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APPLICATIONS

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1967 Review of Analytical Applications

REVIEWS

AUTHORS' BIOGRAPHIES

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The authors of the 1967 Application Reviews include representatives from government, industry, and universities. Many of them have written reviews for ANALYTICAL CHEMISTRY in past years

KINGSLEY

ALTSHULLER

The Authors

Air Pollution

A. P. Altshuller is the Chief, Chemical and Physical Research and Development Program, National Center for Air Pollution Control, Public Health Service, 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 research 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 of the Public Health Service in Cincinnati. He assumed his present position in January 1967. He has published about 100 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. He received the Eighteenth Annual (1967) Cincinnati Chemist Award of the Cincinnati Section, American Chemical Society.

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 Chemists.

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 21 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 48 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 19 papers.

George G. Esposito, research analytical chemist at Aberdeen Proving Ground, has been working with resinous coating materials for the past 16 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 32 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 Executive Vice President and Technical Advisor 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 ac-


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tivities 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.

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 electronics 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 and is now serving as referee on agricultural liming materials and associate referee on sampling of fertilizers. His research interests include the development of quantitative gas chromatographic methods for amino acids, purines, pyrimidines, nucleosides, fatty acids, and pesticide residues; characterization and interactions of proteins; and automation of analytical methods for nitrogen, phosphorus, and potassium in fertilizers and for biologically important molecules. He is author of at least 75 publications in analytical and biochemistry. Dr. Gehrke has been an invited scientist on gas chromatography of amino acids to the Polytechnique Ecole in Paris, Edmonton, Alberta, and a number of institutions in the U.S.; as well as on automation in analytical chemistry to Germany, France, and England.

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. He has developed automatic analytical methods for K_2O and P_2O_5 in fertilizers, and is currently doing research on the automation of the Kjeldahl method and other biochemical molecules in plant and animal materials. He is author of nine scientific papers on new methods of fertilizer analysis.

BORKER

HINES

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FOLTZ

PASZTOR

Food

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 Analytical Chemists and a member of ACS, AAAS, Institute of Food Technologists, and ASTM Committee E-15.

Katherine G. Sloman, Research Specialist, Analytical Chemistry, 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.

Arthur K. Foltz, Senior Chemist, Analytical Services Group, General Foods Technical Center, received a B.S. in 1958 from Queens College. After an analytical position with D.C.A. Food Industries, he joined General Foods Corporate Research in late 1958. Most of his experience has been in the field of food analysis, particularly involving instrumental methods of measurement, and especially gas chromatography in the past few years. Lately he has been involved in methods development for food additives and trace contaminants as well as for trace materials in biological systems. Memberships include the ACS and ASTM Committee E-19 on Gas Chromatography.

Ferrous Metallurgy

Laszlo C. Pasztor, Research Supervisor at the Graham Research Laboratory of Jones & Laughlin Steel Corporation, Pittsburgh, Pa., was born in Hungary. He was graduated (1943) and received his M.S. (1954) from the University of Science of Budapest and did further graduate work at the University of Pittsburgh. He joined J&L Steel Corporation—Graham Research Laboratory—in 1957 as a research chemist and his work has involved mainly analytical research. He was promoted to research supervisor in 1963. His publications have been in the field of ferrous metallurgical analysis including papers on solvent extraction separations, spectrophotometric methods, isolation and determination of nonmetallic inclusions in steels and neutron activation analysis. Laszlo C. Pasztor is a member of the ACS, the Society of the Sigma Xi, the SAS, Spectroscopy Society of Pittsburgh, the Society for Analytical Chemists of Pittsburgh, the ASTM, the Society of Chemical Industry (England), and of the Analytical Chemistry Section of IUPAC.

Charles R. Hines, Senior Research Chemist with the Jones & Laughlin Steel Corp., Pittsburgh, Pa., re-

OLIVER

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BACHMAN

ceived his A.B. degree from Dartmouth. He served two years in the Army Chemical Corps as an analytical chemist assigned to Pine Bluff Arsenal, Arkansas, and the Army Chemical Center, Maryland. He worked for Shell Chemical Co. and United States Steel prior to joining Jones & Laughlin in 1963. He is a member of the Society for Applied Spectroscopy, the Spectroscopy Society of Pittsburgh, and the Society for Analytical Chemists of Pittsburgh. At Jones & Laughlin's Graham Research Laboratory he is currently in charge of the spectroscopy laboratory which includes optical emission, atomic absorption, and x-ray fluorescence.

Nonferrous Metallurgy I. Light Metals

Richard T. Oliver is a Section Head in the Analytical Chemistry Division of the Alcoa Research Laboratories, New Kensington, Pa. He was graduated from Central Connecticut State College in 1953, received his M.S. from Northeastern University in 1955 and his Ph.D. in Analytical Chemistry from Iowa State University in 1959. Since receiving his doctorate, he has been employed as a research chemist for Alcoa. His work over the last seven years has involved research and development of analytical techniques in the area of acid–base titrations in nonaqueous solvents, ion exchange separations, specific ion electrodes, and electrochemical methods. During the past year, his activities have centered around the automatic control of chemical processes in the aluminum industry. He is a member of the ACS, the Society for Analytical Chemists of Pittsburgh, and is Chairman of the 1968 Pittsburgh Conference.

Ernest P. Cox is employed at the Alcoa Research Laboratories, New Kensington, Pa., as a research analytical chemist. During undergraduate work, he was an NSF research participant at the University of Arkansas. After receiving a B.S. in Chemistry from the University of Tennessee in 1965, he was an analytical chemist at the American Cyanamid Central Research Laboratories, Stamford, Conn., prior to coming to Alcoa. His principal research interests are in development of methods for analysis of trace impurities in aluminum as well as in evaluation of proposed ISO methods. He is a member of the American Chemical Society, the Society for Analytical Chemists of Pittsburgh, and the Pittsburgh Chemists Club.

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 11 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


BANKS


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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 group leader, Senior Chemist, and Section Chief 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 general analytical chemistry of such elements as U, Th, rare earths, Ti, V, Cr, Zr, Nb, Mo, Hf, Ta, and W. He has published well over 100 research papers over the past 20 years. Dr. Banks is a member of several honorary and scientific societies including the ACS and AAAS.

Simultaneous Determination of Oxygen and Nitrogen in Metals by Carrier-Gas Fusion Extraction

Wayne E. Dallmann is a chemist with the Ames Laboratory of the U. S. Atomic Energy Commission, located at Iowa State University. He received the B.A. degree in chemistry from Mankato State College in 1960, after which he began his appointment at the Ames Laboratory. In 1964 he received the M.S. degree in analytical chemistry from Iowa State University. His principal interest is in the determination of gases in metals and metal salts, on which subject he has coauthored several papers with Dr. V. A. Fassel. He has been concerned primarily with the development and refinement of high temperature fusion–extraction analytical procedures and apparatus, and for the past several years has been in charge of the extensive experimental facilities at the Ames Laboratory for the determination of the gaseous elements in metals. He is a member of the Phi Lambda Upsilon honorary chemical society.

Velmer A. Fassel is Professor of Chemistry at Iowa State University and Section Chief with the Ames Laboratory of the Atomic Energy Commission. His undergraduate training was received at Southeast Missouri State College and he holds the Ph.D. degree in physical chemistry from Iowa State University. His research interests are in the application of atomic and molecular emission and absorption spectroscopy to analytical problems, correlation of infrared spectra with molecular structure, and high temperature–high vacuum analytical techniques. He has published more than 85 research papers, and served as coeditor of *Spectrochimica Acta* from 1952 to 1965. He now holds the office of Secretary of the Commission on Spectrochemical and Other Optical Methods of Analysis of the International Union of Pure and Applied Chemistry. The annual Medal Award of the Society for Applied Spectroscopy was presented to Professor Fassel in 1964 and he has been elected to Fellow membership in the Optical Society of America and the American Association for the Advancement of Science.

Pesticide Residues

Sidney Williams is Assistant 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 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.

J. William Cook is Deputy Director of the Division of Food Chemistry, Bureau of Science, 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 becom-

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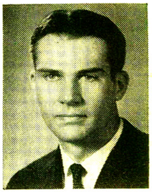

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ing a member of the Food and Drug Administration in 1939. Since then he has served in various capacities in FDA, the past 15 years in pesticide chemicals. For a number of years he was responsible for the evaluation of the chemistry, methods of analysis, 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 is a titular member of the Pesticide Section of the Division of Applied Chemistry, International Union of Pure and Applied Chemistry.

Petroleum

Frederick Donald Tuemmler, Head of Analytical Standardization Department, Shell Development Co., has been active in analytical work since 1932. At present he is secretary coordinator of analytical standardization for the petroleum and chemical laboratory of Shell Oil Co. He attended Shurtliff College (B.S., 1929), University of Illinois (M.S., 1930) and Harvard University (M.A., 1935, Ph.D., 1937). He served as Secretary of the Analytical Section of the International Union of Pure and Applied Chemistry (1952–56). He is currently associated with American groups supporting the actions of International Standards Organization Technical Committee 28 on Petroleum Products and Technical Committee 48 on Aromatic Hydrocarbons and has played a major role in the preparation of two ISO Recommended Practices. In the American Society for Testing and Materials, he has been particularly active in the work of its Committee D-2 on Petroleum Products, as well as in the work of several other Committees; for this activity he received the ASTM Award of Merit (1950). As a specialty he has been a part of the development of volume correction tables for liquid petroleum and chemical products, being the most active participant in all national and international standards promulgated on this subject to date. His work in the American Chemical Society was contributed to the northern California Section as a member of its steering committee. The Petroleum Reviews, the work of sixteen specialists in the field of petroleum analysis, have been coordinated by Tuemmler.

Pharmaceuticals and Related Drugs

J. G. Theivagt is Group Leader, Formulation Methods, in the Analytical Research Department of Abbott Laboratories. He received his B.S. from the University of Illinois in 1952. 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 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.

D. C. Wimer is Group Leader, Investigational Drugs-Research, 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 acid-base interactions, functional group analysis particularly of organic nitrogen compounds, and nonaqueous solvent chemistry. He is a member of the ACS, AAAS, and Sigma Xi.

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 been with the Research Division of the Goodyear Tire and Rubber Co. in Akron, Ohio, where he is a section head in analytical chemistry. He is a member of ACS, its Analytical Division, and ASTM.

Gordon S. Trick is a research scientist with Goodyear Tire and Rubber Co., Akron, Ohio. Dr. Trick earned his B.S. degree at McGill University in 1949, his M.S. at the University of Western Ontario in 1950, and his Ph.D. at McGill University in 1952. He was also a Ramsay Fellow at King's College,


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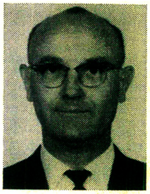
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University of London in 1953. Before joining Goodyear in 1956, Dr. Trick was employed by Defence Research Board of Canada. His major research interests are in gas and liquid phase kinetics, physical properties of chemical warfare agents, mass transfer, development and application of experimental techniques to characterize high polymers and relate to observed physical behavior. Dr. Trick is a member of the Polymer and Rubber Divisions of ACS and a member of the Chemical Institute of Canada.

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, 1958, and 1966, and in Germany in 1964, and is Chairman 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 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. H
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Billy P. Robinson received his B.
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of natural water, and disposal
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Maryland R. Midgett received h
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leigh, N. C. in 1961 and transferre
to Denver, Colo., in 1965 where he
currently engaged in analytic
methods research. His research in
terests include developments
methods for the determination
trace elements in water by atomi
absorption, and development an
improvement of existing techniqu
for major elements.

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ANALYTICAL REVIEWS

1967

APPLICATIONS

1967 Review of Analytical Applications

The articles that comprise this nineteenth Annual Review issue of Analytical Chemistry were prepared, upon invitation of the Editors, by authorities drawn from the highest ranks of universities, industry, and government. There is no doubt that reviews on timely subjects are in great demand. However, both editors and authors are constantly seeking ways to present *meaningful* critical reviews, especially in those areas of burgeoning research that cause one to question how any review at all can be useful, regardless of its criticality or the care which goes into its preparation. The job of the authors in this respect is the most difficult: they must husband their resources, sift through enormous compilations for the most notable developments, and report in the briefest of forms the efforts of their labors. In turn, the Editors must continue to seek the most appropriate topics for future presentation, and, more important, for authors with the highest qualifications to prepare the reviews. We hope you will find in this issue, as in the many that preceded it, that the efforts of the authors have led to articles of inestimable value to scientists interested in applied analysis.

H. A. Laitinen

Air Pollution

A. P. Altshuller, Bureau of Disease Prevention and Environmental Control, National Center for Air Pollution Control, U. S. Department of Health, Education, and Welfare, Public Health Service, 4676 Columbia Parkway, Cincinnati, Ohio 45226

MOST OF THE WORK discussed in this review was published or presented between November 1964 and November 1966. Some references are included even though they were published before November 1964 because the work appeared in journals that are not available on a current basis. Conversely, references from readily available journals are included through December 1966. The previous review (*5*) included papers published or presented between October 1962 and October 1964, with a few more recent references. Presentations cited in the previous review ordinarily are not cited again as publications in the present review.

Comparison with the previous review shows that a small decrease in analytical chemical research activity has occurred. This decrease amounts to about 15% if the field studies references are not included in the comparison. Even a small decrease in effort is unexpected, since an overall increase in technical activity and public concern about air pollution has occurred in the U. S. in the last few years.

Methods for analysis of inorganic pollutants greatly outnumber those for organic pollutants in the present review. A large number of publications were concerned with analysis of polynuclear aromatic compounds, of sulfur dioxide and hydrogen sulfide, and of auto exhaust emissions.

As usual, many of the papers at the symposia and general sessions on air pollution held at the spring and fall meetings of the American Chemical Society in 1965 and 1966 were in the field of analytical chemistry. Papers reporting analytical chemical applications were presented at the 1965 and 1966 meetings of the Air Pollution Control Association and the American Industrial Hygiene Conference. The California State Department of Public Health sponsored a conference including analytical aspects of air pollution in January 1965.

Several review articles were published on special topics in the analysis of air pollutants (*118, 138, 264*). A collection of selected methods for the measurement of air pollutants was made available (*212*). The formation of an Analytical Methods Evaluation Service for air pollutants within the Public Health Service was announced (*226*). A 12th volume of translations of the U.S.S.R. literature consists of the 1963 volume of *Gigiena i Sanitariya* (*148*). Air quality measurements on suspended particulates, 17 metals, three nonmetals, and six gases during 1964–65 in U. S. cities have been summarized (*209*). Continuous air monitoring data for gases in Cincinnati and Washington, D. C., in 1962–63 have been tabulated and discussed (*210, 211*).

BIOLOGICAL INDICATORS

Various biological substrates have been used as indicators in laboratory studies. Additional studies have been reported of the ability of various components of the organic fraction of air particulate matter to immobilize *Paramecium caudatum*. The use of eye irritation panels as subjective indicators in laboratory investigations of photooxidation reactions is continuing.

The use of plants as indicators of air pollution has been reviewed (*98*). The relationships between tobacco fleck injury and ozone levels in the field and greenhouse have been reviewed (*100*). A synergistic effect of sulfur dioxide on ozone damage to tobacco plants has been reported (*172*). Ozone-type injury was not observed with fumigation by 3 pphm of ozone, but injury was observed when 3 pphm of ozone was present concurrently with about 25 pphm of sulfur dioxide. The responses of tobacco wrapper Bel-W_3 and pinto bean to various ozone concentrations ranging from 10 to 55 pphm for 0.5 to 4 hours of fumigation have been reported (*99*). Both ozone and peroxyacetyl nitrate can oxidize the reduced forms of nicotinamide adenine dinucleotides (*180*). Peroxyacetyl nitrate can inactivate enzymes by oxidation of enzyme sulfhydryl groups and also can inhibit incorporation of acetate into fatty acids (*180*). Preliminary results have been reported on the relation of ozone and peroxyacetyl nitrate concentrations to the corresponding types of plant damage as a result of fumigations with photooxidized propylene–nitrogen oxide mixtures (*7*).

The bioassay of 15 benzene extracts of atmospheric particulates collected in various U. S. cities for carcinogenic activity was reported in terms of the immobilization of *Paramecium caudatum* exposed to ultraviolet radiation in the 3600 Å region (*74*). High activity was shown by an aromatic fraction, high to moderate activity by oxygenated fractions (not containing benzo[*a*]pyrene), and intermediate activity by the crude benzene extracts. Promising results were obtained in the comparison of this same bioassay technique with an assay technique for carcinogenic activity in neonatal mice (*73*).

Possible methods for the determination of lacrimatory potential have been considered (*140*). A polarographic determination involving catalytic reaction of sulfhydryl groups at a dropping mercury electrode in a cobalt-containing buffer solution was applied to a variety of lacrimators. Eye irritation intensity responses were discussed as a function of hydrocarbon and nitrogen oxide concentrations in the photooxidation of the propylene–nitric oxide system (*134*). Only part of the intensity of response could be accounted for by the formaldehyde produced, and the remainder of the response was not attributable to peroxyacyl nitrate at the concentrations present in the reaction mixtures. Data on eye irritation responses as a function of time were obtained in other work on the photooxidation of propylene–nitrogen oxide mixtures and of diluted auto exhaust mixtures (*221*). The effects of reducing hydrocarbon, nitrogen oxide, or both reactants were discussed for the reactant concentration ranges investigated. Results of previous irradiation chamber experiments have been examined by statistical procedures to determine the effects of hydrocarbon and nitrogen oxide concentrations on eye irritation responses (*188*).

SAMPLING AND CALIBRATION METHODS

The high sulfate concentrations obtained in shorter-time sampling of air on glass-fiber filters result from oxidation of sulfur dioxide to sulfate on the filters (*144*). The discrepancy is reduced with longer-time sampling because the sulfate from sulfur dioxide reaches a saturation level.

The efficiencies of the midget impinger and a new 2-ml micro-impinger were compared under various sampling conditions (*150*); the microimpinger is equivalent or superior to the midget impinger in sampling for sulfur dioxide, aniline, and dust. Improved collection efficiency in atmospheric sampling by the stacking of three fritted filters was reported (*256*). A sixfold sampler with sintered glass filters has been discussed (*15*). Collection efficiencies in the sixfold absorber were consistently higher than those in the normal fritted bubbler for hydrogen sulfide, nitrogen dioxide, carbon dioxide, benzene, and butanone. The use of hypodermic needles as critical orifices in air samples has been evaluated (*155*).

In an investigation with $^{35}SO_2$ as a tracer, significant loss of sampling efficiency below 10 pphm in both the conductometric and colorimetric pararosaniline methods was reported (*281*). In another study with the same tracer technique, contradictory results were obtained. A 99% collection efficiency was found for sulfur dioxide in tetrachloromercurate solution at 1.2 liters per minute in the range from 1 to 10 pphm (*23*). In this same study 98% and higher collection efficiencies were found for sulfur dioxide in hydrogen peroxide solution at rates as high as 6 liters per minute in the range from 0.3 to 17 pphm sulfur dioxide. Another group of investigators also has reported efficiencies near 99% in the 1- to 25-pphm range for sulfur dioxide into hydrogen peroxide solution at a flow rate of 1.4 liters per minute (*27*).

The absorption efficiencies of 6- to 60-turn spiral contact columns were evaluated at several air and liquid flow rates with two formulations of the Saltzman-type nitrogen dioxide reagent (*203*). In another investigation the Saltzman and Jacobs-Hochheiser colorimetric procedures for nitrogen dioxide were compared for collection efficiencies under various conditions (*231*). Further evaluation of glass-fiber filters has been reported in the sampling of hydrogen fluoride (*197*). Calibration of limed filter papers has been discussed for measuring short-term hydrogen fluoride dosages (*206*).

Interest in charcoal sampling tubes for sampling gases and vapors continues. The effects of sampling rate and volume collected on efficiency of collection on charcoal sampling tubes prior to gas chromatographic analysis was determined for methyl ethyl ketone, toluene, trichloroethylene, butyl acetate, 2-methylcyclohexanol, and styrene (*80*). The absorption and recovery efficiencies of acrolein, methyl sulfide, and *n*-propyl mercaptan were evaluated on two different activated carbons (*31*). Sampling of the tracer substance, sulfur hexafluoride, onto carbon followed by desorption by heating permitted subsequent electron-capture gas chromatographic analysis to the 10^{-9}-ppm level (*46*).

The use of activated silica gel as an adsorbent for atmospheric contaminants has been reviewed with specific consideration of studies of adsorption of aromatic hydrocarbons, halogenated hydrocarbons, and acetone (*34*). Measurements of collection efficiencies on silica gel as a function of flow rate and sample volume were reported for benzene, toluene, trichloroethylene, perchloroethylene, carbon tetrachloride, butyl acetate, ethyl acetate, formaldehyde, nitrogen dioxide, and other vapors (*179*). Silica gel tubes have been used for the collection of solvents such as perchloroethylene, mineral spirits, aromatic solvents, and ethyl acrylate from stacks (*204*). Dimethyl sulfoxide was satisfactory for recovery of a number of solvents, with subsequent gas chromatographic analysis (*77*). For Stoddard solvent, naphtha, and higher-boiling solvents, carbon disulfide and carbon disulfide–water were used for desorption, with subsequent infrared analysis (*77*).

A chromatographic method for the concentration of trace impurities in the atmosphere has been developed, based on the theory of phase equilibrium (*189*). Short tubes containing the gas chromatographic substrates silicone elastomer E-301 and polyethylene glycol 400 on Celite 545 were used to evaluate the method.

The precision available in the use of plastic bags was determined to average ±3% with 50 to 150 ppm of trichloroethylene (*11*). Pressure-volume characteristics of plastic bags have been reported (*25*).

Use of the permeation rates of gases from sections of plastic tubing permits the dispersing of nanogram quantities with precision at constant temperature (*193*). Permeation rates as functions of temperature were obtained for sulfur dioxide, nitrogen dioxide, propane, and butane. Methods for the fabrication, calibration, and use of permeation tubes were described (*193*). In a later report, modifications were suggested for extending the range of use of permeation tubes (*192*). Permeation tubes containing sulfur dioxide were studied in more detail; in this study the gravimetric method of calibration compared favorably with colorimetric and coulometric methods (*247*). Permeation tubes made of Teflon should be useful for preparing accurate concentrations of atmospheric pollutants, for calibrating instruments, and for developing or comparing analytical methods.

A portable gas dilution apparatus has been developed and applied to generation of sulfur dioxide, hydrogen sulfide, nitric oxide, and nitrogen dioxide (*276*). In this same work, the satisfactory generation of sulfur dioxide at concentrations from 0.01 to 32 ppm by the permeation tube technique was confirmed. A simple compact dilution device can be used to dilute gaseous samples 3000 to 1 with accuracy within ±1% (*227*). An automated variable-level dispersing system for hydrogen fluoride has been developed (*114*). An ultraviolet-type ozone generator has been used for producing ozone over a wide concentration range (*257*).

Calibration of several coulometric oxidant analyzers for ozone response by infrared techniques has been discussed (*259*). Methods for the synthesis and purification of peroxyacyl nitrates by preparative-scale gas chromatography have been described, along with the storage and handling characteristics of the products diluted in nitrogen (*261*). A dynamic calibration procedure for an acid aerosol analyzer has been reported (*246*).

PARTICULATES AND AEROSOLS

Inorganic Particulates. An automatic air sampling system has been combined with a direct-reading spectrograph to analyze beryllium in particulates every 75 seconds with a sensitivity of 0.5 μg per cubic meter (*224*). After lead is collected on an iodine column or millipore filter and extracted, lead can be determined by atomic absorption spectroscopy with a sensitivity of 0.013 ppm at 2170 Å by use of an oxyhydrogen flame and T-piece adapter (*39*). Anionic interferences were eliminated by use of EDTA. A rapid analysis for lead on filter paper has been obtained by use of diphenylcarbazide in ethanol (*94*). Analyses of lead in particulates by the spectrographic, polarographic, spectrophotometric (tetrahydroxyquinone), and turbidimetric procedures (sodium rhodizonate or potassium dichromate) have been compared (*218*). Application of the Goetz aerosol spectrometer to particle-size distribution of lead in particulates has been discussed (*157*). Zinc can be determined by paper chromatography and complexometric analysis in the range of 5 to 150 μg (*222*). Application of the ring-oven technique to the identification and determination of airborne particulates has been reviewed (*288*).

Fluorides have been measured in the range of 2 to 10 μg per cubic meter of air by the alizarin-complexion method after collection in a series of three silver tubes filled with silver balls (*35*). The determination of fluoride in air and plant tissues by the Willard-Winter and semiautomated methods was discussed (*120*). Analyses of the subcellular distribution of fluoride in orange leaves have been reported (*40*). An improved diffusion chamber, elimination of transfer steps, and application of sodium 2-(*p*-sulfophenylazo)-1,8-dihydroxynaphthalene-3,6-disulfonate zirconium lake have been developed for spectrophotometric determination of microgram amounts of fluoride in samples of biological material (*41*). A method has been made available for the analysis of chloride, bromide, and iodide in atmospheric samples; the method involves neutron activation followed by chemical separations (*69*). An improved method has been reported for determination of micro-sized sulfate particles (*154*). Sulfates have been determined by use of x-ray emission spectrometry (*285*). Macro- and microscopic applications of dispersion staining in air pollution have been described (*55*).

Aerosols. A review of current problems in atmospheric aerosol research considered such topics as sampling procedures, distribution of chemical components with particle size, size analysis of acid aerosols, and gas-particle interactions (*284*). A number of aerosol generators have been developed. Three generators, an atomizer-impactor combination and two spinning disk systems, have been used for preparing homogeneous, monodisperse, spherical, and electrically neutral solid particles of a fluorescent dye (*291*). Homogeneous aerosol production of solid particles by electrical atomization also has been reported (*296*). Monodisperse aerosols have been generated with various shape factors (*153*). A new impaction device was developed for determination of aerosol concentration and composition (*83*).

Size analysis of acid aerosols by a metal film technique has been discussed (*110*). Microscopic and electron-microscopic techniques can be used to identify sulfuric acid aerosol in presence of other substances in particulates (*131*). Hydrogen-flame emission spectrophotometry has been used in the monitoring of sulfuric acid aerosol (*54*). Hydrogen-flame ionization analyzers were shown theoretically and experimentally to have potential for detecting and sizing organic aerosols (*81, 191*). A radiotracer study of lower-molecular-weight olefins irradiated with and without sulfur dioxide proved that no organic aerosols are formed in these systems (*96*).

Organic Particulates. The nature of the benzene-soluble fraction of air particulates has been discussed (*272*). The *n*-alkanes in air particulates can be separated and analyzed by removing unsaturates by bromination and by separating *n*-alkanes and isoalkanes by molecular sieves, combined with gas-liquid partition chromatography (*171*). Benzo[*a*]pyrene is extractable from bituminous coal (*279*). The adsorption and extraction of benzo[*a*]pyrene from soots has been investigated (*137*). The degree of volatilization of polycyclic hydrocarbons has been related to particle size and temperature (*223*).

Ultraviolet irradiation of benzo[*a*]pyrene and perylene in diluted particulate matter caused 35 to 65% of these hydrocarbons to disappear (*274*). Similar losses did not occur in the dark. Further work has been reported on possible modes of disappearance of benzo[*a*]pyrene (*275*). Photochemical oxidation of benzo[*a*]pyrene in benzene solution with oxygen present produced at least three polynuclear aromatic quinones, which were identified by melting point, by elemental analysis, and by infrared or ultraviolet spectra (*164*).

Studies continue on various modifications of chromatographic, spectrophotometric, and fluorescent procedures for separation and analysis of benzo[*a*]pyrene and other polynuclear aromatic hydrocarbons. A combination column and paper chromatographic system has been used for the separation of benzo[*a*]pyrene (*290*). Liquid–liquid partition chromatography on silica gel for concentration followed by column chromatography on alumina and spectrophotometric analysis has been used to determine 13 polycyclic hydrocarbons (*87*). The spectrophotometric determination of benzo[*a*]pyrene in the presence of pyrene has been discussed (*186*). Thin layer chromatography on alumina followed by low-temperature fluorescence spectrometry has been used to analyze for benzo[*a*]pyrene (*121*). The determination of 1,2,4,5-dibenzopyrene and of 3,4,9,10-dibenzopyrene in atmospheric samples can be accomplished by column or thin layer chromatography combined with fluorescent spectroscopy (*62*). Fluorescent spectrometry also has been applied in the measurement of benzo[*a*]pyrene and benzo[*k*]fluoranthrene by use of appropriate excitation wavelengths (*66*). Recoveries and background problems in analysis of polycyclic hydrocarbons by fluorescent spectroscopy have been studied (*297*). Programmed-temperature operation of glass capillary columns coated with SE-52, in combination with flame ionization or electron-capture detectors, provided separation and analysis of many polycyclic hydrocarbons, including partial separation of benzo[*a*]pyrene and benzo[*e*]pyrene (*38*). A dual-column, dual-flame ionization detector apparatus has been used in the separation and analysis of six polycyclic hydrocarbons in air particulate samples (*58*). Partial separation of benzo[*a*]pyrene from benzo[*e*]pyrene was reported. Several alkylated derivatives of polycyclic hydrocarbons have been identified in air particulate samples (*245*). Identifications were made by gas–liquid partition chromatography followed by fluorometric measurement of the bands, and by mixed-adsorbent two-dimensional thin layer chromatography followed by fluorometric measurement of the spots.

Direct fluorometric scanning of thin layer chromatograms has been applied to mixtures of polycyclic hydrocarbons, mixtures of polynuclear aza heterocyclics, and coal-tar pitch fumes (*242*). Eight polycyclic hydrocarbons and 25 aza heterocyclic hydrocarbons were identified by their fluorescent spectra and by quenchofluorometric techniques after column and thin layer chromatography of coal-tar pitch, petroleum refinery incinerator effluents, and coal combustion stack effluents (*241*). A column chromatographic procedure has been developed for separating basic polynuclear aromatic compounds in complex mixtures (*240*). Several procedures for fluorometric analysis of benz[*c*]acridine and other aza heterocyclic compounds after thin layer separation have been evaluated for use with air particulate and coal-tar samples (*243*). The chromatographic separation and spectral analysis of polynuclear aromatic amines and heterocyclic imines have been investigated (*235*).

Spectral characterization of nitroarenes and amines and of polycyclic aldehydes, ketones, and quinones has been reported (*122*). Eight fluorescent quenching techniques have been applied in the direct analysis of thin layer chromatographic spots of various acridines, quinolines, and large-ring carbonyls (*234*).

Column and thin layer chromatographic procedures were investigated for the separation of polynuclear ring carbonyl compounds (*244*). Phenalen-1-one and 7-H-benz[*d,e*]anthracen-7-one in air particulate and source effluent samples have been identified and analyzed through use of one- and two-dimensional thin layer chromatography and direct spectrophotofluorometric examination of the thin layer plates (*236*). Various modifications of these thin layer chromatographic–fluorometric procedures for 7-H-benz[*d,e*]anthracen-7-one and phenalen-1-one were compared (*236*). Appropriate reagents have been discussed for making fluorescent formaldehyde and especially nonfluorescent aromatic substances that are precursors to formaldehyde on paper and thin layer chromatography (*258*). Based on indications of monocyclic carbonyl compounds in auto exhaust, an investigation has shown that 1,4-cyclohexanedione can be condensed with *o*-phthalaldehyde to form pentacenquinone, which can be determined spectrophotometrically as the neutral compound or dicationic salt in sulfuric acid solution and also spectrophotofluorometrically in acid solution (*232*).

The purification of allergenic macromolecular compounds isolated from air particulates was accomplished by thin layer and preparative chromatography (*84*). A highly sensitive and selective colorimetric method has been developed for amino acids or primary amine precursors following thin layer chromatography (*233*).

INORGANIC GASES

The sensitivity limitations involved in the direction of atmospheric vapors by surface-related effects have been considered (*65*). The condensation nuclei counter and its application to gas analysis was discussed, with detailed consideration of sulfur dioxide, nitrogen dioxide, and phosgene analysis (*282*). Improved sampling and analysis procedures have been reported for measurement of sulfur dioxide, nitrogen dioxide, ammonia, and hydrogen sulfide in the National Air Sampling Network of the Public Health Service (*178*). Interferences with various methods for analysis of inorganic gases in air have been reviewed (*183*). A simple modification of a commercial sulfur dioxide analyzer allows its use for a variety of other atmospheric pollutants (*187*). A new colorimetric monitoring instrument for the sequential determination of sulfur dioxide, nitric oxide, nitrogen dioxide, hydrogen sulfide, chlorine, ozone, and fluoride has been described (*82*).

Carbon Monoxide and Carbon Dioxide. An extensive bibliography on carbon monoxide presents abstracts of analytical methods (*50*). Carbon monoxide analyses by a Hersch-type galvanic cell and by gas chromatography have been evaluated (*68*). Carbon monoxide also was analyzed gas chromatographically in a laboratory photooxidation reaction study (*6*). A gas chromatographic procedure for carbon dioxide in air has been described (*184*).

Sulfur Dioxide. An extensive bibliography on sulfur oxides presents abstracts of analytical methods (*51*). Several groups of investigators have compared two or more methods for the analysis of sulfur dioxide. Closely comparable results were obtained in the comparison of the silica gel procedure of Stratmann with the colorimetric pararosaniline method with collection in tetrachloromercurate solution (*265*). Comparison of this spectrophotometric method, the hydrogen peroxide method, and an electroconductometric instrument in analyses of ambient air samples showed significant variations from city to city that suggested varying interference patterns (*109*). A laboratory comparison of the conductometric, titrimetric, and spectrophotometric procedures for sulfur dioxide in air showed no significant difference in responses among the several methods (*276*).

Several investigations assessed the use of the lead dioxide cylinders for measurement of sulfur compounds. The critical parameters considered in design of lead dioxide cylinders included optimum loadings, lead dioxide particle size, use of various binders, and presence of reducing-type sulfur compounds (*105*). A statistical analysis has been reported after 4 years of measurement at 20 sites. Results from adjacent lead dioxide cylinders and hydrogen peroxide–type instruments were compared (*70*); no generally applicable direct calibration between these two methods could be obtained. A titrimetric method for sulfate in lead dioxide candles involved conversion of lead sulfate to ammonium sulfate followed by titration with barium perchlorate with Thoron as an indicator (*217*).

Several suggested modifications in the colorimetric pararosaniline method for sulfur dioxide include a procedure for purification and standardization of the pararosaniline dye, use of phosphoric acid to control acidity, and use of sulfamic acid to eliminate nitrogen dioxide interference (*249*). The same authors previously reported a study of reagent purification, pH effects, stability of complex, and stability of colored product (*248*). With the sulfamic acid modification, application of the pararosaniline method to a large group of samples from U. S. cities resulted in sulfur dioxide concentrations averaging 30% higher than the concentrations obtained in the absence of sulfamic acid (*178*). In another study with synthetic gas mixtures, very little decrease in absorbance was reported for the pararosaniline method with nitrogen dioxide added as compared to absorbances measured with no nitrogen dioxide added (*276*). The use of the reagent *p*-aminoazabenzene has been investigated for the determination of sulfur dioxide (*133*). Sulfur dioxide has been analyzed by x-ray emission spectrometry (*285*). Hydrogen flame emission spectrophotometry also has been applied to the analysis of sulfur dioxide in atmospheric samples (*54*).

A number of new or modified monitoring instruments for sulfur dioxide have been evaluated. A modified *Auto-Analyzer* was used to determine sulfur dioxide as sulfate at concentrations from 0 to 10 μg per ml by spectrophotometric titration with barium perchlorate and Thoron in dioxane–water solvent (*202*). Collection was in hydrogen peroxide rather than tetrachloromercurate solution because of somewhat better storage properties. A portable apparatus based on the pararosaniline reagent was developed for determining sulfur dioxide. Sampling time was 8 minutes, with a 15-minute period for color formation; accuracy was ±15% (*102*). Modifications of the Cummings-Redfern sulfur dioxide instrument (based on the reaction with the starch–iodine complex) allow recording of results, shorten the response time, and provide multiple full-scale ranges (*12*). Another instrument, also based on the starch–iodine complex reaction, prints out 0.5-hour mean values (*19*). Since the starch–iodine reaction has poor specificity, a platinum catalyst with added hydrogen was built into the apparatus to eliminate interference by ozone and nitrogen dioxide. A galvanic-coulometric monitoring instrument based on the iodine–iodide reaction has been developed for sulfur dioxide in air (*103*). The method has high sensitivity, and a response time of several minutes, but shows poor specificity without a scrubber. Ozone interference is eliminated by a silver screen–granular silver column; interference by nitrogen dioxide is claimed to be only 2%, with hydrogen sulfide, mercaptans, organic sulfides, and aldehydes interfering to varying extents (*103*, *167*). A combination sulfur dioxide and ozone analyzer has been constructed; the system is based on oxidation of iodide to iodine by ozone in one channel and consumption of iodine by sulfur dioxide in the other channel, with amperometric measurements of the iodine concentration (*250*). Ozone is removed from the air going to the sulfur dioxide channel by a bed of ferrous sulfate crystals, and sulfur dioxide is removed from the ozone channel by a bed of quartz chips soaked in a solution of chromium trioxide in aqueous phosphoric acid. Sensitivity of this instrument is reported to be high, but its response time is no better than that of several other sulfur dioxide instruments. Potential interferences are discussed but not investigated (*250*).

