salicylideneaminophenol; 8-(*p*-tolenesulfonylamino) quinoline was used to determine zinc and cadmium. They also proposed the use of 3,6-dihydroxy-2,4-bis[*N*,*N*-bis(carboxymethyl) aminomethyl]fluorescein as a fluorescent indicator for the chelatometric determination of trace amounts of calcium. Shigematsu *et al.* (*25V*) determined p.p.b. amounts of scandium in sea water by precipitating calcium and scandium oxalates, redissolving the ignited precipitate, and successively extracting the scandium, using first thenoyltrifluoroacetone in acid solution, then 5,7-dichloro-8-quinolinol in first acid then alkaline solution. Lombardi (*21V*) reported preliminary results of an investigation comparing di-β-naphthylthiocarbazone (dinaphthizone) and dithizone as analytical reagents for determining a number of trace heavy metals in brines, sea water, and fresh water. The high molar absorptivity of dinaphthizone and its low acidity, as compared with dithizone, seem to offer advantages over the latter reagent when extracting certain metal complexes from slightly alkaline samples. Neither reagent is clearly preferable to the other for all elements tested or for all types of samples. With but few exceptions, dinaphthizone is generally superior when extractions are made on alkaline brines, but impractical when extractions are made below pH 8. Attempts to determine several metals in a single sample aliquot by first extracting all reacting metals and then successively stripping one or a group from the extractant using appropriate complexing agents were not entirely successful, but did point to the possibility of using such a technique for the analysis of sea water.

Color and turbidity of water may be determined photometrically but appreciable color or turbidity each affects the accuracy of the photometrically-determined value of the other. Acuna (*1V*) developed nomograms which conveniently permit a proper correction to be applied in such determinations.

A new model of a null-type photoelectric colorimeter for field use was described by Sinyukov and Skopintsev (*26V*). A wavelength range of from 313 to 720 mμ is covered with nine filters. It accommodates 300-mm. long cuvets of 45-ml. capacity and provides increased sensitivity for nitrogen, phosphorus, and silicon in sea water (0.5 mg., 2 to 3 mg., and 50 mg. per cu. meter, respectively). Faganelli (*10V*) reported on field methods used to determine silicon, phosphorus, and nitrate-, nitrite-, and ammonia-nitrogen in sea water as a part of the investigations of the International Geophysical Year. Certain aspects of the use of ion-exchange resins and chromatography in the analysis of water, waste water, and radioactive waste water were discussed by Albersmeyer (*2V*).

Phenomena observed during the potentiometric titration of oilfield waters using a platinum electrode were noted by Yavorskii and Timofeeva (*30V*); the oxidation of reducing agents and of iodide was observed. Zhukova (*31V*) described a method for calculating the correction for the gaseous component, in solutions of 0.7 to 3.0 mg. of ammonia per liter at pH 7 to 10, when determining the salt concentration in steam condensate electrometrically. The corrections are presented graphically.

LITERATURE CITED

Introduction

(1) Ballinger, D. G., *J. Water Pollution Control Federation* **35,** 116–22 (1963).

(2) Borovik-Romanova, T. F., Kutsenko, Yu. I., *Spektral'n. Opred. Redkikh i Rasseyan. Elementov, Akad. Nauk SSSR, Inst. Geokhim. i Analit. Khim.* **1962,** 204–38; *C.A.* **57,** 11831*f* (1962).

(3) Collins, A. G., Pearson, C., Attaway, D. H., Ebrey, T. G., *U. S. Bur. Mines, Rept. Invest.* **6087** (1962).

(4) Constable, F. H., *Istanbul Univ. Fen Fak. Mecmuasi Ser. C* **28** (3–4), 130–4 (1963) (in English); *C.A.* **61,** 4061*a* (1964).

(5) Dall'Aglio, M., Tonani, F., *Studi Ric. Div. Geomineraria, Comit. Nazl. Ric. Nucl.* **3,** 329–51 (1960); *C.A.* **58,** 9956*b* (1963).

(6) Goldberg, E. D., *Microchem. J. Symp. Ser.* 1, 55–68 (1961).

(7) Goya, H., Zigman, P., Lai, M., Mackin, J., *U. S. Dept. Com., Office Tech. Serv.,* **AD 404,884** (1963).

(8) Humphrey, W. H., *Effluent Water Treat. J.* **2,** 505–8 (1962).

(9) *J. Water Pollution Control Federation* **34,** 419–27 (1962).

(10) *Ibid.,* **35,** 553–9 (1963).

(11) *Ibid.,* **36,** 535–72 (1964).

(12) Konovalov, G. S., *Sovrem. Metody Analiza Prirodn. Vod., Akad. Nauk SSSR* **1962,** 143–57; *C.A.* **58,** 13608*f* (1963).

(13) Leddicotte, G. W., Moeller, D. W., *U. S. At. Energy Comm.* **CF-61-5-118** (1961).

(14) Lishka, R. J., Kelso, F. S., Kramer, H. P., *J. Am. Water Works Assoc.* **55,** 647–56 (1963).

(15) Mackereth, F. J. H., *Freshwater Biol. Assoc., Sci. Publ.* **21,** 70 (1963).

(16) Matveev, A. A., *Sovrem. Metody Analiza Prirodn. Vod., Akad. Nauk SSSR* **1962,** 5–11; *C.A.* **59,** 11099*h* (1963).

(17) Mazel, M. I., Akulovich, V. M., *Tr. Soveshch. po Ispel'z. i Obogashch. Kaliinykh Solei Belorussii* **1961,** 203–16; *C.A.* **58,** 9606*g* (1963).

(18) Pohl, F. A., *Z. Anal. Chem.* **197,** 193–7 (1963); *C.A.* **60,** 12*e* (1964).

(19) Selz, J., Haerdi, W., Monnier, D., *Chimia* **17,** 354–5 (1963) (in French); *C.A.* **60,** 6197*h* (1964).

(20) Skougstad, M. W., Fishman, M. J., ANAL. CHEM. **35,** 179R–204R (1963).

(21) Sokolov, I. Yu., Brodskii, A. A., Miller, A. D., Reznikov, A. A., *Byul. Nauchn.-Tekhn. Inform. Min. Geol. i Okhrany Nedr. SSSR* **10,** 90–5 (1962); *C.A.* **60,** 14252*c* (1964).

(22) Sokolov, I. Yu., Polyakov, V. A., Lushnikov, V. V., *Tr. Vses. Nauchn.-Issled. Inst. Gidrogeol. i Inzh. Geol.* **19,** 183–8 (1961); *C.A.* **58,** 2272*a* (1963).

(23) Sussman, S., Portnoy, I. L., Jacobson, J., *Am. Soc. Testing Mater., Spec. Tech. Publ.* **337,** 6–13 (1962).

(24) Terhaag, L., *Arch. Hyg. Bakteriol.* **146,** 268–91 (1962); *C.A.* **57,** 16337*e* (1962).

Alkali Metals

(1A) Berezina, M. D., *Tr. Gos. Inst. po Proektir i Issled. Rabotam Neftedobyvayushchei Prom. "Giprovostokneft"* **3,** 85–9 (1961); *C.A.* **58,** 345*f* (1963).

(2A) Chow, T. J., *Anal. Chim. Acta* **31,** 58–63 (1964) (in English).

(3A) Chow, T. J., Goldberg, E. D., *J. Marine Res.* **20,** 163–7 (1962).

(4A) Collins, A. G., *U. S. Bur. Mines, Rept. Invest.* **6047** (1962).

(5A) Collins, A. G., ANAL. CHEM. **35,** 1258–61 (1963).

(6A) Derecki, J., Geisler, J., Jankovic, D., Jaworowski, Z., *Nukleonika* **8,** 69–74 (1963) (in English); *C.A.* **59,** 12161*b* (1963).

(7A) Djavad-Nia, D., *Ann. Pharm. Franc.* **20,** 105–8 (1962); *C.A.* **58,** 350*c* (1963).

(8A) Dobremyslova, I., Dvorak, J., Novobilsky, V., *Chem. Prumysl* **12,** 287–9 (1962); *C.A.* **57,** 13553*h* (1962).

(9A) Dolezal, J., Novozamsky, I., Zyka, J., *Collection Czech. Chem. Commun.* **27,** 1830–4 (1962) (in German); *C.A.* **57,** 13179*e* (1962).

(10A) Efendi, M. E., Sokolovich, V. B., *Izv. Tomsk. Politekhn. Inst.* **102,** 138–40 (1959); *C.A.* **58,** 4361*d* (1963).

(11A) Feldman, C., Rains, T. C., ANAL. CHEM. **36,** 405–9 (1964).

(12A) Ferraris, M. M., *Comit. Nazl. Energia Nucl.* **RT/PROT 13,** 8 (1963); *C.A.* **60,** 15119*h* (1964).

(13A) Frantsuzova, T. A., *Sb. Nauchn. Tr. Nauchn.-Issled. Gornorudn. Inst.*

Ukr. SSR **5,** 284–91 (1962); *C.A.* **58,** 13129*a* (1963).
(14A) Golchert, N., Sedlet, J., *U. S. At. Energy Comm.* **OP-831** (1962).
(15A) Golding, R. M., Speer, M. G., *New Zealand J. Sci.* **4,** 203–13 (1961).
(16A) Goremykin, V. E., *Sovrem. Metody Analiza Prirodn. Vod., Akad. Nauk SSSR* **1962,** 22–31; *C.A.* **60,** 3871*d* (1964).
(17A) Gupta, R. S., Ramaswamy, D., *Leather Sci.* (Madras, India) **10,** 255–8 (1963); *C.A.* **59,** 11101*e* (1963).
(18A) Ivanova, L. M., Shvedov, V. P., *Radiokhimiya* **5,** 185–9 (1963); *C.A.* **60,** 9017*a* (1964).
(19A) Jonsson, G., *Aktiebolaget Atomenergi, Stockholm* **AE-105,** 9 (1963) (in English); *C.A.* **59,** 8472*h* (1963).
(20A) Kalhousova, H., *Vodni Hospodarstvi* **13,** 108–9 (1963); *C.A.* **59,** 1378*f* (1963).
(21A) Kriventsov, M. I., *Sovrem. Metody Analiza Prirodn. Vod., Akad. Nauk SSSR* **1962,** 20–1; *C.A.* **59,** 11101*a* (1963).
(22A) Li, L., Huang, C., *Hua Hsueh Tung Pao* **1964,** 50–2; *C.A.* **61,** 444*g* (1964).
(23A) Mamedov, I. A., Emirdzhanova, A. A., *Izv. Vysshikh Uchebn. Zavedenii, Neft i Gas.* **6,** 84 (1963); *C.A.* **60,** 10387*e* (1964).
(24A) Mohanrao, G. J., Folsom, T. R., *Analyst* **88,** 105–8 (1963).
(25A) Morgan, A., Arkell, G. M., *Health Phys.* **9,** 857–62 (1963).
(26A) Shvedov, V. P., Ivanova, L. M., Zharikov, V. I., *Radiokhimiya* **5,** 182–5 (1963); *C.A.* **59,** 13687*a* (1963).
(27A) Uzumasa, Y., Nasu, T., Terae, K., *Nippon Kagaku Zasshi* **83,** 1253–7 (1962); *C.A.* **58,** 9956*c* (1963).
(28A) Yamagata, N., *Nature* **200,** 157–8 (1963).
(29A) Yofe, J., Stiller, M., *U. S. At. Energy Comm.* **IA-712** (1962).