A sulfur-specific microcoulometric cell for use in gas chromatography was modified to provide improved response characteristics, longer electrolyte life, and higher sensitivity (*2*). The detection limits of the detector are 5 pphm for sulfur dioxide and 1 pphm for hydrogen sulfide; the detector also responds to mercaptans and organic sulfides. This same cell has been used for direct analysis of sulfur dioxide, hydrogen sulfide, and organic sulfur compounds by use of selective prefilters to remove all but one of the sulfur compounds before the sample stream reaches the detector (*4*).

Hydrogen Sulfide. The absorption efficiency of the cadmium hydroxide suspension that has been used prior to the spectrophotometric methods for hydrogen sulfide has been determined by the $H_2{}^{35}S$ isotopic tracer technique (*24*). When three types of collectors were evaluated, the impinger with sintered glass disk gave the best efficiencies, averaging 97%, at flow rates up to 6 liters per minute at hydrogen sulfide concentrations between 1 and 30 pphm. Various methods for analyzing hydrogen sulfide have been reviewed, and the methylene blue procedure recommended for atmospheric analysis (*119*). The flow rates suggested in this latter discussion, especially at lower hydrogen sulfide concentrations, are higher than those recommended as a result of the $H_2{}^{35}S$ isotopic tracer study (*24*). A study of the light sensitivity of the cadmium sulfide suspension formed after collection of hydrogen sulfide in cadmium hydroxide indicates the need to protect the cadmium sulfide suspension from exposure to light (*139*).

Another investigation showed that in collecting hydrogen sulfide in cadmium hydroxide the midget impinger could be used at 3 liters per minute, and that a multijet bubbler could be used at 15 liters per minute (*276*). In this same work, sulfur dioxide at 33 pphm increased the absorbance by 15%, ozone at 24 pphm decreased the absorbance slightly, and nitrogen dioxide had little or no effect. A portable hydrogen sulfide analyzer has been developed which achieves good sensitivity by use of tetraacetoxymercurifluorescein as a fluorescent indicator for hydrogen sulfide (*9*). A potentiometric method for low concentrations of hydrogen sulfide that uses sulfide-sensitive electrodes was reported (*190*). Membrane filters impregnated with lead acetate have been used in another method for hydrogen sulfide (*196*). Sulfur dioxide interference was eliminated by use of a potassium bicarbonate–impregnated filter; however, nitrogen dioxide also can interfere above 20 pphm.

Fading of the lead sulfide spots in the lead acetate–impregnated paper-tape samplers can be caused by oxidation by air or ozone or by the action of sulfur dioxide and light (*230*). *o*-Phenyl phenol was an effective antioxidant, but use of this compound is not recommended for quantitative determination of hydrogen sulfide (*230*). In another investigation parallel fading characteristics of the lead sulfide spots also were observed, and a lack of reliability of lead acetate paper tapes in automatic samplers was suggested (*200*). A mercuric chloride reagent for paper-tape sampling was developed which does not fade in light or under prolonged exposure to 10 ppm of ozone, nitrogen oxides, and sulfur dioxide (*200*).

Nitrogen Oxides. Glass-fiber disks impregnated with acid dichromate were evaluated for use in oxidizing nitric oxide to nitrogen dioxide (*124*). Although this process is 25% more efficient than the standard permanganate bubbler at relative humidities between 19 and 46%, the efficiency of the acid dichromate decreased rapidly as the relative humidity was increased above 46% (*123*). A modification of the permanganate oxidation involving permanganate reagent on glass beads was useful at concentrations up to 4 ppm of nitric oxide at relative humidities between 10 and 70% (*36*).

A study of the stoichiometry and of potential interferences with the reagent formulated by Saltzman has been reported. The investigators disagree with the 0.72 stoichiometry factor reported earlier (*229*), and find instead 1 mole of nitrogen dioxide equivalent to 1 mole of nitrite (*266*). These workers found no interference from fluoride, formaldehyde, or nitric oxide but claimed some interference from sulfur dioxide, which could be eliminated by addition of sodium tetrachloromercurate. No significant sulfur dioxide interference with this type of coupling reaction with nitrogen dioxide has been found in other recent investigations (*183*, *276*).

The method based on the Saltzman-type reagent and the Jacobs-Hochheiser method were compared by analysis of atmospheric samples for nitric oxide and nitrogen dioxide (*107*). A colorimetric reagent with rapid color development and with improved intensity and stability has been formulated for nitrogen dioxide analysis; sodium 2-naphthol 3,6-disulfonate (R-salt) is used as a promoter and tartaric acid is substituted for acetic acid (*162*). Because it requires only a few minutes for full color development this reagent is of special interest for use in monitoring instrumentation. In other work, acetic acid was omitted from the Saltzman reagent formulation for use in a continuous analyzer; results were satisfactory, and the reagent is less toxic, less corrosive, and less expensive (*71*).

A more detailed study of various azo-dye reagent formulations resulted in the use of a reagent containing citric acid, and sulfanilamide with *N*-(1-naphthyl)-ethylenediamine in water (*182*). In analysis of nitrogen dioxide by an *Auto*-Analyzer unit the reagent contained phosphoric acid, sulfanilamide, and *N*-(1-naphthyl)ethylenediamine in water (*178*).

A method was developed for the analysis of both nitric oxide and nitrogen dioxide in the 100- to 5000-ppm range by formation of the $FeSO_4 \cdot NO$ complex and spectrophotometric measurement at 445 or 580 mμ (*136*). Another procedure for nitric oxide and nitrogen dioxide involved collecting in alkaline solution, acidifying, adding hydrogen peroxide, making basic and evaporating, reacidifying, adding hydrogen peroxide, making basic and evaporating, reacidifying, adding *o*-hydroxybenzoic acid, diluting with alkali, and reading color intensity (*135*).

Ozone and Oxidants. A group of laboratory analytical methods including the colorimetric potassium iodide, phenolphthalein, sodium diphenylamine sulfonate, fluorescein, and 4,4′-dimethoxystilbene reagents and the nitrogen dioxide equivalent and rubber-cracking methods were evaluated for collection and analysis of low concentrations of ozone (*101*). The nitrogen dioxide equivalent method was preferred for ozone, but rubber cracking proved convenient for field survey work.

Another comparison study evaluated the utility of the colorimetric potassium iodide, coulometric, and galvanic-type analyzers, an ultraviolet photometer, and rubber cracking in application to atmospheric analysis (*42*). Differences in response to atmospheric samples were attributed to differences in response to interferences. In a third study of various ozone and oxidant methods, a wide variety of existing and new methods were considered with respect to sensitivity and specificity for ozone, nitrogen dioxide, hydrogen peroxide, alkyl hydroperoxides, peroxyacyl nitrates, acyl peroxides, and peracids (*215*). These methods were evaluated for their effectiveness in differentiating the various types of oxidant material.

A newly developed spectrophotometric method for ozone involves reaction of ozone with 1,2-di-(4-pyridyl)-ethylene in glacial acetic acid and formation of pyridine-4-aldehyde, which is reacted with 3-methyl-2-benzothiazolinone hydrazone hydrochloride (3-MBTH) to form an azine that can be determined at 442 mμ (*97*). Calibration, precision, and sensitivity, air sampling, and interferences have been considered. Sulfur dioxide, nitrogen dioxide, peroxyacyl nitrate, and other oxidants do not interfere appreciably (*97*). However, hydrogen peroxide will interfere significantly if present in concentrations comparable to those of ozone (*215*). A new method for atmospheric oxidant is based on the oxidation in ethanol of 9,10-dihydroacridine to the fluorescent acridine, acidification with acetic acid, and measurement at 482 mμ (*288*). The sensitivity of the method is high, and the product is stable within 1 hour after sampling. Sulfur dioxide and hydrogen peroxide do not interfere, but nitrogen dioxide interferes significantly. A chemiluminescence method for ozone involving use of gallic acid with rhodamine B has been investigated (*16*). A recording potentiometric analyzer for oxidants using $NH_4Fe(C_2O_4)_2$ as a reducing agent has been reported (*268*).

Other Inorganic Gaseous Pollutants. Phosgene has been determined in the part-per-billion range and higher by electron-capture gas chromatography (*208*). Various colorimetric reagents for phosgene were evaluated in liquid solution, impregnated on paper, and on granular solids (*151*). Hydrogen cyanide has been determined electrometrically by bromine titration with biamperometric indication at concentrations from 0 to 1 μg per liter (*263*).

ORGANIC GASES AND VAPORS

Applications of gas chromatography and infrared spectroscopy to the determination of organic pollutants have been reviewed (*88*). Portable instruments have been fabricated and evaluated for total organics and gas chromatographic analysis of trace constituents (*89*). Preliminary results in evaluating substrates for subtractive analysis or reactive organics in air have been reported (*194*). Combinations of various subtractive techniques, column substrates, and gas chromatographic detectors were used to identify various hydrocarbons, chlorinated hydrocarbons, organic sulfur compounds, and organic nitrates in atmospheric samples (*292*).

A wide variety of silica gels were evaluated with respect to average pore diameter, water content, and added organic liquid phases in the separation of C$_1$ to C$_5$ paraffins, C$_2$ to C$_4$ olefins, and acetylene in atmospheric samples (*13*). Aromatic hydrocarbons in the C$_6$ to C$_{10}$ range have been concentrated and analyzed by flame ionization after separation on an open-tubular-column substrate (*185*). Several groups of investigators have discussed concentration and separation techniques for hydrocarbons (*78*, *127*, *128*, *260*) similar to those previously reported (*5*). A semiautomatic gas chromatographic system was developed for determining *n*-paraffins and hydrocarbon types (*86*).

Collection efficiencies and molar absorptivities have been measured for acetaldehyde and propionaldehyde in the 3-methyl-2-benzothiazolone hydrazone method for aldehydes in air (*47*). A procedure was reported for reducing the interference from oxides of nitrogen in the chromotropic acid method for formaldehyde (*286*). An evaluation of colorimetric methods for trace analysis of acrolein indicated that the 4-hexylresorcinol method was superior to the tryptophan method in sensitivity, precision, and selectivity (*126*).

A procedure has been developed for neutron activation analysis of chlorinated hydrocarbons collected from the atmosphere on charcoal (*177*). The Fujiwarz reaction for the colorimetric analysis of chlorinated hydrocarbons was evaluated for determination of carbon tetrachloroethane in air (*160*). An electron-capture gas chromatographic method has been developed for the ultramicroanalysis of bromotrifluoromethane and octofluorocyclobutane as well as sulfur hexafluoride as meteorological tracer substances in the atmosphere (*228*).

Aliphatic alcohols in the C$_1$ to C$_{10}$ range in air can be separated and determined by paper chromatography (*205*). A colorimetric determination of phenols in air utilizes a stabilized diazonium salt (*33*). An evaluation of substrates and detector characteristics has been reported for the electron-capture gas chromatographic analysis of peroxyacyl nitrates in air (*14*). An electron-capture gas chromatographic method was developed for nitroethylene and 1-nitro-1-propane in air with a detection limit of 0.001 ppm (*262*). The microcoulometric detector either with gas chromatographic or subtractive separation of sulfur compounds has been evaluated for the organic sulfur compounds methyl mercaptan, methyl sulfide, and dimethyl sulfide (*2*, *4*).

ANALYSES OF SOURCES OF POLLUTION

Vehicular Emissions. Various problems associated with the sampling and holding of automobile exhaust gases have been discussed (*79*, *253*). Losses in containers, lines, and traps and by chemical reactions can occur.

The responses of the nondispersive infrared analyzer and the flame ionization analyzer for various hydrocarbons were measured and compared (*115*). It was concluded that the flame ionization analyzer, when operated at conditions giving approximately equal responses to hydrocarbons and low oxygen interference, gave a better approximation to total hydrocarbon concentration in automobile exhaust samples than did the nondispersive infrared analyzer.

A rapid-response flame ionization detector has been constructed, with a response time of less than 1 second (*85*). An apparatus has been developed for analyzing hydrocarbon classes in less than 5 minutes by use of subtractive column substrates with flame ionization detection (*132*). This technique was evaluated in analyses of samples from large groups of automobiles on the road and on dynamometers (*22*, *255*).

Analyses of a variety of aliphatic and aromatic hydrocarbons have been accomplished by temperature programming of open tubular colums from subambient temperatures (*117*, *166*). One of these gas chromatographic systems also required the analysis of each sample for all components and then use of mercuric perchlorate to remove all but the paraffinic hydrocarbons before a second analysis for paraffinic components only (*166*). This system has been used in an evaluation of engine modifications on exhaust composition and in a comparison of several automobiles with engine modification and air injection control systems (*116*). The other system (*117*) has been used to evaluate the effect of gasoline composition on exhaust emissions of a vehicle with an air injection device (*198*).

In another gas chromatographic system several larger-diameter open tubular column substrates were used under isothermal conditions along with a silica gel substrate for light hydrocarbons (*13*, *255*). This system has been employed in a series of studies of automobiles whose exhausts were sampled on the road under normal driving conditions (*254*), of automobiles' exhaust emissions after both hot and cold starts in winter and summer, with sampling on the road (*169*), and of exhaust emissions from groups of automobiles equipped with air injection or engine modification devices or unequipped, again with sampling on the road (*255*).

A gas chromatographic system involving temperature programming of packed columns previously discussed (*5*) has been used in an evaluation of exhausts from various models of device-equipped and unequipped automobiles operated on dynamometers (*22*). Unsaturate compositions of exhaust emissions with gasolines of different compositions were determined in another investigation by gas chromatography with a subtractive technique (*112*). In this study the exhaust compositions have been related to several reaction parameters in photochemical irradiation chamber measurements on the diluted exhaust emissions.

Several methods for analyzing nitric oxide in automobile exhaust emissions have been evaluated; these methods involved conductivity, spectral measurement of nitrogen dioxide after oxidation of nitric oxide, and direct mass spectrometric analysis (*37*). The mass spectrometric method was evaluated for interferences under different engine conditions. Difficulties in obtaining high precision in nitrogen oxide analyses even under steady-state operation were shown to result from engine variations. This mass spectrometric method compared favorably with the usual colorimetric analysis for nitrogen oxides. Another preliminary evaluation of mass spectrometric analysis of nitric oxide has been reported (*60*). Colorimetric analyses of nitrogen oxides in automobile exhaust by several different procedures were compared (*63*). Water vapor, surface effects, and reactions between nitrogen dioxide and certain unsaturated hydrocarbons influenced losses of nitrogen oxides in these procedures.

Carbonyl compounds in automobile exhaust emissions have been collected in bubblers containing aqueous 2,4-dinitrophenylhydrazone reagent. The hydrazones were filtered, extracted, and dissolved in carbon disulfide and then analyzed by gas chromatography with a flame ionization detector (*181*). Sampling efficiencies, analytical errors, and potential interferences were evaluated. In another investigation of the determination of formaldehyde in liquid mixtures, the 2,4-dinitrophenylhydrazone was used similarly and the possible application to air pollutants suggested (*146*). Carbonyls in auto exhaust emissions can be identified by collection in bisulfite reagent followed by combined gas–liquid chromatography and infrared techniques (*72*).

Emissions from a group of automobiles and tracks have been analyzed for carbon monoxide, carbon dioxide, nitrogen oxides, total hydrocarbons, benzene-soluble particulates, and 10 of the polynuclear aromatic hydrocarbons (*93*). The presence of aza heterocyclic hydrocarbons such as benzo[*h*]quinoline, benzo[*c*]acridine, and indenoquinolines in automobile exhaust has been demonstrated (*238*). The ratio of aza heterocyclics to polynuclear aromatic hydrocarbons in automobile exhaust is much smaller than in other emission sources, an indication that automobile exhaust is only a very minor contributor to atmospheric levels of aza heterocyclic compounds.

Diesel exhaust emissions from two- and four-cycle engines have been analyzed for light hydrocarbons, nitrogen oxides, formaldehyde, acrolein, total aliphatic aldehydes, total particulates, and polynuclear aromatic hydrocarbons (*219*). Emissions from truck-type diesel engines were analyzed for nitrogen oxides, for formaldehyde, and for hydrocarbons including those from the unburned fuel (*113*). The gas chromatographic method used for higher-molecular-weight hydrocarbons in this latter study has been reported (*251*). An investigation was made into the effect of barium additives used to suppress smoke from diesels on the concentrations of hydrocarbons, oxides of nitrogen, and aldehydes in diesel exhaust from a two-cycle and a four-cycle engine (*252*).

Other Sources of Emissions. A portable combustion analyzer for hydrocarbons, carbon monoxide, and carbon dioxide has been developed for use in air pollution source testing (*20*). Solvent vapors have been analyzed directly on polyethylene glycol 400 or squalane substrates with an argon ionization detector (*75*). Solvent analysis has been done in another investigation by use of both gas chromatographic and infrared analysis (*77*). The formation of nitrogen oxides in combustion of pulverized coal has been studied to determine effects of excess air and of recirculation of combustion gas (*17*).

Four methods for analyzing sulfur dioxide in flue gases were compared and found to give comparable results (*125*). Sulfur dioxide can be determined in flue gases by a colorimetric method employing alizarin and thorium salts (*111*).

Polynuclear aromatic hydrocarbons have been analyzed in emissions from several industrial processes including asphalt air blowing, asphalt hot-road mix, and manufacture of carbon black, steel, coke, and chemicals (*283*). Effluents from the stack of a coal-burning residence, from two industrial sources, and from air contaminated with coal-tar pitch fumes have been analyzed for 12 polynuclear aromatic hydrocarbons and 11 aza heterocyclics including the animal carcinogens benzo[*a*]pyrene, benz[*a*]anthracene, dibenz[*a,h*]acridine, and dibenz[*a,j*]acridine (*239*). The concentrations of benzo[*a*]pyrene measured in gas-works retort houses were 100 to 10,000 times the levels present in the ambient atmosphere of London (*141*).

The amounts of total hydrocarbon, various aliphatic hydrocarbons, carbon monoxide, carbon dioxide, and nitrogen oxides were determined when a special tower built to simulate agricultural burning was used with various agricultural materials (*57*). The results indicated that the contribution of those pollutants associated with photochemical air pollution from agricultural burning was much less than from automobile exhaust emissions.

Methods for sulfur compounds and odorants from kraft pulp mill manufacturing processes were reviewed (*95*). Gas chromatographic analyses of sulfur-containing gases associated with kraft mills included analyses for oxygen, nitrogen, carbon monoxide, water vapor, hydrogen sulfide, sulfur dioxide, and methyl mercaptan using flame ionization, electron-capture, coulometric, thermionic emission and thermal conductivity detectors (*3*). Analytical methods have been presented for a wide variety of gases from kraft pulp mill operations including hydrogen sulfide, sulfur dioxide, methyl mercaptan, ethyl mercaptan, dimethylsulfide, and other sulfides and disulfides (*269*).

A PHS report has been published on atmospheric emissions from sulfuric acid manufacturing processes (*214*). Several methods of analysis for nitrogen oxides in gases from a pressure nitric oxide plant were evaluated (*76*).

FIELD STUDIES

Work continues on the development of methods for atmospheric tracer substances to be used in meteorological investigations. The use of fluorescent particles as atmospheric tracers has been reviewed (*145*). Problems of calibration, separation, field operations, and background levels in the use of the gaseous tracers sulfur hexafluoride and dichlorodifluoromethane have been discussed (*278*). The atmospheric stabilities of sulfur hexafluoride, bromotrifluoromethane, and octafluorocyclobutane have been evaluated with respect to reactions with various atmospheric pollutants, effects of ultraviolet radiation, and washout (*228*). These three substances have high stability in such processes.

The effects of air pollutants on exposed cotton fabrics have been measured (*32*). Atmospheric corrosion studies have been made in two metropolitan areas (*280*). The horizontal and vertical distribution of corrosion rates were determined in an industrialized city on the seacoast (*129*).

Trends in suspended particulates in the air of U. S. cities for 1957 to 1964 have been reviewed (*170*). Total solids, iron, manganese, copper, tin, lead, and zinc were determined in the Paris area and the values compared to those in U. S. cities (*53*). Sodium, potassium, calcium, and magnesium were determined along with sulfate, ammonium, nitrate, and nitrite and related to meteorological variations in areas of Asahikowa, Japan (*195*).

Much activity has occurred in discussions and measurements of lead as an atmospheric pollutant. Sources of atmospheric lead and of atmospheric lead levels were reviewed in a PHS publication (*165*). Concern has been expressed about possible biological effects of the lead levels presently existing in urban atmospheres (*201*). In another review of biological aspects no hazard was considered to exist (*92*). A survey of the lead content of the atmospheres of three U. S. cities has been made (*159*) and the results presented in a PHS publication (*213*). Several thousand analyses for lead by a polarographic method were obtained on samples from 18 cities in Ontario, Canada (*67*). Lead analyses were made as part of more comprehensive analytical studies of the atmospheric composition in London (*286*), Paris region (*53, 277*), Japan (*273*), and Cambridge, Mass. (*152*). Lead isotopic composition of aerosols was determined in Los Angeles and found similar to that in rural snow (*43*). The high lead content in rainwater and on the surface of the sea were attributed to lead from automobile exhaust emissions.

The chloride, bromide, and iodide content of cascade impactor samples were determined by neutron activation analysis at two sites in Cambridge, Mass. (*152*). The results for chlorides and iodides related well to measurements made in unpolluted areas, while the bromide content was similar to that in polluted environments. Strong correlation of lead and bromide contents during low visibility situations and in curbside samples suggested a common source in the particulate from automobile exhausts. Several studies involving samples from Los Angeles Basin, San Francisco Bay area, Chicago, and Cincinnati all gave mass median diameters of 0.3 to 0.4 micron for sulfate-containing particulate (*143, 158, 220*). These investigations, as did one conducted in Pittsburgh (*52*), indicated that 80 to 90% of the sulfate-containing particulate was in the respirable size range. Some increase in mass median diameter occurred with increasing relative humidity (*143*). Seasonal variations were reported for sulfate concentrations in samples collected in Budapest (*173*). Fluoride concentrations were measured in an industrial region and surrounding rural area in France (*26*).

The carcinogen benzo[*a*]pyrene continued to be one of the most widely measured atmospheric pollutants. Analyses for benzo[*a*]pyrene by fluorescent or spectrophotometric techniques frequently with thin layer chromatographic separation were reported for samples from London (*286*), Paris (*142, 277*), Vienna (*199*), several cities in Germany (*104*), Leningrad (*56*), several cities in South Africa (*156*), Tokyo (*225*), Sidney, Australia (*45*), a number of cities in Ontario, Canada (*176*), and Pittsburgh (*59*). In the study of German cities, a number of other polynuclear aromatic hydrocarbons were analyzed including anthracene, phenanthrene, pyrene, fluoranthene, 1,3-benzanthracene, chrysene, benzo[*e*]pyrene, 1,2,5,6-dibenzanthracene, and coronene (*104*).

In Sidney, after extraction with acetone or cyclohexane and separation on long alumina columns, samples were analyzed by ultraviolet spectroscopy for pyrene, fluoranthene, 1,2 benzanthracene, chrysene, benzo[*a*]pyrene, benzo[*e*]pyrene, 1,12-benzoperylene, and coronene (*45*). Possible interferences and unresolved fractions also are discussed. In the Pittsburgh study, gas–liquid partition chromatography was used to separate and measure pyrene, 1,2-benzanthracene, chrysene, benzo[*k*]fluoranthene, benzo[*e*]pyrene, and benzo[*a*]pyrene (*59*).

In a sample of Nashville air, 19 polynuclear quinolines, isoquinolines, and acridines were identified (*237*). In this same study, 11 polynuclear aromatic hydrocarbons, three benzacridines, and three quinolines were measured in samples from Atlanta, Cincinnati, Los Angeles, Nashville, New Orleans, and Philadelphia. A composite from 100 U. S. cities was analyzed for nine polynuclear aromatic hydrocarbons, 12 *n*-alkanes, three benzacridines, and three quinolines. The weight ratio of aliphatic hydrocarbons to aromatic hydrocarbons to aza heterocyclics measured in this composite sample was 300:24:1 (*237*). In a sample of particulates scraped off walls in a Naples tunnel, extraction, column chromatographic separation, followed by thin layer chromatography on several substrates, resulted in the identification of 74 compounds (*64*).

Chlorinated hydrocarbon and thiophosphate type pesticides have been analyzed in particulate samples obtained from agricultural communities during spraying operations and from communities with pest control programs (*270*). Use of the electron-capture and the sodium thermionic detectors made it possible to measure a variety of chlorinated hydrocarbons including DDT, chlordane, aldrin, toxaphrene, malathion, and traces of several thiophosphates. Dust transported over long distances was analyzed by electron-capture and microcoulometric chromatographic methods; analyses indicated the presence of DDT, chlordane, DDE, and ronnel and smaller concentrations of heptachlor epoxide, 2,4,S-T, and dieldrin (*48*). Total organic chlorine was 1.3 ppm and total organic sulfur, 0.5 ppm. A rotating-disk impactor and a midget impinger have been used to collect both aerosol and gaseous fractions of airborne di- and trichlorophenoxyacetic acid and derivatives (2,4-D) (*1*). Following analyses by the electron-capture chromatographic method over a 100-day period, the 24-hour average concentrations of aerosol and gaseous 2,4-D compounds were reported. Airborne particulates in Pittsburgh have been analyzed for DDT by gas chromatography (*10*).

A brief review has been presented on automatic and manual data logging systems for air quality measurements of gases (*149*). The results from the Continuous Air Monitoring Program (CAMP) of the Public Health Service in six U. S. cities have been summarized for 1962–64 (*161*). One- and two-year means; maximum monthly, daily, hourly, and 5-minute values; and seasonal and diurnal variations were reported for sulfur dioxide, nitric oxide, nitrogen dioxide, oxidant, carbon monoxide, and total hydrocarbons. The results of 5 years operation of another Public Health Service network in 50 U. S. cities in which sulfur dioxide and nitrogen dioxide have been collected in impinges on a 24-hour basis also have been reported (*271*). Where possible these 24-hour results were compared with results from the continuous air monitoring program. Details on sampling and analyzing the sulfur dioxide and nitrogen dioxide are given (*271*).

In a more recent presentation on this gas network, the details of *Auto*Analyzer analysis have been given for sulfur dioxide, nitrogen dioxide, hydrogen sulfide, and aldehydes (*178*). A monitoring system for sulfur dioxide, nitrogen oxides, oxidants, and aldehydes has been developed in New Jersey with direct analysis by *Auto*Analyzer units (*293*, *294*). Reagent consumption rates, operating costs, on-stream factors, and a lost-time analysis have been discussed (*294*).

A variety of gaseous pollutants including carbon monoxide, carbon dioxide, sulfur dioxide, nitrogen dioxide, oxidants, and formaldehyde have been determined on a toll road and in a parking area in Japan (*273*).

Considerable work has been done on the analysis of carbon monoxide both in the ambient atmosphere and in possible high-risk situations inside automobiles, at traffic intersections, and in tunnels. Analyses were made inside the passenger compartment over a 30-mile commuter route in Los Angeles at various times of the day (*90*). Average carbon monoxide concentrations above 50 ppm for 40- to 80-minute driving periods were reported, with peaks up to 120 ppm. During the winter on this route, carbon monoxide concentrations exceeded 30 ppm for 50% of the time and exceeded 45 ppm for 10% of the time. Similar but more extensive measurements of carbon monoxide have been made in a number of U. S. cities both inside motor vehicles and alongside traffic routes (*30*). For the various routes used in these cities, 50% of the 20 to 30 minute integrated carbon monoxide values exceeded 20 to 40 ppm; 10% of these values exceeded 30 to 60 ppm. Comparison with carbon monoxide values from CAMP stations in the same cities showed the ratio of in-traffic to station values varied from 1.3 to 6.8 (*30*).

Mean values for carbon monoxide during peak traffic hours at the largest traffic section in Dayton, Ohio, were near 40 ppm whereas at all other times the mean values for carbon monoxide were 26 ppm (*216*). Carbon monoxide values exceeded 100 ppm about 10% of the time at this intersection during peak traffic hours. Comparisons also were made with relation to traffic volume, meteorological variations, and concentrations at other urban sites (*216*).

Measurements of carbon monoxide in the central districts of Paris showed that the highest average values were in the range of 26 to 47 ppm during 1960 to 1964 (*277*). Concentrations of carbon monoxide have been reported in several tunnels (*28*, *91*). Carbon monoxide measurements have been made in several cities by use of nondispersive infrared analyzers; results have been integrated at 30-minute intervals for comparison with two modified detector tube methods. (*108*). In general, higher carbon monoxide levels were obtained by the nondispersive infrared measurements than by the detector tube procedures.

Carbon dioxide measurements with a nondispersive infrared analyzer have been obtained in several U. S. cities (*44*). It was demonstrated that the variations in urban carbon dioxide concentrations above the background level result from both combustion and natural sources. Since it is not yet possible to predict the contribution from natural sources to urban carbon dioxide levels measurements of urban carbon dioxide were considered to have only limited utility as an index of air pollution (*44*).

A statistical approach to determining the frequency distribution of sulfur dioxide concentrations has been shown to fit the experimental values obtained at 20 measuring places (*267*). The empirical function obtained can be used in assessment of emission limits for sulfur dioxide. The role of wind parameters has been considered in determining sulfur dioxide concentrations in New Jersey (*147*). Investigators in England and in the U.S.S.R. have been concerned with the determination of sulfur dioxide distributions about power plants (*21*, *163*). A detailed study has been reported on sulfur dioxide levels in an industrial region and adjacent rural area during 1961–63 (*26*). In measurements during the winter in the center of Gothenburg, Sweden, good agreement was obtained for the pararosaniline method, a conductometric method, and a new modification of the hydrogen peroxide method (*202*).

Measurements for hydrogen sulfide by the method involving lead acetate–treated filter paper in a community containing a Kraft pulp mill gave values from 0 to 10 pphm (*106*). Analyses also were made with the methylene blue method. These analytical results were compared with results from exposure of silver specimens (*106*). In another study testing for hydrogen sulfide with lead acetate paper, unglazed tiles and visual examination were used to evaluate paint darkening and to develop a dosage relation for nonhealth air pollution effects of hydrogen sulfide (*292*).

Analyses for nitric oxide and nitrogen dioxide were made by both the Saltzman and the Jacobs-Hochheiser methods in several cities (*107*). Field experience with the mast oxidant analyzer has been discussed in terms of calibration, sampling, maintenance, collection of data, and costs of operation (*207*).

Continuous monitoring for methane, total hydrocarbon, and nonmethane hydrocarbon has been carried out in Los Angeles and Cincinnati by use of modified flame ionization analyzers (*8*). Although methane values are similar in the two cities, nonmethane levels were 4 times as high in Los Angeles as in Cincinnati. The distribution patterns indicated a different and more constant source for methane than for the other pollutants. Hydrocarbons have been measured on 20- to 30-minute integrated samples from insides of vehicles in several U. S. cities and compared with the levels recorded at CAMP stations (*30*). The hydrocarbon values were 1.4 to 2.7 times higher inside vehicles than at the monitoring site. In several cities, the upper 10% of hydrocarbon values exceeded 10 carbon ppm (*30*).

The concentrations of a number of six- to 10-carbon aromatic hydrocarbons have been measured in samples collected at various locations in the Los Angeles Basin (*185*). The concentrations of a number of aliphatic hydrocarbons were determined for samples collected in the Los Angeles Basin (*260*). These samples were subjected to photochemical irradiation in the laboratory to obtain the rates of consumption of the individual hydrocarbons in atmospheric samples. Aliphatic hydrocarbons have been analyzed in 200 samples collected at 21 locations in Tokyo during 1964 and 1965 both at ground level and from aircraft (*127*). It was concluded that the main source of pollution in Tokyo is automobile exhaust gases. Measurements for aliphatic hydrocarbons have been reported in an industrial area in England (*124*). Although nitroolefins can be analyzed at concentrations as low as 0.001 ppm in the atmosphere, the first two members of the nitroolefin series could not be detected in samples collected in the Los Angeles Basin (*262*).

An air pollution evaluation of Titan II testing firings has been made (*61*). The air pollution control program at Cape Kennedy has been reviewed (*168*).

Work continued on attempts to associate asthma episodes with specific atmospheric pollutants. Measurements have been made of aerosols, air ions, and gaseous pollutants in order to isolate causes of asthma episodes in New Orleans (*130*). In Minnesota, measurements were obtained on particulates, sulfur dioxide, and nitrogen dioxide to relate these pollutants to occurrences of bronchial asthma (*174*).

Pollutant concentrations inside homes or apartments have been compared to ambient levels. In New York City, the concentrations of hydrocarbons and nitrogen oxides inside an apartment house adjacent to an expressway were significantly higher than at the air pollution laboratory sampling site, especially on days of high pollution (*29*). In measurements made in Tokyo, particulates and sulfur dioxide levels outside were higher than inside, but nitrogen dioxide and formaldehyde levels were nearly the same inside and outside (*175*). Samples collected in living rooms of homes in Rotterdam showed on the average 80% of the particulate and 20% of the sulfur dioxide levels measured in the outside air (*18*).

Fleck damage on tobacco in Southern Ontario was shown to relate to ozone levels of equal or greater than 20 pphm-hour although measurements were made for a number of other pollutants (*49*).

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Clinical Chemistry

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THE AUTHOR's last review of publications of significant developments in clinical chemistry (*10A*) was continued and covers the period from December 1964 to December 1966.

REVIEWS AND NEW BOOKS

During the past two years increasing interest has been shown in automation, quality control, data handling, and better methods of standardization. Practically all of the representative clinical chemistry procedures have been adapted to automation, and for some procedures many different modifications have been made. Increasing competition in automation equipment is apparent. The Robot Chemist has been developed to accept conventional wet chemistry methods and is adaptable to the performance of several (*16*) different procedures but not simultaneously. Protein does not interfere with most of the tests. Other automated systems such as the Mark IX (Hycel) have been introduced but as yet are not clinically evaluated. Evaluation surveys concerned with quality control and improvement of methodology continue to appear in the literature. Laboratory and hospital management has shown an increasing interest in data handling as an adjunct to automation of methodology. Clinical chemists are concerned with the need for better standards, both primary and secondary (serum).

Hamilton (*7A*) in a review of the literature covering biochemical analyses over the years 1964–65 covered some of the following topics: new journals, ultrafiltration, enzymic methods, electrophoresis (paper, thin-layer, and column), ion exchange, spectrophotometry, instrumentation, and automation. Adams and Mattenheimer (*1A*) reviewed the literature dealing with simplified clinical-chemical test methods. Methods of biochemical analysis for body constituents, hormones, and vitamins were reviewed by Niwa (*14A*). The possibilities and limitations of separation methods in biochemical analysis, such as solvent extraction and distribution, absorption and ion exchange, chromatography, electrophoresis, and gel filtration were reviewed by Pfleiderer (*15A*). A few routine clinical chemistry procedures were discussed by Turpin and Balows (*20A*). Natelson (*13A*) reviewed microtechniques of clinical chemistry. Grunbaum and Pace (*6A*) described simplified microchemical methods of urinalysis. Shively (*17A*) outlined items to be considered in the evaluation of methodology in clinical chemistry. Reviews of specific subjects of interest to clinical chemists were made by Strickland (*19A*) on electrophoresis, Heftmann (*8A*) on chromatography, Walton (*21A*) on ion exchange chromatography, Margoshes and Scribner (*12A*) on emission spectrometry, Boltz and Mellon (*3A*) on light absorption spectrometry, Crummett (*4A*) on ultraviolet spectrometry, Evans (*5A*) on infrared spectrometry, White (*23A*) on fluorometric analysis, Hercules (*9A*) on fluorescence and phosphorescence analysis, Ma and Gutterson (*11A*) on organic microchemistry, West (*22A*) on inorganic microchemistry, Skougstad and Fishman (*18A*) on water analysis, and Wimer, Theivagt, and Papendick (*24A*) on pharmaceuticals and related drugs.

Pushkina (*16A*) presented "Biochemical Methods of Investigation," a handbook for medical hygienists and pathologists. "New Methods of Biochemical Photometry" was published by Asatiani (*2A*). The procedures and articles presented in the fifth volume of "Standard Methods of Clinical Chemistry" are referred to in the appropriate sections of this review.

APPARATUS AND EQUIPMENT

Haley and Thomas (*15B*) clinically evaluated the new Warner-Chilcott Robot Chemist for speed of operation, ease of change-over from one method to another, and service problems, and compared its work capacity and precision with a competitive multichannel automated instrument. Natelson (*35B*) described an automatic chemical analyzer and sample dispenser which can process a large number of samples and give results in a short time. The Steroscan electron microscope which produces an excellent "three-dimensional" image was described by Müller (*32B*). A new precision spectrophotometer with improved photometric stability and accuracy which permits measurement of concentration over a wide range without dilution was developed by Gilford, Emary, and Martens (*14B*). Müller (*31B*) described a compact low-cost infrared spectrophotometer (IR–102 Microspec) equipped with circular variable narrowband interference filters and a recorder. Matsuyama (*28B*) developed an apparatus for rapid determination of organic iodine in biological materials by burning the sample in a stream of oxygen bubbled through 25% HCl. Kaiser (*24B*) described the uses of and the instrumentation involved in scintillation spectrometry. The problems in analysis of biological materials by atomic absorption spectroscopy were discussed by Willis (*47B*). Veillon *et al.* (*45B*) used a 150-watt xenon arc continuous source, a total-consumption atomizer-burner, and a low resolution monochromator for low-limit detection of 13 metallic elements by atomic fluorescence flame spectrophotometry. The Beckman Electroscan 30 designed for 15 electroanalytical techniques, which "combines in one complete self-contained electrochemical system flexibilities and capabilities previously obtained only with a combination of instruments," was described by Müller (*30B*). Spiegel (*41B*) used a new glass electrode for the anaerobic measurement of blood pH. Electrodes suitable for measuring blood pH were assembled by Hroch von Dalebor (*20B*) in a hypodermic syringe and needle for continuous measurements in both situ and vivo. Segal and Wodley-Smith (*38B*) described an apparatus for the continuous electrolytic determination of small quantities of gaseous ammonia. Shapira and Wilson (*40B*) developed a micro absorptiometric flow cell with 1-mm circular aperture with in-beam sample volume of 8 μl and sample volume of 40 μl.

Gelman (*12B*) reviewed the historical development and applications of microporous membrane technology and Friedlander and Rickles (*11B*) the theory and development of the technology of those membrane processes of most concern to analytical chemists. An apparatus employing a Bac-T-Flex membrane was described by Leaver (*25B*) for large-scale purification of proteins by membrane electropartition. A dialysis method without use of membranes was developed by Zascpa and Dobry-Duclaux (*52B*) by diffusion at the interface between solution and solvent in a vertical long narrow tube filled with solvent, in which the solution flows down the axis. The structure and principal properties of plastic materials and their applications in the field of medical biochemistry, pharmacology, and nutrition were described by Ghersa (*13B*). Electrophoresis of protein or enzyme was performed by Dubbs (*6B*) in a gel medium across which, transverse to the direction of protein migration, a continuous pH gradient was extending. El-Marsafy and Abdel-Gawad (*8B*) made an economical modified inverted V-type electrophoresis cell with which sharp resolution was achieved in 4 hours. Methods of detection of fluorescent substances separated by "small-volume" electrophoresis were described by Fischl and Segal (*10B*). Winsten *et al.* (*49B*) used large scale continuous flow electrophoresis for enzyme fractionation. Efimov (*7B*) designed an apparatus for the automatic application of solution to paper for chromatographic separation. Microgram samples of proteins, nucleic acids, and polysaccharides separated by liquid chromatography were detected by Stouffer, Kersten, and Krueger (*43B*) by use of a H-flame ionization detector in which a continuous platinum chain was passed through the flame. Mather and Assimos (*27B*) evaluated a quick screening method for detection of abnormal levels of organic blood volatiles by direct gas-liquid chromatography of blood. Ornstein (*36B*) reviewed the background and theory of disk electrophoresis and Mori and Hashimoto (*29B*) thin-layer zone electrophoresis. Müller (*33B*) described the Dupont 310 (analog-computer) curve resolver which permits synthesis of a complex curve exactly matching the observed curve. Lotito, McKay, and Seligson (*26B*) described an analog computer for the computation of stained paper electrophoresis patterns of serum proteins.

Jewell (*23B*) developed an apparatus for stoppering multiple tubes in one motion. A stopped-flow apparatus was described by Sturtevant (*44B*) which can be used for spectrophotometry or fluorometry. Sequeira (*39B*) developed an automatic multiple pipetting device that can be operated with program cards. Natelson (*34B*) reviewed recent developments in x-ray spectrometry as applied to clinical chemistry. A new instrument, the Bilirubinometer Mk III, described by Jackson (*22B*), is a differential spectrophotometer for reading the difference between the absorbance at 450 and 540 mμ. Ferrari, Catanzaro, and Russo-Alesi (*9B*) developed a new system for Kjeldahl digestion which involves the use of superheated walls of a revolving glass helix. Puro (*37B*) developed a fluorometric scanner-recorder system for the Turner fluorometer. Hovnanian, Brennan, and Botan (*19B*) adapted an ultraviolet television microscope to a quantitative fluorescent microscope for the detection of biologically crucial molecules. Herrmann and Roetger (*16B*) described a modified plasma arc burner for the chemical determination of a number of elements in biological fluids. Wistreich (*50B*) developed an apparatus for quantitative determination of fatty material and water in biological tissue. Vysotskaya and Krivopalov (*46B*) designed a fluorometer for the detection of catecholamines in living cells. Deaver, Swedlung, and Bradley (*5B*) constructed a new superconducting circuit with superconducting magnetic shields and persistent current magnets to measure magnetic properties of some macromolecules of biological interest. Belliveau, Grayson, and Wenandy (*3B*) evaluated a hollow plastic pneumatic ram for separation of serum from blood clotted in a test tube. A high pressure filtration device was described by Bagshawe, Kemble, and Thomas (*2B*) for the concentration of gonadotropic hormones from urine by absorption onto and elution from kaolin. Ashmead (*1B*) developed a liquid sampling valve which introduces measured volumes of samples into an analytical apparatus. Holmes (*18B*) proposed a closed-system aeration-diffusion apparatus for the determination of blood ammonia. An extractor was devised by Holmes (*17B*) for blood cholesterol determination, which dries and extracts the sample on filter paper in one continuous operation with one solvent. Automated instrumentation for the determination of prothrombin time was described by Cutler-Hammer (*4B*), Sterling *et al.* (*42B*), Wylie and Fraser (*51B*), Huff (*21B*), and Winsten (*48B*).

AUTOMATION

The Technicon Symposium was held in September 1965 in New York, at which time 43 papers on automated methods in clinical chemistry were presented. These papers were published in "Automation in Analytical Chemistry" (1965) (*82C*). A larger symposium was held in November 1966 in New York. Müller (*62C*) reviewed automation in clinical chemistry, especially the SMA-12 *Auto*Analyzer. Skeggs (*80C*) reviewed the principles of colorimetric chemical analysis as performed by the *Auto*Analyzer and described its modules and multiple analytical systems. Skeggs emphasized the need of automated systems capable of analyzing a single serum for 20 to 40 constituents for better diagnosis of the patient (*79C*). Newman *et al.* (*67C*) described the theory of operation and function of the Robot Chemist, which consists of three basic modules, a sample presentation and sampling unit, a sample processing unit, and a sample sensing unit. Loeb (*50C*) described the automated Mecolab system which is based on discrete analysis. The sample is prepared and measured in individual test tubes as in manual methods, and consists of several separate modules, such as sampling, reagent addition, supernatant, autocolorimeter, and automatic indexing units. The Hycell Mark IX (Hycel, Inc., Houston, Tex.) which performs 10 different analyses on 40 specimens per hour by conventional wet chemistry methods has been announced by the manufacturer but has not been clinically evaluated. An automatic analytical apparatus was developed by Baruch and Travaglio (*4C*) for the preparation of protein-free filtrates. Cole, Kirsch, and Thiers (*13C*) evaluated two types of parameters of continuous flow systems, those which describe the steady state, and those which describe the transient state.

Hicks *et al.* (*32C*) described LINC, a small, inexpensive, high-speed digital computer, programmed to operate in the "conventional mode" and requiring no knowledge of computer programming. Cotlove (*17C*) outlined a system for computer data processing on line from automatic analytical instruments and considered some aspects of computer integrated automation in a clinical chemistry laboratory (*18C*). Young and Cotlove (*95C*) studied the factors which influence the reported concentration of samples analyzed by means of an *Auto*Analyzer, and also peak recognition and identification for real-time on-line data acquisition (*95C*). Constandse (*16C*) defined a laboratory data acquisition system and discussed the various instruments which could be connected to such a system. Mabry *et al.* (*53C*) modified the *Auto*Analyzer system to perform nine frequently performed biochemical analyses on 50 to 100 μl of serum. Hicks and Nalevac (*33C*) described a programmer for the automatic stepwise addition of solutions to chromatographic columns consisting of five parts: column follower electrodes, solution level-sensing circuit, time delay circuit, stepping-relay circuit, and solenoid valves. Marmasse (*55C*) described automatic equipment for multichannel recording of oxidation-reduction potentials in biological media. Uchiyama, Morimoto, and Yoshimura (*89C*) radiotelemetered an implantable glass pH electrode to record the pH of blood in a living animal.

Eggstein *et al.* (*24C*) compared *Auto*Analyzer data with clinical chemical values obtained by conventional techniques and found the precision of the automatic determination as good as the manual. Benson and Patterson (*5C*) accelerated the automatic chromatographic determination of amino acids by use of a spherical particle resin in the Beckman amino acid analyzer. Satake, Matsuo, and Take (*77C*) used a method based on dinitrophenylation with 1-fluoro-2,4-dinitrobenzene for automatic determination of amino acids and peptides. Kurtzman, Smith, and Snyder (*47C*) developed an automatic colorimetric method based on the Sullivan-Hess method for the determination of cystine with the *Auto*Analyzer. The fluorometric phenylalanine method of McCaman and Robins was adapted to automation by Hill *et al.* (*35C*). Hill (*34C*) developed an automated fluorometric method for serum calcium using fluorescein-complexone as the fluorophore. Arvan (*1C*) investigated the effect of magnesium on some automated calcium methods. Smith, Kurtzman, and Ambrose (*82C*) adapted the indicator dye Eriochrome Blue S.E. calcium method to automation, and Manston (*54C*) automated a simultaneous method for calcium and phosphorus. Serum magnesium methods were automated by Breen and Marshall (*8C*) using *0,0*-dihydroxyazobenzene for fluorescence, by Bossche and Wieme (*7C*) by adapting the Mann and Yoe method, by Gitelman, Hurt, and Lutwak (*30C*) by use of the Eriochrome Black T colorimetric method, and by Oklander and Klein (*68C*) by adapting the fluorometric Hill procedure. Nevius and Lanchantin (*66C*) substituted natural gas for propane in the oxygen-fuel supply for the *Auto*Analyzer flame photometer. The Technicon serum electrolyte method was modified by Dawborn, Patalinghug, and Black (*22C*) to measure urine electrolyte in concentrations of 0 to 200 meq per liter. Jacobson (*39C*) employed an automatic continuous method for the urinary determination of Na and K in the presence of ammonia with a glass electrode. Gambino and Schreiber (*29C*) prevented significant loss of CO$_2$ from serum in open *Auto*Analyzer cups by addition of 0.035 ml of 1N NH$_4$OH to 1 ml of serum. A completely automated system for the determination of enzyme activity by initial reaction rates (increase or decrease of absorbance at 340 mμ), was designed by Murray and Harmon (*63C*). Automated colorimetric methods for LDH were described by Capps *et al.* (*10C*) by adaptation of the tetrazolium salt, INT (2-*p*-indophenyl-3-*p*-nitrophenyl-5-phenyl) tetrazolium chloride with phenazine methosulfate as electron carrier; by Morgenstern, Flor, and Kessler (*59C*) by adaptation of the cupric-neocuproine reduction with NADH to the Robot Chemist; and by Plaut (*73C*) by a simple automated colorimetric method. Strandjord and Clayson (*85C*) developed an automated method for determining total LDH and heat-stable LDH 1. Brooks and Olken (*9C*) determined serum LDH by a simple automated fluorometric method by measuring the fluorescence of DPNH formed in the forward lactate → pyruvate reaction. Morgenstern *et al.* (*61C*) automated the glutamic oxaloacetic *trans*-aminase method employing Fast Ponceau L to measure the enzymically generated oxaloacetic acid. Klein, Auerbach, and Morgenstern (*42C*) automated both the phenyl phosphate and 1-naphthyl phosphate substrate methods for determination of acid phosphatase with the *Auto*Analyzer and Robot Chemist (*45C*). Alkaline phosphatase methods were automated by Sterling *et al.* (*84C*) utilizing *p*-nitrophenyl phosphate, by Coleman and Stroje (*14C*) using thymolphthalein monophosphate to eliminate the need for bilirubin blank, by Morgenstern *et al.* (*60C*) who adapted the *p*-nitrophenyl phosphate substrate method, and by Comfort and Campbell (*15C*) who utilized the phenolphthalein diphosphate substrate. An automatic continuous flow dialysis system was used by Stein and Lewis (*83C*) to study the hydrolysis of acetylcholine iodide by acetylcholinesterase. Humiston and Wright (*38C*) automated the determination of cholinesterase by reaction of cholinesterase with acetylthiocholine to release thiocholine which reacts with 5,5′-dithiobis(2-nitrobenzoic acid) to produce color. Fleisher (*27C*) automated the determination of serum creatine kinase activity by reactivation of creatine kinase with cysteine and subsequent reaction with ADP and phosphocreatine. Siegel and Cohen (*78C*) automated the serum creatine phosphokinase method which employs adenosine diphosphate, and mercaptoethanol as a source of sulfhydryl groups, dialysis against recipient solution of *N*-ethyl maleimide, and color development with 1-naphthol and diacetyl. An automated method for the determination of ornithine carbamoyl transferase activity was demonstrated by Strandjord and Clayson (*86C*).

Hill and Sammons (*36C*) described an automated procedure employing the *Auto*Analyzer for the estimation of serum 5′-nucleotidase, in which the enzyme activity was measured as the nickel-sensitive hydrolysis of adenosine 5′-monophosphate. Polar and Metcoff (*74C*) described a semiautomated method for the determination of "true" creatinine. Pino, Benotti, and Gardyna (*72C*) adapted a urine creatinine method to the *Auto*Analyzer which did not require a dialyzer module, and Bittles, Bell, and Neill (*6C*) also described a simple urinary creatinine method for this instrument. Wilcox *et al.* (*92C*) and Wilson (*93C*) adapted the Berthelot reaction to the *Auto*Analyzer for urea N determination. Ceriotti and Spandrio (*12C*) used the cations Fe^{3+} and Ce^{4+} to accelerate the urea-diacetylmonoximephenazone reaction for application to the *Auto*Analyzer. The phosphotungstic hydroxylamine method was automated by Musser and Ortigoza (*64C*). Galli and Jeanmarie (*28C*) adapted the thiobarbituric acid reaction for the estimation of fructose with the *Auto*Analyzer. Nagler (*65C*) presented an automated method based on the production of furfural derivatives for the determination of glycogen. An automated microdetermination of blood glucose employing the glucose oxidase method was described by Faulkner (*26C*), using the peroxidase-*o*-tolidine colorimetric method, and by Lim (*49C*) by conductometric measurement of the gluconic acid formation. Swann and Balazs (*87C*) automated the *p*-dimethyl aminobenzaldehyde method for the determination of *N*-acetylhexosamines. Dawborn (*21C*) applied an insulin method based on hydrolysis of inulin to fructose before dialysis to the *Auto*-Analyzer. Details of an automated trihydroxyindole procedure for the differential estimation of epinephrine and norepinephrine in tissues, urine, and blood were presented by Robinson and Watts (*75C*). Klein and Oklander (*43C*) automated a serum and urine acetoacetate method based on the Rothera reaction. Hochella and Weinhouse (*37C*) adapted two lactic acid methods to the *Auto*Analyzer: the Barker-Summerson and the lactic dehydrogenase coupling through diaphorase to the oxidoreduction dye, 2-(*p*-iodophenyl)-3-(*p*-nitrophenyl)-5-phenyltetrazolium chloride. Minaire, Studievic, and Foucherand (*58C*) determined lactic acid with the *Auto*Analyzer by measuring the absorbance of NADH at 340 mμ. Mathieu, Treuil, and Revol (*56C*) adapted the color reaction of diazotized *p*-nitroaniline for the determination of phenols with the *Auto*Analyzer. Balazs *et al.* (*3C*) described an automated method for the determination of hexuronic acids based on the carbazole reaction. Young (*94C*) adapted the Delsal and Manhouri inorganic phosphate method to the *Auto*Analyzer. Lorch and Gey (*52C*) by a simple arrangement, analyzed free fatty acids by recording with the *Auto*Analyzer the color change of an indicator-buffer system. Whitley and Alburn (*91C*) and Kraml (*46C*) reported semiautomated serum phospholipid methods for the *Auto*Analyzer. A procedure was described by Lofland (*51C*) for the determination of triglycerides in serum by 2-propanol extraction with the *Auto*Analyzer. Young and Hicks (*96C*) determined serum iron with the *Auto*Analyzer by dialysis of reduced iron and complexing with tripyridyltriazine for colorimetric measurement. A similar method was described by Zak and Epstein (*97C*), who developed color with 4,7-diphenyl-1,10-phenanthroline sulfonate. Kemp (*41C*) estimated hemoglobin by the cyanmethemoglobin method using the *Auto*-Analyzer.

A fluorometric system for automatically separating serum protein fractions based on a DEAE-Sephadex column was described by Toporek and Philipp (*88C*). Roubal and Tappel (*76C*) separated proteins on a Sephadex (G-100) column for molecular weight determination and monitored the column effluent with *Auto*Analyzer components. Crowley (*19C*) made simultaneous determinations of serum total protein and albumin with an *Auto*Analyzer. An automated procedure for serum albumin was presented by Klein and

Oklander (*44C*) based on the binding of albumin with 1-anilinonaphthalene-8-sulfonic acid which gives linear fluorescence with albumin concentration. Glenn (*31C*) and Jones (*40C*) described a method for the determination of serum globin using an *Auto*Analyzer, based on the measurement of turbidity when serum is mixed with 27.4% sodium sulfate solution. An automated method for the determination of prothrombin time was described by Miale *et al.* (*57C*). Cawley *et al.* (*11C*) used a semiautomated immunoelectrophoresis system based on the utilization of agar-coated Cronar and Mylar sheets. Welshman, Bell, and McKee (*90C*) described an automated method for the estimation of serum protein-bound iodine following alkaline incineration. Crowley and Jensen (*20C*) evaluated a completely automated *Auto*Analyzer system for protein-bound iodine. Piette *et al.* (*71C*) determined total and hormonal iodine in serum with the *Auto*Analyzer using a Dowex (50-X2) column for separation of hormonal activity. Pedersen (*70C*) invented a semiautomatic programmed apparatus for the determination of protein-bound iodine. Austin and Koepke (*2C*) compared an automated colorimetric and an acid distillation method for total organic serum iodine and found good agreement. Oppenheimer, Martinez, and Bernstein (*69C*) determined the maximum binding capacity of thyroxine-binding prealbumin in serum by an improved method using conventional paper electrophoresis and glycine acetate buffer at pH 9.0. Leithoff (*48C*) made an automatic determination of blood ethanol by reduction of ethanol with alcohol dehydrogenase in presence of DPN and colorimetric determination of excess DPN. A semiautomatic determination of lead in whole blood was described by Delves and Vinter (*23C*). Falk and Kelly (*25C*) adapted the Bratton-Marshall sulfonamide method to automation.

CONTROL AND PRECISION OF CLINICAL CHEMISTRY METHODS

Winsten (*16D*) outlined sound rules for the collection and preservation of biological specimens such as sampling after a minimum 4-hour fast, use of serum free of hemolysis, prompt clot separation, refrigeration, and analysis within 24 hours. Blaivas (*2D*) described the requirements for stabilization of blood samples for mail transport. Caraway (*4D*) summarized the specific sources of error in particular determinations in clinical chemistry. Wirth and Thompson (*17D*) studied the effect of a large variety of factors both chemical and physical in nature, and conditions and substances, on the results of laboratory procedures. Straumfjord and Copeland (*14D*) made a survey of day-to-day clinical chemistry quality control values in 33 university medical school hospitals. An "average of normals" method of quality control was suggested by Hoffmann and Waid (*7D*) which was carried out by averaging all patients' test values that fall within the normal range (daily) and plotting this "average of normals" on a control chart. Shively (*11D*) tabulated the different methods used for 20 chemical tests performed in clinical laboratories by means of a questionnaire submitted to members of the American Society of Clinical Pathology. Slutskii (*12D*) discussed the errors in the application of biochemical analytical methods. Copeland (*5D*) outlined the accuracy and precision requirements for medical diagnosis and therapy and stated that standards should be described using statements of precision and accuracy associated with method and causes of variability. Plym (*10D*) compared the coefficient of variation of various concentrations of 14 constituents in serum and determined which to include in the quality control program of the laboratory. Van Peenen and Lindberg (*15D*) demonstrated the limitations of laboratory quality control with reference to the "number plus" method. Sparapani and Berry (*13D*) evaluated standard deviations from repeated determinations on frozen serum pools and found a coefficient of variation of 5 to 8% consistent with laboratory experience. Peacock *et al.* (*9D*) studied the flow of data in the clinical chemistry laboratory and made recommendations for data processing. Hill and Brown (*6D*) provided a guide to applications of statistics in chemistry and chemical engineering. Bowers and Kelley (*3D*) made precision estimates in clinical enzymology and studied variability related to enzyme source and concluded that poor precision in clinical laboratories reported in past enzyme surveys may be related to problems inherent in the test specimen as well as to human, instrumental, and procedural inadequacies. Barnett (*1D*) proposed a statistical scheme by which a test method can be compared to a reference method. Mather (*8D*) reported a panel discussion on the standardization of nomenclature in clinical chemistry.

AMINO ACIDS

Matthias and Wagner (*14E*) surveyed methods using paper, thin-layer, gas, and column chromatography for the separation and determination of amino acids in protein-containing materials. Artis (*2E*) developed test indicators for quantitative determination of L-amino acids in fluids. McEvoy-Bowe and Thevi (*13E*) determined the following amino acids in 0.1 ml of plasma: alanine, arginine, glycine, lysine, serine, taurine, threonine, and valine, using a micro-Sephadex G-25 column to separate plasma proteins from amino acids. Ambert *et al.* (*1E*) described three techniques for determination of urinary amino acids: electrophoresis at pH 5.4, two-dimensional thin-layer silica gel chromatography, and electrophoresis in one direction followed by chromatography in a second direction. A procedure for bidimensional high-voltage electrophoresis and chromatography on filter paper was adapted to 3 × 3 inch cellulose thin layers by Samuels and Ward (*15E*). Troughton, Brown, and Turner (*17E*) described a method for separation of amino acids in unmodified urine combining high voltage electrophoresis on thin-layer plates in one direction and with thin-layer chromatography in the other direction. Gerritsen, Rehberg, and Waisman (*6E*) determined free amino acids in serum and tissue extracts by ion-exchange chromatography after precipitating proteins by picric and sulfosalicylic methods, and after precipitating part of the proteins at pH 2.2 and centrifugation at 18,000 g. Jacobs (*11E*) reviewed the use of cation and anion exchange resins and the spectrophotometric determination of amino acids. Hill, Summer, and Roszel (*7E*) described a simple quantitative method for determining tryptophan derivatives in urine by thin-layer chromatographic separation, localizing their fluorescence, and eluting in alkali. Belen'kii and Orestova (*3E*) reported a method for rapid microdetermination of α-amino nitrogen in amino acids, polypeptides, and proteins by a gas chromatographic method. Izumi (*10E*) improved the Sakaguchi reaction for arginine by pretreatment of amino acid mixtures with acetic anhydride to block amino groups. Wolosowicz and Niewiarowski (*19E*) determined ϵ-aminocaproic acid in urine by a simple method based on its antifibrinolytic properties in which the prolongation of fibrinolysis time was measured in a standard clotting system. Hunter (*9E*) determined histidine in urine by an improved bromine colorimetric method in which histidine was selectively adsorbed on a weak acid cation exchanger (Amberlite IRC-50), eluted by acid, and determined colorimetrically. A simple, rapid spectrophotometric determination of methionine with pentacyano-amminoferrate was described by LaRue (*12E*). An accurate method for the determination of proline in serum was adapted by Summer and Roszel (*16E*) to a micro scale. Wren and Wiggall (*21E*) reported an improved colorimetric method for the determination of proline in the presence of other ninhydrin-positive compounds. Wong, O'Flynn, and Inouye (*20E*) described spectrophotofluorometric micromethods for the determination of phenylalanine and tyrosine in 25 μl of serum. Faulkner (*5E*) modified the phenylalanine method of McCaman and Robins, which is based on the observation that fluorescence obtained from the reaction of phenylalanine and ninhydrin is greatly enhanced in the presence of glycyl-DL-phenylalanine and is more specific at pH 5.8. Wapnir and Bessman (*18E*) reported a method for the assay of free tryptophan in urine by means of high-voltage paper electrophoresis. Methods utilizing 2-hydroxy-5-nitrobenzyl bromide for the determination of free and peptide-linked tryptophan were reported by Dasgupta, Rothstein, and Boroff (*4E*) and by Horton and Koshland (*8E*) for residues in proteins.

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

Gambino (*7F*) reviewed the principles of pH, calculated P_{CO_2}, interpolated P_{CO_2}, and direct measurement of CO_2, and presented technical details of procedures for pH and P_{CO_2} with suggested modifications. Storage of blood at 0° to 4° for measurement of pH, P_{O_2}, and P_{CO_2} was investigated by Portnoy, Thomas, and Gurdjian (*12F*), who found no significant change in pH for 3 hours or change in P_{O_2} and P_{CO_2} for 8 hours. Yee (*14F*) described the calibration of a gas chromatographic determination of CO_2 and O_2 in blood and found no significant difference when compared to the Van Slyke manometric procedure. A method for the collection and equilibration of capillary blood samples for the measurement of pH, P_{CO_2}, and standard HCO_3^- by the interpolation method was described by Jones, Kamath, and Kay (*8F*). Arndt and Doelle (*4F*) compared the Astrup microequipment and a quick recording CO_2 electrode for measurement of P_{CO_2} in human blood and found lower standard error by the electrode method. Afonso (*1F*) studied the theory of "closed space" CO_2 equilibration as a basis for micro- and ultramicromethods for the determination of the acid-base status of blood. Linden, Ledsome, and Norman (*10F*) described simple new methods for the determination of CO_2 and oxygen in blood using the electrode system. Mayers and Forster (*11F*) measured blood oxygen content by determining the physically dissolved oxygen released from hemoglobin by ferricyanide with a membrane-covered oxygen electrode. Albers and Fahri (*2F*) described a rapid accurate gas-chromatographic method for determination of the oxygen content of blood. Lankford (*9F*) described a quantitative technique for measuring fibrinolytic and fibrinogenolytic activity. Andreenko *et al.* (*3F*) made a comparative study of three different methods of heparin determination in blood and found the method of titration with thrombin in the presence of heparin-cofactor the best for laboratory practice. Sawinski, Goldberg, and Loiselle (*13F*) measured the osmolality of human saliva with a vapor pressure osmometer. Butler and Watkins (*6F*) described a micromethod for the estimation of plasma volume using the iron-dextran complex Imferon. Boutwell and Stewart (*5F*) added a simple transistor amplifier stage to an inexpensive gamma-radiation detector to measure small losses of radioactive iodenated serum albumin to increase the reliability of routine blood volume measurements.

CARBOHYDRATES

Rogers, Chambers, and Clarke (*18G*) dehydrated carbohydrates in strong HCl to produce furfurals which condense with resorcinol to yield fluorescent derivatives which are quantitatively related in nanograms to the carbohydrates. A colorimetric method for the determination of neutral monosaccharides utilizing a reagent composed of aniline —CH_3COOH—H_3PO_3 was reported by Walborg and Christensson (*21G*). Reisz (*17G*) determined aldohexoses by reacting with *p*-aminosalicylic acid to produce a colored complex. Yaphe and Arsenault (*24G*) described an improved resorcinol reagent for the determination of fructose and 3,6-anhydrogalactose in polysaccharides. Zender and Falbriard (*25G*) proposed a method for the determination of fructose inulin in plasma and urine with a reagent composed of thiobarbituric acid in acidic medium. An enzymic method using galactose oxidase for the determination of galactose in blood by incubation of sample with enyzme in the presence of peroxidase and benzidine was described by Roth, Segal, and Bertoli (*19G*). Comstock and Comstock (*7G*) stabilized for 12 months 20-μl blood samples by application to filter paper and drying. Meites (*13G*) suggested the use of aqueous dilution of whole blood for 2-hour stability of glucose. Ware and Marbach (*22G*) measured the H_2O_2 formed in the serum glucose oxidase ultramicromethod by reacting with iodide in the presence of a catalyst to yield molecular iodine for photometric measurement. Keller (*10G*) utilized the enzymes, hexokinase and glucose-6-phosphate dehydrogenase, in a new method to determine urinary glucose by mercury NADPH absorbance at 340 mμ. Leske and Mayersbach (*11G*) removed reducing substances in urine with $HClO_4$ for employment of the glucose-oxidase-peroxidase method. Bergmeyer and Moellering (*5G*) reported the enzymatic determination of glucose with acyl phosphate-D-glucose-6-phosphotransferase and found the method more specific than the hexokinase method. Alexander and Garbutt (*3G*) used sorbitol as a satisfactory internal standard for the determination of D-glucose by gas-liquid chromatography. Kadish and Hall (*9G*) reported a new method for the continuous monitoring of blood glucose by measurement of dissolved oxygen with a polarographic oxygen analyzer in the glucose oxidase procedure. A simple rapid colorimetric blood sugar determination was reported by Lorentz (*12G*) based on the formation of red formazan from triphenyltetrazolium chloride. Methods for the determination of glucose in biological fluids employing *o*-toluidine in acid medium were reported by Torlotin (*20G*), Raisis and Ustinova (*15G*), Mueller (*14G*), Ahlert, Hofer, and Bestvater (*2G*), Braun and Hofmann (*6G*), and Hofer and Ahlert (*8G*). Wells, Sweeley, and Bentley (*23G*) reviewed the gas chromatographic measurement of carbohydrates. Baehner (*4G*) determined the free hexosamines in serum and urine with Ehrlich's reagent. Adachi (*1G*) determined lactulose by first separating by thin-layer chromatography and applying the cysteine-carbazole-sulfuric acid reaction. Reiner and Cheung (*16G*) adapted the pentose method for animal tissue of Roe and Rice to the determination of pentose in blood and urine.

CATIONS AND ANIONS

The Ad Hoc Committee on Acid Base Methodology chairmanned by Gambino *et al.* (*20H*) with representatives from Denmark, Great Britain, and the United States defined acceptable methods of blood sampling and handling, proper buffers, pH, and normal values. Holasek (*25H*) reviewed the chelatometric methods for the determination of K, Na, Ca, Mg, and $H_2PO_4^-$.. Sakanoue (*39H*) described ultramicroanalytical techniques for the determination of Ca, Mg, and P in small volumes of extracellular fluid. Bethard, Olehy, and Schmitt (*10H*) used neutron activation analysis to determine Mg, Mn, Cu, and Zn in plasma and erythrocytes of human blood. Alexander (*2H*) developed an x-ray fluorescence method for the determination of Ca, K, Cl, S, and P in dried biological tissues and fluids. Valberg *et al.* (*44H*) determined Na, K, Ca, Mn, Cu, and Zn in normal human erythrocytes by spectrochemical analysis. Sunderman and Carroll (*42H*) described a procedure for determination of Ca and Mg by atomic absorption spectrometry, in which interferences of sulfate and phosphate were avoided by additions of strontium chloride. Webster and Kern (*46H*) determined calcium in urine by the colorimetric chloranilate method by elimination of magnesium prior to calcium determination. The use of hydroxy naphthol blue as an indicator in the microtitration of calcium was suggested by van Haga (*45H*). Butcher, Eastis, and Clark (*13H*) found the Nuclear Fast Red dye method applicable for on-board determination of calcium in serum, parotid fluid, and urine in the weightless state. Catledge and Biggs (*14H*) reported that hydroxy naphthol blue as an indicator in the chelometric measurement of calcium with EDTA provides a distinct end point titration. Connerty and Briggs (*16H*) described a spectrophotometric method for determination of serum calcium, using the metal complexing dye, *o*-cresolphthalein complexone. Serum calcium was determined by Connerty and Briggs (*17H*) by means of sodium alizarinsulfonate and by Howell, Pita, and Marquez (*26H*) by an ultramicromethod employing chlorophosphonazo III as a colorimetric reagent. Lamkin and Williams (*29H*) determined the total magnesium and calcium in serum by chelating with buffered Arsenazo, and sequestering calcium in one half of the specimen with EGTA. Bellinger and Campbell (*7H*) and Mager and Farese (*32H*) determined serum calcium by glyoxal bis(2-hydroxyanil) chelation. A new indicator, di-(2-hydroxyphenylimino)-ethane, for the EDTA titration of serum calcium was reported by Skerry (*41H*) and a new reagent, 2-chloro-5-cyano-3,6-dihydroxybenzoquinone, for the spectrophotometric determination of calcium was described by Rehwoldt, Chasen, and Li (*38H*). Koval (*28H*) determined cesium in urine by flame photometry using an 852-mμ filter and correcting for the presence of known amounts of potassium.

Wendland and Winstead (*47H*) reviewed the problems of blood chloride determination. Gilbert (*21H*) determined chlorine by indium chloride band emission in a van der Smissen burner in which chlorine was converted to indium chloride vapor with a sharp band spectrum at 360 mμ. Hamilton (*23H*) developed a single reagent for direct photometric serum chloride determination, which contained ferric ions, mercuric thiocyanate, mercuric ions, and mercuric chloride. Chudnovskaya and Bykova (*15H*) titrated serum chloride with $AgNO_3$ using dichlorofluorescein indicator. Scott, Jones, and Simcock (*40H*) studied the efficiency of iodide trapping by anion exchange resins. Harden, Mason, and Buchanan (*24H*) compared four methods for the estimation of plasma inorganic iodine. Fairman (*19H*) determined inorganically bound iodine-131 in urine by ion exchange separation on a AgCl column, elution as HIO_3, oxidation and reduction, and precipitation as AgI and estimation by beta and gamma count. Boyle *et al.* (*12H*) estimated plasma inorganic iodine by isotope dilution, and by comparison of serum and urine iodine and creatinine ratios, and found good correlation. Benotti *et al.* (*8H*) determined total iodine in urine, stool, diets, and tissue by a procedure requiring a minimum number of reagents. Mitchell (*33H*) determined iodine in feces by combustion in a bomb colorimeter.

Alcock and MacIntyre (*1H*) reviewed the principles of the preparation of samples, and procedures for atomic absorption, flame emission, gravimetric, colorimetric, and volumetric methods. MacDonald and Watson (*31H*) determined magnesium in fluids by dilution with a solution of strontium chloride and application of atomic absorption spectrophotometry. Pruden, Meier, and Plaut (*37H*) compared the atomic absorption flame emission, Titan yellow, Magon photometry, 8-hydroxyquinoline and 8-hydroxyquinoline sulfonate fluorometry methods for magnesium. Natelson *et al.* (*34H*) made microestimations of Na, Mg, and Ba with the x-ray spectrometer on 50-μl serum samples. Berlin (*9H*) employed Eriochrome Black T as an indicator for free Mg ions. Basinski (*5H*) made a systematic study of the Titan yellow magnesium method to establish its optimum conditions. Fluorometric magnesium methods were employed by Thiers (*43H*) using 8-hydroxy-5-quinolinesulfonic acid and by Batsakis *et al.* (*6H*) who used 8-hydroxyquinoline in ethanolic solutions. Cotzias, Miller, and Edwards (*18H*) determined manganese in blood and serum by neutron activation analysis. A new micromethod for the determination of inorganic phosphate was worked out by Itaya and Ui (*27H*) on the principle that Malachite green at a lower pH forms a complex with phosphomolybdate. A method for determination of phosphorus, which was especially advantageous for measurement of enzyme activities, was reported by Baginski, Foa and Zak (*4H*).