Hardness, Alkaline Earth Metals

(1B) Bakacs-Polgar, E., Kurcz-Csiky, I., *Z. Analit. Khim.* **18,** 1206–10 (1963); *C.A.* **60,** 2311*d* (1964).
(2B) Baumgartel, K., Kolb, W., *J. Geophys. Res.* **68,** 3833–4 (1963); *C.A.* **59,** 7238*c* (1963).
(3B) Boni, A. L., ANAL. CHEM. **35,** 744–7 (1963).
(4B) Bozhevol'nov, E. A., Kreingol'd, S. U., *Zh. Analit. Khim.* **17,** 560–4 (1962); *C.A.* **58,** 2834*c* (1963).
(5B) Bozhevol'nov, E. A., Kreingol'd, S. U., *Metody Analiza Khim. Reaktivov i Preparatov, Gos. Kom. Sov. Min. SSSR po Khim.* **4,** 85–8 (1962); *C.A.* **61,** 3675*a* (1964).
(6B) Collins, A. G., Pearson, C. A., ANAL. CHEM. **36,** 787–9 (1964).
(7B) Demmitt, T. F., Burt, M. C., *U. S. At. Energy Comm.* **HW-77538** (1963).
(8B) Diehl, H., Olsen, R., Spielholtz, G. I., Jensen, R., ANAL. CHEM. **35,** 1144–54 (1963).
(9B) Dunaeva, Yu. N., *Gigiena i Sanit.* **28,** 58–61 (1963); *C.A.* **60,** 6622*f* (1964).
(10B) Dziedzianowicz, W., *Acta IMEKO (Intern. Messtech. Konf.)* **1961, 4,** 356–70 (in German); *C.A.* **59,** 8118*g* (1963).
(11B) Elfers, L. A., Hallbach, P. F., Velten, R. J., ANAL. CHEM. **36,** 540–3 (1964).
(12B) Fritz, J. S., Garralda, B. B., *Ibid.,* **36,** 737–41 (1964).
(13B) Gloyna, E. F., Bhagat, S. K., Felsing, W. A., Jr., *J. Water Pollution Control Federation* **35,** 893–902 (1963).
(14B) Guerello, L. O., *Org., Direc., Nacl. Geol. Mineria* **1963,** 8 [Separate]; *C.A.* **60,** 9017*c* (1964).
(15B) Hemmeler, A., Bernardini, P., *Studi Urbinati, Fac. Farm.* **35,** 70–8 (1961); *C.A.* **59,** 3647*c* (1963).
(16B) Herring, H., *Wasser Luft Betrieb.* **6,** 465–7 (1962); *C.A.* **59,** 12159*d* (1963).
(17B) Herszberg, B., Dutailly, M. T., Burg, C., *Min. Sante Publ.* (France), *Rappt. SCPRI* 81 (1962); *C.A.* **59,** 12508*a* (1963).
(18B) Januszkiewicz, T., *Gospodar. Wodna* **1960,** 513–16; *C.A.* **57,** 14895*b* (1962).
(19B) Katz, H., Navone, R., *J. Am. Water Works Assoc.* **56,** 121–3 (1964).
(20B) Lacy, J., *Talanta* **10,** 1031–40 (1963) (in English).
(21B) Litovchenko, G. D., Shipitsyn, S. A., *Mikroelementy v Pochvakh, Vodakh i Organizmakh Vost. Sibiri i Dal'nego Vostoka i ikh Rol'v Zhizni Rast., Zhivotnykh i Cheloveka, Akad. Nauk SSSR, Sibirsk. Otd., Tr. Pervoi Konf., Ulan-Ude* **1960,** 251–7 (Pub. 1961); *C.A.* **59,** 13292*g* (1963).
(22B) Lukin, A. M., Smirnova, K. A., Zavarikhina, G. B., Zh. *Analit. Khim.* **18,** 444–9 (1963); *C.A.* **59,** 3301*e* (1963).
(23B) Melamed, S., *Uch. Zap. Mordovsk. Gos. Univ.* **1962,** 52–6; *C.A.* **60,** 10385*e* (1964).
(24B) Morgan, G. B., Galbraith, E. H., Gilcreas, F. W., *Water Works Eng.* **116,** 33, 53 (1963).
(25B) Olsen, R., Diehl, H., ANAL. CHEM. **35,** 1142–4 (1963).
(26B) Osmond, R. G., Healy, C., Marshall, G. F., *Analyst* **86,** 616–18 (1961).
(27B) Rial, J. R. B., Molins, L. R., *Bol. Inst. Espan. Oceanog.* 111 (1962); *C.A.* **58,** 6572*d* (1963).
(28B) Rowe, J. J., *Geochim. Cosmochim. Acta* **27,** 915–23 (1963).
(29B) Serebryakova, G. V., Bozhevol'nov, E. A., Godlina, G. S., *Metody Analiza Khim. Reaktivov i Preparatov, Gos. Kom. Sov. Min. SSSR po Khim.* **4,** 92–5 (1962); *C.A.* **61,** 3681*a* (1964).
(30B) Shigematsu, T., Tabushi, M., Isojima, F., *Bunseki Kagaku* **11,** 752–6 (1962); *C.A.* **57,** 9594*i* (1962).
(31B) Stone, R. G., *Analyst* **88,** 56–8 (1963).
(32B) Svishchev, G. M., Nikolaev, Yu. M., Novikova, N. Ya., Golutvina, M. M., *Gigiena i Sanit.* **29,** 65–71 (1964); *C. A.* **61,** 4060*h* (1964).
(33B) Szmytowna, M., Latour, J., *Poznan. Towarz. Przyjaciol Nauk, Wydzial Lekar., Prace Komisji Farm.* **1,** 31–8 (1962); *C.A.* **58,** 8774*b* (1963).
(34B) Uesugi, K., Tabushi, M., Murakami, T., Shigematsu, T., *Bunseki Kagaku* **13,** 440–4 (1964);. *C.A.* **61,** 6777*h* (1964).
(35B) *U. K. At. Energy Authority, Prod. Group Rept.* **PG 311(W)** (1962).
(36B) Visapaa, A., *Teknillisen Kemian Aikakauslehti* **20,** 146–51 (1963) (in English); *C.A.* **59,** 8121*c* (1963).
(37B) *Ibid.,* **20,** 563–8 (1963); *C.A.* **60,** 7793*b* (1964).
(38B) Wuenscher, H. J., Hoffmann, H., *Chem. Tech.* (Berlin) **15,** 492 (1963); *C.A.* **59,** 15028*e* (1963).
(39B) Zizin, V. G., Proskuryakov, L. M., Yakovets, V. V., Shklovskii, Ya. A., *Tr. Bashkirsk. Nauchn.-Issled. Inst. po Pererabotke Nefti* **5,** 296–8 (1962); *C.A.* **58,** 4301*g* (1963).

Iron, Manganese, Chromium, Aluminum, and Rhenium

(1C) Albinati, J. F. P. de, *Anales Assoc. Quim. Arg.* **51,** 96–122 (1963); *C.A.* **60,** 1096*h* (1964).
(2C) Bahensky, Vl., Zika, Zd., Lastovkova, O., *Korose Ochrana Mater.* **1962,** 96–7; *C.A.* **58,** 13597*d* (1963).
(3C) Barker, M. R., *Proc. Soc. Water Treat. Exam.* **12,** 233 (1963).
(4C) Goto, K., Komatsu, T., Furukawa, T., *Anal. Chim. Acta* **27,** 331–4 (1962) (in English).
(5C) Gustin, V. K., Sweet, T. R., ANAL. CHEM. **36,** 1674–6 (1964).
(6C) Hellwig, D. H. R., Steenderen, R. A. van, *Water Waste Treat. J.* **9,** 118–20 (1963).
(7C) Ito, M., Musha, S., *Bunseki Kagaku* **13,** 6–10 (1964); *C.A.* **60,** 9017*c* (1964).
(8C) Klump, W., Busch, H., *Mitt. Ver. Grosskesselbesitzer* **78,** 215–17 (1962); *C.A.* **58,** 3198*e* (1963).
(9C) Kostrikin, Yu. M., Kalinina, N. M., Dzysyuk, A. A., *Teploenergetika* **9,** 37–42 (1962); *C.A.* **57,** 14898*a* (1962).
(10C) Kreingol'd, S. U., Bozhevol'nov, E. A., Lastovskii, R. P., Sidorenko, V. V., *Zh. Analit. Khim.* **18,** 1356–61 (1963); *C.A.* **60,** 6204*f* (1964).
(11C) Krusenstjern, A. v., Harst, H., *Metalloberflaeche* **13,** 315–16 (1959); *C.A.* **60,** 2631*c* (1959).
(12C) Lisenko, N. F., Mustafin, I. S., Molot, L. A., *Izv. Vysshikh Uchebn. Zavedenii, Khim. i Khim. Tekhnol.* **5,** 712–16 (1962); *C.A.* **58,** 8771*g* (1963).
(13C) Mokievskaya, V. V., *Tr. Inst. Okeanol., Akad. Nauk SSSR* **54,** 115–22 (1962); *C.A.* **58,** 7719*f* (1963).
(14C) Nechaeva, A. A., *Inform. Sb. Vses. Nauchn.-Issled. Geol. Inst.* **51,** 151–4 (1961); *C.A.* **59,** 8473*f* (1963).
(15C) Noll, C. A., Stefanelli, L. J., ANAL. CHEM. **35,** 1914–16 (1963).
(16C) Plsko, E., *Chem. Anal.* (Warsaw) **7,** 239–44 (1962) (in German); *C.A.* **57,** 12271*d* (1962).
(17C) Plsko, E., Blazej, A., *Veda Vyzkum Prumyslu Kozedelnem.* **7,** 133–42 (1963); *C.A.* **60,** 11743*c* (1964).
(18C) Shidlovskaya-Ovchinnikova, Yu. S., *Gidrokhim. Materialy* **24,** 33–5 (1955); *C.A.* **51,** 9981*d* (1957).
(19C) *Ibid.,* **35,** 168–76 (1963); *C.A.* **59,** 8472*f* (1963).
(20C) Solomin, G. A., Fesenko, N. G., *Ibid.,* **33,** 128–33 (1961); *C.A.* **57,** 9596*g* (1962).
(21C) Solomin, G. A., Fesenko, N. G., *Sovrem. Metody Analiza Prirodn. Vod., Akad. Nauk. SSSR* **1962,** 57–60; *C.A.* **58,** 11097*d* (1963).
(22C) Tetlow, J. A., Wilson, A. L., *Analyst* **89,** 442–52 (1964).
(23C) Wakimoto, S., *Bunseki Kagaku* **10,** 968–71 (1961); *C.A.* **58,** 6573*d* (1963).
(24C) Wilson, A. L., *Analyst* **88,** 389–401 (1964).
(25C) *Ibid.,* pp. 402–10.
(26C) Zavodnov, S. S., *Sovrem. Metody Analiza Prirodn. Vod., Akad. Nauk SSSR* **1962,** 61–2; *C.A.* **58,** 12295*a* (1963).

Copper, Zinc, Lead, Cadmium, and Nickel

(1D) Ariel, M., Eisner, U., *J. Electroanal. Chem.* **5,** 362–74 (1963).
(2D) Ballinger, D. G., Hartlage, T. A., *Water Sewage Works* **109,** 338–41 (1962).
(3D) Billikova, A., Billik, V., *Vodni Hospodarstvi* **13,** 437–8 (1963); *C.A.* **60,** 10366*c* (1964).
(4D) Boichinova, E. S., Aleskovskii, V. B., *Tr. Leningr. Tekhnol. Inst. im. Lenosveta* **48,** 94–100 (1958); *C.A.* **59,** 9308*d* (1963).
(5D) Bougis, P., *J. Conseil, Conseil Perm. Intern. Exploration Mer* **28,** 171–4 (1963); *C.A.* **60,** 10386*b* (1964).
(6D) Bozhevol'nov, E. A., Kreingol'd, S. U., *Metody Analiza Khim. Reaktivov*

i Preparatov, Gos. Kom. Sov. Min. SSSR po Khim. **4,** 96–9 (1962); *C.A.* **61,** 2467*f* (1964).
(7D) Chistyakov, N. M., Blagoveshchenskaya, Z. I., *Gigiena i Sanit.* **28,** 58–61 (1963); *C.A.* **59,** 7238*g* (1963).
(8D) Comte, A., Ducluzeaux, F., Boucherle, A., Badinand, A., *Bull. Trav. Soc. Pharm. Lyon* **6,** 55–60 (1962); *C.A.* **59,** 11100*c* (1963).
(9D) Dobychin, S. L., Aleskovskii, V. B., *Tr. Leningr. Tekhnol. Inst. Lensoveta* **48,** 45–8 (1958); *C.A.* **58,** 1901*g* (1963).
(10D) Evstifeev, M. M., Kovalenko, P. N., *Elektrokhim. i Optich. Metody Analiza Sb.* **1963,** 78–81; *C.A.* **61,** 444*c* (1964).
(11D) Fabricand, B. P., Sawyer, R. R., Ungar, S. G., Adler, S., *Geochim. Cosmochim. Acta* **26,** 1023–7 (1962) (in English).
(12D) Hissel, J., Cadot-Deithier, M., *Tribune CEBEDAU* **15,** 549–54 (1962); *C.A.* **58,** 9956*g* (1963).
(13D) Ishibashi, M., Shigematsu, T., Tabushi, M., Nishikawa, Y., Goda, S., *Nippon Kagaku Zasshi* **83,** 295–8 (1962); *C.A.* **57,** 10994*b* (1957).
(14D) Kreingol'd, S. U., Bozhevol'nov, E. A., *Metody Analiza Khim. Reaktivov i Preparatov, Gos. Kom. Sov. Min. SSSR po Khim.* **4,** 100–7 (1962); *C.A.* **61,** 2468*a* (1964).
(15D) Kyuregyan, E. A., *Izv. Akad. Nauk Arm. SSSR, Geol. i Geogr. Nauki* **16,** 163–6 (1963); *C.A.* **60,** 3870*h* (1964).
(16D) Loveridge, B. A., Owens, A. F., *At. Energy Res. Estab.* (Gt. Brit.) **AERE-R-3945** (1962).
(17D) Lukin, A. M., Chernaya, L. S., Petrova, G. S., Sosnina, A. I., *Zavodsk. Lab.* **28,** 398–401 (1962); *C.A.* **58,** 2838*f* (1963).
(18D) Markova, A. L., *Zh. Analit. Khim.* **17,** 952–4 (1962); *C.A.* **58,** 11097*h* (1963).
(19D) Natelson, S., Leighton, D. R., Calas, C., *Microchem. J.* **6,** 539–56 (1962).
(20D) Nehring, D., *Z. Fischerei* **10,** 379–81 (1961); *C.A.* **58,** 8774*d* (1963).
(21D) Rona, E., Hood, D. W., Muse, L., Buglio, B., *Limnol. Oceanog.* **7,** 201–6 (1962).
(22D) Rozenfel'd, A. S., *Tr. Leningr. Sanit.-Gigien. Med. Inst.* **69,** 144–9 (1961); *C.A.* **58,** 12294*e* (1963).
(23D) Sambueva, A. S., Shipitsyn, S. A., *Mikroelementy v Pochvakh, Vodakh i Organizmakh Vost. Sibiri i Dal'nego Vostoka i ikh Rol v Zhizni Rast. Zhivotnykh i Cheloveka, Akad. Nauk SSSR, Sibirsk. Otd., Tr. Pervoi Konf., Ulan-Ude* **1960,** 263–7 (Pub. 1961); *C.A.* **59,** 8473*a* (1963).
(24D) Snow, E. A., *J. New Engl. Water Works Assoc.* **74,** 250–8 (1960).
(25D) Wilson, A. L., *Analyst* **87,** 884–94 (1962).