Portnoy and Gurdjian (*35H*) modified a routine pH meter with a glass electrode to measure Na and K. Annino (*3H*) determined sodium in urine and Boenzi and Materese (*11H*) determined sodium in serum, CSF, and urine with glass electrodes. Portnoy, Gurdjian, and Henry (*36H*) found favorable comparison of the glass electrode and flame photometry methods for Na and K determination in serum. Lund and Mathies (*30H*) devised an x-ray spectrographic procedure for micromeasurement of potassium in tissue and serum. Guyon and Lorah (*22H*) reported a sulfate micromethod based on the release of fluorescent salicylfluorone from its nonfluorescent thorium complex in the presence of sulfate.

LIPIDS

Thin-layer chromatography of lipids was reviewed by Padley (*17J*) and Nakazawa (*15J*). Levin and Head (*14J*) described a method for the quantitative separation of tissue neutral lipid fractions. Gloster and Fletcher (*5J*) determined the normal values for the major neutral and phospholipid classes of serum using a simple method of thin-layer chromatography. Neudoerffer and Lea (*16J*) described a method for retarding autoxidative degradation of lipids during chromatographic analysis. Wagener (*24J*) described techniques for detection and documentation of lipids after thin-layer chromatography.

Keler-Bacoka and Dujmovic (*12J*) performed electrophoretic analyses of human serum lipids in three buffer systems—veronal-acetate, phosphate, and borate-acetate—at pH 8.6. Gruene (*6J*) cited the advantages of membrane films as supporting material in the electrophoresis of human serum lipids. Thompson and Smith (*23J*) applied on-column acid-base reactions for the gas chromatic separation of free fatty acids and amines.

Pokrovskii (*18J*) reviewed modern methods for determinations of neutral fat in blood and tissues. Kaplan and Lee (*11J*) determined triglyceride in 0.1 ml of serum by extraction, saponification, oxidation to HCHO, and colorimetric determination with chromotropic acid. Ignatowska (*9J*) used a similar procedure. Eggstein and Kruetz (*4J*) and Spinella and Mager (*21J*) used modified enzymatic procedures for the determination of blood and plasma neutral fats. Laurell and Tibbling (*13J*) developed a simple and rapid method for plasma or whole blood glycerol by a fluorometric adaptation of enzymic conversion of glycerol to form DPNH. The analysis of fatty acids and derivatives by gas chromatography was reviewed by Supina (*22J*). Plasma triglycerides were isolated by Bowers *et al.* (*2J*) by using chromatography on silica gel–impregnated glass paper, and internal standard and gas-liquid chromatography. Hagenfeldt (*7J*) described a method in which heptadecanoic acid was used as an internal standard for the direct gas chromatographic determination of individual free fatty acids in plasma. Sheath (*20J*) estimated plasma nonesterified fatty acids and triglyceride fatty acids by thin-layer chromatography and colorimetry. Rovescalli (*19J*) adapted Duncomb's method to the Spinco ultramicrosystem to permit determination of free fatty acids on 20 μl of plasma. Hoeflmayr and Fried (*8J*), using 0.1 ml of serum, determined the fatty acid esters in aqueous instead of alcoholic medium. Itaya and Ui

(*10J*) extracted free fatty acids from biological fluids by using phosphate or Tris maleate buffer at pH 6.2 and $CHCl_3$ as extraction agent and determined them colorimetrically. Wood and Sodhi (*26J*) determined plasma free fatty acids by the isolation of their 14 CH_3 esters by silicic acid column chromatography and measurement of their radioactivity by scintillation counting. Anstall and Trujillo (*1J*) adapted a microcolorimetric procedure for measurement of long-chain fatty acids in pure solution by elimination of phospholipid interference to determine free fatty acids in serum. De Koning (*3J*) developed a new procedure for estimating sphingomyelin by determining sphingomyelin-choline in phospholipid mixtures. Wolochow *et al.* (*25J*) reported a rapid method for quantitative determination of fecal fat based on the principle of electrical capacitance.

ENZYMES

Guilbault (*24K*) reviewed the developments and use of enzymes in analytical chemistry. Searcy, Hayashi, and Berk (*46K*, *47K*) described a saccharogenic serum amylase micromethod in which glucose was measured with alkaline dinitrosalicylic acid. Wilding (*52K*) described a saccharogenic method for the assay of amylase in fractions of serum obtained by electrophoresis on paper and polyacrilamide gel. Serums buffered with citrate at pH 6.2 were found by Doe, Mellinger, and Seal (*21K*) to preserve acid phosphatase at 25°C for 7 days. Seal, Mellinger, and Doe (*45K*) found that 1-naphthyl phosphate substrate was as specific as tartrate-inhibited phenyl phosphate for the determination of prostatic acid phosphatase. Rudolph (*40K*) and Babson and Phillips (*7K*) found that diazotized 5-nitro-*o*-anisidine was the most sensitive coupling agent for 1-naphthol formed in the serum acid phosphatase determination. Coleman (*20K*) found the phenolphthalein monophosphate assay method the method of choice for acid phosphatase, as it was sensitive and free from bilirubin interference, and produced a stable color. Berger (*10K*) and Bowers and McComb (*14K*) modified the *p*-nitrophenyl phosphate substrate method of Bessey for acid and alkaline phosphatase determination. Babson *et al.* (*5K*) described a rapid and precise procedure for alkaline phosphatase which employed phenolphthalein monophosphate substrate. Shepherd (*48K*) stabilized the reagents for a microenzymatic method for the determination of ceruloplasmin in serum. McComb, LaMotta, and Wetstone (*32K*) developed a method for demonstrating atypical serum cholinesterase by using *o*-nitrophenyl as substrate. Johnson and Whitehead (*29K*) determined serum cholinesterase activity by incubating acetylcholine bromide with serum within the capillary of the Astrup electrode for measurement of liberated acetic acid. Guilbault and Kramer (*25K*) described two new fluorogenic substrates for the determination of cholinesterase: resorufin butyrate and indoxyl acetate. A simple cholinesterase method in which cholinesterase acts on acetylthiocholine to release thiocholine which reacts with 5,5-dithiobis-(2-nitrobenzoic acid) to produce a yellow color was described by Garry and Routh (*23K*). Nuttall and Wedin (*35K*) modified the Tanzer and Gilvary creatine kinase activity method to obtain a simple rapid colorimetric method. Fluorometric creatine kinase activity methods were described by Sax and Moore (*44K*), who measured creatine liberated by formation of a fluophor with ninhydrin in alkaline solution and by Sax and Moore (*43K*) who determined the creatine liberated in the reaction ADP + creatine phosphate $\rightleftharpoons$ ATP + creatine. Sardesai and Provido (*42K*) described a simple fluorometric method for plasma trypsin in which plasma was incubated with *p*-tosyl arginine methyl ester and the methanol formed by hydrolysis was measured fluorometrically. Rutkowski (*41K*) measured plasma trypsin-like esterase activity with benzoyl arginine ethyl ester. Behrman (*9K*) measured serum formylhydrate dehydrogenase by a method which couples the oxidation of aldehyde to carboxy acid by enzyme action and reduction of 2,6-dichlorobenzene. Fluorometric methods for the estimation of *N*-acetyl-β-glucosaminidase and β-galactosidase in plasma were described by Woollen and Walker (*54K*) using 4-methylumbelliferyl glycosides as substrates. Caygill and Jevons (*17K*) measured β-glucosaminidase activity in body fluids using the synthetic substrate *p*-nitrophenyl 2-acetylamino - 2 - deoxy - β - D - glucopyranoside. A fluorometric method for estimation of β-glucuronidase in plasma, using methyl umbelliferyl β-glucuronide as substrate, was described by Woollen and Walker (*55K*) and Woollen and Turner (*53K*). Rognoni and Ronchini (*38K*) developed an enzymatic method for the determination of glucose-1-phosphate based on its conversion to glucose-6-phosphate by phosphoglucomutase. Hue and Free (*27K*) determined guanase in serum by measuring the amount of guanine converted to xanthine. Caraway (*16K*) determined serum guanase activity colorimetrically by measuring the ammonia formed by deamination of guanine with the phenate-hypochlorite reaction. Bonner and Cantey (*13K*) determined serum hyaluronidase activity colorimetrically by a method based on the quantitation of *N*-acetylglucosamine end groups liberated from hyaluronic acid.

Simple rapid colorimetric methods for assay of serum lactic acid employing tetrazolium salts were described by Babson and Phillips (*6K*), Chapman and Boutwell (*19K*), and Briere, Preston, and Batsakis (*15K*). Guilbault and Kramer (*26K*) described a fluorometric procedure for determination of lactic acid dehydrogenase by conversion of resazurin to fluorescent resorufin. Techniques for the separation of lactic dehydrogenase isoenzymes on cellulose acetate were described by Preston, Briere, and Batsakis (*36K*), Lance (*30K*), and Barnett (*8K*), and on agar gel by Starkweather *et al.* (*49K*) and Wieme (*51K*). Martinek, Berger, and Broida (*33K*) modified the serum and urine leucine aminopeptidase method in which L-leucylnaphthylamide substrate was used. Remp (*37K*) reported a rapid method for determination of serum lipase employing a substrate containing purified olive oil, sodium deoxycholate, and Tris buffer. Romel and LaMancusa (*39K*) described a procedure for the separation of glutamic oxalacetic transaminase by electrophoresis on cellulose polyacetate and staining with a diazonium salt. Babson (*4K*) described a serum transaminase method in which the oxalacetic acid formed as a product of the reaction was measured by coupling with 6-benzamido-4-methoxy-*m*-toluidine diazonium chloride. Amador, Franey, and Massod (*2K*) surveyed the diagnostic accuracy of the spectrophotometric and dinitrophenylhydrazine transaminase methods. A method for ornithine carbamyltransferase was described by Ceriotti and Gazzaniga (*18K*) based on citrulline determination by the diacetylmonoxime-phenazone reaction. Methods for the estimation of plasma plasminogen were presented by Mitchell and Cope (*34K*) who used casein as substrate and euglobulin as plasminogen source, and by Hummel *et al.* (*28K*) who used azocasein as substrate. Al-Khalidi *et al.* (*1K*) determined xanthine oxidase activity in blood serum and tissue by a sensitive radioactive method employing $8-^{14}C$ xanthine as substrate. Dubbs (*22K*) found selective activation of a serum cholinesterase isoenzyme by ultrasonic treatment. Antonis, Clark, and Pilkington (*3K*) developed semiautomated enzymatic methods for fluorometric estimation of plasma levels of various intermediary metabolites. Blakley (*12K*) estimated the enzymic formation of purine and pyrimidine deoxyribonucleotides by the use of the diphenylamine reagent. Le Pecq and Paoletti (*31K*) utilized the increased fluorescence of ethidium bromide when it binds to double-stranded regions of

DNA or RNA for their determination. Bergmeyer and Bernt (*11K*) described enzymic methods for the quantitative determination of acetoacetate and D(−)-3-hydroxybutyrate in blood. Walsh (*50K*) determined plasma pyridoxal phosphate with wheat germ glutamic-aspartic apotransaminase.

FUNCTION TESTS

Kidney. A number of methods and modifications for the determination of serum creatinine were compared for their reproducibility, accuracy, and sensitivity by Haury (*9L*). Nakadate, Maki, and Kimura (*17L*) determined urinary creatinine by a method based on the reaction of creatinine with 2,2′,4,4′-tetranitrobiphenyl in the presence of KOH. Slot (*24L*) described a new more specific method for creatinine based on the fact that the color produced by creatinine is less resistant to acidification than the color produced by other Jaffe chromogens. Pataki (*18L*) determined urinary creatinine by thin-layer chromatography. Kibrick and Milhorat (*12L*) determined serum creatine with creatine phosphokinase based on the method of Tanzer and Gilvarg. Cutler and Glatte (*2L*) described a radioactive isotope technique for the simultaneous measurements of glomerular filtration rate and effective renal plasma flow.

Liver. Gambino (*6L*) described a total bilirubin reference method in which serum was added to a sodium acetate–caffeine sodium benzoate reagent to buffer and accelerate the coupling of bilirubin with diazo reagent. MacDonald (*14L*) made a micromodification of the Malloy and Evelyn total and direct bilirubin method. Colorimetric micromethods for serum bilirubin were described by Ferro and Ham (*4L*) and Rutkowski and deBaare (*21L*). Raia (*19L*) used a modified diazo reagent containing 2,4-dichloroaniline for histochemical demonstration of conjugated and unconjugated bilirubin. Van den Bossche (*25L*) used acetamide as a water-soluble accelerator of the coupling reaction of free bilirubin with diazotized sulfanilic acid. Heirwegh, Jansen, and Van Roy (*10L*) determined urinary bilirubin as the zinc(II)-azobilirubin complex. Girard, Paolaggi, and Frappier (*7L*) employed methyl isobutyl ketone as an extraction solvent in the determination of bilirubin. Shvartsman (*23L*) described a paper chromatographic method for determination of the fractions of serum bilirubin. Blondheim and Kaufmann (*1L*) reported that exposure of unconjugated bilirubin in albumin solutions to light resulted in an increase in the direct diazo reaction. Meites and Traubert (*15L*) suggested the use of a stable stock solution of bilirubin in chloroform to standardize the determination of bilirubin in serum as azobilirubin. Kuenzle *et al.* (*13L*) presented a method for the chromatographic separation and determination of bile pigments using reversed-phase partition chromatography of serum and bile on silicone-treated kieselguhr columns.

Improved extraction methods for the determination of urinary porphyrins were described by Fernandez, Henry, and Goldenberg (*3L*) and Schlenker, Taylor, and Kitchell (*22L*). Herbert (*11L*) presented an analytical scheme for the fractionation and quantitative determination of fecal porphyrins. Methods for the determination of urinary porphobilinogen were described by Moore and Labbe (*16L*) and Gutniak and Krawczyk (*8L*). Ferro and Ham (*5L*) investigated the use of a stable latex particle suspension as a thymol turbidity standard. Rosenberg, Eimann, and O'Leary (*20L*) modified the Hanger cephalin cholesterol flocculation test by measuring turbidity after 4-hour cold incubation, centrifugation, and turbidity measurement of resuspended sedimented particles. Winkler *et al.* (*26L*) determined hepatic blood flow with five test substances using simultaneous sampling of hepatic venous blood from 2 parts of the liver.

HEMOGLOBIN

Skendzel and Copeland (*14M*) reported the results of surveys in 1962–1964 of hemoglobin measurements in hospital laboratories, which showed that the best performance was among laboratories which used a commercially purchased cyanmethemoglobin standard. The National Academy of Sciences–National Research Council [Cannan (*4M*)] specifically recommended the cyanmethemoglobin method as a common method and a cyanmethemoglobin solution as a standard. Lewis (*9M*) presented a quantitative method for the estimation of hemoglobin in serum and urine using *o*-tolidine as the H donor in a peroxidase system. A method for the determination of total blood hemoglobin by conversion to azido-methemoglobin was described by Vanzetti (*18M*). Miles Laboratories (*10M*) prepared a reagent to determine traces of blood, which contained cumene, gelatin, polysaccharide dialdehyde, a buffer (pH 4 to 7), and *o*-toluidine as colorimetric indicator and quinine as an activator. Andersen and Shoemaker (*1M*) compared a colorimetric method for analysis of the red cell content of tissues with an independent radio-assay method. An equation was developed by Bjure and Nilsson (*2M*) for the determination of hemoglobin O saturation in the presence of carboxyhemoglobin from the ratio between the absorbancies of a hemolyzed blood sample at two different wavelengths. A new, simple, rapid, and accurate method for determining hemoglobin-binding capacity and haptoglobin type simultaneously was described by Ferris *et al.* (*8M*) by acrylamide gel electrophoresis. Valeri *et al.* (*16M*) determined hemoglobin-binding capacity by using cellulose acetate membrane electrophoresis. Farago (*7M*) detected minute traces of blood by thin-layer chromatography. Petz and Economidou (*12M*) presented a simplified technique of starch block electrophoresis for the quantitative determination of hemoglobin. Blackwell and Huang (*3M*) used hemolyzates of unwashed red cells for screening populations for abnormal hemoglobins by starch-gel electrophoresis. Colfs and Verheyden (*5M*) proposed a support medium of cellulose acetate for rapid determination of serum haptoglobin by electrophoresis. Morrison and Horie (*11M*) determined the heme A concentration in cytochrome preparations by the hemochromogen method. Van Kampen and Zijlstra (*17M*) reviewed hemoglobin and its derivatives, the absorption spectra and extinction coefficients of Hb, HbO$_2$, HbCO, Hi, HiCN, and the spectrophotometric determination of the O$_2$ saturation, HbCO, Hi, and sulfhemoglobin. Subramanian, Padmanaban, and Sarma (*15M*) devised a simple method for the estimation of siderochromes based on their reaction with Folin-Ciocalteu reagent to give a blue complex under alkaline conditions. Salvati, Tentori, and Vivaldi (*13M*) outlined a technique for the determination of the extinction coefficient of human hemoglobin-cyanide. Dubowski (*6M*) reviewed the clinical methods for the determination of carboxyhemoglobin, methemoglobin, sulfhemoglobin, and methemalbumin on fresh blood specimens.

METALS

Determination of metals in biological material was reported by Parker and Humoller (*19N*), who measured Zn and Cu, and by Rousselet (*22N*) who reported techniques for estimation of Mg, Ca, Fe, Cu, and Zn. A chemical kinetic method using coordination chain reactions was proposed by Margerum and Steinhaus (*15N*) for ultra trace determination of metals. Trace elements, Ni, Zr, Ag, Cu, Sn, Ti, Al, Pb, Mn, and Cr, were determined by Lifshits (*14N*) in erythrocytes and human plasma by spectrographic determination. Hashmi *et al.* (*9N*) reported that formic acid hydrazide reacts with Au, Cu, and Co in acid medium to give violet, pink, and blue colors which may be used for the determination of these metals. Improved sensitivities for the measurement of Mg, Ag, Zn, and Ca were reported by Ellis and Demers (*6N*) using the hydrogen–entrained-air flame with a more intense continuum source in atomic fluorescence flame spectrometry. Samsahl, Brune, and Wester (*23N*) determined 30 trace elements in malignant and nonmalignant human tissues by neutron-activation analysis. Smales and Mapper (*25N*) presented procedures for the determination in biological materials of the trace elements: Ge, As, Sb, Cu, Cr, Mo, Ag, In, Zn, and Pd by neutron activation. Haven, Haven, and Dunn (*10N*) determined Ca, Cu, Mn, and Mg in serum, simultaneously by activation analysis. Elhanan and Cooke (*5N*) studied the factors affecting line intensities in the flame spectrometry of metals in organic solvents.

Sass-Kortsak (*24N*) reviewed the methods of determination of copper, ceruloplasmin, and bound copper in biological materials, and the use of radioactive copper in these methods. Matsuba (*16N*) determined copper in serum and urine by formation of a yellow-green complex with *N,N,N′,N′*-tetraethylthiuram disulfide. Herrmann and Lang (*13N*) measured copper by extraction of the diethyldithiocarbamate complex with $CHCl_3$, applying this extract directly to the absorption flame photometer. Butler (*2N*) described an improved absorptiometric method for the determination of traces of copper in biological materials with dithizone. Bizollon (*1N*) determined serum copper by radioisotope dilution and incomplete extraction. Ness and Dickerson (*18N*) determined serum iron without deproteinization by use of nitroso R salt. Rogerson and Helfer (*21N*) determined serum iron by atomic spectrophotometry and eliminated the effect of interfering substances by using standards containing equal concentrations of protein. Stamm, Staudinger, and Weis (*27N*) and Deggau *et al.* (*3N*) suggested the use of a new complexing agent, 2,6-di-2-pyridyl-4-(*p*-methoxyphenyl)pyridine in a micromethod for serum iron determination. Yamamura and Sikes (*31N*) described the use of citrate-EDTA masking for the selective determination of iron with 1,10-phenanthroline. Goodwin, Murphy, and Guillemette (*8N*) measured serum iron directly by incubation in acetate buffer with reducing agent and sulfonated bathophenanthroline. Williams and Conrad (*30N*) described a single-tube method for measuring both serum iron concentrate and iron-binding capacity using 2,4,6-tripyridyl-*s*-triazine chelating agent. Zettner, Sylvia, and Capacho-Delgado (*32N*) employed absorption spectroscopy to determine serum iron and iron-binding capacity. Stojceski, Malpas, and Witts (*28N*) determined iron-binding capacity by a colorimetric method, by a radioisotopic technique, and by a method employing immunodiffusion. Dubinskaya and Pelekis (*4N*) determined iron in blood by neutron activation using 50 mg of blood.

Heidrich (*11N*) determined serum magnesium with Titanium yellow and stabilized the dye-$Mg(OH)_2$-lake with poly(vinyl alcohol) for photometric measurement. Moav (*17N*) determined manganese in urine by neutron activation analysis, by irradiating the sample 5 minutes at a thermal flux of 7×10^{12} n/sq cm/sec, precipitating MnO_2 with added carrier by boiling with HNO_3 and $KClO_3$, and counting the precipitate with a scintillation spectrometer. Galli (*7N*) estimated molybdenum in urine colorimetrically by the dithiol reaction. Atomic absorption spectrophotometry was used by Sunderman (*29N*) and Sprague and Slavin (*26N*) to determine nickel in urine, and by Prasad, Oberleas, and Halsted (*20N*) to determine zinc in biological fluids. Helwig *et al.* (*12N*) described a uniform procedure for determination of zinc in multiple samples of serum and urine.

NITROGEN COMPOUNDS

Enzymic methods for the determination of ammonia in biological fluids, based on the reaction: ketoglutaric acid + H^+ + NH_3 $\rightleftharpoons$ L-glutamic acid + NAD + H_2O, using glutamic dehydrogenase were described by Mondzac, Ehrlich, and Seegmiller (*14P*), Reichelt, Kvamme, and Tveit (*19P*), and Schmidt and Schwarz (*22P*). Kaplan (*10P*) made a critical evaluation of the Berthelot reaction (indophenol blue) for the determination of serum and urinary ammonia. Reardon, Foreman, and Searcy (*18P*) suggested the use of sodium salicylate and dichloroisocyanurate instead of phenol and sodium hypochlorite in the Berthelot reaction. Horn and Squire (*9P*) reported increased sensitivity and accuracy of the indophenol blue reaction if sodium hypochlorite of pH 12.5 was added to an ammonia solution before the addition of sodium phenate. Chung (*4P*) reported the stabilization of blood ammonia if buffered to pH 10.1 with borate. Foreman (*6P*) determined plasma NH_3 by a rapid ion-exchange technique. Buys Ballot and Steendijk (*1P*) determined blood ammonia after microdiffusion with the aid of the rubazonic acid reaction.

Kibrick and Milhorat (*11P*) described a method for the determination of serum creatine with creatine phosphokinase. Wallace, Biggs, and Dahl (*24P*) determined diphenylhydantoin by ultraviolet spectrophotometry in blood in the presence of other drugs without separation by extraction. Preuss, Bise, and Schreiner (*17P*) described a method for determination of glutamine in plasma and urine in which ammonia produced after the addition of glutaminase is a measure of glutamine present. A colorimetric micromethod for determination of tissue glutathione was described by Owens and Belcher (*16P*), in which 5,5′-dithiobis (2-nitrobenzoic acid), EDTA, and glutathione reductase were used. Lyle (*12P*) developed a specific, sensitive, and simple method for determination of glutathione by reaction of reduced glutathione with *o*-phthalaldehyde for fluorometric measurement. Reynolds and Thomas (*21P*) described a colorimetric method for the determination of hydrazine and monomethyl hydrazine in blood. Mueting and Burgard (*15P*) determined indican in serum and urine by hydrolysis of indican to indoxyl, which reacted with thymol and $FeCl_3$ in acid to give a red-violet color. Bryan (*2P*) reported a new, rapid, and precise colorimetric method for urinary indoxyl sulfate based on column chromatography on a cation-exchange resin followed by diazotization and coupling. Zemlyans'ka (*25P*) presented a colorimetric method for determination of total nicotinic acid in blood based on the color reaction of nicotinic acid with cyanogen bromide in the absence of aromatic amines. Thompson (*23P*) described a method for determination of nicotinamide-adenine dinucleotide phosphate in blood as the oxidized (NADP) and reduced ($NADPH_2$) forms.

Thin-layer chromatography of nucleic acid derivatives was reviewed by Honjo (*8P*). Hilgertova (*7P*) presented a fluorometric method for the simultaneous determination of oxidized and reduced forms of pyridine nucleotides in liver tissue. Reiss, Muraine, and Dainciart (*20P*) made a comparative study of different ultramicrotechniques for quantitation of urea and concluded that urease methods gave best results. A method for determining urea in blood and urine with diacetyl monoxime-glucuronolactone reagent was presented by Momose, Ohkura, and Tomita (*13P*). Filho (*5P*) reported that urea and diacetyl monoxime react in acid medium with intensified color formation when Fe^{3+} traces and phenol are present. Christian (*3P*) determined serum nonprotein nitrogen by digesting protein-free filtrate with H_2SO_4, $HgSO_4$, and K_2SO_4 and titrating the $(NH_4)SO_4$ formed with electrogenerated hypobromite.

HORMONES

Kilshaw (*19Q*) surveyed endocrinology methods. Thin-layer chromatography of steroid hormones and their clinical application were reviewed by Ibayashi (*15Q*). Benraad, Verwilghen, and Kloppenborg (*3Q*) investigated isotope fractionation as a source of error in the determination of aldosterone by isotope dilution. Benraad and Kloppenborg (*2Q*) described a double isotope dilution derivative assay for aldosterone determination in urinary extracts. Fluorometric plasma and urine cortocoid methods, procedural variations sensitivity, specificity, and precision were reviewed by Silber (*36Q*). A simple fluorometric method for the determination of unconjugated cortisol in human urine was described by Pal and Smith (*29Q*). Methods for the extraction and measurement of pituitary gonadotropin were described by Watson and Knouse (*41Q*) and Keller and Rosemberg (*18Q*). Snook and Cole (*38Q*) developed a bioassay for antiserum against human chorionic gonadotropin (HCG) based upon the inhibition of the uterine response to HCG in the immature female rat. A simple technique for the radioimmunoassay of human growth hormone (HGH) using HGH^{131}I, rabbit anti-HGH serum, and an anion exchange resin to separate the bound and free HGH^{131}I was developed by Lazarus and Young (*20Q*). Coward and Smith (*6Q*) reported a new rapid screening test for phaeochromocytoma. Fleming and Clark (*11Q*) developed a method for a single solvent extraction and simultaneous determination of serotonin, dopamine, and norepinephrine in less than 1 gram of brain tissue. Mattok, Wilson, and Heacock (*23Q*) described a method for determination of adrenaline, noradrenaline, dopamine, metanephrine, and normetanephrine in urine, in which the amines present in urine were adsorbed on Amberlite IRC-50. Methods for the determination of catecholamines, based on the trihydroxyindole method, were described by Kahane and Vestergaard (*17Q*) for urine and by Fiorica (*10Q*) for plasma. Ruthven and Sandler (*33Q*) described a procedure for measuring the urinary concentration of total metadrenalines and 4-hydroxy-3-methoxyphenyl glycol. Methods for the determination of urinary 3,4-hydroxymandelic acid were reported by Annino, Lipson, and Williams (*1Q*) and Tautz, Voltmer, and Schmid (*39Q*) who used thin-layer chromatography, by Williams and Greer (*42Q*) who used gas chromatography, by Mills (*25Q*) and Sato (*34Q*) who used anion exchange-isotope dilution and adsorption on Dowex AG-1 chlorideform, by Eichhorn and Ruthenberg (*8Q*), and by Men'shikov and Bol'shakova (*24Q*) who used electrophoresis. Two-dimensional chromatography was found more reliable and accurate for determination of urinary vanilmandelic acid than colorimetric or electrophoretic methods by Wocial (*43Q*). Crymble (*7Q*) compared and evaluated colorimetric procedures for 3-methoxy-4-hydroxymandelic acid. Mustala, Tuomisto, and Airaksinen (*27Q*) modified the Udenfriend method for greater specificity for the determination of 5-hydroxyindoleacetic acid. Berry and Krupanidhi (*4Q*) described a simple and sensitive method for estimation of 5-hydroxyindoleacetic acid in urine by paper chromatography.

Rudy (*31Q*) presented a quality control program for the determination of protein-bound iodine, in which the statistical analysis reflects both the precision and accuracy of the procedure. Hoeman *et al.* (*14Q*) compared three methods for the determination of the ratio of labeled protein-bound iodine PB^{131}I over the total radioactive iodine in blood plasma. Methods employing ion-exchange resins for the determination of serum thyroxine were reported by Ruiz and Somellera (*32Q*) who used Amberlite IRA-400, by Nakajima *et al.* (*28Q*) who employed a ^{131}I-T$_3$ resin, and by Murphy (*26Q*) who used an anion-exchange resin and radioactively labeled thyroxine. Ingbar *et al.* (*16Q*) described a new method for measuring the free thyroid hormone in human serum. Liewendahl and Lamberg (*21Q*) determined free thyroxine in serum by dialysis and sephadex G-25 filtration. A polyacrylamide electrophoretic study of thyroxine binding to human serum by Marshall and Levy (*22Q*) indicated the separation of four distinct zones. Faircloth, Williams, and Florsheim (*9Q*) developed a simple bidimensional thin-layer chromatographic procedure for the determination of iodinated amino acids in thyroid hydrolyzates. Scriba (*35Q*) determined the binding property of triiodothyronine of serum protein by dextran gel filtration, and Toro-Goyco and Matos (*40Q*) separated the abnormal iodoproteins in serum by gel filtration. Cavalieri and Searle (*5Q*) utilized equilibrium dialysis of a mixture of plasma or serum and radiometric triiodothyronine (T$_3$) to determine the fraction of T$_3$ which is diffusible. Gottlieb and Herbert (*12Q*) employed dextran-coated charcoal impregnated with I^{131}-triiodothyronine as an alternative to the coated charcoal T$_3$ test. Herbert *et al.* (*13Q*) also used hemoglobin-saturated charcoal to separate free T$_3$ from T$_3$ bound to serum protein. Richards and Mason (*30Q*) described the gas chromatographic separation of organic compounds T$_3$ and T$_4$ of serum by forming *N,O*-bistrifuroacetyl methyl esters and detection by flame ionization. Smith, Mozley, and Wagner (*37Q*) determined protein-bound iodine in human plasma by thermal neutron activation analysis.

ORGANIC ACIDS

A gas chromatographic method for the determination of acetic acid in biological materials was described by Medzihradsky and Lamprecht (*16R*). Goedde and Brunschede (*7R*) employed a thin-layer chromatographic method for the quantitative estimation of β-aminoisobutyric acid in urine. Young and Renold (*24R*) described a microfluorometric enzymatic procedure for the determination of ketone bodies in blood. Kleeberg (*13R*) reported a semiquantitative colorimetric test for diacetic acid in blood and urine. Nanogram quantities of EDTA were determined spectrophotometrically by Kratochvil and White (*14R*) by reaction with bis(2,4,6-tripyridyl-*s*-triazine) iron. Johnstone, Kemp, and Hibbard (*12R*) described a colorimetric method for the estimation of urinary formiminoglutamic acid. Ishidate, Matsui, and Okada (*11R*) determined D-glucaric acid in urine by ion-exchange column separation and formation of the intensely colored 1,5-diphenyl formazan derivative. Zarembski and Hodgkinson (*25R*) determined glyoxylic acid in blood and urine by complexing glyoxylic acid and resorcinol in acid to form a product which fluoresces in alkali. Mustala, Tuomisto, and Airaksinen (*18R*) estimated 5-hydroxyindoleacetic acid in urine by its reaction with 1-nitroso-2-naphthol in HONO. Holcomb, McCann, and Boyle (*9R*) presented a simple method for the estimation of *p*-hydroxyphenylpyruvic acid in urine by formation of *p*-hydroxybenzaldehyde and spectrophotometric measurement at 275 mμ. Rutkowski and DeBaare (*20R*) described an ultramicroprocedure for determination of serum L-lactate without removal of protein. Schon (*22R*) described a sensitive enzymic method for lactic acid by dehydrogenation by NAD-lactic dehydrogenase which reduced Fe^{3+} to form *o*-phenanthroline-Fe^{2+} complex. Savory and Kaplan (*21R*) determined lactic acid in blood by gas chromatography in which lactic acid was oxidized by ceric sulfate to acetaldehyde. Cresta (*4R*) employed a new reaction for colorimetric determination of lactic acid by decarboxylation in hot 50% H$_2$SO$_4$ and coupling ethanol formed with tyroxine to give an orange-red color. Rosenberg and Rush (*19R*) used an enzymic-spectrophotometric method to determine pyruvic and lactic acid in blood. Stoner and Blivaiss (*23R*) determined homogentisic acid in urine by oxidizing to 1,4-benzoquinone-2-acetic acid and conjugating with 2,4-dinitrophenylhydrazine.

Granerus and Magnusson (*8R*) determined 1-methyl-4-imidazoleacetic acid in urine semiquantitatively by converting the acid to ethyl ester. Urinary methylmalonic acid was determined by Giorgio and Plaut (*6R*) by isolation on anion-exchange resin and diazotization with *p*-nitroaniline to form a green color. Munro and Fleck (*17R*) reviewed methods of extraction, separation, and analytical measurement of nucleic acids. Zarembski, Hodgkinson, and Knowles (*26R*) developed a more rapid gel filtration (Sephadex G-25) for the rapid determination of oxalic acid in biological materials. Isdale, Buchanan, and Rose

(*10R*) and Eichhorn and Rutenberg (*5R*) determined uric acid in serum specifically by incubating serum for 1 to $1^1/_2$ hours at 56° to 60° C to remove nonspecific reducing substrates prior to uric acid estimation. Caraway and Marable (*2R*) found good agreement between carbonate and uricase-carbonate serum uric acid methods. Marymount and London (*15R*) used H_2O_2 formed in enzymatic conversion of urate to allantoin to oxidize *o*-dianisidine for colorimetric determination of serum uric acid. Bergmann and Kalina (*1R*) separated uric acid and its thio derivatives from plasma proteins by Sephadex prior to their spectrophotometric determination. Cohen, Fishman, and Jenkins (*3R*) described a method based on extraction with isobutyl alcohol followed by fluorometry for determination of xanthurenic acid in urine.

ORGANIC COMPOUNDS

Arsenault and Yaphe (*1S*) developed a new, sensitive test to determine acetaldehyde by use of the resorcinol-HCl acid test. Schilke and Johnson (*10S*) determined serum acetoacetate by using alkaline aminoacetic acid and sodium nitroprusside and measuring color produced at 550 mμ. A simple method for determination of small amounts of citrate was described by Kraus (*7S*), in which citrate was converted to pentabromoacetone and reacted with sodium sulfide for color production. Chapman, Smith, and Simmons (*4S*) proposed a simple procedure for determination of 2 - amino - 2 - hydroxymethyl - 1,3-propanediol in urine by a modification of the formaldehyde titration. Aungst (*2S*) described a specific and sensitive method for the detection of ferritin in body fluids by a modification of the Ouchterlong technique using constant feed reservoirs. Jones, Murray, and Burt (*5S*) determined hypoxanthine in tissues by $HClO_4$ extraction and automation of the xanthine oxidase spectrophotometric assay. Bryan (*3S*) determined urinary indoxyl sulfate by preliminary column chromatography on cation-exchange resin followed by diazotization and coupling for color development. Petersen, Jorni, and Jorgesen (*8S*) described an enzymatic spectrophotometric method for separate determination of hypoxanthine and xanthine in human urine. A simple, rapid, sensitive procedure for determination of indoxole in biological specimens was developed by Kaiser, Bowman, and Forist (*6S*) based on ethyl acetate extraction of the alkaline specimens and fluorometric analysis of the ethanolic extract. Rinderknecht and Rinderknecht (*9S*) presented a simple rapid paper chromatographic method for the determination of urinary pseudo-uridine.

PROTEINS

Roman (*46T*) reviewed the methods of separating and identifying serum protein. Dubowski (*11T*) surveyed various methods (specific gravity, absorbance, and paper electrophoresis) for physical-chemical measurements of serum proteins. Muirhead (*33T*) reviewed x-ray analysis of proteins. Watson (*55T*) reviewed the techniques for separation of albumin from globulin and their determination. Globulins were separated from serum by precipitation with HCl-ethanol and the albumin remaining in supernatant with alcoholic sodium acetate precipitation by Fernandez, Sobel, and Goldenberg (*14T*). Rodkey (*44T*) precipitated serum albumin with 2% trichloroacetic acid in ethanol. Christian, Knoblock, and Purdy (*8T*) made direct coulometric titrations of Kjeldahl digests to determine protein nitrogen. De la Huerga, Smetters, and Sherrick (*10T*) made a critical survey of biuret methods of serum protein analysis. Gosiar (*19T*) reviewed the quantitative color reactions for proteins and reported the reagents, *o*-diacetylbenzene, mercurial sulfhydryl, and hydroxynaphthaldehyde as the most promising. Leffler (*27T*) described a procedure for colorimetric determination of serum protein nitrogen by the Berthelot reaction. Strukova and Fedorova (*52T*) determined Kjeldahl nitrogen without ammonia distillation by precipitation and separation of the ammonia salt with sodium tetraphenylborate. Barskii and Ivanov (*4T*) estimated protein in tissues with luminescence produced by Procion Yellow 4RS. Pastewka and Ness (*38T*) recommended human mercaptalbumin and albumin-Fraction V standardized by Kjeldahl as reference standards for serum protein analysis. Fessel and Feichtmeir (*15T*) compared four serum albumin methods—biuret, electrophoresis, colorimetric, and ultracentrifugation—and found that each does not measure the same factor. Keyser (*25T*) reviewed the dye-binding methods for the estimation of serum albumin. A method of improved reliability for the direct determination of serum albumin with 2-(4′-hydroxybenzeneazo) benzoic acid was described by Ness, Dickerson, and Pastewka (*35T*). The decrease in absorbance when bromocresol green combines with albumin was used by Rodkey (*45T*) for its measurement. Boehringer and Soehne (*6T*) obtained a patent for an albumin test paper using various bromo dyes. The ceruloplasmin-*p*-phenylenediamine color was intensified by coupling with 2-cyanoacetylcoumarone by Curtain (*9T*).

Atencio, Burdick, and Reeve (*3T*), using I^{131}-fibrinogen, determined plasma fibrinogen by an accurate isotope dilution method. Goodwin (*18T*) compared heat precipitation, thrombin precipitation, and sulfite fractionation methods for fibrinogen estimation and found good agreement. Morrison (*32T*) described a fluorometric micromethod for detection of 0.01 μg of heme protein in tissues. Hatch (*21T*) reviewed the nature and composition of various classes of serum lipoproteins. Rabinovitz and Schen (*40T*) used zinc tetracycline as a fluorescent stain for serum lipoproteins in immunoelectrophoresis. Narayan and Kummerow (*34T*) reported that Sudan Black B was satisfactory to detect lipoproteins in the presence of other proteins. Reissell, Hagopian, and Hatch (*41T*) described a thin-layer electrophoretic method for semiquantitative study of serum lipoproteins. A new technique for the determination of serum mucoproteins was developed by Guglielmone and Marenzi (*20T*) in which both hexose and sialic acid were determined. McNeil *et al.* (*30T*) found the serum sialic acid method to be a clinically adaptable method for the estimation of serum glycoprotein.

Rice (*42T*) evaluated and standardized the turbidimetric trichloroacetic method for spinal fluid protein determination. Friedman (*16T*) made a critical evaluation and modification of the copper - Folin - Ciocalteu spinal fluid total protein method. Evans and Quick (*13T*) reported that polyacrylamide gel electrophoresis of spinal fluid protein produced 15 constant bands. Gammaglobulins of spinal fluid were determined by MacPherson (*31T*) by a microprecipitation technique using rabbit antiserum and by Einstein, Richard, and Kwa (*12T*) using chromatographic separation with DEAE-Sephadex anion exchanger. Kyle and McGuckin (*26T*) separated macroglobulins from myeloma proteins in serum by Sephadex G-200 gel filtration.

Battistini (*5T*) surveyed the recent advances in agar, continuous cellulose acetate, and immunoelectrophoresis. Sunderman and Johnson (*53T*) made electrophoretic fractionation of serum proteins by the automated Skeggs-Hochstrasser technique and observed incomplete separation of alpha$_1$ globulin from serum albumin. Methods of serum protein fractionation on cellulose acetate were described by Webster (*56T*), Briere and Mull (*7T*), Paget and Coustenoble (*36T*), Kaplan and Savory (*23T*), and Wheat and Walther (*57T*). Giovanelli (*17T*) reported that alpha$_2$-globulins were 22% greater when separated on cellulose acetate than on paper. Paget and Coustenoble (*37T*) performed microelectrophoresis of serum glycoproteins on cellulose acetate gels and on cellulose polyacetate. Kelsey, de Graffenried, and Donaldson (*24T*) stained serum glycoproteins separated by electrophoresis on cellulose acetate with the periodic acid–Schiff reaction and Rice and Bleakney (*43T*) stained spinal fluid proteins separated in this manner with Nitrosin dye. Lima, Smith, and Stahlmann (*28T*) reported that chromatography was superior to gel electrophoresis for the fractionation of the lower molecular weight peptides. Serum proteins were fractionated by Sasahira, Kakusui, and Shimmon (*49T*) by gel filtration in Sephadex G 200, by Sorkina (*51T*) by filtration with DEAE-Sephadex A25, and by Adams, Yoder, and Free (*1T*) with DEAE-cellulose column. Sarnecka-Keller, Noworytko, and Kos (*48T*) fractionated urinary peptides on Sephadex G-25. A method for location and detection of small amounts of protein separated by agar-gel electrophoresis with copper rubeanate was described by Wagner (*54T*). Searcy *et al.* (*50T*) reported pronounced decoloration of protein zones in starch agar gel electrophoretic patterns when treated with iodine. Saifer and Pascal (*47T*) made immunochemical studies of human serum components obtained with urea-starch gel electrophoresis. Pastewka, Ness, and Peacock (*39T*) resolved 200 μg of serum proteins into 20 to 30 discrete bands by disk electrophoresis in polyacrylamide gel. Thin-layer separation of proteins by electrophoresis was achieved by Lopiekes, Dastoli and Price (*29T*) by using 20 × 20 cm alumina G plates equilibrated with $0.1M$ phosphate buffer. Heinzel *et al.* (*22T*) presented a micromethod for the quantitation of antibody content in immunoprecipitates to determine antibody and antigen protein. An improved method giving clear undistorted lines for quantitative immunoelectrophoresis of serum proteins was described by Afonso (*2T*).

STEROLS

Richter (*25U*) reviewed the pitfalls and trends in steroid analyses. Vanden Heuvel and Horning (*28U*) reviewed the separation, identification, and estimation of steroids. Ulick and Vetter (*27U*) estimated the secretory rates of aldosterone and 18-hydroxycorticosterone by the single injection isotope dilution technique for the activities of their metabolites, tetrahydroalderstone and 18-hydroxytetrahydro A. Methods for the colorimetric determination of serum cholesterol were reviewed by Etienne, Etienne, and Cottet (*16U*). Radin (*24U*) presented procedures for the purification of cholesterol and characterization for establishment as a primary standard. Badzio (*2U*) showed that marked differences may occur in the molar absorptivity of free and esterified cholesterol when $FeCl_3$ reagent is used. Sax and Daughetee (*26U*) reported that the Babson iron–sulfuric acid cholesterol procedure can be improved by using isothermal reaction and extracting cholesterol from sera in the presence of a silver salt of an ion exchange resin. Altescu (*1U*) found that substitution of $CuCl_2$ for $FeCl_3$ in the cholesterol reagent resulted in lower blanks, color stability, and reduced effect of temperature. Nishina, Hariya, and Kimura (*21U*) developed a new colorimetric micromethod for serum cholesterol based on the oxidation of cholesterol to cholest-4-ene-3,6-dione and formation of *p*-nitrophenylhydrazone derivative for color development. Curtius, Buergi, and Keller (*9U*) obtained 6% lower serum cholesterol values by gas chromatography than by the digitonide method. Badzio and Boczon (*3U*) described optimum conditions for determination of free and esterified blood cholesterol by thin-layer chromatography. Cejka and Venneman (*7U*) isolated urinary aldosterone as its diacetate and ketolactone monoacetate by column chromatography. Devis (*11U*) made a critical study of the chemical and physical methods of estimating plasma and urinary corticosteroids. Patti, Frawley, and Stein (*23U*) cited many advantages of gas-liquid chromatography over paper chromatography for the determination of 17-hydroxycorticoids in urine and blood. Copinschi, Cornil, and Franckson (*8U*) described a new method for the measurement of cortisol secretion rate by the isotope dilution technique. Doerner and Stahl (*12U*) described a routine procedure for the fluorometric determination of nonconjugated cortisol and corticosterone in body fluids. Plasma cortisol was separated from corticosterone by Eechaute (*13U*) by means of adsorption chromatography on a silica gel column.

Henry (*18U*) reviewed physical and chemical methods of estimating 17-ketosteroids in plasma and urine. Feher (*17U*) described an improved method for the determination of individual urinary 17-oxosteroids. Vestergaard and Sayegh (*29U*) compared the bismuthate and periodate oxidation methods for assay of 17-ketogenic steroids and suggested improvements after chromatographic studies of the analytical steps. Metcalf (*20U*) reported that oxidation with metaperiodate of urinary 20-diols to 17-oxogenic steroids was complete in 10 minutes at 55°C. A comparison of the total 17-ketosteroids by gas-liquid chromatography and by the Zimmerman reaction was made by Darcey and Evenson (*10U*). Cawley *et al.* (*6U*) evaluated the urinary 17-ketosteroid gas-liquid chromatographic patterns and observed a number of peaks with retention times shorter than that of 17-ketosteroids. Berrett and McNeil (*5U*) developed a method for measuring the major urinary 17-ketosteroids. Emsun and Aras (*15U*) developed a new chemical reaction, sulfuric acid–ethyl acetate, for the determination of progesterone, dehydroepiandrosterone, estrone, and estradiol. Oertel and Groot (*22U*) developed a urinary pregnanediol and pregnanetriol method consisting of enzymatic hydrolysis, extraction, purification of extracts, spread-layer chromatography, and color reaction quantitation. Merck (*19U*) used thin-layer chromatography to determine the pregnane-3α, 20-α-diol content of urine, after special extraction with cyclohexane. Ehrlich (*14U*) described a method for measuring urinary pregnanediol which utilized thin-layer chromatography and an isotype derivative dilution technique. Barry (*4U*) compared four urinary pregnanediol methods and found quantitation by means of gas chromatography to be the preferred method.

TOXICOLOGY

Altshuller (*1V*) reviewed the literature on air pollution under the general subjects: biological indicators, sampling and calibration methods, particulates and aerosols, inorganic gases, organic gases, and analyses of source of pollution. Recent developments in the application of neutron activation analysis techniques to forensic problems were reviewed by Guinn (*30V*). Said and Fleita (*54V*) determined acetaldehyde in 0.5-ml samples of blood and urine. A spectral absorbance method for blood involving a shift of absorbance curves in the presence of strong alkali was developed by Routh *et al.* (*53V*) to measure the effect of acetylsalicylic acid alone at 300 mμ and the additional effect of salicylic acid at 319 mμ. A simple method for the quantitative determination of *N*-acetyl-*p*-aminophenol was described by Welch and Conney (*67V*) based on acid hydrolysis to *p*-aminophenol and coupling with phenol in the presence of hypobromite to form indophenol dye for spectrophotometric measurement. Fike and Sunshine (*24V*) identified antihistamine quantitatively in biological material in 3 hours by thin-layer chromatography. Bjerre and Porter (*5V*) described a rapid method based on ultraviolet spectrophotometry for simultaneous determination of salicylates and barbiturates in serum. Campion and North (*8V*) studied the effect of protein binding of barbiturates on their rate of removal during peritoneal dialysis by ultrafiltration experiments. Fastlich, Searle, and Davidow (*22V*) identified eight different barbiturates in blood by paper chromatography. Barbiturates in biological fluids were identified by thin-layer chromatography by Walker and Wood (*63V*), by paper chromatography by Waddell (*62V*), and by micro gas chromatography by Anders (*2V*).

Eskes (*20V*) determined alcohol in blood specifically by ester formation with vanadium 5,7-diiodo-8-hydroxyquinolate anhydride. Van Gent (*61V*) determined blood alcohol with vanadium oxurate, which forms a colored complex. De Oliveira (*18V*) determined ethanol by its reaction with methylene blue periodide to free methylene blue for photometric measurement. Borkenstein (*6V*) determined ethyl alcohol in exhaled air by passing through sulfuric acid–dichromate reagent and catalyzing with Ag, Pd, or Pt salts and measured the decrease in dichromate colorimetrically. Routine assay methods by gas chromatography for ethanol, methanol, acetone, and 2-propanol in body fluids were described by Natelson and Stellate (*49V*). Gas chromatographic methods for the determination of ethanol in biological specimens were employed by Davis (*17V*), Entenman, Jaeger, and Skahen (*19V*), Freudiger and Vignau (*26V*), Fritz *et al.* (*27V*), Mather and Assimos (*46V*), and Stone (*57V*). Harms (*33V*) identified urinary morphine and codeine by thin-layer chromatography and ultraviolet spectrophotometry. Methods for the determination of glutethimide (Doriden) were reported by Winsten (*68V*) who used gas-liquid chromatography, by Korzun *et al.* (*41V*) who employed both thin-layer and gas-liquid chromatography, and by Dauphinais and McComb (*16V*) who used solvent extraction. Wallace, Biggs, and Dahl (*64V*) determined diphenylhydramine in biological materials by acid hydrolysis of ether linkages, oxidation of the resulting secondary alcohol, steam distillation, and ultraviolet measurement. Dal Cortivo and Broich (*15V*) identified and estimated lysergic acid diethylamide by thin-layer chromatography and fluorometry. Hynie, Konig, and Kacl (*35V*) used a thin-layer system in $CHCl_3$ and $(CH_3)_2CO$ (4 to 1) and color detection of spots with *p*-(dimethylamino)benzaldehyde in HCl to detect meprobamate in human urine. Conklin and Hollifield (*13V*) determined nitrofurantoin in urine by direct extraction with nitromethane and addition of alkaline reagent to the extract to produce color. Grafnetterova (*28V*) determined penicillins in biological material by hydrolysis of the extracted penicillin to penicillamine and titration of the -SH group of the latter. Santos, Marques, and Ralha (*55V*) found paper and thin-layer chromatography, ultraviolet and infrared spectroscopy, and x-ray diffraction more sensitive than the classical color reaction for the identification of strychnine. Stewart (*56V*) described techniques of breath analysis to detect solvent poisoning. Kakutani, Yamano, and Fukuda (*36V*) devised a simple apparatus for gas chromatographic determination of carbon monoxide in blood. Ayres, Criscitiello, and Giannelli (*3V*) measured small amounts of blood CO by gas chromatography following vacuum extraction in a Van Slyke manometric apparatus. Colorimetric determination of blood and breath CO was made by Feldstein (*23V*) by passing samples into an alkaline solution of silver sulfamoyl benzoate and measuring the resulting colloidal silver colorimetrically.

Hainline (*31V*) modified the methemoglobin method of Evelyn and Malloy by use of a triton-borate solution to hemolyze and clear the diluent to remove turbidity caused by the stroma of the red cells. Krishnan and Erickson (*42V*) developed a rapid ion-exchange separation technique and incorporated it into the neutron activation method for determination of arsenic in biological samples. Guilbault and Kramer (*29V*) developed an ultrasensitive specific method for cyanide using *p*-nitrobenzaldehyde and *o*-dinitrobenzene in which cyanide is regenerated and acts as a catalyst. Petrie, Locke, and Meloan (*50V*) described a specific fluorometric method for cyanide by reaction with quinone monoxime benzene sulfonate ester, which yielded a highly fluorescent product. Chang and Thompson (*10V*) determined fluoride in submicrogram amounts in biological samples by diffusion and colorimetric measurement with sodium-2-(*p*-sulfophenylazo)-1,8-dihydroxynaphthalene-3,6-disulfonate-zirconium lake. Weinstein *et al.* (*66V*) described a semi-automated method for fluoride analysis in biological materials in which final color development was attained by reaction of volatile F with alizarin complexon. Hall (*32V*) used the latter reaction for microdetermination of fluorine in biological specimens. MacDonald (*45V*) investigated and standardized the method of Knights, MacDonald, and Ploompuu for the determination of salicylate. Kinser, Keenan, and Kupel (*39V*) determined antimony in biological materials by spectrochemical determination and Matulis and Guyon (*47V*) by spectrophotometric measurement. Monte-Bovi, Sciarra, and Lynch (*48V*) described a method for determination of borates in urine based on the blue color produced when a solution of poly(vinyl alcohol) and iodine reacts with boric acid. Konikowski and Farr (*40V*) presented a method for determination of microgram quantities of inorganic boron in mammalian tissues, in which a deproteinized extract was made to react with quinalizarin in concentrated H_2SO_4 to produce an intense color. Kinser (*38V*) determined bismuth and tellurium in animal tissue by atomic absorption spectrophotometry in microgram amounts. Christian (*11V*) described a polarographic method for the determination of as little as 1 μg of gold in blood and serum. A spectrophotometric method for the determination of gold based on measuring the absorbance of the tetraazidogold complex was described by Clem and Huffman (*12V*).

Rice *et al.* (*52V*) standardized a modification of the diphenylthiocarbazone lead method in which 2 ml of blood or 10 ml of urine are required. Forman and Garvin (*25V*) reported a rapid screening method for determination of urinary lead by chelation separation and dithizone colorimetric determination. Atomic absorption spectrophotometry was used by Berman (*4V*) to determine lead in blood and urine, and by Pierce and Cholak (*51V*) to determine Pb, Cr, and Mo in urine and blood. Browett (*7V*) described a procedure for the determination of Hg and Pb in the atmosphere and in blood and urine. Kudsk (*43V*) reported an improved method for the determination of 4 to 100 nanograms of mercury in a dithizone extract by ultraviolet photometry. Kellershohn, Comar, and Le Poec (*37V*) measured 10 to 3 μg of mercury in human blood by activation analysis. Sunderman (*58V, 59V*) measured nickel in biological materials by atomic absorption spectrometry and by spectrophotometry using diethyldithiocarbamate complex. Cummins, Martin, and Maag (*14V*) devised an improved method for determination of selenium in biological material which required less than 1 hour. Taussky *et al.* (*60V*) determined selenium traces in biological materials by the reaction of Se with 3,3-diaminobenzidine and fluorometric measurement. Submicro amounts of selenium in biological material were determined with 2,3-diaminonaphthalene by Watkinson (*65V*). Estes and Faust (*21V*) and Hozdic (*34V*) determined microgram amounts of silicon in biological materials by the silicomolybdenum blue reaction. A rapid x-ray fluorescence method was used by Champion, Taylor, and Whittem (*9V*) to determine traces of strontium in biological samples. Livingston and Smith (*44V*) estimated vanadium in biological material by neutron activation analysis.

VITAMINS

Thabet and Tabbian (*11W*) proposed a microtest for vitamin A, based on the observation that vitamin A after oxidation to vitamin A aldehyde reacted with *o*-aminophenol in acid to produce an intense red color. Awdeh (*1W*) separated vitamin A from carotenoids by means of a microcolumn on 325-mesh silicic acid. Myint and Houser (*9W*) proposed a simplified thiochrome method to determine small amounts of thiamine in small amounts of whole blood and serum which eliminated deproteinization, sample dilution, and purification by adsorption column. Green (*4W*) proposed a spectrophotometric assay method for avidin and biotin based on binding of dyes by avidin. Skeggs (*10W*) reviewed the discrepancies in assay results, choice of organism, and factors controlling vitamin B_{12} assays. Lau *et al.* (*7W*) presented a method for measurement of serum B_{12} level using radioisotype dilution and charcoal coated with albumin or hemoglobin. Frenkel, Keller, and McCall (*2W*) described a radioisotopic assay of vitamin B_{12} in serum based on the principle of saturation analysis, addition of cobalt-57 B_{12} to the unknown sera as a tracer for the subsequent determination of per cent recovery of endogenous B_{12}. Kutter (*6W*) prepared an indicator powder for determination of ascorbic acid in urine by grinding 1 part of $AgNO_3$ and 5 parts of $(NH_4)_2CO_3$ together to obtain a white mixture which was darkened according to ascorbic acid concentration when urine was added. Vann (*12W*) described a new method for the determination of ascorbic acid based on the quantitative reduction of ferric to ferrous ion by ascorbic acid and the colorimetric measurement of the ferrous - bathophenanthroline complex. Fujii and Tatsukawa (*3W*) modified the L-ascorbic acid method of Hiraoka by using a pH 8 phosphate buffer containing glycerol as a stabilizer for the reduced 2,6-dichloroindophenol mixed with the oxidized form. Martinek (*8W*) investigated the use of tripyridyltriazine as a chromogenic reagent for the colorimetric determination of serum vitamin. Hansen and Warwick (*5W*) determined free and total tocopherol in serum by a micro ultraviolet fluorometric method by use of 295-mμ ultraviolet light and fluorescent measurement at 340 mμ.

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Coatings

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THIS BIENNIAL REVIEW covers the period from January 1965 through December 1966 and includes the authors' choice of important contributions to the analysis of coating materials. It is hoped that the attempt to be selective has not caused omission of commendable contributions. There is a noticeable increase in foreign publications concerning coating analysis much of which appears to duplicate work that was reported in earlier years.

Other reviews of a similar nature have appeared in this period (*110, 111*) in addition to reviews on special analytical or coating subjects. The annual reviews of the literature (*93*) on the composition and characteristics of fats and oils was prepared for 1965 but omitted in 1966. A 20-page review of quantitative methods of analysis of certain plastics (*8*) contained procedures for such polymers as polyvinyl, polyamide, and cellulose-type materials, including the identification of plasticizers, antioxidants, stabilizers, and monomers.

The largest number of reviews to appear in this period concerned the subject of gas chromatography and its application to analysis of coating materials. The most recent is comprehensive in subject matter (*51*); another treats the analysis of solvents and resins only (*43*), and a third (*96*) concerns all types of chromatography as related to the paint field. The review by Kelly (*74*) outlines the procedures used for solvents, monomers, additives, polyols, carboxylic acids, oils, and resins. The gas-liquid chromatographic analysis of polymers after pyrolysis, along with solvents and fatty acids was outlined (*48*). A review of the application of gas chromatography to the analysis of rubbers and synthetic resins (*106*) contained a table of pyrolysis products of a wide range of polymers. Arndt (*4*) discussed the history, basic principles, and recent advances in gas chromatography and summarized its application to problems encountered in the paint industry.

A larger number of books than usual concerning various phases of coating analysis have appeared in this period. A book by Haslam and Willis (*53*) on the identification and analysis of plastics contains many procedures applicable to coatings and includes such subjects as polymers, copolymers, plasticizers, stabilizers, and antioxidants, in addition to a chapter on gas–liquid chromatography and a library of nearly 100 infrared spectra. Ke (*71*) edited a book presenting newer methods of polymer characterization and another (*72*) on thermal analysis of high polymers. The latter included synthetic fibers, linear polyesters, polyurethane elastomers, polybutene, and polyethylene. Mitchell and Billmeyer (*101*) edited a book on the analysis and fractionation of polymers and the third volume of the 6th edition of "Standard Methods of Chemical Analysis" (*142*) appeared in this period. Part II of this volume on instrumental analysis, contains a chapter on paint, varnish, and lacquer. Hummel (*62*) included polymers, resins, waxes, plasticizers, monomers, and solvents in his publication of infrared spectra in the medium and long wavelength regions. A chapter on the analysis of vegetable oils appears in the 4th edition of Williams' book (*144*) on the examination of oils, fats, and fatty foods. A chapter on the analysis and characterization of polyunsaturated acids appears in volume 9 of the "Chemistry of Fats and Other Lipids" edited by Holman (*60*). In "Plasticization and Plasticizer Processes" as edited by Gould (*46*), one of the chapters evaluates the various forms of chromatography as methods for the analysis of plasticizers and for the identification of the saponification products of ester-type plasticizers. The 7th volume of a series of abstracts on gas chromatographic methods, was edited by Knapman (*77*). This series, started in 1958, contains many subjects of interest to the industry, such as the analysis of solvents, polyols, plasticizers, terpenes, fatty acids, and pyrolyzed resins.

GENERAL ANALYTICAL SCHEMES

The general principles of gas chromatography and the mechanism of pyrolysis were discussed by Guillet (*49*) who presented examples of chromatograms obtained with polymers. The complementary nature of infrared spectrometry and gas phase chromatography was stressed in a publication (*50*) that summarized and illustrated the application of both techniques to polymer analysis. Jain and associates (*66*) illustrated the identification of 41 paints by the combination of pyrolysis and gas chromatography. The behavior of certain chemical groups during pyrolysis as determined by gas chromatography was described in a paper (*34*) that also contained characteristic chromatograms obtained from many polymers. The applications of gas chromatography in the paint field were described in an article (*42*) that stressed the examination of hydrocarbon solvents and polymers following pyrolysis. Cianetti and associates (*23*) described a method of thermal degradation and the chromatograms that resulted from its application to various members of the cellulose family of plastomers. Berton (*14*) studied the pyrolysis products of plastics and other materials by gas chromatography and demonstrated the use of selective detectors for such products as saturated, aromatic, and olefinic hydrocarbons, aldehydes, alcohols, ketones, and halogenated compounds. He applied his technique to a broad spectrum of materials including paint. Scholz and associates (*121*) investigated the characterization of polymeric compounds by gas chromatographic analysis after oxidative degradation.

A new detector was described by Ford and Kennard (*38*) which can be used in conjunction with column chromatography. They described applications to the field of coatings including analysis for monomer, dimer, and trimer acids.

The basic technique and research applications of thin layer chromatography were discussed by Privett and Blank (*115*) including the application to the analysis and fractionation of fats and oils. A bibliography of literature on the thin layer chromatographic technique was compiled (*75*) that included 1041 references and 121 classifications including sections on pigments, resins, fatty acids, and plastics. This work is updated semi-annually.

Paper chromatography has been applied to the identification of such waterproofing agents and paper coatings as urea- and melamine-formaldehyde, styrene and copolymers, and protein resins (*89*).

Page and Bresler (*109*) applied nuclear magnetic resonance spectroscopy to the determination of molecular weight and the acid end-group content of glycol polyesters.

Foster *et al.* (*40*) examined various polymers as films in the 1.0 to 2.7-micron spectral region and reported on its usefulness for reaction kinetics, degradation studies, and the quantitative measure of polymer blends. Low and Inoue (*88*) obtained infrared emission spectra on the surfaces of paint, paper, rubber, and polyethylene and concluded that empirical methods could be developed for specialized applications, such as quality control of pigments and coatings. Although not so versatile as various better-known techniques of absorption spectrometry, it offers the advantages of simplicity of sample preparation and examination. A technique was outlined (*90*) for the preparation of wedge-shaped KBr disks which can be used in differential spectroscopy. Spectra were illustrated for plasticizer mixtures, a plasticized poly(vinyl acetate), and an alkyd where one or more components of each are placed in the wedge which in turn is positioned in the reference beam so as to compensate for the corresponding absorbance arising in the disk of the sample.

Rozentals (*119*) obtained spectra of the gaseous products of pyrolysis of various elastomers in the ultraviolet spectral region, 185–260 mμ. Characteristic spectra were obtained for most of the synthetic rubbers and polystyrene and the author recommended extending the procedure to the examination of other materials. References to the analysis of paint, dyes, pigments, and ink were included in a new book (*143*) on the subject of reflectance spectroscopy. Rahder (*116*) has recommended the application of dry distillation with lime as a preliminary test in analyzing synthetic resins and included his preference for qualitative tests to be applied to the solution of the volatile products in water. A paper was published (*6*) that covered in detail the use of modern analytical techniques for identifying traces of paint. Differential thermal analysis has been applied (*134*) to the identification of synthetic coating resins in an investigation that included alkyds, amino-alkyds, epoxy esters, amino-epoxy esters, vinyl and related polymers, and ester gum. Atlas and Mark (*5*) surveyed modern methods of determining polymer properties in solution and in solid state. Characterization in the solid state included the use of x-ray, electron beam diffraction, infrared and ultraviolet spectrophotometry, differential thermal analysis, etc.

SPECIFIC CLASSES OF HIGH POLYMERS

Most of the contributions that appeared in this period on the analysis of specific polymers, concerned rosin products and most of these utilized gas chromatography. Some papers treated only the methods of preparing the esters for separation. The retention time of the methyl esters of rosin acids were tabulated (*19*) using flame ionization type of detector. Chang and Pelletier (*20*) demonstrated the separation of seven of the most common resin acids as methyl esters using a column prepared with QF-1 (trifluoropropylmethyl silicone). Brooks *et al.* (*17*) used a polyamide liquid phase (Versamid 900). Another investigation (*138*) reported relative retention data for methyl esters of ten resin acids on columns of polar polyesters and nonpolar Apiezon N grease (10%) on hexamethyldisilazane-treated Chromosorb W. Hetman and associates (*54*) formed methyl esters of rosin acids directly in the injection port of the chromatograph at 400° C by decomposition of the tetramethylammonium salts. In order to study the relationship of the dimer content of rosin to crystallization time, Leonard *et al.* (*84*) separated the methyl esters on a low-loaded, deactivated column. The column was prepared by treating diatomaceous earth with dimethyldichlorosilane and trimethylchlorosilane which reacted with the reactive sites on the column and at the same time deposited a small amount of silicone to serve as the liquid phase. In a similar investigation (*120*), the composition of various types of rosin was determined by gas chromatographic separation of the methyl esters and related to crystallizing tendencies.

Hummel and Pohl (*63*) published a paper that discusses the infrared absorption of abietic acids and their derivatives in an attempt to show how exact knowledge of the bands for different carbonyl functions in the derivatives, could simplify subsequent analytical work. Resin acid methyl esters have also been separated by thin layer chromatography (*148*) using alumina impregnated with silver nitrate. The developing solvent was 25% ethyl ether in light petroleum.

The detection of melamine in lacquers or dried films was accomplished by paper chromatography (*147*). The sample was first hydrolyzed with hydrochloric acid, dried, and dissolved in ammonium hydroxide. The developing solvent was a 3:1 mixture of phenol and dilute aqueous formic acid, and the paper was sprayed with aqueous ammoniacal copper acetate. Phthalic acid was also detectable if the sample contained alkyd resin.

Infrared spectrometry was used (*18*) to identify the diol components of polyester resins prepared from the condensation of mixed maleic and phthalic anhydrides with ethylene, diethylene, and propylene glycols. The spectral region from 7.1 to 7.5 microns was used. Another technique (*86*) for identifying the components of unmodified polyesters and polyester-type urethanes, made use of GLC after pretreatment and separation of diamines from the polyols.

In order to determine the epoxy value of materials not suited to the hydrobromic acid titration, Fioriti and associates (*37*) used picric acid as colorimetric reagent and included two commercial epoxy resins in the variety of samples used to demonstrate the applicability of the method. Weatherhead (*141*) used thin layer chromatography for the analysis of bisphenol–epichlorohydrin resins. Using silica gel G on glass plates, chloroform as developing solvent, and chromic acid as spray reagent, he was able to distinguish between similar resins from different manufacturers, measure the monomer content quantitatively, and isolate the hydrolyzable chlorine. Spell and Eddy (*130*) separated the various molecular species of epoxy resins very sharply by thin layer chromatography and characterized each from the infrared spectra.

Cellulose esters were determined without removal of plasticizers or stabilizers by quantitative methanolysis followed by separation of the methyl esters with a gas chromatograph having a flame ionization detector (*140*).

Infrared spectrometry was used (*135*) to determine the poly(vinyl acetate) content of the copolymer with vinyl chloride; the calculation was based on absorbance intensities of the bands at 5.74 microns and 7.02 microns. A potentiometric method was also described (*128*) for measuring the vinyl acetate in a copolymer with vinyl chloride. In this method, the sodium acetate formed by saponification of the vinyl acetate was titrated with hydrochloric acid. In another, very similar procedure (*83*), the copolymer is first refluxed with acetone to induce swelling before treatment with alkali. Heylmun *et al.* (*55*) determined the vinyl content of silicone gums by treatment with phosphorus pentoxide and measured the ethylene formed with gas–liquid chromatography. An infrared spectrometric study of propylene–vinyl chloride copolymers was made (*94*) and spectra of the polymer films were presented.

Senn (*127*) analyzed butadiene–styrene copolymers by nuclear magnetic resonance spectroscopy. Pyrolysis combined with gas chromatography was employed (*69*) to determine the styrene content of styrene–polyethylene graft copolymers. Debowski (*27*) applied infrared spectrometry to the determination of styrene in the ternary copolymer styrene–vinyl acetate–maleic anhydride. He used the band at 14.1 microns and worked with samples dissolved in acetone. Post (*114*) outlined an absorbance ratio method for the determination of styrene at the 80–100% level in butadiene copolymers using the absorbance at 3.25 and 10.3 microns. She described her technique for isolating and purifying the copolymer.

Secrest (*124*) made a comprehensive study of the infrared spectra of phenolic resins and discussed in detail the structures, assignment of absorption bands, and the use of assigned frequencies. He was able to distinguish heat reactive forms from nonreactive resins, establish relative degree of substitution, and identify resins after reaction with oils.

An investigation was conducted (*44*) to determine the yields of methacrylate and acrylate monomers from their copolymers by pyrolysis. The monomers were measured by gas chromatography, the correction factors were assigned, and quantitative results were reported. A method for measuring methyl methacrylate in the presence of methyl acrylate (*112*) was based on saponification of the former, followed by oxidation with permanganate to form formaldehyde which was determined colorimetrically with Schiff reagent. There was no interference from methyl acrylate. The composition of chloropyrene–methyl methacrylate was determined (*99*) from the infrared spectra using characteristic bands at 5.80 and 6.02 microns.

SPECIFIC CONSTITUENTS

Bright and associates (*16*) measured styrene monomer in polyesters by gas–liquid chromatography and from ultraviolet absorption. In the former, they used the supernatant liquid from methanol precipitation of the polymer; in the spectrophotometric method, they used the bands at 290–293 mμ with a monomer-free sample of polymer in the reference beam. The gas chromatographic separation was also used in another investigation (*30*) in which larger amounts of styrene monomer, ranging from 13–47% were present as cross-linking agents. The combination of ultraviolet absorption and gas chromatography was also used by Crompton *et al.* (*26*) to calculate the styrene monomer and other aromatic volatiles in conventional and modified polystyrene. Monomeric styrene, methylstyrene, and dimethylstyrene were determined potentiometrically (*7*) by titration of the acetic acid formed when the monomers reacted with mercuric acetate in methanol. A temperature programmed gas–liquid chromatographic method was described (*39*) for measuring *n*-butyl acrylate, vinyl acetate, and 2-ethylhexylacrylate residual monomers in vinyl acetate polymer emulsions. An internal standard was used for quantitative work. Crompton and Buckley (*25*) published a polarographic procedure for both the residual acrylonitrile and styrene monomers in the copolymers.

MacWilliams and associates (*92*) extracted acrylamide monomer from the polymer and copolymer using solvent, treated with an ion exchange resin, then provided both polarographic and spectrophotometric procedures. The former method was considered more specific. The working range was from 0.01 to 0.1% of the monomer.

Jankowski and Garner (*68*) determined the carboxylic acids present as esters in plasticizers and polymers by transesterification and gas chromatographic separation. Sodium methoxide in methanol and methyl acetate as reagent gave quantitative yield of the esters which were extracted and chromatographed with diphenyl ether as internal standard. Ten dicarboxylic and four fatty acids were included but no mention was made of maleic or fumaric, and it is presumed that they could not be differentiated by the technique. Dicarboxylic acids have also been identified (*102*) as their methyl esters by GLC using glass beads. Novak (*108*) supplied procedures for the polarographic analysis of three combinations of acids in alkyds and polyesters. Included were phthalic, maleic, fumaric, citraconic, and mesaconic acids. Traxton (*136*) evaluated published polarographic methods for determining dicarboxylic acids in various coating resins and presented his preferred technique for determining maleic, fumaric, and phthalic acids in polyester resins and plasticizers. He also studied the degree of isomerization of maleic to fumaric acid during hydrolysis of resins prior to polarographic determination. Another polarographic method was published (*78*) for trimellitic, pyromellitic, tetrachlorophthalic, and chlorendic acids without interference from phthalic, maleic, and fumaric acids. Thin layer chromatography has been applied to the problem of separating mixed acids (*80*) and polyhydric alcohols (*79*). Five different developing systems were studied and the work included 30 dicarboxylic and monocarboxylic acids and 17 alcohols. By the comparison of two potentiometric titration curves of polyesters in acetone–ethanol solution and in acetone–water mixture, it was possible to calculate the proportion of acid anhydrides, free acids, and carboxyl end-groups (*35*). A gravimetric method for determining the amount of phthalic acid in various products was described (*47*) in which the acid is precipitated as the mercurous salt. The isolated depotassium salts in water are acidified with acetic acid, mercurous nitrate is added in excess, the sample is boiled, and the salt collected, dried at 130° C, and weighed. An alternate technique was described in which the excess mercurous nitrate was titrated. Characteristic bands in the infrared spectra of acids separated by saponification, have been used to detect alkoxyethanedicarboxylic acids in polyesters (*76*). The diols and polyols used in making polyesters have been identified (*36*) by paper chromatography in the solution remaining after removal of the dicarboxylic acids by saponification. April (*3*) estimated the ratio of maleic to phthalic anhydrides in polyesters and alkyds by a combination of infrared spectroscopy and gas chromatography.