Silver, Gold, and Mercury

(1E) Markova, A. N., Astaf'eva, E. A., *Byul. Nauchn.-Tekhn. Inform. Min. Geol. i Okhrany Nedr SSSR* **1962,** 70–1; *C.A.* **60,** 14253*b* (1964).
(2E) Pakhomova, K. S., Vysotskaya, T. A., *Tr. Yakutskogo Filiala Sibirsk. Otd. Akad. Nauk SSSR, Ser. Geol.* **16,** 75–80 (1963); *C.A.* **59,** 9309*b* (1963).
(3E) Uman, G. A., *J. Am. Water Works Assoc.* **55,** 205–8 (1963).
(4E) Vasilevskaya, A. E., Shcherbakov, V. P., Levchenko, A. V., *Zh. Analit. Khim.* **18,** 811–15 (1963); *C.A.* **59,** 8473*b* (1963).
(5E) Weiss, H. V., Lai, M., *Anal. Chim. Acta* **28,** 242–8 (1963) (in English).

Vanadium, Molybdenum, Titanium, Zirconium, Niobium, Tin, Germanium, and Uranium

(1F) Alberti, G., Grassini, G., Di Ferrante, E., *Studi Ric. Div. Geomineraria, Comit. Nazl. Ric. Nucl.* **3,** 447–54 (1960); *C.A.* **58,** 9957*g* (1963).
(2F) Brudz, V. G., Shafran, I. G., Smirnova, K. A., Drapkina, D. A., Zelichenok, S. L., Podol'skaya, B. L., *Tr. Vses. Nauchn.-Issled. Inst. Khim. Reaktivov* **25,** 17–23 (1963); *C.A.* **60,** 15138*h* (1964).
(3F) Fishman, M. J., Skougstad, M. W., Anal. Chem. **36,** 1643–6 (1964).
(4F) Grassini, G., Alberti, G., *Microchem. J. Symp. Ser.* **2,** 285–91 (1962).
(5F) Hamaguchi, H., Kawabuchi, K., Onuma, N., Kuroda, R., *Anal. Chim. Acta* **30,** 335–45 (1964) (in English).
(6F) Hampson, B. L., *Analyst* **88,** 529–33 (1963).
(7F) Herrmann, M., *Z. Anal. Chem.* **190,** 78–80 (1962); *C.A.* **58,** 5037*g* (1963).
(8F) Ilica, D. D., *Rev. Chim.* (Bucharest) **13,** 377 (1962); *C.A.* **58,** 350*b* (1963).
(9F) Konovalov, G. S., *Sovrem. Metody Analiza Prirodn. Vod., Akad. Nauk SSSR* **1962,** 52–6; *C.A.* **58,** 12295*c* (1963).
(10F) Koyama, K., Michelson, C. E., Alkire, G. J., *U. S. At. Energy Comm.* **HW-30148** (1953) (Declassified 1960).
(11F) Nikolov, K., Kamburov, Khr., *Izv. Fiz. Inst. s ANEB, Bulgar. Akad. Nauk* **10,** 67–73 (1962); *C.A.* **58,** 6573*g* (1963).
(12F) Reznikov, A. A., Mulikovskaya, E. P., *Inform. Sb. Vses. Nauchn.-Issled. Geol. Inst.* **51,** 135–41 (1961); *C.A.* **58,** 13610*b* (1963).
(13F) *Ibid.,* **51,** 143–9 (1961); *C.A.* **58,** 13610*a* (1963).
(14F) Singer, E., Marecek, J., *Z. Anal. Chem.* **196,** 321–3 (1963); *C.A.* **59,** 11101*f* (1963).
(15F) Suzuki, M., Takeuchi, T., *Kogyo Kagaku Zasshi,* **66,** 191–4 (1963); *C.A.* **59,** 12175*b* (1963).

Boron, Selenium, and Tellurium

(1G) Brouchek, F. I., *Tr. Gruzinsk. Politekhn. Inst.* **5,** 27–32 (1962); *C.A.* **60,** 7793*g* (1964).
(2G) Collins, A. G., Waters, C. J., Pearson, C. A., *U. S. Bur. Mines, Rept. Invest.* **6474** (1964).
(3G) Eristavi, D. I., Brouchek, F. I., *Tr. Gruzinsk. Politekhn. Inst.* **5,** 17–26 (1962); *C.A.* **60,** 7793*e* (1964).
(4G) Eristavi, D. I., Brouchek, F. I., *Soobshch. Akad. Nauk Gruz. SSR* **30,** 565–72 (1963); *C.A.* **60,** 1410*h* (1964).
(5G) Eristavi, D. I., Brouchek, F. I., Cheishvili, L. I., *Tr. Gruzinsk. Politekhn. Inst.* **5,** 3–15 (1962); *C.A.* **60,** 7793*c* (1964).
(6G) Glebovich, T. A., *Tr. Vses. Nauchn.-Issled. Inst. Gidrogeol. i Inzh. Geol., Sb.* **19,** 172–83 (1961); *C.A.* **58,** 1240*a* (1963).
(7G) Greenhalgh, R., Riley, J. P., *Analyst* **87,** 970–6 (1962).
(8G) Korobov, D. S., Lipovskii, M. A., *Geokhim. Sb. Saratovsk. Univ.* **1963,** 177–80; *C.A.* **61,** 6676*e* (1964).
(9G) Murakami, T., *Himeji Kogyo Daigaku Kenkyu Hokoku* **14,** 108–15 (1961); *C.A.* **58,** 924*h* (1963).
(10G) Rainwater, F. H., Thatcher, L. L., *U. S. Geol. Surv., Water-Supply Paper* **1454** (1960).
(11G) Roquebert, J., Vitte, G., *Bull. Soc. Pharm. Bordeaux* **101,** 29–32 (1962); *C.A.* **57,** 1996*e* (1962).
(12G) Rowe, J. J., *U. S. Geol. Surv., Profess. Paper* **450-B,** 114–16 (1962).
(13G) Vera, M. A., *Anales Fac. Quim. Farm., Univ. Chile* **14,** 149–55 (1962); *C.A.* **60,** 6202*h* (1964).

Chloride, Bromide, and Iodide

(1H) Bystritskii, A. L., Aleskovskii, V. B., Bardin, V. V., *Izv. Vysshikh Uchebn. Zavedenii, Khim. i Khim. Tekhnol.* **6,** 31–4 (1963); *C.A.* **59,** 9323*d* (1963).
(2H) Ceausescu, D., *Rev. Chim.* (Bucharest) **11,** 241 (1960); *C.A.* **57,** 9596*i* (1962).
(3H) Gladyshev, V. P., Kalvoda, R., *Zavodsk. Lab.* **27,** 1456–8 (1962).
(4H) Hadjiioannou, T. P., *Anal. Chim. Acta* **30,** 488–94 (1964) (in English).
(5H) Iwasaki, I., Utsumi, S., Kang, C., *Bull. Chem. Soc. Japan* **36,** 325–31 (1963); *C.A.* **58,** 13109*g* (1963).
(6H) Jungreis, E., Gedalia, I., *Mikrochim. Acta* **1960,** 145–9 (in English).
(7H) Kalvoda, R., Gladyshev, V. P., *Collection Czech. Chem. Commun.* **27,** 2365–71 (1962) (in German); *C.A.* **58,** 2846*c* (1963).
(8H) Kobyak, G. G., Bondareva, E. G., *Uch. Zap. Permsk. Gos. Univ.* **25,** 81–3 (1963); *C.A.* **61,** 1606*a* (1964).
(9H) Koske, P. H., *Kiel. Meeresforsch.* **19,** 182–8 (1963); *C.A.* **60,** 13017*e* (1964).
(10H) Milicevic, V., Knezevic, A., Custovic, F., *Glasnik Drustva Hemicara Technol. Nr Bosne Hercegovine* **10,** 63–6 (1961); *C.A.* **61,** 6776*h* (1964).
(11H) Miller, A. D., Shneider, L. A., *Zh. Analit. Khim.* **18,** 371–6 (1963); *C.A.* **58,** 13609*d* (1963).
(12H) Palei, P. N., Udal'tsova, N. I., *Zavodsk. Lab.* **30,** 151–3 (1964); *C.A.* **60,** 13017*d* (1964).
(13H) Panasyuk, V. J., Yaroshevich, R. A., *Metody Issled. i Kontrolya Steklovolokn. Materialov Sb.* **1963,** 62–6; *C.A.* **61,** 413*e* (1964).
(14H) Proskuryakova, G. F., Shveikina, R. V., Chernavina, M. C., *Izv. Vysshikh Uchebn. Zavedenii, Khim. i Khim. Tekhnol.* **6,** 729–34 (1633); *C.A.* **60,** 9017*d* (1964).
(15H) Stolc, V., *Mikrochim. Ichnoanal. Acta* **1963,** 894–900; *C.A.* **60,** 4781*e* (1964).
(16H) Tomonari, A., *Nippon Kagaku Zasshi* **82,** 864–9 (1961); *C.A.* **57,** 11858*a* (1962).
(17H) *Ibid.,* **83,** 693–5 (1962); *C.A.* **57,** 10529*a* (1962).
(18H) Woelk, H. U., *U. S. At. Energy Comm.* **HMI-B7** (1959).
(19H) Yatsimirskii, K. B., Budarin, L. I., Blagoveschenskaya, N. A., Smirnova, R. V., Fedorova, A. P., Yatsimirskii, V. K., *Zh. Analit. Khim.* **18,** 403–8 (1963); *C.A.* **59,** 1081*b* (1963).
(20H) Zimmerman, M., Stocmeier, L. v., *Mitt. Ver. Grosskesselbesitzer* **60,** 203–4 (1960); *C.A.* **60,** 10385*h* (1964).
(21H) Zitomer, F., Lambert, J. L., Anal. Chem. **35,** 1731–4 (1963).

Fluoride

(1J) Banerjee, G., Chowdhury, A. N., *Indian Minerals* **6,** 186–7 (1962); *C.A.* **59,** 4902*c* (1963).
(2J) Beyermann, K., *Z. Anal. Chem.* **194,** 1–19 (1963); *C.A.* **59,** 2152*c* (1963).
(3J) Bhakuni, T. S., Sharma, N. N., *Environ. Health* (India) **4,** 6–10 (1962).
(4J) Kelso, F. S., Matthews, J. M., Kramer, H. P., Anal. Chem. **36,** 577–9 (1964).

(5J) Khosht, K. B., Lyashcheva, K. V., *Sb. Tr. Odessk. Med.. Inst.* **15**, 149–54 (1961); *C.A.* **57**, 9601*f* (1962).
(6J) Kobayashi, S., *Bunseki Kagaku* **10**, 1004–8 (1961); *C.A.* **59**, 6127*e* (1963).
(7J) Li, L., *K'o Hsueh Tung Pao* **1961**, 45–6; *C.A.* **58**, 13608*g* (1963).
(8J) Meyling, A. H., Meyling, J., *Analyst* **88**, 84–7 (1963).
(9J) Palin, A. T., *J. Inst. Water Engrs.* **17**, 347–55 (1963).
(10J) *Ibid.*, **18**, 47–52 (1964).
(11J) Quentin, K. E., *Vom Wasser* **29**, 91–118 (1962); *C.A.* **61**, 444*a* (1964).
(12J) Sarma, P. L., Anal. Chem. **36**, 1684–5 (1964).
(13J) Shapiro, M. Ya., Kolesnikova, V. G., *Zh. Analit. Khim.* **18**, 507–9 (1963); *C.A.* **59**, 3647*a* (1963).

Sulfate and Sulfide

(1K) Bolberitz, K., Csanady, M., *Hidrol. Kozl.* **42**, 524–9 (1962); *C.A.* **61**, 5360*g* (1964).
(2K) Bozhevol'nov, E. A., Kreingol'd, S. U., *Metody Analiza Khim. Reaktivov i Preparatov, Gos. Kom. Sov. Min. SSSR po Khim.* **4**, 131–3 (1962); *C.A.* **61**, 3691*f* (1964).
(3K) Bulusu, K. R., Sen, A. K., *Environ. Health* (India) **6**, 1–6 (1964); *C.A.* **60**, 14253*c* (1964).
(4K) Chaudhuri, J. C., Purohit, A. D., Bhargava, T. N., *Current Sci.* (India) **31**, 418 (1962); *C.A.* **58**, 5380*c* (1963).
(5K) Dashevskii, L. I., *Tr. Vses. Nauchn.-Issled. Inst. Sakhar. Svekly* **34**, 327–8 (1959); *C.A.* **57**, 13182*h* (1962).
(6K) Erdey, L., Gegus, E., Vandorffy, M. T., *Magyar Kem. Lapja* **17**, 277–81 (1962); *C.A.* **57**, 12265*e* (1962).
(7K) Grabarov, P. G., *Izv. Akad. Nauk Kaz. SSR, Ser. Botan. i Pochvoved.* **1961**, 90–3; *C.A.* **57**, 9597*i* (1962).
(8K) Gupta, R. S., Ananthanarayanan, S., *Indian J. Chem.* **1**, 403–5 (1963); *C.A.* **60**, 2641*c* (1964).
(9K) Hsieh, C., Jen, C., *Hua Hsueh Pao* **27**, 106–12 (1961); *C.A.* **59**, 12177*d* (1963).
(10K) Johnson, C. R., McClelland, P. H., Boster, R. L., Anal. Chem. **36**, 300–2 (1964).
(11K) Kralova, Z. K., Shibaeva, N. P., *Zh. Analit. Khim.* **19**, 258–60 (1964); *C.A.* **60**, 15593*c* (1964).
(12K) Matveev, A. A., Nechiporenko, G. N., *Gidrokhim. Materialy* **33**, 134–7 (1961); *C.A.* **57**, 10946*g* (1962).
(13K) Meditsch. J. de O., Castiel, V., *Anais Assoc. Brasil. Quim.* **20**, 49–53 (1961); *C.A.* **58**, 20*e* (1963).
(14K) Moghe, V. B., Talati, N. R., Mathur, C. M., *Current Sci.* (India) **33**, 262 (1964); *C.A.* **61**, 34*f* (1964).
(15K) Nechiporenko, G. N., *Gidrokhim. Materialy* **33**, 143–50 (1961); *C.A.* **57**, 10946*e* (1962).
(16K) *Ibid.*, **33**, 151–4 (1961); *C.A.* **57**, 10946*f* (1962).
(17K) Nechiporenko, G. N., *Sovrem. Metody Analiza Prirodn. Vod., Akad. Nauk SSSR* **1962**, 32–46; *C.A.* **59**, 9675*f* (1963).
(18K) Nogina, A. A., Kobyak, G. G., *Uch. Zap., Permsk. Gos. Univ.* **25**, 73–8 (1963); *C.A.* **61**, 1611*a* (1964).
(19K) Novak, J., *Vodni Hospodarstvi* **12**, 486–7 (1962); *C.A.* **59**, 1378*g* (1963).
(20K) Palaty, V., *Sb. Vysoke Skoly Chem.-Technol. Praze, Oddil Fak. Technol. Paliv Vody* **3**, 55–67 (1959); *C.A.* **57**, 16338*a* (1962).
(21K) Pungor, E., Balazs, L., *Mikrochim. Acta* **1960**, 118–26.
(22K) Quentin, K. E., Pachmayr, F., *Vom Wasser* **28**, 79–93 (1961); *C.A.* **61**, 5360*h* (1964).
(23K) Quentin, K. E., Pachmayr, F., *Z. Anal. Chem.* **187**, 425–9 (1962); *C.A.* **60**, 9017*f* (1964).
(24K) *Ibid.*, **200**, 250–6 (1964); *C.A.* **60**, 10387*h* (1964).
(25K) Rapp.-Sik, S., *Magy. Allami Foldt. Int. Evi. Jelentese* **1957/58**, 377–83 (1961); *C.A.* **57**, 14895*g* (1962).
(26K) Sietz, F. G., *Z. Anal. Chem.* **190**, 418–19 (1962); *C.A.* **58**, 5380*d* (1963).
(27K) Supatashvili, G. D., *Gidrokhim. Materialy* **33**, 138–42 (1961); *C.A.* **57**, 10946*i* (1962).
(28K) Tolnay, V., Rapp.-Sik, S., *Hidrol. Kozl.* **41**, 512–13 (1961); *C.A.* **61**, 445*b* (1964).
(29K) Ugol'nikov, N. A., Galkin, D. E., *Tr. Tomskogo Gos. Univ., Ser. Khim.* **154**, 262–3 (1962); *C.A.* **60**, 6214*g* (1964).
(30K) Wayman, C. H., Anal. Chem. **35**, 768–9 (1963).
(31K) *Ibid.*, **36**, 665–6 (1964).
(32K) Yanysheva, V. S., Sazonova, Z. A., *Metody Analiza Khim. Reaktivov i Preparatov, Gos. Kom. Sov. Min. SSSR po Khim.* **4**, 133–5 (1962); *C.A.* **61**, 3695*a* (1964).
(33K) Zavodnov, S. S., *Sovrem. Metody Analiza Prirodn. Vod., Akad. Nauk SSSR* **1962**, 63–6; *C.A.* **58**, 13609*b* (1963).
(34K) Zavodnov, S. S., *Gidrokhim. Materialy* **35**, 203–6 (1963); *C.A.* **59**, 9674*h* (1963).

Silica, Phosphorus, and Arsenic

(1L) Abbott, D. C., Emsden, G. E., *Proc. Soc. Water Treat. Exam.* **12**, 230–3 (1963).
(2L) Abbott, D. C., Emsden, G. E., Harris, J. R., *Analyst* **88**, 814–16 (1963).
(3L) Allen, R. C., *Am. Soc. Testing Mater., Spec. Tech. Publ.* **337**, 67–71 (1962) (Pub. 1963).
(4L) Aunins, E., Tisheizers, E., *Tr. Gos. Okeanogr. Inst.* **68**, 159–72 (1962); *C.A.* **59**, 12507*g* (1963).
(5L) Ballinger, D. G., Lishka, R. J., Gales, M. E., Jr., *J. Am. Water Works Assoc.* **54**, 1424–8 (1962).
(6L) Breitling, V., *Mitt. Ver. Grosskesselbesitzer'* **89**, 109–11 (1964); *C.A.* **61**, 6777*c* (1964).
(7L) Datsko, V. G., Semenov, A. D., *Sovrem. Metody Analiza Prirodn. Vod., Akad. Nauk SSSR* **1962**, 112–14; *C.A.* **58**, 9957*f* (1963).
(8L) Harvey, H. W., *J. Marine Biol. Assoc. U. K.* **27**, 337–59 (1948); *C.A.* **42**, 7464*e* (1948).
(9L) Heron, J., *Limnol. Oceanog.* **7**, 316–21 (1962).
(10L) Herrig, H. J., *Tech. Ueberwach.* **4**, 285–6 (1963); *C.A.* **59**, 13690*c* (1963).
(11L) Hofmann, P., *Ochistka Prom. Stochnykh Vod., Akad. Stroit. i Arkhitekt. SSSR, Vses. Nauchn.-Issled. Inst. Vodosnabzh., Kanaliz., Gidrotekhn. Sooruzhenii i Inzh. Gidrogeol., Tr. Soveshch., Moscow* **1958**, 246–53 (Pub. 1960); *C.A.* **57**, 10945*i* (1962).
(12L) Johnson, W. C., Jr., *U. S. At. Energy Comm.* **HW-73972** (1962).
(13L) Jones, P. G. W., Spencer, C. P., *J. Marine Biol. Assoc. U. K.* **43**, 251–73 (1963); *C.A.* **59**, 6127*g* (1963).
(14L) Kemula, W., Rosolowski, S., *Chem. Anal. (Warsaw)* **7**, 915–24 (1962); *C.A.* **58**, 1907*b* (1963).
(15L) Krapivina, S. S., Khachvankyan, M. A., *Inform. Metod. Materialy po Vopr. Gidrogeol. v Bal'neotekhn. Lechebn. Vod i Gryazei, Moscow* **2**, 75–80 (1960); *C.A.* **57**, 9591*a* (1962).
(16L) Mazo, L. Ya., Shafran, I. G., *Tr. Vses. Nauchn.-Issled. Inst. Khim. Reaktivov* **25**, 283–5 (1963); *C.A.* **61**, 443*f* (1964).
(17L) Molinski, St., Szporek-Dybkowska, W., *Chemik* (Gliwice) **12**, 336–7 (1959); *C.A.* **59**, 9675*c* (1963).
(18L) Morrison, I. R., Wilson, A. L., *Analyst* **88**, 88–99 (1963).
(19L) *Ibid.*, **88**, 100–4 (1963).
(20L) *Ibid.*, **88**, 446–55 (1963).
(21L) Nakaya, S., *Bunseki Kagaku* **12**, 241–7 (1963); *C.A.* **59**, 7238*d* (1963).
(22L) Semenov, A. D., Semenova, I. M., Datsko, V. G., *Sovrem. Metody Analiza Prirodn. Vod. Akad. Nauk SSSR* **1962**, 110–11; *C.A.* **59**, 7238*h* (1963).
(23L) Sono, K., Watanabe, H., Mitsukami, Y., *Bunseki Kagaku* **12**, 352–8 (1963); *C.A.* **59**, 6128*b* (1963).
(24L) Spillner, F., *Mitt. Ver. Grosskesselbesitzer* **80**, 366–8 (1962); *C.A.* **58**, 4301*f* (1963).
(25L) Szekielda, K. H., *Kiel. Meeresforsch.* **19**, 16–19 (1963); *C.A.* **60**, 7794*f* (1964).
(26L) Umezaki, Y., *Nippon Kagaku Zasshi* **82**, 856–9 (1961); *C.A.* **57**, 10946*d* (1962).

Nitrate, Nitrite, and Nitrogen Compounds

(1M) Abbott, D. C., *Proc. Soc. Water Treat. Exam.* **13**, 154–6 (1964).
(2M) Acuna, M. S. I., *Rev. Obras Sanit. Nacion* (Buenos Aires) **25**, 209–17 (1961); *C.A.* **57**, 14431*g* (1962).
(3M) Apostolache, S., *Rev. Chim.* (Bucharest) **13**, 615–17 (1962); *C.A.*, **58**, 10727*f* (1963).
(4M) Armstrong, F. A. J., Anal. Chem. **35**, 1292–4 (1963).
(5M) Bark, L. S., Higson, H. G., *Talanta* **11**, 621–31 (1964).
(6M) Bucksteeg, W., Kalweit, G., Dietz, F., *Z. Anal. Chem.* **200**, 54–6 (1964); *C.A.* **60**, 10386*c* (1964).
(7M) Chow, T. J., Johnstone, M. S., *Anal. Chim. Acta* **27**, 441–6 (1962).
(8M) Dal Pont, G. Newell, B., Staniforth, J., *Australian J. Marine Freshwater Res.* **14**, 37–43 (1963); *C.A.* **59**, 15029*a* (1963).
(9M) Datsko, V. G., Kaplin, V. T., *Sovrem. Metody Analiza Prirodn. Vod., Akad. Nauk SSSR* **1962**, 118–22; *C.A.* **58**, 9957*d* (1963).
(10M) Demmitt, T. F., *U. S. At. Energy Comm.* **HW-79296** (1963).
(11M) Dudova, M. Ya., *Tr. Vses. Nauchn.-Issled. Inst. Gidrogeol. i Inzh. Geol., Sb.* **19**, 163–71 (1961); *C.A.* **58**, 6568*f* (1963).
(12M) Edwards, G. P., Pfafflin, J. R., Schwartz, L. H., Lauren, P. M., *J. Water Pollution Control Federation* **34**, 1112–16 (1962).
(13M) Fadrus, H., Maly, J., *Z. Anal. Chem.* **202**, 164–76 (1964); *C.A.* **61**, 444*d* (1964).
(14M) Fishman, M. J., Skougstad, M. W., Scarbro, G. F., Jr., *J. Am. Water Works Assoc.* **56**, 633–8 (1964).
(15M) Fleps, W., *Gesundh.-Ingr.* **84**, 209–12 (1963); *C.A.* **60**, 14247*h* (1964).
(16M) Frazier, R. E., *J. Am. Water Works Assoc.* **55**, 624–30 (1963).
(17M) Goldman, E., *Am. Soc. Testing Mater., Spec. Tech. Publ.* **337**, 57–66 (1962) (Pub. 1963).
(18M) Goodman, B. L., *Water Sewage Works* **110**, 27–8 (1963).
(19M) Henrioul, F., *Rev. Ferment. Ind. Aliment.* **18**, 51–9 (1963); *C.A.* **61**, 1171*b* (1964).
(20M) Husmann, W., Malz, F., Bortliss, H., *Tech.-Wiss. Mitt.* (V.D.I.) **1960**, 69–88; *C.A.* **61**, 1599*g* (1964).
(21M) Jenkins, D., Medsker, L. L., Anal. Chem. **36**, 610–12 (1964).