Schrotter (*122*) demonstrated the technique of acetylation in ethyl acetate at room temperature in the presence of perchloric acid as a catalyst for obtaining the hydroxyl value of alkyd resins. The reaction is complete in 6 minutes and the unchanged acetic anhydride is measured in the usual manner. Sullivan and Hahn (*133*) tested various standard methods for determining hydroxyl content on copolymers of styrene with allyl alcohol, but obtained results lower than theoretical in all cases. They developed a suitable procedure based on esterification with *n*-decanoic acid.

Hydroxymethyl groups were determined (*146*) in phenolic resins by the difference between a total hydroxy group determination and titration of the phenolic hydroxy groups. The former analysis was made by infrared in the 2.78 to 3.33-micron region. A procedure was outlined (*118*) for determining the amount of free phenol in phenolic resins and varnishes. After steam distillation of a weighed sample and diluting the distillate to definite volume, the absorption was measured at 269 mμ.

Gas chromatography was used by Stevens (*131*) to extend a previous method (*132*) to include the analysis for the unreacted aldehydes in phenol–acrolein and phenol–furfural resins.

The first procedure to appear in the literature (*1*) for determining isophthalic acid in lacquers, utilized the infrared spectra of both the solvent-free film and of an acetone solution of air-dried film to give values for nitrocellulose, isophthalic, and for phthalic anhydride if present. Clarkson and Robertson (*24*) explained a refined calculation for nitrogen in nitrocellulose when determined from the infrared absorption. Doerffel (*28*) reported the determination of nitrile groups in polymers from infrared spectra through the use of KBr tablets. He also investigated the use of potassium ferrocyanide and thiocyanate as internal standards. Ultraviolet spectrophotometry was used for the analysis of methylphenylsiloxane resins to measure their phenyl group content (*139*). Chloroform was used as solvent and the extinction was measured at 260, 266, and 272 mμ. A report is available (*73*) that describes a method for the hydrolysis of polyamide and amine–adduct curing agents for epoxies that permits subsequent quantitative separation and identification of the constituents.

A procedure was developed (*21*) for determining both fluorine and chlorine in the same sample of polymer by the gamma-activation method. The amount of each halogen was calculated from the total induced activity following irradiation for 20 minutes. Small quantities of both free chlorine and resin-bound chlorine were measured (*137*) in epoxy resins by potentiometric titration with silver nitrate in 85% acetic acid. The sample was dissolved in dioxane containing anhydrous acetic and the same technique was applied directly to the untreated sample and again after hydrolysis to obtain total chlorine. A report was prepared (*31*) for the analysis of five reactive diluents used in solventless epoxy compounds. The analysis is applied directly to the product tested in most cases and programmed-temperature gas chromatography was used for the separation. The method is quantitative for the diluents with the exception of butanediol diglycidyl ether.

OILS AND FATTY ACIDS

Four methyl ester primary standards were used by Pons and Frampton (*113*) to study the precision and accuracy of gas chromatographic methods for the separation of fatty methyl esters. Hofstetter *et al.* (*59*) determined the equivalent chain length on 79 methyl esters and 7 ethyl esters of unsaturated fatty acids with gas chromatography. Nikelly (*107*) presented chromatograms and quantitative analytical data for the separation of free fatty acids on a micro glass bead column containing a liquid phase along with an acidic material. A comparison was made (*103*) of chromatograms obtained from untreated, unsaturated fatty esters with those produced by hydrogenated methyl esters, for identification purposes. A special hydrogenation accessory was located in the injection port. The fatty acids from soya bean and fish oils were used.

Jamieson and Reid (*67*) studied methods of transesterification used for the analysis of oils and fats. A rapid method was published (*100*) for saponification followed by rapid esterification for use with triglycerides and lipids. McGinnis and Dugan (*91*) described a rapid method for preparing methyl esters of fatty acids. They first form a sulfuric–fatty acid complex at the temperature of a dry ice–acetone bath which then forms methyl esters when methanol is added.

Litchfield and associates (*87*) made a study to determine the optimum operating conditions for affecting quantitative recovery of model triglyceride mixtures by gas chromatography. Similarly, Kuksis (*82*) reviewed available techniques and discussed optimum separation and recovery conditions. In another paper (*70*), thin layer chromatography was demonstrated with sunflower oil as a means of separating triglycerides.

Wood *et al.* (*145*) compared the retention times of derivatives of hydroxy fatty acids. They prepared trimethylsilyl ether derivatives of the methyl ester of riconoleic acid, found the formation to be quantitative, and the derivatives to emerge from the gas chromatographic column in much less time than the acetates. Paper chromatography was used to separate and identify fatty acids (*22*), with benzyl and phenacyl halides used as reagents. Evans and associates (*33*) made use of column chromatography to investigate the nature of the polymeric products formed in oils by the action of heat and oxygen.

Reversed-phase paper chromatography was used (*105*) to separate the acids of tall oil. In another publication, ultraviolet spectrophotometry was used (*98*) to determine the concentration of rosin acids when mixed with fatty acids.

Examination of the ultraviolet spectrum from 220 to 360 mμ, and measurement of the absorbance at 315 mμ, was employed (*41*) to determine the amount of fish oil in linseed oil. The method was applicable to quantities not exceeding 18% fish oil.

A review of known methods for the iodometric determination of peroxide in fats was made (*126*) and the causes and elimination of error in determining peroxide number of oils were discussed. A procedure was published (*64*) for determining the iodine number of fats and oils by the use of *N*-bromosuccinimide.

ASSOCIATED MATERIALS

The use of thin layer chromatography was described (*15*) for separating and identifying plasticizers. Hancock and associates (*52*) used gas chromatography, column chromatography, and a gravimetric procedure for determining the diethyl phthalate in lacquers, varnishes, and paints. Gas chromatography was used (*45*) for the detection of plasticizers used for vinyl chloride polymers and copolymers. Included were such materials as adipates, azelates, citrates, sebacates, phthalates, and fatty acid esters.

Ultraviolet spectra have been used (*129*) to identify and determine mixtures of phenolic antioxidants and ultraviolet absorbers in polypropylene. Commercial grades of chlorendic anhydride have been analyzed (*29*) for constituents through the use of potentiometric nonaqueous titration. The degree of resinification and the effects of various storage conditions on turpentine was studied (*104*) using thin layer chromatography.

A highly selective liquid phase was used (*32*) to separate aromatic hydrocarbons from paint thinners, by gas–liquid chromatography. Benzene can be used as internal standard. Toluene and xylene are readily identified and other aromatics can be classified and measured. Hudy (*61*) described a technique for analyzing lacquer solvents by gas chromatography without prior removal of the vehicle or pigment, but presented no quantitative data. Another paper (*85*) concerned the analysis of the composition of individual technical grade lacquer solvents by GLC, using toluene as standard.

High-resolution spectra of inorganic pigments and extenders in the mid-infrared region were recorded and discussed (*2*). The usefulness of the method for qualitative analysis and the technique for preparing disks are described in detail. There were 78 pigments included in this work and the authors describe an absorbance ratio method for quantitatively determining the per cent of rutile in mixtures with anatase titania. Jackson and Whitehead (*65*) determined 27 trace elements in titanium dioxide by spark-source mass spectrography. The interest in the trace elements is due to the effect they have on the relative brightness of the pigment. Seivers (*125*) measured the titanium dioxide content of bauxite by gas chromatography. The oxide was converted to the chloride by reaction with carbon tetrachloride at elevated temperatures. Berger and Cadoff (*11*) compared their polarographic technique to chemical methods for measuring lead and zinc in white pigments containing white lead and zinc oxide. The acetic acid–ammonium acetate buffer they used served as both digestion medium and subsequent electrolyte. The same buffer was used for the polarographic determination of titanium dioxide in paint pigments. After formation of the EDTA complex with titanium. The analysis of mixed paint pigments for both chromium and zinc content has been combined into a single procedure (*81*) which utilizes the complexometric method for the zinc and iodimetric titration of the chromium. Another complexometric procedure for determination of lead in lead pigments has been published (*95*). Ion exchange chromatography was used (*13*) to separate alkaline-earth metals from zinc oxide as a preliminary step to the colorimetric determination of the magnesium and calcium in mixed paint pigments. Rathi (*117*) presented tests for distinguishing similar pigments of similar colors, mostly organic-type pigments. A technique was outlined (*10*) for measuring the apparent density of organic pigments with an accuracy of approximately 0.4%.

Barker *et al.* (*9*) published routine methods for determining toxic metals in toys, school materials, and paint, including such metals as antimony, arsenic, barium, cadmium, chromium, and lead. Berger (*12*) supplied a method for the spectrophotometric analysis of mercury in latex paints in which the metal is complexed with diphenylthiocarbazone after treatment with sulfuric acid. A number of procedures for fungicidal preparations in paint were presented by Hoffmann and associates. An iodimetric method was used for phenylmercuric compounds (*58*), ultraviolet spectrophotometry for salicylanilide and toluene-*p*-sulfonamide (*57*), and chlorine analysis was combined with spectrophotometry for pentachlorophenol and

N-(trichloromethylthio) phthalimide (*56*).

Three different procedures were described by Sciarra (*123*) for determining the volatile content of aerosol products. A rapid total solids procedure (*97*) was based on the use of a hot-wire drying element in place of the usual oven. The tared drying element is dipped into the sample solution, quickly reweighed, then plugged into the apparatus described and the heating rate adjusted to maximum drying rate without decomposition.

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Essential Oils and Related Products

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THIS 11th review of analytical procedures for essential oils and related products covers the literature for September 1964 to August 1966 inclusive. It follows the general pattern previously established (*194*).

During this time, many analytical methods have been refined and applied with increasing skill to the investigation of essential oils and related products. This advance has been greatly aided by the improved instruments which have become available.

The combining of techniques, such as gas chromatography with mass spectrometry or nuclear magnetic resonance spectroscopy, which was in the development stage only a few years ago, has now become standard procedure. Other techniques which have been combined with increasing frequency are thin-layer or paper chromatography with infrared or ultraviolet spectroscopy. Such combined procedures have enabled analysts to solve difficult problems of identification, structural determination, and quantitative assay.

As a result of this progressive use of modern techniques, the secrets of the composition of many essential oils have been further penetrated in spite of their complexities and the difficulties involved in their analysis.

OFFICIAL COMPENDIA

The Seventeenth Edition of the "United States Pharmacopeia" and the Twelfth Edition of the "National Formulary" became official on September 1, 1965. Four oils were omitted from the new U.S.P., and, as customary, these were included in the N.F:

Juniper tar oil (Cade oil)
Lavender oil (Lavender flowers oil)
Myristica oil (Nutmeg oil)
Rose oil (Otto of rose)

Seven items, previously in the N.F., were omitted from the latest edition:

Bergamot oil
Capsicum oleoresin
Bay oil (Myrcia oil)
Pimenta oil
Sassafras oil
Rectified turpentine oil
Benzyl benzoate

Benzyl benzoate was in turn taken into the U.S.P.

The Scientific Committee of the Essential Oil Association of the U.S.A. has published specifications for five additional essential oils or natural products and 18 aromatic chemicals, increasing the total number of specifications from 245 to 268 as of January 1967. The natural products monographs added since the last review are:

No.	
251	Oil bay
252	Oil turpentine rectified
253	*d*-Limonene
255	Oil pimenta berries
256	Oil bergamot expressed

Specifications for the following aromatics were issued:

No.	
246	Ethyl phenyl glycidate
247	Diethyl succinate
248	Amyl caproate
249	Amyl propionate
250	Isoborneol
254	Allyl cyclohexyl propionate
257	Methyl ionone, alpha type and gamma type
258	Butyl isobutyrate
259	Isobutyl butyrate
260	Ethyl isovalerate
261	Ethyl decanoate
262	Methyl heptenone
263	4-*Tertiary* butyl cyclohexyl acetate
264	Amyl cinnamate
265	Amyl octanoate
266	Benzyl isovalerate
267	Benzyl isobutyrate
268	2,6-Dimethyl heptenal

Specifications were rewritten for:

No. 61 Ionones

and there was a refractive index revision on:

No. 3 Oil petitgrain Paraguay

Gas chromatographic (GC) methods were published for:

No.	
36A	Cedrol in oil cedarwood Texas
36B	Cedrol in oil cedarwood Virginia
17	Citronellol
125	Citronellyl acetate
162	Citronellyl formate
11	Geranyl acetate
206	Geranyl formate
61	Alpha ionone in ionones
61	Beta ionone in ionones
257	Alpha type methyl ionone in methyl ionones
257	Gamma type methyl ionone in methyl ionones
2, 135	Linalool in oil bois de rose Peru or Brazil
7	Citral in oil lemongrass
202	Isoeugenol isomers in isoeugenol

This brings the total GC methods to 15.

The general standardized gas chromatographic method for the analysis of essential oils was published (*272*).

Infrared spectra for identification purposes have been included in 25 additional monographs, bringing the total to 108.

The Index of Specifications has been revised to include common synonyms of the aromatic chemicals and to list those which have GRAS status.

Essential Oils. GENERAL PROCEDURES. The character of essential oils, the problems confronting the analyst in determining their composition, and the newer analytical apparatus and techniques presently employed to solve these problems were discussed by Naves (*418*).

The value and interpretation of gas chromatography for the analysis of essential oils was discussed by Kingston (*303*). He stressed that some of the inherent difficulties have been diminished by newer chromatograph designs, which reduced the heating period and introduced various improved capillary columns. A number of polyesters were evaluated as stationary phases in gas chromatography of terpenoids by Bapat, Ghatge, and Bhattacharyya (*27*), who derived a mathematical formula to express the relative efficiency of each polyester. Vernin (*691*) discussed gas chromatographic techniques applied to essential oils and synthetic products, and tested the use of two stationary phases of different polarity. Von Rudloff (*534*) obtained reproducible characteristic gas chromatograms of the oil from a single conifer needle using a solid sample injector.

Several analytical studies have been reported in which gas chromatography was employed in an unusual or novel manner. Teranishi, Buttery, and Mon (*656*) ran direct analyses of volatile vapors, using hydrogen flame ionization detectors to minimize the interference from oxygen and water present in the vapors. Sfiras and Demeilliers (*567*) studied the vapors emitted from a perfumed soap, and noted changes brought about by aging. A method of direct analysis of essential oils from the plant material which avoids steam distillation to obtain the oil, was used by Schratz and Wahlig (*558*). By this method, chromatograms more truly representive of the oils as they exist in the plant were obtained. Rifino, Monte-Bovi, and Sciarra (*516*) applied gas chromatography to the analysis of mixtures of oils in aerosol formulations. Zolotovich and Hickethier (*726*) found that earlier selections of desirable plants for breeding improved varieties of essential oil bearing plants could be made with the aid of gas chromatographic examination of small amounts of material from individual plants. Hayashi *et al.* (*212*) reported certain catalytic effects due to the support substance in gas chromatography. Such effects can lead to the erroneous interpretation of results.

Gas chromatographic investigations of groups of essential oils, which cannot readily be reviewed under the individual oils, were reported by several authors. Eight oils, the main constituents of which belong to different chemical classes, namely, oils of rosemary, cinnamon, anise, wintergreen, caraway, clove, eucalyptus, and chenopodium, were investigated by El-Deeb *et al.* (*142*). They proposed that the method be officially adopted in Egypt for the analysis of major constituents. Oils of anise, fennel, star anise, lavender, peppermint, and geranium were gas chromatographed by Schantz (*552*), who reported that methyl chavicol was found in oil of star anise, but not in oil of anise. The quantitative determination of the major components of the oils from *Artemisia porrecta, Artemisia absinthum, Juniperus oblonga,* and *Juniperus depressa* was reported by Goryaev, Lishtvanova, and Bazalitskaya (*179*). The oils of *Pinus sibirica, Larix sibirica,* and *Picea obovata* were chromatographed by Vol'skii *et al.* (*698*), who reported the presence of 18 monoterpene hydrocarbons in various proportions in each of the oils. Kuechler, Buechi, and Iconomou (*332*) reported quantitative data for menthol, neomenthol and menthyl esters in peppermint oil. They also compared synthetic and natural wintergreen oils and found that the natural oil does not meet the requirements of the Swiss Pharmacopeia V. The oils of *Baccharis rosmarinifolia, Escallonia subra, Fabiana imbricata, Myrceugenia exsucca,* and *Laurelia serrata,* all obtained from plants native to the region of the Nahuel Huapi National Park, were investigated by Montes (*395, 396, 398, 399*).

Simultaneous dual column gas chromatography was achieved by Mitzner and Jones (*388*) by placing a tee immediately in back of the injection port of a commercially available apparatus.

A practical approach to a standard qualitative characterization of essential oils by programmed temperature gas chromatography was proposed by Rasquinho (*506*) after careful trial of various conditions.

Preparative gas chromatography using large-bore open tubular columns and spring packed columns was found efficient for the separation of terpenoid compounds for subsequent infrared and proton magnetic resonance analysis by Teranishi, Flath, and Mon (*658*). Teranishi *et al.* (*659*) also described simple and efficient devices for the collection of 0.5 to 5 and 5 to 100 mg of chromatographically purified materials. Beroza (*41*) used another simple trap for transferring µl quantities to other instruments. The trap allows direct addition of an internal standard and may be modified to retain high-boiling fractions. A method for evaluating the efficiency of preparative gas chromatographic columns based on effective peak number was devised by Mitzner and Jones (*389*). Nano, Sancin, and Martelli (*412*) illustrated an improved method for collecting gas chromatographic fractions, and for using thin-layer chromatography in their identification.

Reaction gas chromatography techniques were comprehensively discussed by Beroza and Coad (*42*), and Beroza and Sarmiento (*43*) described a reaction gas chromatography apparatus and procedure for the instantaneous hydrogenation of a variety of unsaturated essential oil components. Hoff and Kapsalopoulou (*236*) used syringe reactions for the identification of low-boiling substances.

A microreactor for hydrogenation, dehydrogenation, esterification, isomerization, oximation, and boration was utilized by Osokin, Fel'dblyum, and Kryukov (*465*) to separate and identify alcohols, oxides, and carbonyl compounds by reaction gas chromatography. Nigam and Levi (*429*) used Se or S as the dehydrogenating agent for sesquiterpenes in a gas chromatographic procedure.

Combinations of other techniques together with gas chromatography have been employed with increasing frequency and success. Such a combination was used by Tsubaki, Nishimura, and Hirose (*677*) to analyze the oils from *Aster indicus, Artemisia princeps,* and *Ambrosia elatior.* Fractional distillation prior to gas chromatography was followed by investigation with infrared, nuclear magnetic resonance, and ultraviolet spectroscopy, and mass spectrometry. Many constituents of the oils were quantitatively determined. Ricciardi and Cassano (*512*) examined several oils from plants of the Argentine seacoast relying primarily on gas chromatography and infrared spectrometry. Broderick (*72*) discussed the assistance which may be rendered to gas chromatographic identifications by the sense of smell, and Ottoson and von Sydow (*467*) compared gas chromatographic peaks with the electrophysiological responses of the olfactory membrane of a decapitated frog and found them to be in substantial agreement.

Column and thin-layer chromatography used by Koul and Nigam (*320*) to isolate main components from the oils of caraway, lemongrass, citronella, dill seed, and patchouly. Retention on chromatographic as well as gas chromatographic columns and R_f values on thin-layer plates for 33 of the chief constituents of essential oils were recorded by El-Deeb, Karawya, and Wahba (*141*).

The use of thin-layer chromatography continues to increase, both by itself and in combination with other techniques, and many improved methods of developing thin-layer chromatographs have been reported. Among these is the use of gases followed by pyrolysis for the development of organic compounds as described by Heibrink (*213*). Jork (*275*) used a direct method of photographing the remission spectrum directly from the chromatoplate instead of scrapping or eluting the spot. Sawicki, Stanley, and Johnson (*551*) cut the chromatogram so that spots could be spectrofluometrically analyzed. Five different detection agents were employed by Tyihak and Vagujfalvi (*680*) in the thin-layer chromatographic analysis of the essential oils from German chamomile, milfoil, wormwood, estragon, fennel, and three varieties of mint. Karawya and Wahba (*290, 291*) separated the main constituents of anise, sandalwood, orange, orange flower, bergamot, and lavender oils. Nigam, Nigam, and Levi (*439*) published data of *Rf* values and color reactions from development agents which may be used to identify many essential oil components. Their method can also be used to detect sophistication or to determine the botanical and geographical origin of essential oils. Thin layer separations of the oils of rue and rose, the concrete of civet, and their clathrates were run by Bhatnagar (*51*). Bolado and Montes (*66*), and Figueroa and Montes (*150*) applied thin-layer chromatography to the separation and identification of the principal components of the oils from Spanish boldus, eucalyptus, pennyroyal, pine, cinnamon, laurel, and salvort. The results indicate that the Spanish oils are comparable to oils from other sources. Thin-layer chromatography was also used by Karawya and Wahba (*292*) for the analysis of oils of eucalyptus and rosemary, and by Zielinska-Sowicka and Szepczynska (*725*) for identification of components in the essential oils listed in the Polish Pharmacopoeia. The *Rf* values of some common oxygenated terpenes were determined by Ikan and Meir (*252*) and by Guven (*203*). Vernin (*692*) detailed experimental conditions for the thin-layer separation and quantitative analysis of various isolates and essential oils. The *Rf* values obtained were tabulated. Functional group reactions, either directly on the plate or in capillaries prior to spotting, were applied to thin-layer chromatography by Mathis (*373*).

Paper chromatography was used by Richter and Muscholl (*515*) to isolate linalool, geraniol, citronellol, and linalyl acetate from oils of lavender, lavandin, geranium, citronella, and melissa. The quantities of the isolates were estimated by colorimetry.

Infrared spectroscopy was used by Vega Morales (*687*) to detect adulterants in the oils of lavender, peppermint, lemon, and orange.

The application of ultraviolet spectroscopy was critically studied by Vernin (*693*). He stressed its importance in the elucidation of molecular structure. The technique was utilized by Koul and Nigam (*322*) for the quantitative determination of a number of components in various oils.

X-ray diffraction powder patterns of 17 isolates and aromatic chemicals were presented by Levi and Cloutier (*347*).

The application of the Raman effect to the analysis of essential oils was studied by Bertolotti (*44*).

A technique for measuring optical rotatory dispersion at various temperatures was illustrated by Horsman and Emeis (*241*) by application to several isolates including *d*-camphor, *l*-α-phellandrene, and *d*-2-butanol.

The quantitative colorimetric estimation of limonene, eugenol, citral, and cineole was achieved by Koul and Nigam (*321*) with the adaptation of known qualitative color reactions. Phenols, aromatic amines, indoles, and certain compounds having conjugated double bonds were detected with ferric ferricyanide by Pesez and Bartos (*475*), and Counts (*116*) also used color reactions to identify many hydroxyl compounds.

Mass spectrometry was applied to structural problems by Weinberg and Djerassi (*706*), who studied the rearrangements of simple terpenes on electron impact.

Two oximation methods were compared in their reaction with carbonyl and other oxygenated functional groups by Aarna, Lippmaa, and Paluoya (*2*). Aarna and Lippmaa (*1*) also studied the acid and base catalyzed acetylation of phenols.

The optical activity of terpenes was related to the location of the growing plants and the conditions of biosynthesis by Pigulevskii (*482*).

Bulgarian oils of peppermint, geranium, lavender, jasmine, and turpentine were examined for their major components and quality by Lozzi (*353*). Northwest Himalayan oils including those from *Ligustum elatum, Heraclium canescens, Sium latijugum,* and *Caucalis anthriscus* were analyzed by Sarin (*549*), who identified their major components. Likewise, Peyron (*477*) analyzed and described several little known essential oils of potential interest for perfumery, including lentiscus oil, Borneo lemon oil, limonette petitgrain oil, and petitgrain mandarin oil.

The cohobated oils obtained by charcoal absorption from the distillation of chamomile, lavender, and peppermint were examined by Juhasz *et al.* (*279*). They (*277*) also investigated oils of lavender and peppermint buds obtained by salting out the cohobation waters. A combined extraction and distillation was employed by Topalov, Kalichkov, and Khadzhiiski (*670*) to obtain oils from cumin, dill, and celery. The same authors (*671*) found that the method yielded better qualities of oil from coriander, anise, and fennel than is normally obtained by distillation alone.

A method for determining the essential oil content of plant material, based on the refractive index of the distillate, was worked out by Podlubnaya *et al.* (*488*). Teppeev and Goryaev (*655*) described a laboratory steam-distillation apparatus which may be used for the same purpose. The change in oil content under air drying and anaerobic conditions was studied by Mush (*410*) for rose, Indian mustard, sage, and lavender.

The analysis of oleoresins by instrumental methods commonly used for essential oils was recommended by Stahl (*612*).

Individual Oils. Oil of *Abies alba* was found by Soroczynska and Jonczyk (*602*) to contain santene, α- and β-pinene, camphene, and limonene. The yield of oil from branches of *Abies nephrolepis* and *A. sachalinensis* was shown by Tomchuk (*667*) to be adequate for commercial use.

The oil of *Achillea fragrantissima* was analyzed by Shalaby and Richter (*569*). Among many other substances, they found linalool and its esters, α-terpineol, and 3-nonanol.

Aframomum mala oil was investigated by Eglinton and Hamilton (*140*). They did not find the C-16 hydrocarbons, α- and β-kayene, which were claimed to be present.

Agastache formosana oil contained, among many other constituents, large amounts of *l*-isomenthone and *l*-pulegone according to Fujita, Ueda, and Fujita (*165*). Fujita and Fujita (*163*) also analyzed oil of *Agastache rugosa.* Some of the constituents identified were 1-octen-3-ol, *p*-methoxybenzaldehyde, *p*-methoxycinnamaldehyde, and methyl chavicol, the main component.

The lower boiling "thymene" fraction of ajowan oil was shown by Bhargava and Haksar (*50*) to consist largely of α- and β-pinene. Kartha and Asim Kidwai (*296*) modified the iodometric technique for the estimation of thymol to permit the analysis of micro and semimicro quantities of the oil.

A constituent of *Ambrosia cumanensis,* cumanin, was shown to be a tricyclic sesquiterpene lactone by Romo, Joseph-Nathan, and Siade (*524*). Herz, Chikamatsu, and Tether (*217*) investigated the composition of *Ambrosia ilicifolia.* The structure of peruvin, another sesquiterpene tricyclic lactone, which was found in *Ambrosia peruviana* was established by Joseph-Nathan and Romo (*276*).

The terpene and sesquiterpene fractions of the oil from *Amorpha fruticosa* was analyzed by Motl, Romanuk, and Herout (*403*). The composition of these fractions was established and the structure of a new bicyclic sesquiterpene, (−)-γ-amorphene, was elucidated. Georgiev (*170*) noted the changes which took place in the oil obtained from this botanical upon aging.

The oil of *Angelica archangelica* was investigated by Nigam, Paul, and Handa (*441*), who separated many components by fractionation, and by Klouwen and ter Heide (*308*) who quantitatively determined pentadecanolide and hexadecanolide. They (*309*) also elucidated the composition of the terpene fraction. The accumulation of coumarins in the fruit was studied by Dranitsyna and Denisova (*136*), and the structure of archangelicin was established by Nelsen and Lemmich (*427*). The lactones in oil of *Angelica sachalinensis* were chemically studied by Nikonov and Pimenov (*449*). Pimenov (*486*) also conducted an investigation of the seasonal changes in coumarin content of some Far Eastern species of Angelica.

Anise seed oil was studied with thin-layer chromatography by Szasz and Szasz (*631*), who observed changes in the content of 4-allyl anisole, anisaldehyde, and methyl chavicol upon aging and oxidation of the oil. Topalov, Kalichkov, and Khadzhiiski (*668*) found that steam distillation did not get all the volatile oil out of anise seeds because the volatile oil is dissolved in the glyceride oil of the endosperm and is therefore partially fixed. El-Hamidi and El-Gangihi (*143*) compared the yield and quality of oil from anise seed obtained from various parts of the world with the yield from seed grown in Egypt.

The loss in yield and quality of oil resulting from the aging of Bulgarian anise seed was studied by Georgiev (*169*), and the quality and quantity of oil obtained under different conditions of steam and water distillation were reported by Nguyen-Nang-Winh and Polyakov (*424*). Suprun (*624*) quantitatively determined anise oil in salmiak-anise drops.

Oil of star anise was exhaustively investigated by Porsch, Farnow, and Winkler (*493, 495*). Eight new constituents, previously not identified in the oil, were reported, while 13 other substances reported by earlier investigators could not be found. Concha and Wungwira (*109*) determined the yield, properties, and some main components of a freshly distilled oil.

An oil from the blossoms of *Anthocephalus cadamba* was analyzed by Bahadur, Gupta, and Nigam (*26*), and geraniol, linalool, and their acetates were found in high percentages.

The oils from eight species of Artemisia were examined as to their content of α-pinene, sabinene, cineol, α- and β-thujone by Goryaev and Gimaddinov (*176*). Several constituents of *Artemisia afra* oil were identified by Cardoso (*94*), and Manjarrez and Medina (*367*) identified the major components from the oils of *A. mexicana* and *A. klotzchiana*. Chandrashekar and Krishnamurti (*99*) isolated umbelliferone and genkwanin from *A. sacrorum*. The constitution of vachanic acid, a sesquiterpene hydroxy acid from the oil of *A. vachanica* was established by Kechatova *et al.* (*298*). Vashist and Handa (*685*) identified the main components of *A. vestita* oil.

The composition of oil of Araucaria was described by von Rudloff (*533*). He reported finding α- and β- eudesmol, γ-eudesmol, and also guaiol, elemol, araucariol and other minor components.

The content of oil of *Asarum europaeum* and its active principle, asaron, in the rhizomes and leaves was studied by Gracza, Csiszer, and Tatar (*188*).

The adulteration of oil of balm with other oils was detected by Phokas (*479*) with thin-layer chromatography.

Bulgarian basil oil was found to resemble closely the American-type oil of sweet basil according to the analysis of Ivanov, Tchorbadjiev, and Iordanov (*263*). The major components of red and white Egyptian basil oil were identified by Hoerhammer, Hamidi, and Richter (*235*).

Bellary leaf oil was obtained in a much better yield from young trees than from old trees in the experiments of Gowda (*186*).

Bergamot oil was characterized by the infrared and ultraviolet spectra of its volatile and nonvolatile fractions as described by Rotondaro (*531*). The technique also has application for lemon and orange oils. A combination of ultraviolet spectrophotometry and gas chromatography for the detection of adulteration of bergamot oil was demonstrated by Calvarano and Calvarano (*91*). Labruto and Di Giacomo (*341*) investigated the composition of the residue from the manufacture of bergamot oil and found bergaptene, as well as a number of terpenes. Muller (*407*) described the production, use, properties, and analysis of bergamot oil.

Bursera graveolens oil was studied by Crowley (*117*). Among other, more usual components, he also found one that is thought to be unique as a plant constituent, being a 2,3-seco triterpene.

The structures of cacalol and cacalone, found in *Caclea decomposita* oil, were established by Romo and Joseph-Nathan (*523*).

The stereochemistry of acorone from calamus oil was clarified by McEachan, McPhail, and Sim (*357*), and the quantitative estimation of asarones in Indian calamus oil by column chromatography was reported by Chopra, Vashist, and Handa (*104*).

Some main components of the oils from two Callistemon species grown in Ghana were estimated by Talalaj (*642*).

Safrole was determined in red camphor oil by Tanaka and Ono (*649*), using infrared spectrometry, after first removing the interfering substances with thin-layer chromatography. Lishtvanova and Goryaev (*349*) analyzed the oil for camphor, using a gas chromatographic technique.

The oxidation products of limonene from caraway oil were studied by Preuss (*499*), and theories were proposed for their natural formation.

Cardamom oil was examined by fractionation and gas chromatography by Nigam *et al.* (*437*). Over 90% of its composition was quantitatively established.

The composition of oils from cultured and wild carrot seed were compared by Stahl (*608*), and Pigulevskii, Kovaleva, and Motskus (*485*) reported the physical and chemical properties of oil of wild carrot seed from eleven districts of the Soviet Union. The composition and yield of oils from seed grown in Moldavia was given by Meshcheryuk (*378*). Levisalles and Rudler (*348*) proved the structure of carotol, and Biernacka *et al.* (*53*) studied the molecular structure of carotol and daucol, both of which are sesquiterpene alcohols found in carrot seed oil.

Oil of catnip was found by Sakan *et al.* (*546*) to contain, in addition to matatabilactone, three new lactones related to nepetalactone, all of which were quite attractive to cats.

The physico-chemical properties of oil of *Cedrela mexicana* from Ghana were reported by Talalaj (*645*). Kapadia *et al.* (*287*) established the structure of mustakone and copaene from *Cedrela toona* wood oil.

From the oil of Himalayan cedarwood, Bisarya and Dev (*57*) isolated allohimachalol, a new type sesquiterpenoid, and established its structure; and Manzoor-I-Khuda and Saelque (*368*) isolated dewardiol and dewarenol.

The steam distillation residue from chamomile was extracted by Juhasz, Udvary, and Tyihak (*278*), and they reported obtaining nearly 0.5% of an oil containing more alcohols and acids than the distilled oil. Blazek and Stary (*59*) checked the oil content and chamazulene in wild and cultivated chamomile. Nobilin, a sesquiterpene lactone with a ten-membered ring, was isolated from Roman chamomile and characterized by Benesova, Herout, and Sorm (*37*).

Gas chromatographic and mass spectrometric studies of the volatiles from chives led to the identification by Wahlroos and Virtanen (*703*) of Tiglaldehyde, 2-methyl-2-pentenal, and several sulfides.

A paper chromatographic technique for the rapid detection of adulteration of oils of Ceylon cinnamon, cassia, cinnamon-leaf, and mixtures containing these oils was developed by Richter (*514*). Kuechler, Buechi, and Iconomou (*331*) ran gas chromatographic analyses of cinnamon water, *Cinnamomum cassia* oil, and *C. chinensis* oil and compared the resulting curves.

The oils from two species of camphor trees, oils of *Cinnamomum glanduliferum* leaves and *C. bodinieri* stems, were examined by Tsai, Ting, and Nieh (*676*), and many major components were quantitatively determined. Other camphor tree oils recently investigated by Tsai *et al.* (*674*) were two leaf oils from subspecies of *Cinnamomum parthenoxylon* and two from *C. molle*.

The formation of essential oil and its components in Ceylon citronella was observed by Sadgopal (*543*), who defoliated and distilled the plant material at monthly intervals. Rajamani *et al.* (*504*) described the properties of citronella oil produced in India. Elemol from the tail fraction of Java citronella oil was stereochemically clarified by Wagh, Paknikar, and Bhattacharyya (*700*).

Citrus oils were subjected to gas chromatographic analysis by Farnow, Porsch, and Raupp (*147*), who, in the investigation of lemon and bergamot oil, used a thermal conductivity detector with packed columns and a flame-ionization detector with various capillary columns. Kesterson *et al.* (*301*) used gas chromatography to identify different species and varieties of citrus from an examination of their leaf oils. To accomplish this they determined 33 compounds in many leaf oils. This work was further expanded by Pieringer, Edwards, and Wolford (*481*), who explored in addition the use of infrared and ultraviolet spectrophotometry for the differentiation of 8 citrus leaf oils and 2 oils from sour orange. They concluded that infrared and ultraviolet spectroscopy appear to be limited to the identification of species. Navarro (*414*) used infrared spectroscopy to differentiate between natural and imitation citrus oils. D'Amore and Calapaj (*120*) used thin-layer chromatography to separate, and ultraviolet spectroscopy to identify fluorescent substances such as coumarins and flavones in various citrus oils. The terpene and sesquiterpene portions of many citrus oils were exhaustively analyzed by Hunter and Brogden (*243*). Wide differences in the composition of the various citrus oils were observed. Hunter and Moshonas (*247*) used gas chromatography and mass spectroscopy to identify the alcohols in grapefruit, lemon, lime, and tangerine. A rapid method for determination of acidity and aldehyde content in citrus oils was developed by Monselise (*394*). Experiments by Ting and Newhall (*666*) indicated that the antioxidant in citrus fruit is a tocopherol. Di Giacomo and Rispoli (*133*) investigated the protective effect of flavonoids in citrus beverages against oxidation catalyzed by ultraviolet light.

The physico-chemical properties of an oil distilled from *Clausena anisata* were recorded by Talalaj (*643*).

The change in the properties of clove bud and stem oils resulting from different conditions of distillation were studied by Belcher (*36*).