(22M) Justatowa, J., *Chem. Anal.* (Warsaw) **8**, 41–50 (1963); *C.A.* **61**, 6395*c* (1964).
(23M) Kaplin, V. T., Fesenko, N. G., *Sovrem. Metody Analiza Prirodn. Vod., Akad. Nauk SSSR* **1962**, 123–6; *C.A.* **58**, 9957*b* (1963).
(24M) Konnov, V. A., *Tr. Inst. Okeanol., Akad. Nauk SSSR* **54**, 123–4 (1962); *C.A.* **58**, 7720*a* (1963).
(25M) Lancy, L. E., Zabban, W., *Am. Soc. Testing Mater., Spec. Tech. Publ.* **337**, 32–45 (1962) (Pub. 1963).
(26M) Makitie, O., *Ann. Agr. Fenniae* **2**, 159–62 (1963) (in English); *C.A.* **60**, 11377*f* (1964).
(27M) Miller, G. W., Long, L. E., George, G. M., Sikes, W. L., ANAL. CHEM. **36**, 980–3 (1964).
(28M) Morris, A. W., Riley, J. P., *Anal. Chim. Acta* **29**, 272–9 (1963) (in English).
(29M) Navone, R., *J. Am. Water Works Assoc.* **56**, 781–3 (1964).
(30M) Newell, B., Dal Pont, G., *Nature* **201**, 36–7 (1964).
(31M) O'Brien, J. E., Fiore, J., *Wastes Eng.* **33**, 238–9 (1962).
(32M) Peach, S. M., *Analyst* **86**, 757–8 (1961).
(33M) Prochazkova, L., *Arch. Hydrobiol.* **56**, 179–85 (1960); *C.A.* **59**, 3647*f* (1963).
(34M) Rapp.-Sik. S., *Magy. Allami Foldt. Int. Evi Jelentese* **1957/58**, 377–83 (1961); *C.A.* **57**, 14895*g* (1962).
(35M) Rosca, G., Vasilescu, E., *Rev. Chim.* (Bucharest) **11**, 241–2 (1960); *C.A.* **57**, 10945*h* (1962).
(36M) Roskam, R. Th., Langen, D. de, *Anal. Chim. Acta* **30**, 56–9 (1964) (in English).
(37M) Rossum, J. R., Villarruz, P. A., *J. Am. Water Works Assoc.* **55**, 657–8 (1963).
(38M) Sawicki, E., Stanley, T. W., Pfaff, J., D'Amico, A., *Talanta* **10**, 641–55 (1963) (in English).
(39M) Tetlow, J. A., Wilson, A. L., *Analyst* **89**, 453–65 (1964).
(40M) Vaccaro, R. F., Thunberg, J. C., Ketchum, B. H., *Limnol. Oceanog.* **7**, 322–9 (1963).
(41M) Williams, P. M., Coote, A. R., *Ibid.*, **7**, 258 (1962).
(42M) Yen, L., *Hai Yang Yu Hu Chao* **5**, 115–23 (1963); *C.A.* **60**, 3871*a* (1964).

Alkalinity and pH

(1N) Barnes, I., *U. S. Geol. Surv., Water-Supply Paper* **1535-H** (1964).
(2N) Disteche, A., Dubuisson, M., *Bull. Inst. Oceanog.* **57**, 8 (1960) (in French); *C.A.* **57**, 13552*c* (1962).
(3N) Hirsch, W. W., *Chem. Tech.* (Berlin) **14**, 36–40 (1962).
(4N) Kabanov, V. V., *Okeanologiya* **2**, 1085–92 (1962); *C.A.* **58**, 7719*h* (1963).
(5N) Kryukov, P. A., *Gidrokhim. Materialy* **33**, 155–9 (1961); *C.A.* **57**, 9589*f* (1962).
(6N) Park, K., Oliphant, M., Freund, H., ANAL. CHEM. **35**, 1549–50 (1963).
(7N) Roberson, C. E., Feth, J. H., Seaber, P. R., Anderson, P., *U. S. Geol. Surv., Profess. Paper* **475-C**, C212-5 (1963).
(8N) Tsurikova, A. P., *Tr. Gos. Okeanogr. Inst.* **68**, 151–8 (1962); *C.A.* **60**, 3870*d* (1964).
(9N) *Vodni Hospodarstvi* **12**, 359 (1962); *C.A.* **58**, 7721*c* (1963).

Oxygen Demand

(1P) Ballinger, D. G., Lishka, R. J., *J. Water Pollution Control Federation* **34**, 470–4 (1962).
(2P) Dobbs, R. A., Williams, R. T., ANAL. CHEM. **35**, 1064–7 (1963).
(3P) Donlan, R. J., *Sanitalk* **6**, 22–5 (1958); *C.A.* **58**, 4294*d* (1963).
(4P) Drozdov, N. P., Vladimirova, V. A., Kuptsova, Z. K., *Gidroliz. i Leskhim. Prom.* **15**, 20 (1962); *C.A.* **57**, 12269*b* (1962).
(5P) Eye, J. D., Reuter, L. H., Keshavan, K., *Proc. Symp. Instr. Methods Anal.* **6**, F4 (1960).
(6P) Greenberg, A. E., Lennette, E. P., *Water Sewage Works* **110**, 166–7 (1963).
(7P) Hart, C. W., Jr., Hart, D. G., Hess, N. E., *Turtox News* **42**, 150–1 (1964).
(8P) Hiser, L. L., Busch, A. W., *J. Water Pollution Control Federation* **36**, 505–16 (1964).
(9P) Zdybiewska, M., *Przemysl Chem.* **41**, 394–7 (1962); *C.A.* **57**, 9601*a* (1962).

Oxygen and Other Gases

(1Q) Aleskovskii, V. B., Koval'tsov, V. A., Fedorov, I. N., Tsyplyatnikov, G. P., *Zavodsk. Lab.* **28**, 1440–2 (1962); *C.A.* **60**, 15593*b* (1964).
(2Q) *Ibid.*, **30**, 105–7 (1964); *C.A.* **60**, 7794*c* (1964).
(3Q) Andreev, S. V., *Med. Radiol.* **9**, 47–51 (1964); *C.A.* **61**, 7694*e* (1964).
(4Q) Apte, V. R., Dabadghao, S. B., Deshpande, W. M., Deshpande, V. D., *Environ. Health* (India) **4**, 202–6 (1962); *C.A.* **60**, 11372*a* (1964).
(5Q) Athavale, V. T., Krishnan, C. V., Subramanian, A. R., *Analyst* **87**, 707–11 (1962).
(6Q) Berka, A., Hofmann, P., *Chem. Prumysl* **13**, 287–90 (1963); *C.A.* **59**, 9675*b* (1963).
(7Q) Berka, A., Hofmann, P., *Anal. Chim. Acta* **28**, 196–9 (1963) (in German).
(8Q) Berndt, H., *Staedtehygiene* **1960**, 224–5; *C.A.* **61**, 8057*g* (1964).
(9Q) Bossy, M., *Tech. Sci. Munic.* **57**, 201–3 (1962); *C.A.* **61**, 4062*b* (1964).
(10Q) Burianek, K., *Sb. Vysoke Skoly Chem.-Technol. Praze, Oddil Fak. Technol. Paliv Vody* **3**, 69–81 (1959); *C.A.* **57**, 14895*i* (1962).
(11Q) Burke, J. D., *J. Elisha Mitchell Sci. Soc.* **78**, 145–7 (1962); *C.A.* **58**, 5380*a* (1963).
(12Q) Cantone, B., Gurrieri, S., *Boll. Sedute Accad. Gioenia Sci. Nat. Catania* **72**, 681–8 (1960); *C.A.* **58**, 5379*f* (1963).
(13Q) Carvalho, A. H. de, Calado, J. G., Moura, M. L. de, *Rev. Port. Quim.* **5**, 15–19 (1963) (in French); *C.A.* **60**, 10386*h* (1964).
(14Q) Chenouard, J., Dirian, G., Gabilly, R., *Chim. Anal.* **44**, 334–9 (1962); *C.A.* **57**, 12266*b* (1962).
(15Q) Eckfeldt, E. L., Shaffer, E. W., Jr., ANAL. CHEM. **36**, 2008–13 (1964).
(16Q) Elliott, J. I., *Brit. Power Eng.* **2**, 52–5 (1960); *C.A.* **57**, 9597*c* (1962).
(17Q) Elliott, J. W., *Progressive Fish Culturist* **25**, 42–5 (1963).
(18Q) Faber, P., Brand, W., *Kerntechnik* **6**, 79 (1964); *C.A.* **61**, 1610*h* (1964).
(19Q) Galster, H., *Z. Anal. Chem.* **186**, 359–64 (1962); *C.A.* **57**, 9597*d* (1962).
(20Q) Gaunt, H., Shanks, C., *Chem. Ind.* (London) **1964**, 651–3.
(21Q) Grasshoff, K., *Kiel Meeresforsch.* **18**, 42–50 (1962); *C.A.* **57**, 13552*a* (1962).
(22Q) *Ibid.*, **18**, 151–60 (1962); *C.A.* **58**, 11096*e* (1963).
(23Q) *Ibid.*, **19**, 8–15 (1963); *C.A.* **60**, 7792*d* (1964).
(24Q) Grasshoff, K., *Umschau Wiss. Tech.* **63**, 480–1 (1963); *C.A.* **60**, 10387*c* (1964).
(25Q) Gubeli, O., *Fundamente Balneo Bioclimatol.* (Stuttgart) **1**, 21–31 (1959); *C.A.* **58**, 346*b* (1963).
(26Q) Gusev, S. I., Ketova, L. A., *Sb. Nauchn. Tr. Permsk. Gos. Med. Inst.* **41**, 143–4 (1962); *C.A.* **61**, 8058*f* (1964).
(27Q) Hissel, I. J., *Tribune CEBEDEAU* **16**, 60–70 (1963); *C.A.* **59**, 2507*e* (1963).
(28Q) Hoak, R. D., Bramer, H. C., *Purdue Univ., Eng. Bull., Ext. Ser.* **112**, 366–76 (1962).
(29Q) Houghton, G. U., *Proc. Soc. Water Treatment Exam.* **11**, 121–6 (1962).
(30Q) Jessop, G., Holy, H. W., *Acta IMEKO* **4**, 473–86 (1961) (in English); *C.A.* **59**, 2509*d* (1963).
(31Q) Kanellopoulos, E. V., *Gt. Brit., Dept. Sci. Ind. Res., Mech. Eng. Research Lab., East Kilbride, MERL Fluids Note* **77**, (1959); *C.A.* **57**, 10945*f* (1962).
(32Q) *Ibid.*, **78** (1959); *C.A.* **57**, 10945*d* (1962).
(33Q) Karge, H., *Z. Anal. Chem.* **200**, 57–68 (1964); *C.A.* **60**, 10386*a* (1964).
(34Q) Katz, S., Neukelekian, H., *Sewage Ind. Wastes* **31**, 1022–33 (1959).
(35Q) Kerenyi, P., *Vodni Hospodarstvi* **13**, 21–3 (1963); *C.A.* **59**, 3646*h* (1963).
(36Q) Kerenyi, P., Kuba, P., *Chem. Zvesti* **17**, 146–51 (1963); *C.A.* **59**, 12507*a* (1963).
(37Q) Kilner, A. A., Ratcliff, G. A., ANAL. CHEM. **36**, 1615–17 (1964).
(38Q) Kliffmueller, R., *Arch. Hydrobiol.* **56**, 113–27 (1959).
(39Q) Kryukov, P. A., Zavodnov, S. S., *Gidrokhim. Materialy* **34**, 114–18 (1961); *C.A.* **57**, 12265*g* (1962).
(40Q) Kutyurin, V. M., *Zh. Analit. Khim.* **18**, 765–8 (1963); *C.A.* **59**, 12507*d* (1963).
(41Q) Lindsay, W. T., Jr., ANAL. CHEM. **35**, 408–9 (1963).
(42Q) Lo, B. K., *Chem. Weekblad* **58**, 223–6 (1962); *C.A.* **57**, 13553*i* (1962).
(43Q) Mancy, K. H., Okun, D. A., Reilley, C. N., *J. Electroanal. Chem.* **4**, 65–92 (1962).
(44Q) Mancy, K. H., Westgarth, W. C., *J. Water Pollution Control Federation* **34**, 1037–51 (1962).
(45Q) Maros, L., Szakacs-Pinter, M., Perl, I., Schulek, E., *Acta Chim. Acad. Sci. Hung.* **33**, 59–65 (1962) (in German); *C.A.* **58**, 8773*a* (1963).
(46Q) Meier-Ewert, H., Bruenner, H., *Staedtehygiene* **1960**, 223–4; *C.A.* **61**, 8057*f* (1964).
(47Q) Meyling, A. H., Frank, G. H., *Analyst* **87**, 60–2 (1962).
(48Q) Montgomery, H. A. C., Thom, M. S., Cockburn, A., *J. Appl. Chem.* (London) **14**, 280–96 (1964).
(49Q) Morgan, P. F., Bewtra, J. K., *J. Water Pollution Control Federation* **34**, 363–75 (1962).
(50Q) Mori, M., Goto, K., Ishihara, Y., *Nagoya Kogyo Gijutsu Shikensho Hokoku* **13**, 109–11 (1964); *C.A.* **60**, 14248*c* (1964).
(51Q) Needleman, M., *J. Appl. Chem.* (London) **12**, 294–305 (1962).
(52Q) Rezayeva, L. T., *Zh. Analit. Khim.* **17**, 874–7 (1962); *C.A.* **58**, 8403*f* (1963).
(53Q) Roskam, R. Th., Langen, D. de, *Anal. Chim. Acta* **28**, 78–81 (1963) (in English).
(54Q) Roumy, R., *Comm. Energie At. Ser. Doc. Ser. Bibliog.* **36**, (1963); *C.A.* **60**, 9017*g* (1964).
(55Q) Samsoni, Z., *Energia Atomtech.* **15**, 251–3 (1962); *C.A.* **57**, 13177*b* (1962).
(56Q) Scott, R. H., *J. Water Pollution Control Federation* **36**, 342–4 (1964).

(57Q) Smit, G. B., Ehrenburg, J. P., *Mitt. Ver Grosskesselbesitzer* **78**, 208–14 (1962); *C.A.* **57**, 11853*f* (1962).
(58Q) Solov'ev, L. G., *Okeanologiya* **4**, 149–55 (1964); *C.A.* **60**, 15592*h* (1964).
(59Q) Speranskaya, A. A., Khaidarov, I. Sh., *Vestn. Mosk. Univ., Ser. III, Fiz., Astron.* **18**, 24–7 (1963); *C.A.* **60**, 7794*f* (1964).
(60Q) St. John, P. A., Winefordner, J. D., Silver, W. S., *Anal. Chim. Acta* **30**, 49–55 (1964) (in English).
(61Q) Stepanova, N. D., Aleskovskii, V. B., *Izv. Vysshikh Uchebn. Zavedenii, Khim. i Khim. Tekhnol.* **7**, 24–8 (1964); *C.A.* **61**, 5360*a* (1964).
(62Q) Taniguchi, T., *Nippon Suisan Gakkaishi* **28**, 448–52 (1963); *C.A.* **60**, 3871*c* (1964).
(63Q) Tanno, K., *Bull. Chem. Soc. Japan* **37**, 804–11 (1964); *C.A.* **61**, 5360*f* (1964).
(64Q) Trotti, L., Sacks, D., *Arch. Oceanog. Limonol.* (*Venice*) **12**, 257–66 (1962); *C.A.* **58**, 12295*d* (1963).
(65Q) Volborth, A., Banta, H. E., Anal. Chem. **35**, 2203–5 (1963).
(66Q) Waclawik, J., Waszak, S., *Chem. Anal.* (Warsaw) **8**, 737–47 (1963); *C.A.* **60**, 7792*e* (1964).
(67Q) Wartburg, A. F., Brewer, A. W., Lodge, J. P., Jr., *Air Water Pollution* **8**, 21–8 (1964).
(68Q) Williams, D. D., Miller, R. R., *U. S. Dept. Com., Office Tech. Serv.*, **AD 265,420** (1961).
(69Q) Wood, A. A., *Effluent Water Treat. J.* **3**, 23–5 (1963).

Detergents

(1R) Abbott, D. C., *Analyst* **88**, 240–2 (1963).
(2R) Bolton, H. L., Webster, H. L., Hilton, J., *Ibid.*, **86**, 719–23 (1961).
(3R) Frankhouser, M. C. T., *Univ. Microfilms* (Ann Arbor, Mich.), *Order No.* **63-3243**; *Dissertation Abstr.* **23**, 4512–13 (1963).
(4R) Gameson, A. L. H., Lewis, V. H., *Inst. Sewage Purif. J. Proc.* **1962**, 283–301.
(5R) Heinerth, E., *Vortraege Originalfassung Intern. Kongr. Grezflaechenaktive Stoffe*, **3**, *Cologne*, 1960, **3**, 292–4 (Pub. 1961); *C.A.* **57**, 10949*d* (1962).
(6R) Karapaev, B. I., Malkina, I. I., *Neftepererabotka i Neftekhim., Nauchn.-Tekhn. Sb.* **1963**, 9–13; *C.A.* **60**, 3864*h* (1964).
(7R) Levchenko, D. N., Khudyakova, A. D., Gavrilova, N. D., *Zavodsk. Lab.* **27**, 408–9 (1961).
(8R) Michelsen, E., Maerki, E., *Mitt. Gebiete Lebensm. Hyg.* **52**, 557–66 (1961); *C.A.* **61**, 8057*h* (1964).
(9R) Niemitz, W., Fuss, K., *Gas-Wasserfach* **104**, 117 (1963); *C.A.* **58**, 11097*h* (1963).
(10R) Sztafrowski, P., *Gaz, Woda Tech. Sanit.* **35**, 147–9 (1961); *C.A.* **59**, 12507*b* (1963).
(11R) Wayman, C. H., *U. S. Geol. Surv., Profess. Paper* **450-B**, 117–20 (1962).

Pesticides end Herbicides

(1S) Abbott, D. C., Egan, H., Hammond, E. W., Thomson, J., *Analyst* **89**, 480–8 (1964).
(2S) Aly, O. M., Faust, S. D., *J. Am. Water Works Assoc.* **55**, 639–46 (1963).
(3S) Ceresia, G., *N. Y. State Dept. Health, Ann. Rept. Div. Lab., Res.* **1962**, 73.
(4S) Erne, K., *Acta Chem. Scand.* **17**, 1663–76 (1963) (in English); *C.A.* **60**, 2330*d* (1964).
(5S) Goodenkauf, A., Erdei, J., *J. Am. Water Works Assoc.* **56**, 600–6 (1964).
(6S) Gutenmann, W. H., Lisk, D. J., *Ibid.*, **56**, 189 (1964).
(7S) Hindin, E., Dunstan, G. H., *Proc. Brit. Insecticide Fungicide Conf., Brighton Eng.* **1961**, 401–3 (Pub. 1962).
(8S) Kahn, L., Wayman, C. H., Anal. Chem. **36**, 1340–3 (1964).
(9S) Skrinde, R. T., Caskey, J. W., Gillespie, C. K., *J. Am. Water Works Assoc.* **54**, 1407–23 (1962).
(10S) Weiss, C. M., Gakstatter, J. H., *J. Water Pollution Control Federation* **36**, 240–53 (1964).

Organics

(1T) Abbott, D. C., *Proc. Soc. Water Treat. Exam.* **13**, 153–4 (1964).
(2T) Antia, N. J., *Can. J. Microbiol.* **9**, 403–9 (1963).
(3T) Babeshkina, Z. M., Kaplin, V. T., Fesenko, N. G., *Gidrokhim. Materialy* **35**, 207–17 (1963); *C.A.* **59**, 11100*f* (1963).
(4T) Babkin, M. P., Voloskovets, A. L., *Neftepererabotka i Neftekhim., Nauchn.-Tekhn. Sb.* **1963**, 7–8; *C.A.* **59**, 12498*h* (1963).
(5T) Barker, L., Hollingworth, N. W., *Chem. Prumysl* **10**, 525–8 (1960); *C.A.* **59**, 14585*a* (1963).
(6T) Bars, E. A., Kogan, S. S., Fikhman, A. N., *Geokhim. i Gidrokhim. Neft. Mestorozhd., Akad. Nauk SSSR, Inst. Geol. i Razrabotki Goryuch. Iskop.* **1963**, 131–44; *C.A.* **60**, 11748*d* (1964).
(7T) Bodyu, V. I., Fel'dman, Ya. S., *Gidrolizn. i Lesokhim. Prom.* **16**, 11–12 (1963); *C.A.* **60**, 2631*b* (1964).
(8T) Boquiaux, J., Mertens, A., *Centre Belge Etude Doc. Eaux, Bull. Mens.* **110**, 42–8 (1960); *C.A.* **58**, 8831*a* (1963).
(9T) Camps, J. M., Arias, E., *Invest. Pesquera* **23**, 125–31 (1963); *C.A.* **59**, 13686*f* (1963).
(10T) Congdon, J. V., Zaiatz, S. B., *Tech. Papers Ann. Meeting Sugar Ind. Technicians, Inc.* **20**, 30–7 (1961).
(11T) Crummett, W. B., Hummel, R. A., *J. Am. Water Works Assoc.* **55**, 209–19 (1963).
(12T) Devlaminck, F., *Centre Belge Etude Doc. Eaux, Bull. Mens.* **101**, 135–8 (1959); *C.A.* **58**, 8772*h* (1963).
(13T) Drugov, Yu. S., *Gigiena i Sanit.* **29**, 56–7 (1964); *C.A.* **60**, 13008*g* (1964).
(14T) Dyatlovitskaya, F. G., Berezovskii, F. I., *Ibid.*, **27**, 50–5 (1962); *C.A.* **58**, 11093*h* (1963).
(15T) Dyatlovitskaya, F. G., Potemkina, S. K., *Ibid.*, **28**, 38–44 (1963); *C.A.* **59**, 3640*e* (1963).
(16T) Effenberger, M., *Ochistka Prom. Stochnykh Vod., Akad. Stroit. i Arkhitekt. SSSR, Vses. Nauchn.-Issled., Inst. Vodosnabzh., Kanaliz., Gidrotekhn. Sooruzhenii i Inzh. Gidrogeol., Tr. Soveshch., Moscow* **1958**, 241–5 (Pub. 1960); *C.A.* **58**, 13597*f* (1963).
(17T) Effenberger, M., Deyl, Z., *Sb. Vysoke Skoly Chem.-Technol. Praze, Oddil Fak. Technol. Paliv Vody* **2**, 225–36 (1958); *C.A.* **57**, 16344*e* (1962).
(18T) Golubeva, M. T., *Lab. Delo* **1964**, 6–9; *C.A.* **60**, 11744*c* (1964).
(19T) Goncharova, I. A., Datsko, V. G., *Sovrem. Metody Analiza Prirodn. Vod., Akad. Nauk SSSR* **1962**, 88–97; *C.A.* **58**, 9957*e* (1963).
(20T) Goncharova, I. A., Stradomskaya, A. G., Datsko, V. G., *Gidrokhim. Materialy* **35**, 156–60 (1963); *C.A.* **59**, 9671*c* (1963).
(21T) Gorizontova, T. N., *Ibid.*, **33**, 180–3 (1961); *C.A.* **57**, 9596*b* (1962).
(22T) Hanya, T., Ogura, N., *Intern. Ser. Monographs Earth Sci.* **15**, 447–56 (1964).
(23T) Hoak, R. D., *Air Water Pollution* **6**, 521–38 (1962).
(24T) Hoggan, D., *Proc., Ann. Instr.-Autom. Conf. Exhibit.* **16**, 83-LA-61 (1961).
(25T) Hrivnak, J., Schiessl, O., *Vodni Hospodarstvi* **14**, 39–40 (1964); *C.A.* **60**, 10379*a* (1964).
(26T) Kanibalodskaya, R. G., Sidorskaya, L. F., Shevlyakov, V. A., *Novosti Neft. i Gaz. Tekhn., Neftepererabotka i Neftekhim.* **2**, 20–1 (1961); *C.A.* **58**, 8761*f* (1963).
(27T) Kaplin, V. T., Fesenko, N. G., *Sovrem. Metody Analiza Prirodn. Vod., Akad. Nauk SSSR* **1962**, 131–5; *C.A.* **58**, 18773*h* (1963).
(28T) Kavan, I., *Vodni Hospodarstvi* **13**, 143–4 (1963); *C.A.* **60**, 5194*b* (1964).
(29T) Khailov, K. M., *Okeanologiya* **2**, 835–44 (1962); *C.A.* **58**, 5379*b* (1963).
(30T) Kogan, S. S., Turaeva, S. N., *Geokhim. Kaustobiolitov i ikh Mestorozhd., Gos. Kom. Sov. Min. SSSR po Toplivn. Prom., Inst. Geol. i Razrabotki Goryuch. Iskop.* **1962**, 193–7; *C.A.* **58**, 3198*a* (1963).
(31T) Komaki, S., *Bunseki Kagaku* **11**, 1197–9 (1962); *C.A.* **58**, 3195*f* (1963).
(32T) Kuchumova, N. A., Verigo, S. I., Mamontova, O. V., *Tr. Vses. Nauchn.-Issled. Inst. po Pererobotke i Ispol'z. Topliva* **12**, 237–45 (1963); *C.A.* **60**, 14248*b* (1964).
(33T) Ladendorf, P., *Vom Wasser* **29**, 119–41 (1962); *C.A.* **61**, 1601*f* (1964).
(34T) Lamar, W. L., Goerlitz, D. F., *J. Am. Water Works Assoc.* **55**, 797–802 (1963).
(35T) Leibnitz, E., Behrens, U., Koll, H., Richter, H., *Chem. Tech.* (Berlin) **14**, 33–6 (1962).
(36T) Lur'e, Yu. Yu., Antipova, P. S., *Nauchn. Soobshch. Vses. Nauchn.-Issled Inst. Vodosnabzh., Kanaliz., Gidrotekhn. Sooruzh. i Inzh. Gidrogeol., Ochistka Prom. Stochn. Vod., Moscow* **1960**, 45–6; *C.A.* **57**, 15809*b* (1962).
(37T) Lur'e, Yu. Yu., Panova, V. A., *Zavodsk. Lab.* **29**, 33–5 (1963); *C.A.* **59**, 308*d* (1963).
(38T) *Ibid.*, **29**, 293–5 (1963); *C.A.* **59**, 1371*h* (1963).
(39T) Magda, M., Chwaszcza, R., Chmielowski, J., *Gaz, Woda Tech. Sanit.* **36**, 313–14 (1962); *C.A.* **60**, 7794*e* (1964).
(40T) Massart, R., Missa, L., *Centre Belge Etude Doc. Eaux, Bull. Mens.* **111**, 162 (1960); *C.A.* **61**, 3693*a* (1964).
(41T) Mitkalev, B. A., Avferenok, S. N., Kozorezov, E. S., *Novosti Neft. i Gaz. Tekhn., Neftipererabotka i Neftekhim.* **12**, 20–2 (1961); *C.A.* **58**, 8763*h* (1963).
(42T) Montgomery, H. A. C., Thom, N. S., *Analyst* **87**, 689–97 (1962).
(43T) Morkowski, J., *Wasser, Luft, Betrib* **6**, 345–8 (1962); *C.A.* **61**, 8059*b* (1964).
(44T) Mrkva, M., *Vodni Hospodarstvi* **13**, 335–6 (1963); *C.A.* **60**, 10387*d* (1964).
(45T) Naucke, W., Beck, H. J., Bressel, H. J., Tarkmann, F. N., Tramitz, G., *Brennstoff-Chem.* **45**, 38–40 (1964) (in German); *C.A.* **60**, 10376*f* (1964).
(46T) Nemtseva, L. I., Semenov, A. D., Datsko, V. G., *Zh. Analit. Khim.* **19**, 383–5 (1964); *C.A.* **60**, 15592*c* (1964).
(47T) Obenaus, R., *Schweiz. Z. Hydrol.* **25**, 9–29 (1963); *C.A.* **60**, 10386*e* (1964).
(48T) Otsuki, A., *Nippon Kagaku Zasshi* **84**, 798–802 (1963); *C.A.* **60**, 2640*e* (1964).
(49T) Palmork, K. H., *Acta Chem. Scand.* **17**, 1456–7 (1963) (in English); *C.A.* **59**, 13686*c* (1963).
(50T) Parsons, T. R., *J. Marine Res.* **21**, 164–71 (1963).