A greenish, readily oxidizable oil of unpleasant odor was obtained in a very low yield from *Comarum palustre* by Naumchik and Rozentsveig (*413*). Several components were identified.

Twin solvents and a nephelometer were employed by Gurvich (*200*) for the microdetermination of linalool in coriander oil. Another microdetermination, depending on hydration and absorbance was devised by Chernysheva (*102*), and Lassanyi (*344*) estimated linalool by measurement of the spot area on a thin-layer chromatoplate. Some differences in the essential oils from nine varieties of coriander were reported by Dublyanskaya, Panfilova, and Chernysheva (*137*), and seasonal changes in the oil were reported by Luk'yanov and Mukhanlva (*356*). Avakova, Kustova, and Shchedrina (*22*) analyzed the terpenes from coriander oil by the usual combination of procedures and identified many components. *trans-2*-Tridecen-1-al was isolated, characterized by ultraviolet, infrared, and nuclear magnetic resonance spectra, and synthesized by Reisch, Schratz, and Qadry (*509*), who found it to be the flavor-determining component of coriander.

A new sesquiterpene acid from costus root oil, costic acid, was structurally characterized by Bawdekar and Kelkar (*34*).

The oil from the wood of *Cryptomeria japonica* was found to be rich in sesquiterpenes by Nagahama (*411*), who identified delta-cadinene, calamene, calacorene, and copaene.

Egyptian cumin oil was analyzed using thin-layer chromatography by El-Hamidi and Richter (*144*), who identified many of its constituents.

Oil of *Cyperus articulatus* was extensively investigated by Couchman, Pinder, and Bromham (*114*). Employing diverse instrumental and chemical techniques, they identified many components. Cyperenone, a new sesquiterpene ketone from oil of *C. scariosus*, was isolated and characterized by Nigam (*428*).

Davana oil was discussed by Gowda and Ramaswamy (*187*), and data on yields and quality were included.

The oil from *Descurainia sophia* seed was chemically analyzed by Baslas (*32*), who quantitatively determined its content of various sulfides and isothiocyanates.

A thin-layer chromatographic technique described by Betts (*45*) can be used to distinguish Indian dill from the dill species cultivated in America and other parts of the world and to detect mixtures of the two types.

Several sesquiterpenes from *Dictyopteris divaricata* were isolated and identified by Irie, Yamamoto, and Masamune (*254*).

The yield of erigeron oil from plants cultivated in Poland was determined at different stages of growth by Kostecka-Madalska and Polanowski (*319*).

Oil of *Escallonia illinita* was distilled by Rojas (*522*) from fresh and dried plant material, and its properties and odor were described.

Oil of *Eucalyptus globulus* from Angola was analyzed by Cardoso do Vale (*95*) and found to be of similar composition to oils from the same plant from other areas. Angjeli (*9*) determined the oil content and cineol percentage of *Eucalyptus globulus* and *E. rostrata* grown in Albania. Porsch, Farnow, and Winkler (*494*) identified the components constituting the major portion of the oil from *Eucalyptus staigeriana*. Fernandes Costa (*149*) determined several major constituents of the oils from Eucalyptus species grown in Angola, and Elkeiy *et al.* (*145*) did the same with Eucalyptus species cultivated in Egypt. The essential oil content of some Eucalyptus species growing in Nigeria was investigated by Osisiogu (*464*).

The quantity of fennel oil in various parts of the fruit was chekced by Topalov, Kalichkov, and Khadzhiiski (*669*), and Vlakhov, Ognyanov, and Tsankova (*697*) determined much of the composition of the oil from Bulgarian sweet fennel.

A new sesquiterpene lactone, gaillardilin, was isolated from two gaillardia species, and its structure was established by Herz *et al.* (*221*).

In oil of galbanum, Teisseire (*652*) identified and estimated the quantity of ten hydrocarbons, and four terpene and five sesquiterpene alcohols.

The oil of garlic was analyzed with thin-layer chromatographic methods by Schultz and Mohrmann (*561, 562*). Many of the components were separated and identified.

Oil of geranium from plants grown in Albania was analyzed by Angjeli (*10*), who reported some of its major components and its physico-chemical properties. The constitution of two new sesquiterpene ketones from geranium bourbon oil, furopelargones A and B, was established by Lukas *et al.* (*354*). The complex combinations of techniques employed are described in detail. Giannotti (*172*) isolated two monoterpene diols from the oil and determined their structure by infrared, mass and nuclear magnetic resonance spectra. Goto (*182, 184*) studied the effect of *Aspergillus niger* on the composition of geranium oil, and also the changes caused by treatment with ion-exchange resin. The structures of α- and β-bourbonene, tricyclic sesquiterpene hydrocarbons, were established by Krepinsky, Samek, and Sorm (*327*) after isolating them from the oil. Corbier and Teisseire (*112*) reported two oxides, 2,2,6-trimethyl-6-vinyl-tetrahydropyran and 2-methyl-2-vinyl-5-isopropenyl-tetrahydropyran in the oil of geranium, and subsequently they (*113*) also identified *l*-neoisomenthol and *l*-copaene.

New trace components in oil of ginger were reported by Nigam, Nigam, and Levi (*440*). Gas chromatographic and infrared criteria of identity were determined, especially for two new alcohols, and these data are important for establishing the authenticity of ginger oil. Talalaj (*647*) described the properties of an oil distilled from roots grown in Ghana. Connell and Sutherland (*110*) isolated a new natural sesquiterpene, *l*-β-sesquiphellandrene. Nigam and Levi (*431*) found that a new sesquiterpene alcohol, β-humulen-7-ol, is formed when oil of wild ginger is chromatographed on an Al_2O_3 column.

Oil of ginger grass was found to contain several previously unreported components, *trans-p*-menth-8-ene-1,2-diol, *cis-* and *trans*-carveol, *cis-* and *trans*-dihydocarvone, piperitenone, limonene oxide, and an aromatic compound. The analysis was conducted by Nigam, Nigam, and Levi (*438*), using primarily gas chromatography and infrared spectroscopy.

The composition of the high-boiling fraction of *Glycyrrhiza triphylla* oil was established by Goryaev, Yurina, and Dembitskii (*180*), employing column and gas chromatography, infrared spectroscopy, and a study of the chemical and physical properties of the separated components.

A new flavor constituent of grapefruit oil, nootkatone was isolated by MacLeod and Buigues (*360*). The sesquiterpene ketone had a distinct taste at 100 ppm, and was also found in bergamot, lemon, lime, orange, and tangerine oils. Its chemical structure was further clarified in a later paper by MacLeod (*359*), and Kesterson *et al.* (*300*) studied the nootkatone content of Duncan grapefruit oil as related to fruit maturity. Hunter and Brogden (*244*) synthesized it by converting valencene obtained from orange oil. The coumarins in grapefruit oil came under intensive study by Fisher and Nordby (*152*) who found nine, five of which had not been previously reported. In a later paper they (*153*) described two of the new coumarins in detail.

Hamanasu absolute oil was subjected to chemical and chromatographic analysis by Nishimura *et al.* (*450*). Many of its constituents were found to be the same as those of Bulgarian rose oil, but were present in different proportions.

The components of the essential oil of water hemlock were identified by Kozhin and Kovaleva (*325*). Though the plant is one of the most poisonous, the oil contains no toxic ingredients.

Heracleum candicans oil, investigated by Sharma, Nigam, and Handa (*574*), contained carveol acetate and dihydrocarveol among other substances identified. The same constituents were also among those identified in *Heraculeum canescens* oil by Sharma and Handa (*573*).

Hiba wood oil was investigated by Ito *et al.* (*260*), who isolated elemenal, γ-cuparenol, and a C-14 ketone. The structures of the first two were established. Thujaplicin and thujopsene were quantitatively separated from the oil with nitromethane extraction by Ito and Abe (*259*). Kitahara and Yoshikoshi (*305*) established the structure of hibaene, and Dauben and Oberhänsli (*124*) isolated and proved the structure of two isomeric cuprenenes.

Hinoki leaf oil was exhaustively analyzed by Thomas (*662*) who identified nearly all the constituents which make up the oil. He (*661*) subsequently characterized 2-methyl-5-isopropenylanisole, a previously unknown ether isolated from the oil, and together with Stoll and Palluy (*663*) he established the structure of another isolate, thujopsenal, and identified elemol. Hayashi, Yano, and Matsuura (*211*) elucidated the composition of the sesquiterpene fraction, and Fujise *et al.* (*162*) identified the components of both hinoki leaf and wood oil by chemical and physical means. *d*-Longifolene, *d*-γ-cadinene, calamenene, and thunbergene were found in the leaf oil.

Oil of hops continued to be the subject of intensive investigation. Burton and Stevens (*82*) isolated hulupinic acid and hard resin from the oil. Burton, Elvidge, and Stevens (*81*) further clarified the structure of humulinic acid and related substances. Buttery, Black, and Kealy (*83*) found 87 constituents, many of which they identified, in the oxygenated fraction of hop oil. A 192 ft. × 0.01 in. stainless steel column was used, and half the effluent was led to a mass spectrometer, the rest being passed to a vacuum ionization gauge detector. Buttery, Lundin, and Ling (*85*) reexamined the sesquiterpene hydrocarbons, and in addition to confirming previous findings, identified three new substances, γ-cadinene, selina-3,7(11)-diene and selina-4(14), 7(11)-diene. Buttery, Black, and Ling (*84*) also identified methyl geranate in the oil, using infrared absorption and mass spectra. Methyl dec-4-enoate and methyl deca-4,8-dienoate are two esters present in hop oil which Nickerson and Likens (*426*) indicated are transesterified in beer and wort to the ethyl esters. The structure of two isohumulinones were elucidated by Ashurst and Whitear (*16*), who (*17*) also studied the effect of hop resins on beer flavor. Ashurst and Elvidge (*15*) elucidated the structure of the autoxidation products of humulone. Spetsig (*604*) isolated and characterized two isohumulone isomers, and McPhail and Sim (*362*) determined the stereochemistry of humulene by x-ray analysis of its silver nitrate adduct. Roberts and Stevens (*520*) found through chromatography and preparative gas chromatography that the major unsaturated esters of hop oil were straight-chain compounds, although a small quantity of methyl geranate was present. Six flavonoid substances, three of which were identified, were isolated by Bhandari (*49*), who showed that these same substances can be chromatographically identified in beer. Brown, Burton, and Stevens (*73*) elucidated the structures of adhulupone and analogues of kulupone, Alderweildt *et al.* (*3*) characterized the isomerization products of humulone, Laws (*345*) established structures for the lupones, and Ashurst *et al.* (*14*) further clarified the structures of hulupone and humulinic acid. Guadagni, Buttery, and Harris (*192*) studied the odor intensities of the components of hop oil.

Oil of horsemint was carefully distilled under a CO_2 atmosphere by Scora (*566*). Most of its composition was determined with the use of two different gas chromatographic columns and infrared spectrometry.

From the oil of *Iva microcephala*, Herz, Hoegenauer, and Romo de Vivar (*218*) isolated and characterized a new tricyclic sesquiterpene lactone, microcephalin, and Herz, Romo de Vivar, and Lakshmikantham (*222*) isolated and established the structure of two new guaianolides, pseudoivalin and dihydropseudoivalin.

The essential oil of jasmin was subjected to repeated thin-layer chromatography by Demole (*126*), who also employed diffusion from one chromatoplate to another and studied the diffusion rates of various components. The recipient plates were eluted and the eluate was analyzed by gas chromatography. Many constituents were thus quantitatively determined. Demole, Willhalm, and Stoll (*127*) determined that the ketolactone discovered by Naves and Grampoloff was the lactone of 5′-hydroxyjasmonic acid. Hill and Edwards (*231*) established the absolute configuration of methyl jasmonate. Calvarano (*90*) found that the composition of 26 absolute essences of jasmine from Calabria corresponded closely with the composition previously reported. Muller (*406*) discussed the yield and composition of jasmine oil.

The terpene fraction components of three juniper oils from *J. communis*, *J. phoenicea*, and *J. macrophoda*, were quantitatively reported as determined with gas chromatography by Klein and Farnow (*306*). Similarly, Karlsen and Svendsen (*293*) reported the composition of the monoterpene fraction of Norwegian juniper oil, noting in particular the relatively high proportion of sabinene. Couchman and von Rudloff (*115*), in a gas chromatographic investigation of the oil from creeping juniper, also found *d*-sabinene to be the major component, and reported many other constituents, including methyl citronellate. Sood (*596*) analyzed the oil from *Juniperus macropoda* from India. Hirose (*232*) confirmed the existence of nootkatinol in the oil from *Juniperus rigida* wood. The composition of the oil from *Juniperus semiglobosa* was quantitatively determined by Ignatova *et al.* (*250*), and von Rudloff and Couchman (*535*) quantitatively reported the composition of another juniper oil from Rocky Mountain plants. They found its composition to vary widely from that of oils obtained from four local ornamental junipers.

The oil of Labdanum was studied by Buenadicha (*78*), who reported physicochemical properties and ultraviolet spectra, and also separated the acidic portion of the oil wherein he identified many individual acids.

The oil of laurel leaf was examined by Chow, Motl, and Lukes (*106*) who identified several new compounds therein. Teisseire (*653*), however, in a concurrent investigation, could not substantiate the presence of all of the reported new compounds. Pruidze (*501*) distilled oil from fresh leaves, aged stems and leaves, and tailings, and compared the properties of the resulting oils.

The composition of over 20 different lavender oils was investigated by Kharchenko (*302*) who ascertained many of their main components. The constituents of Bulgarian lavender oil were quantitatively determined by Ognyanov *et al.* (*457*), who found that the oil differs from the French oil by containing about 12% 1-terpinen-4-ol. Peyron and Benezet (*478*) reported finding hexyl butyrate in oils of lavender and lavandin for the first time. Many other components, previously reported, were also determined. A new diterpene alcohol was isolated from lavender oil by Kal'yan and Lazur'evskii (*284*). Goto (*183*) incubated lavender oil with *Aspergillus niger*, and found, among other changes, a partial allylic rearrangement of geraniol to *d*-linalool. Calvarano (*89*) investigated lavender oil from Calabria and found it comparable with good quality oils from other regions. He also reported two new components, γ-terpinene and nonanal. Taddei (*632*) investigated the yield of oil as related to the time of day at which the plant material was harvested.

The oils of lavandin from plants growing at various elevations were compared by Calvarano (*88*). Each oil was quantitatively analyzed and the presence of sabinene, myrcene, 3-carene, and γ-terpinene was reported, possibly for the first time. Vinot and Bouscary (*694*) studied the optical rotation of lavender oils, and Devetak and Cenci (*129*) gave physico-chemical properties of lavender oils produced in Yugoslavia.

A lemon oil from Argentina was chromatographed by DiGiacomo and Rispoli (*134*) who quantitatively established the composition of its terpene portion. Di Giacomo, Pennisi, and Rispoli (*132*) also determined the oxygen-containing substances from Sicilian lemon oil. In this study a new modification of the gas chromatographic technique was employed. Rispoli and Di Giacomo (*518*) employed a similar technique to investigate the sesquiterpene fraction of Sicilian lemon oil, and found it to consist of α-bergamotene, caryophyllene, bisabolene, and small quantities of some other unidentified components. Pennisi and Di Giacomo (*474*) compared the yields of oil from various types of fruit, and reported their properties and composition, and Rispoli, Di Giacomo, and Tracuzzi (*519*) developed a rapid method for the infrared spectroscopic determination of the quality and freshness of lemon oil. D'Amore and Sergi (*121*) used thin-layer chromatography to identify the chalcones in lemon oil, and MacLeod, Lundin, and Buigues (*361*) identified benzyl ether as an adulterant in an abnormal oil.

The yields and physico-chemical properties of lemongrass oil obtained at different seasons from grass grown in Ghana were reported by Talalaj (*639*). The citral content was normal.

The composition of the oxygenated fraction of oil of *Libanotis transcaucasia* was reported by Pigulevskii and Bakina (*483*). Among the compounds isolated were acorenone, bisabolol, farnesol, elemicin, and 3,4,5-trimethoxybenzaldehyde.

From the oil of *Ligularia sibirica* Ishii, Tojio, and Minato (*257*) isolated ligularol and ligularone and established their structures.

Many components of the oil of *Ligustum elatum* growing in the Western Himalayan region were quantitatively determined by Talwar, Nigam, and Handa (*648*).

Constituents of an oil obtained from concrete of lily of the valley were identified by Wakayama and Namba (*704*).

The presence of p-α-dimethylstyrene in distilled lime oil and its absence in expressed lime oil was demonstrated by Loori and Cover (*350*), who also postulated a mechanism for its formation.

An oil from *Limnophila rugosa* was reported to have an anise-like odor by Gulati and Duhan (*196*). Its properties were given.

Mexican oil of linaloe was analyzed with thin-layer chromatography by Rodes, dos Santos, and Harada (*521*). Indian linaloe seed oil was distilled by Gowda and Ramaswamy (*171*), who ascertained the physico-chemical properties of the oil.

Detailed investigation of the components of *Lindera strychnifolia* was continued by Takeda and his coworkers. Takeda, Minato, and Ishikawa (*636*) isolated and determined the structure of linderalactone and isolinderalactone. Takeda *et al.* (*637*) identified two new sesquiterpenes, lindestrene and linderene acetate; Takeda, Ikuta, and Miyawaki (*633*) revised the accepted structure of linderene; and Takeda *et al.* (*634*) further proved this structure by studying the dihydro derivative.

Oil of *Lippia multiflora* from Ghana was distilled by Talalaj (*640*) who reported some of its properties.

An oil from the berries of *Litsea citrata* was distilled by Sood (*599*), who reported its properties and some of its components. Gupta and Garg (*198*) identified the major components of an oil from the seeds of *Litsea consimilis;* Fuerst and Feustel (*160*) identified the carbonyl compounds in oil of *Litsea cubeba* by paper chromatography of their dinitrophenylhydrazones; and an oil from *Litsea lanuginosa* was obtained by Sood (*601*), who determined its properties and main constituents.

Several components from the oil of *Lochnera Rosea*, including 2-heptanol, were isolated and identified by Oliveros-Belardo *et al.* (*463*).

The volatile fraction of mandarin oil was analyzed by Kugler (*333*). Using gas chromatography, he identified 48 compounds. Verderio and Venturini (*689*) subjected the oil to thin-layer chromatography under two different solvent systems.

An oil distilled from marihuana was analyzed by Nigam *et al.* (*436*). Linalool, *trans*-linalool oxide, sabinene hydrate, terpinen-4-ol, and caryophyllene oxide were among 18 previously unreported constituents which they identified.

Prepared mate yielded an oil which Montes (*397*) investigated. Many of its components, especially the acids, were identified.

Melaleuca leucadendron oil was examined by Sood (*600*). Its properties, composition, infrared spectrum, and gas chromatogram were given.

The essential oils from 11 species of the genus Mentha were analyzed as to chemical composition by Handa *et al.* (*208*). The occurrence of isopiperitone as a major constituent of *M. rotundifolia* and the presence of several terpenoids as trace constituents in Mentha species were reported for the first time. Nikolaev (*447*) studied the variation in the terpenoid composition of oils from various hybrid Mentha plants and related his findings to the effect of cross breeding. With a view to improving the organoleptic qualities of the oil from Chinese menthol mint, Nikolaev and Semenova (*448*) examined the oil from self-pollinated plants and found that plants having rose colored blossoms gave an undesirable oil without l-menthol, while plants with white blossoms gave the desired characteristic oil. Shimizu, Karasawa, and Ikeda (*581*) found that pinocamphone is the major constituent of the oil from a new variety of mint, *Mentha aquatica*.

The oil from wild *Mentha arvensis* from Piedmont was shown by Sacco and Shimizu (*542*) to consist of two distinct varieties whose properties and composition they differentiated. Gupta (*199*) found that drying Japanese mint in the shade improves the quality of the oil. The classical distinction between *Mentha arvensis* and *M. piperita* is no longer tenable, since Nigam and Levi (*433*) reported the presence of menthofuran in oil of *Mentha arvensis*. Shimizu *et al.* (*580*) reported that the oil from a variety of European *Mentha arvensis* contains 3-octanone as the principal component. Chopra, Vashist, and Handa (*105*) disclosed the composition of oil of *Mentha sylvestris*, the main constituent of which was piperitol.

Oils of milfoil from many localities in Poland were checked for azulene content by Oswiecinska (*466*), and a great variation was reported in the percentage content of this component.

Monodora myristica oil from Ghana was distilled and its properties were ascertained by Talalaj (*644*).

The physico-chemical properties of an oil distilled from *Mosla ocymoides* were determined by Gulati (*195*).

The oil from mustard seed was shown by Kirk, Black, and Mustakas (*304*) to contain CS_2 and allyl thiocyanate. Barothy and Neukom (*30*) isolated p-hydroxybenzyl isothiocyanate from white mustard seeds.

Much of the composition of *Nardostachys jatamansi* oil was chromatographically determined by Shanbhag *et al.* (*570*).

Nepeta ciliaris oil was examined by Gulati and Shah (*197*), who found phenols and aldehydes, but no ketones, to be present. Singh (*590*) distilled the oil from plants growing wild in the Naini Tal district. He found the pleasant aroma promising for use in perfumery.

Neroli oil prepared from bitter orange trees grown in Calabria was investigated and described by Muller (*408*).

From Nutmeg oil Shulgin and Kerlinger (*584*) isolated methoxyeugenol and *trans*-isoelemicin.

The physico-chemical characteristics of an oil obtained from Indian *Ocimum canum* were determined by Dwivedi, Chandra, and Gupta (*138*). They also published the infrared spectrum of the oil.

An oil containing 72% eugenol was obtained by Kostecka-Madalska, Bankowski, and Kuduk (*318*) from plants which were a cross between *Ocimum gratissimum* and *O. methaefolium.* The main composition of oil of *Ocimum kilimandscharicum* was established with gas chromatographic analysis by Nigam, Handa, and Gupta (*435*). Talalaj (*641*) reported the physico-chemical properties of oil of *Ocimum viride* grown in Ghana.

The quantity of oil in onion and garlic tissues was photometrically estimated with a color reaction by Korableva and Potapova (*317*). Wilkens (*707*) isolated and identified several components of onion including the lacrymogenic compound thiopropionaldehyde s-oxide. Das and Rao (*122*) identified phenolic acids in the plant, and Saghir, Mann, and Yamaguchi (*544*) determined the volatiles in the oil as related to habitat, stage of growth, and plant part.

The terpenes and sesquiterpenes in orange oil were identified by Hunter and Brogden (*242*), using gas chromatography for separation, and mass and infrared spectroscopy for confirmation. Among numerous compounds, a new sesquiterpene, valencene, related to nootkatone, was identified. Attaway, Hendrick, and Wolford (*19*) described a technique for transferring the effluent from the gas chromatography of orange oil directly to a thin-layer chromatoplate, and compared it with another technique wherein the effluent is first condensed in a V-tube and then transferred. The latter is more nearly quantitative. Kesterson and Hendrickson (*299*) found a direct relationship between the aldehyde content of Valencia orange oil and the rainfall in the growing area. Using capillary gas chromatography in combination with fast-scan mass spectrometry, Schultz *et al.* (*563*) conducted an exhaustive analysis of the volatile constituents of orange juice. Major esters which they identified were ethyl 3-hydroxyhexanoate and ethyl acetate. Other compounds found for the first time were ethyl hexanoate, methyl ethyl ketone, dimethyl vinyl carbinol, 2-hexenol, perillaldehyde, butyraldehyde, and methyl isobutyl ketone. Stevens, Lundin, and Teranishi (*614*) determined the structure of sinesal, a sesquiterpene aldehyde isolated from orange oil. Attaway and Wolford (*20*) detected more than 50 compounds in recovered orange essence, using a sophisticated combination of the modern techniques. Hunter and Brogden (*245*) isolated and structurally characterized β-ylangene, a new sesquiterpene from orange oil, and Hunter and Moshonas (*246*) identified 19 alcohols in Valencia orange oil. Ten of these alcohols had not previously been found in orange oil, and three had not been found in nature. Benk and Bergmann (*38*) described chemical and electrophotometric methods for detecting the addition of dyes to orange oil. Swift (*626*) identified a number of flavones from orange peel.

Bitter orange oil was investigated, using infrared spectrography and thin-layer chromatography, by Kunkar (*337*), who also compared various methods for determining aldehydes. In addition, he (*338*) identified several of the coumarins present in the evaporation residue. Mehlitz and Minas (*376*) identified many of the oxygenated constituents of the oil, first separating them into classes by the formation of derivatives. Diverse components, such as enanthaldehyde, trans-2-hexenal, carvone, α-ionone, pentanol, and butanol, were found. In a later publication they (*377*) reported further findings in this investigation.

The water soluble constituents of palmarosa oil were examined by Shah and Gulati (*568*), using paper chromatography.

The structure of two new alkaloids, patchoulipyridine and epiquaipyridine from patchouli oil, was established by Buechi, Goldman, and Mayo (*77*).

Five major constituents of *Pelea christophersenii* oil were quantitatively determined with gas chromatography, nuclear magnetic resonance, and infrared spectroscopy by Scheuer and Hudgins (*555*).

The yield and physico-chemical properties of pennyroyal oil from plants harvested in Soke were reported by Tanker and Sezik (*650*). Oil from plants grown in Jammu and Kashmir were chromatographed by Chopra, Vashist, and Handa (*103*), and their major constituents were quantitatively determined.

Black pepper oil was investigated by Wrolstad and Jennings (*715, 716*), who determined the composition of the monoterpene fraction and studied the isomerization which takes place during isolation by thin-layer chromatography. Nigam and Handa (*434*) used gas chromatography to separate and identify the constituents of the oil, and Kamenshchikova, Mokhnachev, and Kuz'min (*285*) used reaction gas chromatography to detect three acids and two carbonyl compounds in the oil.

The gas chromatographic determination of oil in peppermint plants and the identification of some chief constituents were reported by Malingre (*366*) who described a method for obtaining an oil coming as close as possible to the oil naturally present in the plant. The effect of the period of planting on the yield of oil was studied by Skrubis (*592*). The composition of the oxygenated portion of low-menthol peppermint oil was analyzed with rectification and thin-layer chromatography by Avakova, Kustova, and Titova (*23*). The composition of Bulgarian peppermint oil was investigated by a combination of instrumental techniques by Ognyanov and Vlakhov (*456*). Among the many constituents identified were *n*- and *iso*-amyl alcohols, 3-hexenol, phenylacetaldehyde, and ethyl amyl ketone. In another publication they (*696*) reported the identification of oxygen-containing constituents, including the acetate and formate of ethyl-*n*-amylcarbinol. The main constituents of Rumanian peppermint oil were separated with thin-layer chromatography by Rothbacher, Crisan, and Bedo (*530*). Rothbacher (*529*) also reported thin-layer techniques for the identification of cineole and limonene, as well as an adaptation of the resorcinol method for the determination of cineole in Rumanian peppermint oil. Cardoso do Vale (*96*) found that the composition of African peppermint is similar to that of normal oils from other locales. Norri (*451*) used gas chromatography to compare the peppermint oils from plants grown from seeds with those from plants grown from cuttings. He found that the former gave heterogeneous oils, whereas the oils from the latter were homogenous. Vashist and Handa (*684*) analyzed an oil from white mint in which the *l*-menthol content was 14.68%. Goto (*181*) used ion exchange resin for the treatment of black mint oil, and studied the result with gas chromatography. He reported the odor of the oil to be sweeter after treatment.

Perilla oil was investigated by Fujita and Ueda (*164*). Many components of the oil were isolated and identified. Ito (*258*) separated perilla ketone and isoegoma ketone from the oil of perilla seed.

The oil of *Petasites albus* was investigated by Novotny, Herout, and Sorm (*452*) who isolated and established the constitution of petasolides, s-petasolides, petasalbine, albopetasine, and hydroxyeremophilenolide; and, from the oil of *P. hybridus,* Novotny, Samek, and Sorm (*453*) isolated and determined the structure of dimethoxydihydrofuroeremophilane.

An investigation of the comparative compositions of various petitgrain oils was reported by Peyron (*476*).

Pine oils obtained from different plant parts which were cut periodically from *Pinus attentuata* and *P. radiata* were studied using gas chromatography by

Blight and McDonald (*60*). The composition of the oils varied very little. Iconomou, Valkanas, and Buechi (*249*) and Iconomou and Valkanas (*248*) determined many components of the oils from Greek Pinus species. Gas chromatography with various columns and different stationary and liquid phases was employed. Oil of *Pinus nigricans* from Bulgaria was analyzed by Ognyanov and Tzankova (*459*) who determined its major components and some minor components. Two new terpene alcohols produced in ponderosa pine by the microorganism *Ips confusus*, were identified by Silverstein and Rodin (*587*). Karnik, Bhatia, and Lal (*294*) reported the physico-chemical properties of an oil distilled from *Pinus roxburghi*. Gas chromatography together with chemical and infrared identification served Ognyanov, Vlakhov, and Tzankova (*458*) to establish the major components of *Pinus sylvestris* oil from Bulgaria. In an extensive investigation of oils from fresh and aged needles and young and old growth of *Pinus sylvestris*, Juvonen (*280*) observed a close inverse relation between the α-pinene content and that of 3-carene.

The fraction of *Piper longum* oil above monoterpenes was shown by Nigam and Radhakrishnan (*443*) to consist mostly of caryophyllene. Handa, Nigam, and Sharma (*207*) isolated two new sesquiterpenes from the oil and suggested their structures. Nigam and Radhakrishnan (*444*) reported a preliminary investigation of a new sesquiterpene hydrocarbon from *Piper longum* oil.

The major constituents of *Pogostemon plectrantoides* oil were isolated and identified by Nigam and Purohit (*442*).

The hydrocarbon compounds in *Rhododendron duaricum* oil were determined gas chromatographically by Pigulevskii and Belova (*484*).

Rose oil has been studied by many investigators. Karpacheva (*295*) developed a rapid interferometric technique for the rapid determination of oil content. Paseshnichenko and Guseva (*471*) determined many components in rose oil with thin-layer chromatography and using an absorbent containing $AgNO_3$, they separated geraniol and citronellol. Guseva and Paseshnichenko (*202*) suggested that terpene alcohols, such as phenethyl alcohol, are increased during rose petal fermentation. Lozzi (*352*) found that oil content in rose blossoms decreased by 50% during one hour after they were freshly opened. He also identified many components including valeraldehyde, phenylacetaldehyde, salicylaldehyde and cinnamaldehyde in the oil. Stoianova-Ivanova and Kouzmanova (*616*) studied the physicochemical properties of waxes from rose oils; Stoianova-Ivanova, Nikolova, and Tzvetkova (*617*) investigated the molecular size of the unsaturated hydrocarbons in the waxes, and Stoianova-Ivanova and Khadzhieva (*615*) identified six straight chain alcohols therein. Wollrab (*712*) used chromatography on heated silica columns, as well as thin-layer and gas chromatography, to separate and identify the straight and branched chain components in rose oil wax. In a later publication Wollrab, Streibl, and Sorm (*713*) reported further quantitative determination of many components of the wax. Igolen (*251*) described the production, properties, and odor qualities of Turkish rose oils, and Svoboda and Matucha (*625*) checked the purity of Bengal rose oil using radioactive labeling.

Rue oils from *Ruta graveoleus*, *R. chalepensis*, and *R. corsica* were examined with paper chromatography by Srepel (*605*), who established the relation of methyl monyl and methyl heptyl ketones in the oils.

The oil content in sage, *Salvia officinalis*, at various stages of development was ascertained by Kolodziejski *et al.* (*314*). Brieskorn and Dalferth (*68*) isolated and structurally identified 2-methyl-3-methylene-5-heptene in Dalmatian sage oil. They (*69*) also identified farnesene, and, in Greek sage oil, caryophyllene. The same authors (*70*), by a combination of fractionation, gas chromatography and infrared spectroscopy, identified a number of components in the oils from six varieties of Salvia. Brieskorn and Grossekettler (*71*) reported the identification of some components of sage seed oil.

The composition of oil of *Santolina chamaecyparissus* was elucidated by Thomas and Willhalm (*665*). Among many components, they isolated and characterized a new triple-unsaturated hydrocarbon, using mass, nuclear magnetic resonance, and infrared spectral data.

The oil of *Schizandrae chinensis* was separated by Shcherbakova (*576*) into hydrocarbons and ketones. Azulenes were formed during the distillation of the hydrocarbon fraction.

Oil of *Senecio neaei* was distilled by Cymerman and Montes (*118*), who used spectroscopic and chromatographic techniques to determine its general composition.

The composition of *Sium latifolium* oil was investigated by Kozhin and Fedorova (*324*), and Clarke, Talwar, and Handa (*107*) distilled and determined the major components of the oil from the leaves and fruit of *Sium latijugum*. Column chromatography was employed to separate the constituents.

In spearmint oil, Burks and Gjerstad (*80*) reported finding, among many other components, three which were hitherto not reported, cineole, α-pinene, and linalool. Sacco and Calvarano (*541*) reported that a new hybrid species, *Mentha lavenduliodora*, derived from *M. viridis*, yields an oil which on analysis proved to be similar to spike lavender oil.

The oils from thirteen species of Picea were analyzed using gas chromatography by Schantz (*553*), who compared the makeup of the various oils.

As the result of studies by Jarvi (*268*) on individual plants, four distinct types of tansy oil were found. According to the principal component of the oil, they were the thujone, camphor, borneol, and cineole types. Von Rudloff and Underhill (*536*) studied the variation in composition of tansy oil depending on the season and found wide differences in the ratio of *l*-thujone to *d*-isothujone. Stahl and Scheu (*609*) identified artemisia ketone as the main constituent of a new strain of tansy. Stahl and Schmitt (*610*) distilled oils from Central European fresh *Tanacetum vulgare* and found that they differed greatly in odor and flavor. Some oils contained no thujone, while others had up to 95% isothujone. The composition of the oils did not appear to depend on the surroundings.

In the oil of black tea, Yamanishi, Sato and Omura (*719*) identified linalool epoxides; Kobayashi *et al.* (*311*) found *cis*-3-hexenoic and *trans*-2-hexenoic acids; Yamanishi *et al.* (*718*) detected *cis*-3-hexenol and many other alcohols, and *trans*-2-hexenal and many other aldehydes; and Kobayashi, Sato, and Yamanishi (*312*) proved the presence of *cis*-2-pentenol. Takeo (*638*) observed the loss of *cis*-2-hexenol and its acetate during refrigerated storage of green tea leaves. Skobeleva and Bokuchava (*591*) isolated a number of esters from tea. Gogiya (*175*) subjected essential oil of tea to paper and thin-layer chromatography and identified many components; and Yamanishi *et al.* (*717*), in a gas chromatographic study of the neutral portion of the oil, identified *cis*-jasmone, and three isomers of linalool oxide, and also suggested the presence of acetates of *trans* and *cis*-3-hexenol among the 42 components separated.

The oil of *Thuja standishii* wood was analyzed by Hirose (*233*) who identified several of its constituents.

The changes in volatile oil and thymol content of thyme were observed during various stages of growth by Muenchow and Pohloudek-Fabini (*405*), using a microanalytical method. Granger, Passet, and Verdier (*189*) demonstrated that α-terpinene is the precursor of *p*-cymene in thyme oil, and studied the seasonal variation in the two components. They (*190*) also studied the variation of linalool content with season and location. Messerschmidt (*380*) determined the effects of cultivation methods, growth stage, and long storage of the herb on the essential oil. Tucakov (*678*) studied the effect of climate and soil on the oil from Yugoslavian thyme.

He found Yugoslavian oil to be similar to French oil. Ivars (*264*) could find no correlation between the essential oil composition and the geographical distribution of two Finnish wild thyme species. Schratz and Qedan (*557*) identified many of 26 constituents separated with thin-layer chromatography from wild thyme oil. Messerschmidt (*381*) showed that thin-layer chromatography is well adapted to the identification of various thyme oils; and he also established the relative proportions of ten constituents in these oils.

The aromatic compounds of tobacco were separated into chemical classes by Chenikov and Shapovalov (*101*). Kapetanovic (*288*) separated the aldehydes, alcohols, and esters from the steam volatile portion of an absolute from tobacco flowers. Johnson and Nicholson (*274*) described a new 13 carbon ketone, solanone, isolated from tobacco. Its structure is at variance with the head-to-tail arrangement predicted by the isoprene rule.

The structure and stereochemistry of four new sesquiterpenes isolated from the wood oil of *Torreya nucifera* was established by Sakai, Nishimura, and Hirose (*545*).

From the oil of *Trichothecium roseum*, Cairns *et al.* (*87*) isolated rosololactone and related diterpenoid lactones, and studied their infrared spectra.