(51T) Parsons, T. R., Strickland, J. D. H., *Ibid.*, **21**, 155–63 (1963).
(52T) Pinkerton, M. K., Lauer, J. M., Diamond, P., Tamas, A. A., *U. S. Dept. Com., Office Tech. Serv., A.D.* **273,986** (1961).
(53T) Povoledo, D., Gerletti, M., *Mem. Ist. Ital. Idrobiol. Dott. Marco de Marchi* **15**, 153–66 (1962); *C.A.* **60**, 11748*f* (1964).
(54T) Przybylski, Z., *Chem. Anal.* (Warsaw) **8**, 601–6 (1963); *C.A.* **60**, 2334*b* (1964).
(55T) Rice, J. K., Simon, D. E., II, Rice, R. C., *Ind. Water Wastes* **7**, 75–8 (1962).
(56T) Scher, A., *Hidrol. Kozl.* **39**, 303–6 (1959); *C.A.* **58**, 11098*b* (1963).
(57T) Scholz, L., *Z. Anal. Chem.* **202**, 425–33 (1964); *C.A.* **61**, 5360*b* (1964).
(58T) Semenov, A. D., Ivleva, I. N., Datsko, V. G., *Gidrokhim. Materialy* **33**, 172–9 (1961); *C.A.* **57**, 9596*d* (1962).
(59T) Semenov, A. D., Ivleva, I. N., Datsko, V. G., *Sovrem. Metody Analiza Prirodn. Vod., Akad. Nauk SSSR* **1962**, 102–3; *C.A.* **58**, 8773*c* (1963).
(60T) *Ibid.*, **1962**, 104–6; *C.A.* **58**, 8772*g* (1963).
(61T) *Ibid.*, **1962**, 107–9; *C.A.* **58**, 8774*c* (1963).
(62T) Semenov, A. D., Ivleva, I. N., Datsko, V. G., *Tr. Komis. po Analit. Khim., Akad. Nauk SSSR, Inst. Geokhim. i Analit. Khim.* **13**, 62–5 (1963); *C.A.* **59**, 9674*d* (1963).
(63T) *Ibid.*, **13**, 66–8 (1963); *C.A.* **59**, 8474*c* (1963).
(64T) Semenov, A. D., Ivleva, I. N., Datsko, V. G., *Gidrokhim. Materialy* **35**, 161–7 (1963); *C.A.* **59**, 8472*d* (1963).
(65T) Sharonova, N. F., Shul'man, Z. F., *Tr. Vses. Nauchn.-Issled. Inst. po Pererabotke i Ispol'z. Topliva* **12**, 230–6 (1963); *C.A.* **61**, 1599*d* (1964).
(66T) Shevchenko, M. A., Goronovskii, I. T., Zabarilo, A. B., *Intensifik. i Avtomatiz. Protsessov Regulirovaniya Kachestva Vody, Kiev, Sb.* **1962**, 110–19; *C.A.* **61**, 443*d* (1964).
(67T) Slowey, J. F., Jeffrey, L. M., Hood, D. W., *Geochim. Cosmochim. Acta* **26**, 607–16 (1962).
(68T) Sproul, O. J., Caskey, J. W., Ryckman, D. W., *Ind. Water Wastes* **7**, 139–45 (1962).
(69T) Stankovic, V., *Chem. Zvesti* **16**, 683–91 (1962); *C.A.* **58**, 7712*a* (1963).
(70T) *Ibid.*, **17**, 274–9 (1963); *C.A.* **59**, 15022*h* (1963).
(71T) Suprunov, A. T., Muravskaya, Z. A., *Tr. Sevastopol'sk. Biol. St., Akad. Nauk SSSR* **16**, 463–6 (1963); *C.A.* **61**, 6778*b* (1964).
(72T) Sztafrowski, P., *Gaz. Woda Tech. Sanit.* **35**, 306–8 (1961); *C.A.* **60**, 3863*e* (1964)
(73T) Ungar, J., Thomas, J. F. J., *Can. Dept. Mines Tech. Surv. Mines Branch, Tech. Bull.* **TB 39** (1962).
(74T) Vaitsekhovskii, R. V., Zasimchuk, T. K., Fainerman, A. E., *Tr. 1-oi [Pervoi] Vses. Nauchn.-Tekhn. Konf. po Ochistke Prom. Stochn. Vod Predpriyatii Khim. Volokon, Kiev* **1961**, 85–90; *C.A.* **58**, 3195*c* (1963).
(75T) Van Hall, C. E., Safranko, J., Stenger, V. A., ANAL. CHEM. **35**, 315–19 (1963).
(76T) Van Hall, C. E., Stenger, V. A., *Water Sewage Works* **111**, 266–70 (1964).
(77T) Weber, W. J., Jr., Morris, J. C., *J. Water Pollution Control Federation* **36**, 573–86 (1964).
(78T) Wierzbicki, T., *Zeszyty Nauk. Politech. Slask.* **4**, 57–61 (1962); *C.A.* **61**, 5352*f* (1964).
(79T) Zdenek, V., Skorepa, V., *Listy Cukrovar.* **78**, 137–41 (1962); *C.A.* **57**, 13554*f* (1962).

Radioactivity and Isotopic Analysis

(1U) Abdullaev, A. A., Khaitov, B. K., Lobanov, E. M., Khaidarov, A. A., *Izv. Akad. Nauk Uz. SSR, Ser. Fiz.-Mat. Nauk* **6**, 40–4 (1962); *C.A.* **58**, 7721*h* (1963).
(2U) Anokhin, V. L., Bylinkina, A. A., Drachev, S. M., *Priemy Sanit. Izuch. Vodoemov Sb.* **1960**, 209–40; *C.A.* **57**, 9593*e* (1962).
(3U) Arnett, E. M., Duggleby, P. McC., ANAL. CHEM. **35**, 1420–4 (1963).
(4U) Barker, F. B., Robinson, B. P., *U. S. Geol. Surv., Water-Supply Paper* **1696-A** (1963).
(5U) Bizollon, Ch. A., Moret, R., *Chim. Anal.* (Paris) **46**, 273–85 (1964); *C.A.* **61**, 5353*e* (1964).
(6U) Blaga, Liviu, Blaga, Lucia, Chifu, A., *Acad. Rep. Populare Romine, Studii Cercetari Fiz.* **15**, 47–53 (1964); *C.A.* **61**, 6776*b* (1964).
(7U) Bosch, J., *Ber. Abwassertech. Ver.* **11**, 20–33 (1959); *C.A.* **59**, 312*a* (1963).
(8U) Buchtela, K., Lesigang, M., *Radiochim. Acta* **1**, 208–12 (1963) (in German); *C.A.* **60**, 2640*g* (1964).
(9U) Butler, F. E., *Health Phys.* **8**, 273–7 (1962).
(10U) Chou, C., *Hai Yang Yu Hu Chao* **5**, 11–17 (1963); *C.A.* **60**, 2640*b* (1964).
(11U) Donlan, R. J., *Sanitalk* **8**, 13–15 (1960); *C.A.* **60**, 304*h* (1964).
(12U) Duquesne, H., Godfroi, E. E., Govaerts, J., Warin, R., Michiels, J., *Health Phys.* **9**, 927–30 (1963).
(13U) Ebersole, E. R., Harbertson, A., Flygare, J. K., Jr., Sill, C. W., *U. S. At. Energy Comm.* **TID-7616**, 147–75 (1962).
(14U) Eichholz, G. G., Hughes, R. B., Nagel, A. E., *Georgia Inst. Technol., Nucl. Eng. Ser., Tech. Rept.* **NE-2** (1964).
(15U) Feldman, M. S., *U. S. At. Energy Comm.* **DP-511** (1962).
(16U) Folsom, T. R., Saruhashi, K., *J. Radiation Res.* (Japan) **4**, 39–53 (1963); *C.A.* **61**, 5358*b* (1964).
(17U) Foti, S. C., Freiling, E. C., *U. S. Dept. Com., Office Tech. Serv., PB Rept.* **156,644** (1960).
(18U) Frantz, A., *Ost. Wasserw.* 13, 45–9 (1961); *Water Pollution Abstr.* **35**, 698 (1962).
(19U) Gebauer, H., Mueller, S., *Atomwirtschaft* **7**, 487–91 (1962); *C.A.* **60**, 11748*h* (1964).
(20U) Gordon, G., Yamatera, H., ANAL. CHEM. **36**, 1866–7 (1964).
(21U) Grune, W. N., Hughes, R. B., Gibson, G. W., *Water Sewage Works* **110**, 34–6 (1963).
(22U) Haughey, F. J., Manganelli, R. M., *J. Water Pollution Control Federation* **36**, 88–95 (1964).
(23U) Hinzpeter, A., *Schriftenreihe Bundesmin. Wiss. Forsch., Strahlenschutz* (Munich) **23** (1963); *C.A.* **61**, 8059*c* (1964).
(24U) Kautsky, H., Schmitt, D. E., *Atompraxis* **9**, 224–7 (1963); *C.A.* **59**, 11100*b* (1963).
(25U) Kerr, J. R. W., Coomber, D. I., Lewis, D. T., *Analyst* **87**, 944–8 (1962).
(26U) Ledbetter, J. O., Gloyna, E. F., *Public Works* **93**, 87–90 (1962).
(27U) Lesigang, M., *Oesterr. Chemiker-Ztg.* **63**, 352 (1962); *C.A.* **58**, 9956*h* (1963).
(28U) Marques, B. E., Grade, M. R., Viana, C. N., *Rev. Port. Quim.* **3**, 9–29 (1961); *C.A.* **60**, 13009*f* (1964).
(29U) Nelepo, B. A., *Tr. Sov. Antarkt. Ekspeditsii* **19**, 169–73 (1961); *C.A.* **58**, 348*h* (1963).
(30U) Nishiwaki, Y., Kawai, H., *Shitsuryo Bunseki* **10**, 83–90 (1962); *C.A.* **60**, 14253*d* (1964).
(31U) Petrow, H. G., Allen, R. J., ANAL. CHEM. **35**, 747–9 (1963).
(32U) Proctor, C. M., Papadopulos, E., Firminhac, R. H., *Limnol. Oceanog.* **7**, 280–6 (1963).
(33U) *Public Works* **93**, 117–18 (1962).
(34U) Reynolds, S. A., *Health Phys.* **8**, 391–6 (1962).
(35U) Schanze, U., *Städtchygiene* **12**, 28–31 (1961); *Water Pollution Abstr.* **35**, 692 (1962).
(36U) Scheidhauer, J., Messainguiral, L., *Chim. Anal.* (Paris) **45**, 452–62 (1963); *C.A.* **59**, 15029*c* (1963).
(37U) Scheidhauer, J., Messainguiral, L., Meiraneisio, A. M., *Comm. Energie At.* (France), *Rappt.* **2354** (1963); *C.A.* **60**, 10387*b* (1964).
(38U) Schoenberg, D., *Isotopen Tech.* **2**, 213–15 (1962); *C.A.* **57**, 13554*e* (1962).
(39U) Sill, C. W., Willis, C. P., ANAL. CHEM. **34**, 954–64 (1962).
(40U) Stradomskii, V. B., *Sovrem. Metody Analiza Prirodn. Vod., Akad. Nauk SSSR* **1962**, 176–86; *C.A.* **58**, 9956*b* (1963).
(41U) Sugimura, Y., Tsubota, H., *J. Marine Res.* **21**, 74–80 (1963).
(42U) Syromyatnikov, N. G., Mukashev, F. A., Kapatsinkaya, L. A., *Radiokhimiya* **5**, 356–60 (1963); *C.A.* **59**, 14573*g* (1963).
(43U) Tolkendorf, E., *Kernenergie* **7**, 108–11 (1964); *C.A.* **60**, 15591*h* (1964).
(44U) Tschang, T., *Ibid.*, **3**, 892–7 (1960); *C.A.* **60**, 6198*g* (1964).

Miscellaneous

(1V) Acuna, M. S. I., *Rev. Obras Sanit. Nacion* (Buenos Aires) **27**, 125–36 (1963) (in Spanish); *C.A.* **60**, 1450*f* (1964).
(2V) Albersmeyer, W., *Staedtehygiene* **10**, 178–80 (1959); *C.A.* **57**, 14899*a* (1962).
(3V) Azcona, J. M., L. de, *Notas Comun. Ist. Geol. Minero Esp.* **4**, 313–18 (1963); *C.A.* **61**, 443*d* (1964).
(4V) Belyaev, V. P., Kalinachenko, V. R., Kuz'min, N. M., Yakimenko, L. M. *Zavodsk. Lab.* **28**, 685 (1962); *C.A.* **58**, 5379*e* (1963).
(5V) Bozhevol'nov, E. A., Serebryakova, G. V., Yanishevckaya, V. M., Kreingol'd, S. U., *Acta Chim. Acad. Sci. Hung.* **32**, 199–206 (1962) (in Russian); *C.A.* **58**, 5026*c* (1963).
(6V) Branche, G., Ropert, M. E., *Colloq. Spectros. Intern., 9th, Lyons,* **1961** **2**, 467–84 (Pub. 1962) (in French); *C.A.* **59**, 1083*g* (1963).
(7V) Britt, R. D., Jr., ANAL. CHEM. **34**, 1728–31 (1962).
(8V) Butler, L. R. P., Brink, D., *S. African Ind. Chemist* **17**, 152–6 (1963); *C.A.* **60**, 7794*b* (1964).
(9V) Dickinson, G. W., Wheeler, G. V., *U. S. At. Energy Comm.* **IDO-14591** (1962).
(10V) Faganelli, A., *Boll. Pesca, Psicicolt. Idrobiol.* **15**, 152–70 (1960); *Ist. Sper. Tolassogr. Publ.* **386** (1962); *C.A.* **59**, 11100*g* (1963).
(11V) German, J. C., *J. Conseil, Conseil Perm. Intern. Exploration Mer* **26**, 21–32 (1960); *C.A.* **60**, 303*g* (1964).
(12V) Giebler, G., Kempf, T., *Z. Anal. Chem.* **199**, 23–34 (1963); *C.A.* **60**, 3870*g* (1964).
(13V) Goldberg, R. D., *ISA, Proc. Ann. Instr.-Autom. Conf. Exhibit* **17**, 17-1-62 (1962).

(14V) Herrin, C. B., *Univ. Microfilms* (Ann Arbor, Mich.), *Order No.* **64-5047;** *Dissertation Abstr.* **24,** 4949 (1964).

(15V) Holden, A. V., *Effluent Water Treat. J.* **2,** 338–40 (1962).

(16V) Jedlewska, A., *Chem. Anal.* (Warsaw) **7,** 381–8 (1962); *C.A.* **57,** 12265*f* (1962).

(17V) Jenkins, D., *Water Waste Treat. J.* **9,** 66–7 (1962).

(18V) Keyser, A. H., *Chem. Eng. Progr.* **60,** 53–6 (1964).

(19V) Klimov, I. T., *Sovrem. Metody Analiza Prirodn. Vod., Akad. Nauk SSSR* **1962,** 67–87; *C.A.* **59,** 9321*c* (1963).

(20V) Libina, R. I., Margolin, L. S., Miller, A. D., Sergeev, E. A., *Tr. Vses. Nauchn.-Issled. Inst. Metodiki i Tekhn. Razvedki, Sb.* **3,** 317–37 (1961); *C.A.* **58,** 7720*d* (1963).

(21V) Lombardi, O. W., ANAL. CHEM. **36,** 415–18 (1964).

(22V) Makinenko, A. I., *Tr. Leningr. Sanit.-Gigien. Med. Inst.* **73,** 11–13 (1961); *C.A.* **58,** 12294*a* (1963).

(23V) Onufrienok, I. P., Glazunova, A. D., Solodovnikova, R. S., *Tr. Mezhved. Soveshch. po Gidrogeokhim. Methodu Poiskov Rudn. Mestorozhd.* **1960,** 215–19 (Pub. 1962); *C.A.* **60,** 9016*e* (1964).

(24V) Pritchard, D. W., *Natl. Acad. Sci.-Natl. Res. Council, Publ.* **600,** 146–54 (1959).

(25V) Shigematsu, T., Tabushi, M., Nishikawa, Y., Hiraki, K., Goda, S., Inoue, R., *Nippon Kagaku Zasshi* **84,** 336–9 (1963); *C.A.* **59,** 12507*f* (1963).

(26V) Sinyukov, V. V., Skopintsev, B. A., *Okeanologiya* **3,** 127–36 (1963); *C.A.* **59,** 4902*a* (1963).

(27V) Smith, A. L., Greenberg, A. E., *J. Water Pollution Control Federation* **35,** 940–3 (1963).

(28V) Williams, J., *U. S. Dept. Com., Office Tech. Serv., PB Rept.* **158,935** (1961).

(29V) Wyckoff, B. M., *Water Sewage Works* 111, 277 (1964).

(30V) Yavorskii, S. I., Timofeeva, V. G., *Zavodsk. Lab.* **28,** 929–30 (1962); *C.A.* **58,** 13608*a* (1963).

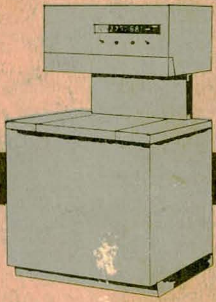
(31V) Zhukova, M. G., *Teploenergetika* 9, 42–4 (1962); *C.A.* **57,** 13554*e* (1962).

for the finest

instrumentation

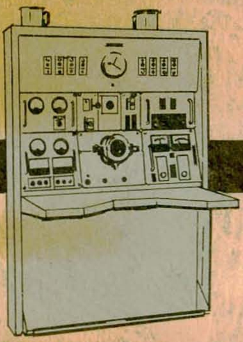
and service

in any of these categories . . .



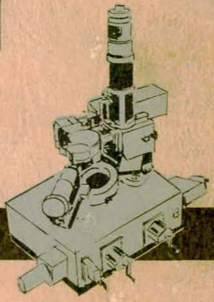
NUCLEAR INSTRUMENTS

such as a
MULTI CHANNEL
LIQUID SCINTILLATION
COUNTER



MASS SPECTROMETERS

such as a
HIGH RESOLUTION
DOUBLE FOCUSSING
GAS SOURCE SPECTROMETER



X-RAY OPTICAL APPARATUS

such as a
SPECTRODIFFRACTOMETER
for fluorescence and diffraction
analysis of the same specimen

ELECTRON MICROSCOPES

such as a HIGH RESOLVING POWER MICROSCOPE
with built in rotating beam deflector stage
and high resolution goniometer stage

look to PICKER nuclear

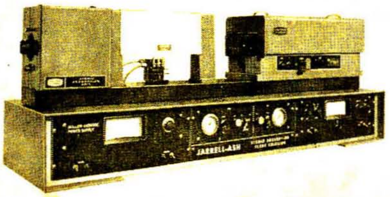
DIVISION OF
PICKER X-RAY
CORPORATION
WHITE PLAINS,
NEW YORK

Representatives in principal cities throughout the Free World

Now you can analyze metallic ions to parts per billion

– by atomic absorption spectroscopy
– by flame emission spectroscopy

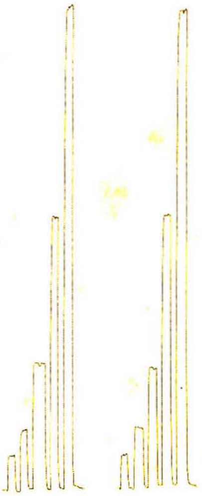
with this one instrument



THE JARRELL-ASH ATOMIC ABSORPTION / FLAME SPECTROMETER

Capacity	120 analyses per hour
Precision	Best precision of all routine techniques at high sensitivity
Convenience	Easily operated by average technician. Direct reading without manipulating knobs during analysis
Stability	Special lamps and power supply provide stable calibration for hours
Installation and Operator Training	Provided by Jarrell-Ash personnel; included in price
Related Jarrell-Ash Instrumentation	Gas chromatograph

Write for Bulletin 82-362



Duplicate calibrating standards analyzed and reproduced within three minutes. Aqueous standards of $MgCl_2$ containing 0.01μg ml — 1.0μg ml Mg analyzed at the 2852A line.

SOME TYPICAL USES

- Blood
- Urine
- Tissue
- Soils
- Fertilizers
- Plants
- Plating Solutions
- Geological Specimens
- Electrolytes
- Cement
- Crude Petroleum
- Petroleum Products
- Oil Additives
- Vegetable Oils
- Animal Fats
- Metallurgical Specimens
- Ores
- Natural Waters
- Plant Effluent
- Animal Feeds
- Sea Water
- Foods
- Beverages
- Beer and Wine
- Plastics
- Chemicals
- Air Pollution Samples
- Water Pollution Samples
- Drugs

Jarrell Ash

ANALYTICAL INSTRUMENTATION · SPECTROSCOPY · GAS CHROMATOGRAPHY · X-RAY DIFFRACTION

JARRELL-ASH COMPANY · 510 Lincoln Street, Waltham, Massachusetts 02154
JARRELL-ASH (Europe) S. A., Rue de la Jacuse 6, Le Locle, Switzerland
NIPPON JARRELL-ASH CO., LTD., Kiyamachi-Sanjyo-Sagaru, Nakagyoku, Kyoto, Japan