Turpentine oil from *Larix europaea* was analyzed gas chromatographically by Matawowski and Stopinska (*372*), who found that the composition is similar to that of the turpentine oil from Siberian larch. The turpentine oils from *Pinus halepensis* and *P. brutia* grown in Greece were compared, using gas chromatography, by Iconomou, Valkanas, and Buechi (*249*). Baboshin *et al.* (*25*) described the application of a gas chromatographic technique at 50–60° below the boiling point of the fraction being chromatographed to the analysis of turpentines. Mirov, Zavarin, and Snajberk (*386*) investigated the chemical composition of some eastern Mediterranean turpentines from pines, in relation to their botanical classification. This technique was applied to pines of southeastern Asia in a further study by the same investigators (*724*).

Valerian oil of Polish origin was treated by Witek and Krepinsky (*710*) to yield a neutral part in which many terpenes, sesquiterpenes, and esters were identified. Three of the hydrocarbons found had not previously been reported. Indian valerian oil was examined by Sood (*597*) who isolated the tricyclic sesquiterpene alcohol, maaliol. Klyne *et al.* (*310*) deducted the absolute stereochemical configuration of valeranone. Kulkarni, Paknikar, and Bhattacharyya (*335*) established the structure of β-bergamotene by physical and chemical methods, and an acetylenic hydrocarbon was found in valerian root oil by Schulte, Reisch, and Busch (*560*). Wild Japanese valerian root oil was distilled and gas chromatographed by Hikino *et al.* (*225*). Among the many components identified were two new compounds, fauronyl acetate and cryptofauronol. The structure and absolute configuration of kanokonol, which appears in valerian oil mainly as the acetate, was established by Hikino, Hikino, and Takemoto (*224*), and Hikino *et al.* (*228*) elucidated the conformation of valeranone. Because a controversy existed, the absolute configuration of valeranone was reexamined by Hikino *et al.* (*226*). The same subject was also studied by Theobald (*660*). Hikino *et al.* (*229*) also established the structure of fauronyl acetate and cryptofauronol, and (*230*) that of β-kessyl ketone and β-kessyl alcohol. Ito *et al.* (*262*) clarified the stereochemistry of α-kessyl alcohol and kessyl glycol. Krepinsky *et al.* (*328*) elucidated the constitution of valerenolic acid, and Bates and Paknikar (*33*) determined the structure of valerenal. In this they disagreed with the earlier findings of Krepinsky and of Stoll.

The structure of a novel aldehyde from vetiver oil, khusilal, was delineated by Kalsi, Chakravarti, and Bhattacharyya (*283*). Trivedi, Kalsi, and Chakravarti (*673*) established the structure and absolute configuration of khusitone. A new sesquiterpene alcohol, khusimol, was isolated and characterized by Umarani, Gore, and Chakravarti (*681*), and the stereochemistry of the derivatives of khusinol was clarified by Chakravarti (*97*). An essential oil from Ghana vertiver was described by Talalaj (*646*), and Nguyen-Trong-Anh and Fetizon (*425*) discussed the isolation, structure, and properties of many sesquiterpenoids from vetiver oil, and reviewed the current knowledge.

The composition of witchhazel oil and its cosmetic effect was studied by Janistyn (*267*).

The components of the neutral fraction of levant wormseed oil were separated and identified by Ishibashi *et al.* (*256*). Rybalko and Ban'kovskii (*539*) identified and confirmed several constituents of levant wormseed oil from *Artemisia cina*, and Sood (*598*) examined an oil from a type of levant wormseed, *Artemisia maritima*, which does not yield santonin. Gas chromatography and infrared spectroscopy indicated a high ketone and low alcohol content.

Wormwood oils from plants growing wild in Hungary and from other areas of Europe were studied colorimetrically and with paper and thin-layer chromatography by Tyihak and Mathe (*679*). Kudrzycka-Bieloszabska and Sawicka (*330*) presented physico-chemical data for an oil of Polish origin and also for oils from other sources.

The composition of ylang-ylang oil was exhaustively explored by Rao, Skakun, and Levi (*505*) in a book titled "Infrared Examination of Ylang-Ylang Oil." Naves (*417*) pointed out the relation of the farnesenes to the dehydration products of farnesol and nerolidol, and showed that sesquiterpenes previously reported in ylang-ylang oil are in fact allofarnesenes.

Oil of *Zanthoxylum hamiltonianum* seed was distilled and analyzed by Sharma, Vashist, and Handa (*575*). Properties and several constituents are reported.

An oil distilled from *Ziziphora clinopodioides* was chromatographed by Goryaev, Gratsianskaya, and Lishtvanova (*177*). Its major components were identified and its physico-chemical properties determined.

Acids. General Procedures. Fatty acids were separated on a gas chromatographic column with a polar liquid phase on glass microbeads by Nikelly (*445*). Acids from C-1 to C-10 were identified by Porter, Bratzler, and Pearson (*497*) in the wood smoke from hard-maple sawdust. Young, Schwartzman, and Melton (*721*) separated the sodium salts of volatile acids by paper chromatography, and Gee (*168*) detailed a method for the methyl esterification of nonvolatile plant acids prior to gas chromatographic analysis.

Individual Compounds. The structure of acoric acid was established by Birch *et al.* (*56*).

Cis- and *trans*-isomers of cinnamic acid derivatives were separated with paper chromatography by Challice and Williams (*98*).

p-Hydroxybenzoic acid and its esters were separated on a thin-layer chromatograph by Salo and Salminen (*548*).

Evidence was presented for the epimeric character of the C-13 substituents in pimaric and isopimaric acids and for the location of the nuclear double bond in isopimaric acid by Antkowiak, Edwards, Howe, and Ap Simon (*11*).

The two dimensional paper chromatograms obtained by gradient elution of the organic acids in 13 commercial and laboratory vanilla extracts were compared by Blomquist, Kovach, and Johnson (*61*).

Aldehydes and Ketones. General Procedures. The gas chromatographic separation of low molecular weight carbonyl compounds was successfully achieved by Mizuno, McMeans, and Chipault (*392*) using nitrobenzene or 2-nitrobiphenyl as the stationary phase. Haken (*206*) correlated the retention data and structural parameters of carbonyl compounds. Gudzinowicz and Johnson (*193*) developed a gas chromatographic determination of 2,3-diketones, using an electron affinity detector. Soukup, Scarpellino, and Danielczik (*603*) separated the 2,4-

dinitrophenylhydrazones of carbonyl compounds directly by gas chromatography using silicone oil as liquid phase.

Thin-layer chromatography was used to separate the oximes of isomeric aldehydes by Pejkovic-Tadic, Hranisavljevic-Jakovljevic, and Nesic (*473*). Camp and O'Brien (*92*) tested various solvent systems for the thin-layer chromatography of the semicarbazones of common aromatic and heterocyclic aldehydes. Zamojski and Zamojska (*723*) tried similar techniques for the identification of volatile aldehydes and ketones, and Denti and Luboz (*128*) described another technique for the thin-layer separation of 2,4-dinitrophenylhydrazones of aldehydes and ketones. Ruffini (*537*) used a variation of the method for the identification of aromatic aldehydes and ketones, and Edwards (*139*) found solvent systems capable of separating the 2,4-dinitrophenylhydrazones of stereoisomeric aldehydes such as the syn and anti forms, and other systems which separated homologous series. Dicarbonyl bis-(2,4-dinitrophenylhydrazones) were separated on thin-layer chromatoplates into groups of 2,3-diketones, α-keto aldehydes, and dialdehydes by Schwartz, Keeney, and Parks (*564*), and isomeric ketones were also separated by a similar method as reported by Shevchenko and Favorskaya (*579*). Vashist and Handa (*683*) developed still another modified method for the thin-layer chromatography of terpene aldehyde and ketone 2,4-dinitrophenylhydrazones. Thin-layer chromatography was also applied to cyclic ketone peroxides by Buzlanova *et al.* (*86*).

Paper chromatography was employed by Fuerst and Feustel (*161*) for the determination of carbonyl compounds as the 2,4-dinitrophenylhydrazones.

A quantitative microanalysis of carbonyl compounds based on column chromatographic separation of their 2,4-dinitrophenylhydrazones and measurement of the absorbance of a treated aliquot was described in detail by Parsons (*470*) and illustrated by application to the determination of carbonyl compounds in butter fat.

The mass spectra of twelve monoterpene aldehydes and ketones were obtained with a combined gas chromatograph and mass spectrometer by von Sydow (*627*), who drew certain general conclusions from the data. Willhalm and Thomas (*708*) examined the mass spectra of menthone which was labeled on each carbon with deuterium, thus facilitating a detailed study of its fragmentation pattern. Isomenthone and carvomenthone were also studied.

The ultraviolet absorptions of 30 mono- and bicyclic terpene ketones were tabulated by Naves, Retamar, and Ricciardi (*422*), and their characteristic displacements were correlated with their structural features.

The continuous conductimetric analysis of an aldehyde was the subject of U.S. Patent 3,140,918 granted to Capuano (*93*). The method is based on the measurement of the conductance of a stream of Schiff reagent before and after reaction with an aldehyde-containing stream.

A spot test which affords a rapid indication of aldehydes in the presence of ketones was devised by Hashmi, Ayaz, and Ahmad (*209*), and another sensitive spot test for aromatic and α,β-unsaturated aldehydes was described by Hashmi *et al.* (*210*). Billman *et al.* (*54*) developed a test for aldehydes and cyclic ketones which contain at least one α-hydrogen. These substances are detected by a color reaction with 12-molybdosilicic acid and its sodium salt. New spot reactions for aromatic aldehydes were reported by Anger and Ofri (*6*). They (*7*) also devised a color reaction for the detection of gamma quantities of aldehyde phenylhydrazones or carboxylic acid hydrazides.

A method for the separation of aldehydes and ketones was patented by Stanley *et al.* (*613*). The carbonyl compounds are first converted to water soluble hydrazones and then recovered by regeneration with water soluble reagents.

A procedure for determining the molecular weight of aldehydes and ketones by titration of their semicarbazones with N-bromosuccinimide was developed by Radecka and Nigam (*503*).

INDIVIDUAL COMPOUNDS. A microdetermination of acetaldehyde in ethyl alcohol with methylbenzothiazolone hydrazone was developed by Verachtert and Frateur (*688*).

Benzaldehyde was gas chromatographically determined in barley sirup by Stacchini and Boniforti (*607*). They (*606*) also applied the technique to oil of almond and almond sirup. Using an electron-capture detector, quantities as low as 1–5 gammas could be determined. Brunelle (*74*) found the official A.O.A.C. method for the determination of benzaldehyde to be less accurate than analysis by ultraviolet spectrometry and 2,4-dinitro-phenyl hydrazine precipitation. Finley and Gilman (*151*) employed three thin-layer chromatographic techniques for the determination of substituted benzaldehyde 2,4-dinitrophenylhydrazones.

A simple polarographic method for the assay of C-14 aldehyde was worked out by Ku and Yu (*329*).

The optical rotatory dispersion of *d*-camphor was studied by Singh and Katzin (*588*). Baboshin *et al.* (*24*) and Goryaev and Lishtvanova (*178*) both made use of gas chromatography for the analysis and control of the products of camphor manufacture.

The configuration of a new sesquiterpene ketone, canarone, isolated from *Canarium strictum*, was established by Wagh, Paknikar, and Bhattacharyya (*699*).

The stereochemistry of 5-caranone and 5-caranol was elucidated by Hendrich and Kuczynski (*215*).

d-4-Caren-3-one and 3-carene-2,5-dione were studied by Zabza and Kuczynski (*722*), who clarified their structures.

Carvone was spectrophotometrically determined in dill and caraway seed by Betts (*46*). Lal and Sen (*343*) assayed carvone in spearmint oil by a potentiometric titration applied to the oximation reaction, and Singh and Gupta (*589*) conducted a critical study of the various methods used for the analysis of carvone and dihydrocarvone in oil of dill.

Citral was assayed, using a reaction with iodine chloride and titration with $Na_2S_2O_3$ by Suprun (*623*). The method is said to be simple, accurate, and reproducible. The molecular polarizability of citrals and the corresponding ψ-ionones in benzene was studied by Chen and Le Fevre (*100*), who reported their dipole moments, molar Kerr constants, and other properties.

The optical rotation of pure *d*-citronellal was estimated to be near 16° in a study by O'Donnell and Sutherland (*454*).

Coniferyl aldehyde was shown by Wahba and Sinsheimer (*702*) to be present in oils containing eugenol.

The structure of the sesquiterpene ketone cyperotundone, isolated from the tubers of nutgrass, was established by Hikino, Aota, and Takemoto (*223*).

Diacetyl and acetyl methyl carbinol were detected in the processing of frozen orange juice by Murdock (*409*), using a color reaction with α-naphthol.

A new triketone, flavesone, was isolated from *Leptospermum flavescens* and *Eucalyptus decorticans* by Bick *et al.* (*52*), who also established its structure.

A gas chromatographic method for the analysis of hydroxy aldehydes was described by Andronov and Norikov (*4*).

The ionones occurring in plant products were discussed by Naves (*419*). Dhont and Dijkman (*130*) separated some isomeric ionones and methylionones by multiple thin-layer chromatography.

The structure of isopatchoulenone, a new sesquiterpene ketone from *Cyperus scariosus* oil was deduced by Nerali *et al.* (*423*).

p-Methoxybenzaldehyde was spectropolarimetrically determined in the presence of the ortho-isomer by Potapov, Terentiev, and Moiseeva (*498*).

The structure of mustakone and copaene was elucidated by Kapadia *et al.* (*287*).

Oplopanone was isolated from *Oplopanax japonicus* by Takeda, Minato, and Ishikawa (*635*), who resolved its structure and absolute configuration.

Some uncertainties relative to the structure of perezone from *Perezia reticulata* were clarified by Wagner *et al.* (*701*).

Rhodinal was separated from mixtures containing citronellal by Eschinasi (*146*), using a patented process which selectively isomerizes citronellal to isopulegol and fixes it as the borate.

The structure of sylvecarvone was established by Kumar and Verghese (*336*) on the basis of the nuclear magnetic resonance spectrum.

The conformation of α- and β-thujones was deduced from nuclear magnetic resonance and ultraviolet spectra by Tori (*672*).

The absolute configuration of *ar*-turmerone was ascertained by Honwad and Rao (*238*).

Ubiquinones were studied with gas chromatography by Morimoto, Shima, and Imada (*401*) who found a proportionality between the log of the retention time and the number of isoprene units.

Vanillin was separated by Blanc *et al.* (*58*) from ethyl vanillin by thin-layer chromatography. Johansen (*273*) employed gas chromatography to identify vanillin and to detect impurities in it. Other analytical procedures relating to vanillin are reviewed in the section on miscellaneous methods dealing with vanilla.

The stereochemistry of zerumbone was clarified by Damodaran and Dev (*119*) who found that it was related to humulene.

Alcohols and Phenols. GENERAL PROCEDURES. Gas chromatography was used for the analysis of 3,5-dinitrobenzoates of alcohols by Galetto, Kepner, and Webb (*167*). Boric acid was employed by Ikeda, Simmons, and Grossman (*253*) to remove alcohols during gas chromatography. The technique was used for the detection of alcohols in peppermint oil. Sato *et al.* (*550*) acetylated alcohols with ketene and immediately subjected the mixture to gas chromatography. Vazquez and Montes (*686*) studied the analysis of phenolic compounds in essential oils by gas, column, and thin-layer chromatography.

Two thin-layer chromatographic methods for the separation of terpene alcohols and their identification by means of a color reaction were described by McSweeney (*363*). Lefebvre *et al.* (*346*) used thin-layer chromatography in two stages to separate the 2,4-dinitrobenzenesulfenates of volatile alcohols. Stahl and Vollmann (*611*) applied special coatings and gradient thin-layer chromatography to separate terpene, sesquiterpene, and diterpene alcohols. Sumere *et al.* (*622*) used a mixture of silica gel and cellulose powder for the thin-layer separation of phenols and coumarins. The technique has the advantages of both thin-layer and paper chromatography. The colored reaction products of phenols with *p*-nitrobenzenediazonium fluoroborate were subjected to thin-layer chromatography by Kondo and Kawashiro (*316*).

Paper solubilization chromatography of alcohols, using ion exchange paper and methanol or acetic acid as the solvent, was described by Sherma and Thompson (*578*). Paper chromatography of terpene alcohols and phenols as the 4-phenylazobenzoates was employed by Pohloudek-Fabini and Muenchow (*489*) for the determination of several essential oil constituents.

Tertiary alcohols were determined, by conversion to alkyl iodides and measurement of the ultraviolet absorbance of the product, in a method developed by Scoggins and Miller (*565*).

The mass spectra of monoterpene alcohols and the use of such spectra in organic analysis was illustrated by Willhalm, Thomas, and Stoll (*709*).

Alcohols in essential oils were assayed with metallic sodium by Teppcev and Goryaev (*654*). After first removing interfering acids and phenols, the hydrogen evolved in the reaction is measured.

Colorimetric microdetermination based on oxidation by $KMnO_4$ of acyclic terpene alcohols was proposed by Guseva and Paseshnichenko (*201*). A series of phenols gave color reactions with choline-tolidine reagent as shown by Weber and Langemann (*705*), who compared the method with other tests and particularly recommended it for developing thin-layer and paper chromatographic separations. Kurmaiah, Satyanarayana, and Rao (*339*) described a sensitive color test for alcohols based on reaction with salicylaldehyde and vanadium.

Interference in the acetylation determination of alcohols is caused by the enolization of ketones, as shown by Patwardhan, Kapoor, and Lal (*472*). Kapoor, Patwardhan, and Lal (*289*) also described this interference in quaternary systems and derived a mathematical formula which may be used to compensate for the experimental error.

A mild, rapid determination of primary alcohols by reaction with 3-nitrophthalic anhydride in dimethylformamide was developed by Floria, Dobratz, and McClure (*158*). The pyridine tosylation index of essential oils in conjunction with pyridine acetylation was proposed as a method for determining primary and secondary alcohols directly, and tertiary alcohols indirectly, by Mesnard, Gibirila, and Bertucat (*379*). Lal and Lal (*342*) applied previously derived formulas to the determination of the percentage of individual components in ternary mixtures containing three alcohols, and quaternary mixtures containing three alcohols and one ester.

Phenylazo esters of phenols were employed for the characterization and quantitative estimation of small amounts of phenols by Muenchow and Pohloudek-Fabini (*404*). Green, Axelrod, and Mislow (*191*) employed asymmetric synthesis of sulfinate esters for the configurational correlation of alcohols. Shumeiko *et al.* (*585*) developed a method based on the formation of borates for the isolation of pure alcohols from mixtures. Berger and Uldall (*40*) proposed *p*-bromophenacyl alkyl xanthates for the identification of alcohols and for distinguishing isomeric alcohols. Jaselskis and Warriner (*269*) described a titrimetric determination of primary and secondary alcohols based on xenon trioxide oxidation.

INDIVIDUAL COMPOUNDS. The constitution of agarospirol, a sesquiterpenoid with a new skeleton, was characterized by Varma, Maheshwari, and Bhattacharyya (*682*).

A mixture of eight amyl, butyl, and propyl alcohols were separated by Fricker (*159*) with gas chromatography, and the isomers of amyl alcohol were determined spectrometrically and colorimetrically by Shmulyakovskii and Anisimova (*583*).

"New borneol" was shown by Bardyshev and Kosnikova (*28*) to be a mixture and not a new chemical entity.

The conformation and stereochemistry of carveols, carvomenthols, and related compounds were studied by Sidhu and Swaleh (*586*), who disagreed with the structures previously proposed by Read and Jonston. Naves (*415*) utilized infrared and nuclear magnetic resonance spectra to study the configuration of carvomenthols. Schroeter and Eliel (*559*) re-examined the subject and found that the original assignments of configuration made by Johnston and Read appear to be correct though contrary to some recent opposing assertions.

The absolute configuration of cedrelanol was revised from that proposed by other investigators in accordance with chemical studies by Smolders (*595*).

Citronellol in the presence of geraniol and linalool was determined by Holness (*237*), based on reactions with *p*-toluenesulfonic acid.

Eugenol was assayed in clove oil with a new semimicro iodometric technique by Kartha and Kidwai (*297*), and Nikiforova and Mel'kanovitskaya (*446*) identified mixtures of eugenol and its isomers with paper chromatography.

Isomeric heptanols were determined in mixtures, based on the calibration of specific peaks in their mass spectra, by Popova, Polyakova, and Khotimskaya (*492*).

The isomerization and racemization of menthols with hydrogenation catalysts were studied by means of gas chromatography by Yoshida, Komatsu, and Indo (*720*). Capillary gas chromatography with a flame-ionization detector was successfully employed by Kogami *et al.* (*313*) to separate and analyze menthol stereoisomers. Thomas and Willhalm (*664*) studied the mass spectra of the menthols, carvomenthols, their acetates and related alcohols. Among other things, they found that the stereochemical isomers of menthol and carvomenthol were readily distinguishable. Electronic pyrolysis was applied to menthol for structure elucidation by Schildknecht and Penzien (*556*). The decomposition products were identified and the method was compared with mass spectrometry.

Phenylethyl alcohol was quantitatively determined in rose oil by Borikhina, Paseshnichenko, and Guseva (*67*), using descending paper chromatography of 3,5-dinitrobenzoates.

The constitution of Pseudoborneol was clarified through chemical studies by Hacker, Keicher, and Miltenberger (*204*).

Diterpenes derived from Sclareol were studied with infrared spectrography by Salei, Lazur'evskii, and Popa (*547*).

The absolute configuration of 4-terpinenol was derived by Ohloff and Uhde (*461*). The infrared spectra of the terpinenols were examined by Mitzner, Lemberg, and Mancini (*391*). Mitzner (*387*) later published a correction to the article.

The chemistry, reactions, and analysis of the components in the production of Terpineol were discussed by Gladden and Watson (*174*), and Mitzner and Lemberg (*390*) analyzed Δ-Terpineol and studied its properties and structure.

A new method for isolating thymol by crystallization from *Trachyspermum copticum* oil was described by Tseitlin (*676*).

The previously proposed stereochemistry of widdrol was modified by Ito, Endo, and Nozoe (*261*) to agree with that suggested by Enzell.

Esters and Lactones. GENERAL PROCEDURES. In the gas chromatography of esters, flame ionization detector response decreases rapidly with short chain methyl esters, as noted in the work by Moore, Richardson, and Amundson (*400*).

A method for the identification of esters by thin-layer chromatography, utilizing Rf values in different solvent systems, as well as color reactions, was developed by Attaway, Wolford, and Edwards (*21*).

Salting-out chromatography of aliphatic esters by elution through a column of ion exchange resin was accomplished by Sherma (*577*). Quereshi and Quereshi (*502*) also developed an ion-exchange method for the detection of esters.

The nuclear magnetic solvent shifts of methyl groups in lactones and carboxylic acid anhydrides were tabulated by Connolly and McCrindle (*111*) for 14 compounds, and they also examined the utility of the rule based on a certain reference plane in predicting such shifts. Rosado-Lojo, Hancock, and Danti (*528*) established correlations between the structure and nuclear magnetic resonance spectra of aliphatic esters including acetates and methyl esters.

Correlations and anomalies in the mass spectra of lactones were discussed by McFadden, Day, and Diamond (*358*). The data presented are useful in the identification of many common lactones. Esters of monoterpene alcohols were examined in a combined gas-chromatograph–mass spectrometer by von Sydow (*629*). The mass spectra of the esters correspond quite closely to those of the related alcohol or hydrocarbon.

The optical rotatory dispersion of a number of aliphatic lactones and some bridged ring lactones were discussed in two papers by Jennings, Klyne, and Scopes (*270*) (*271*).

INDIVIDUAL COMPOUNDS. The structure of alantolactone was determined by Marshall and Cohen (*369*), using nuclear magnetic spectroscopy.

Ambrosiol isolated from *Ambrosia psilostachya* was characterized by Mabry *et al.* (*365*).

A revised structure for aristolactone was proposed by Martin-Smith *et al.* (*370*).

Sesquiterpene lactones from certain varieties of Artemisia were identified and chemically studied by Rybalko and Ban'kovskii (*538*).

The mechanism of hydrolysis of bornyl and isobornyl acetate in acid and alkaline media were described by Bunton, Khaleluddin, and Whittaker (*79*).

Butyl acetate from various raw materials was analyzed by gas chromatography by Shaposhnikov *et al.* (*572*).

Carabrone, a new sesquiterpene lactone, was isolated from the fruits of *Carpesium abrotanoides*, and its structure was established by Minato, Nosaka, and Horibe (*384*).

Coumarin was identified with a new spot test by Anger (*5*), and Anger and Richter (*8*) developed a new colorimetric method for its determination. Several coumarins were chromatographically isolated and identified in Heracleum species by Komissarenko, Chernobai, and Kolesnikov (*315*). From other Heracleum species, Ognyanov *et al.* (*455*) also identified a number of known coumarins and demonstrated the presence of two others. Pimenov (*487*) investigated the coumarin content of 97 species of Umbellales, detecting the compounds with diazo reagents. Prokopenko and Kolesnikov (*500*) used a similar technique together with column chromatography to detect coumarins in plants of the family Umbelliferae. Coumarins from some species of Umbelliferae were also investigated by Kowalska and Skrzypczakowa (*323*) who separated and identified them with thin-layer chromatography.

Chaparrin, a naturally occurring diterpenoid bitter principle, was characterized by Porter (*496*).

The geometry of the double bonds in the ring of costunolide was established by Suchy, Herout, and Sorm (*621*).

Cyclocostunolides were investigated by Kulkarni, Kelkar, and Bhattacharyya (*334*).

The structure of dehydrocostus lactone was clarified by Mathur *et al.* (*374*).

Ethyl butyrate was detected as an artificial addition to a blend of oils with a gas chromatographic technique described by Ratay (*507*).

Furocoumarins were mixed with other substances and subjected to thin-layer chromatography by Beyrich (*48*). Kreier and Sokolov (*326*) isolated furocoumarins from *Heracleum sosnowskyi* fruits, roots, and leaves.

Geigerin and its derivatives were studied through chemical reactions by Barton, Pinhey, and Wells (*31*).

Grosshemine was isolated and characterized by Rybalko and Sheichenko (*540*).

The structures of the guaianolides, artabsin and arborescin, were chemically ascertained by Suchy, Herout, and Sorm (*619*).

Sesquiterpene lactones from *Helenium thurberi* were isolated by Herz and Lakshmikantham (*219*), and the stereochemistry of bigelovin was established. Herz, Lakshmikantham, and Mirrington (*220*), in further explorations of isolates from the Helenium species, established the stereochemistry of mexicanin A.

The structure of hysterin, a sesquiterpene lactone newly isolated from *Panthenium hysterophorus*, was proved by Romo de Vivar, Bratoeff, and Rios (*526*).

Jatamansin, a new terpenic coumarin from *Nardostachys jatamansi*, was extensively studied by Shanbhag *et al.* (*571*) who established its structure.

A rapid and specific gas chromatographic technique for the determination of methyl and ethyl esters of anthranilic acid was described by Brunelle, Martin, and Ohanesian (*75*).

Mexicanin H was studied as to its structure by Romo, Romo de Vivar, and Joseph-Nathan (*525*).

A new oxalactone, one of the macrocyclic musks, was investigated by Berends (*39*).

An investigation of the eight pentyl acetate isomers was reported by Teranishi *et al.* (*657*) who utilized gas chromatography, infrared spectrophotometry, proton magnetic resonance, and mass spectrography, as well as organoleptic evaluation.

The isolation and structure elucidation of psilostachyin and psilostachyin B and C, sesquiterpene dilactones from *Ambrosia psilostachya*, was the subject of three papers by Miller *et al.* (*382*), Mabry, Kagan, and Miller (*364*), and Kagan *et al.* (*281*).

Salonitolide, a sesquiterpene lactone of the germacrane type from *Centaurea salonitana* was isolated and structurally delineated by Suchy, Herout, and Sorm (*620*).

Another new sesquiterpene lactone, santamarine, was isolated from *Chrysanthemum parthenium* and assigned its structural configuration by Romo de Vivar and Jimenez (*527*).

The structure and stereochemistry of xanthumin, a stereoisomer of xanthinin, was established by Minato and Horibe (*383*).

Ethers, Oxides, and Peroxides. General Procedures. Mass spectrometry of the oxides of some bicyclic terpenes was conducted by Arbuzov, Efremov, and Tal'roze (*12*), who derived some general conclusions and proposed specific analytical methods for individual compounds.

Individual Compounds. The structure of alloocimene dioxide suggested by Desalbres was further supported by the findings of Doyle *et al.* (*135*). Naves and Ardizio (*420*) also substantiated previous findings with nuclear magnetic resonance spectra. In a later paper they (*421*) established that the compounds are *trans* and not *cis-trans* stereoisomers.

Atractylon was investigated by Hikino, Hikino, and Yoshioka (*227*) who re-examined its molecular structure and autoxidation products.

The conformation of carvenone and piperitone epoxides was determined by Jackson and Zurgiyah (*266*).

Caryophyllene oxide was shown by Nigam and Levi (*432*) to undergo decomposition and rearrangement when subjected to gas chromatography.

C-6 substituted 4-methoxy-α-pyrones were separated, using thin-layer chromatography on polyacrylonitrile powder with ethyl acetate-cyclohexane, by Haensel and Rimpler (*205*).

Configurations were assigned to rotundifolone and *l*-piperitone oxide, based on optical rotatory dispersion studies, by Shimizu, Katsuhara, and Inouye (*582*).

Terpenes. General Procedures. Sucrose acetate diisobutyrate and dicyclohexyl phthalate were compared as packing for the gas chromatography of terpenes by Armando, Ricciardi, and Cassano (*513*). The data presented demonstrate the superiority of the dicyclohexyl phthalate. Bardyshev and Vedeneev (*29*) tried several stationary phases and conditions for the gas chromatography of mixtures of terpenes present in the products from the manufacture of synthetic camphor. Lukes and Komers (*355*) applied gas chromatography to the separation of sesquiterpene hydrocarbons. Nigam and Levi (*429*) described the use of gas chromatography for the analysis of mixtures of sesquiterpene dehydrogenation products and illustrated its usefulness in studying degradation and rearrangement products. The same authors (*430*) also reported characteristic gas chromatographic criteria of identity for 34 authenticated sesquiterpene hydrocarbons. The technique was illustrated by application to oil of copaiba, and ylangene, α-bergamotene, and Δ-cadinene were reported in this oil for the first time. Gas chromatography was used also by Klein, Farnow, and Rojahn (*307*) to analyze the autoxidation products of various terpenes. Alpha-olefins were analyzed by Teeter *et al.* (*651*), who used gas chromatography together with mass spectrometry with and without catalytic hydrogenation. von Rudloff (*532*) employed a permanganate-periodate oxidation reaction for the convenient identification of gas chromatographically isolated acyclic monoterpenes.

A thin-layer chromatographic technique which had been successfully employed in conjunction with gas chromatography for the identification of volatile terpene hydrocarbons was described by Attaway, Barabas, and Wolford (*18*). The separation of monoterpenes with silver nitrate-silica gel thin-layer chromatography was accomplished by Schantz and Juvonen (*554*), and Vlad (*695*) gave details for the separation of diterpenes on aluminum oxide chromatoplates.

Chromatostrip isomerization studies of terpenes as reported by Wrolstad and Jennings (*715*) were useful for the interpretation of isomerization effects which sometimes result from gas chromatography.

The infrared absorption spectrum of unsaturated terpene hydrocarbon were related to the classes of their structure by Ricciardi (*510*).

Mass spectrometry of terpenes was discussed by von Sydow (*628*).

Conjugated dienes and other unsatturated substances can be colorimetrically determined in mixtures of hydrocarbons by the method described by Marxmeier, Pfeil, and Wolf (*371*).

Pure α-compounds of acyclic monoterpenes were prepared by Ohloff, Seibl, and Kovats (*460*), and their properties were compared with those of the β-compounds.

Sources of monoterpene hydrocarbons were investigated by Wrolstad and Jennings (*714*), who listed many oils as sources and also analyzed commercial terpenes for secondary components.

Individual Compounds. The configuration of the diterpene from *Araucaria imbricata* was determined by Bruns and Weissmann (*76*).

Mixtures of azulenes were separated with paper chromatography by using a viscous hydrocarbon as the stationary phase according to Giannotti and Palaud (*173*). Pallaud and Huynh-An-Hoa (*469*) applied hydrogen transfer reactions to the structural elucidation of certain azulenes, and Lorant and Nadori (*351*) determined azulene in cosmetic preparations by measuring its extinction value.

Refinements in the technique for the isolation of β-cadinene were submitted by Herout, Banassek, and Romanuk (*216*).

The chemistry and analysis of 3-carene was discussed by Verghese (*690*), and the absolute configuration of *d*-4-carene was derived by Piatkowski, Kuczynski, and Kubik (*480*).

The ultraviolet spectrum of a purified caryophyllene was found to differ from that previously reported and to be more in agreement with the accepted structure, according to the report by Ricciardi (*511*). Horeau and Sutherland (*240*) investigated the absolute configuration of several secondary alcohols derived from caryophyllene and confirmed the previous findings of Barton and Nickon.

The absolute stereochemistry of copaene was established by de Mayo *et al.* (*125*).

The constituents of the cotton bud terpene hydrocarbons were separated and identified by Minyard *et al.* (*385*), who employed distillation and column chromatography, as well as infrared, nuclear magnetic resonance, and ultraviolet spectroscopy.

l-α-Curcumene was studied by Honwad and Rao (*239*), who deducted its stereochemical configuration.

The isomers of cymene were gas chromatographed by Rihani and Froment (*517*).

The structure of *d*-elemene from *Dysoxylum fraseranum* oil and from gurjun balsam was elucidated by Gough and Sutherland (*185*).

On the basis of chemical reactions and nuclear magnetic resonance spectra, eremophilene was assigned a structure by Hochmannova and Herout (*234*).

Three previously unknown diterpenes from *Erythroxylun monogynum* were isolated and characterized by Kapadi, Sobti, and Dev (*286*). They also presented a schematic biogenetic sequence for the formation of these and other substances in the plant.

Guaiazulene was isolated from the roots of *Ficus carica* and identified by Fukushi and Tanaka (*166*).

From *Laurencia glandulifera*, Irie *et al.* (*255*) isolated and established the structure of a new sesquiterpene hydrocarbon, laurene.

The autoxidation products of limonene were isolated and identified by Blumann, Farnow, and Porsch (*62*). Perillyl alcohol was one of the products identified.

The structure of longifolene and its reaction products was established by Helmlinger and Ourisson (*214*).

The nuclear magnetic resonance spectra of six racemic forms of menthane and their conversion to various stereoisomeric forms of carane were discussed by Cocker, Shannon, and Staniland (*108*).

Pure α- and β-methylcyclogeraniolenes were prepared by Naves (*416*), in order to clarify the uncertainties relating to their properties. Details of the nuclear magnetic resonance and infrared spectra were given.

An electron diffraction study of the structure of α-pinene was reported by Arbuzov and Naumov (*13*).

The stereochemistry of thujane was elucidated by Ohloff *et al.* (*462*), and the *cis* and *trans* forms of thujane were studied by von Dieffenbacher and von Philipsborn (*131*) through the application of nuclear magnetic resonance.

The structure of thujopsene was confirmed through a total synthesis by Buechi and White (*47*), the absolute configuration of this tricyclic sesquiterpene was estalbished by Dauben and Oberhänsli (*123*).

The configuration of the sesquiterpene hydrocarbon, ylangene, was determined by Motl, Herout, and Sorm (*402*).

Miscellaneous. A rapid pyrolytic determination of oxygen in organic materials was described by Kuznetsolva, Stolyarova, and Dobychin (*340*). The compound is pyrolyzed at 900° in helium and the resulting carbon monoxide is determined with gas chromatography.

The interrelation between structure and biosynthesis in terpene chemistry was discussed by Birch (*55*).

The possible application of hydrogen transfer reactions to structure studies on tepenes was explored experimentally by Pallaud and Huynh-An-Hoa (*468*).

Amino terpenoids from the series of sclareol and resin acids were separated with ascending paper chromatography by Popa and Russo (*491*).

A selective color spot test for organic sulfur compounds was developed by Bayfield, Clarke, and Cole (*35*).

Another new color reaction was found for the detection of mercapto derivatives by Pohloudek-Fabini and Papke (*490*).

The analysis of vanilla extracts has been studied by many investigators. Gas chromatography was employed by Jackson (*265*) to distinguish among Bourbon, Mexican, and Tahitian vanilla extracts. The technique also permitted easy detection of adulteration. The results of a second collaborative study of the official thin-layer chromatographic method for the detection of flavor additives to vanilla extracts were reported by Kahan and Fitelson (*282*). Significant variations were noted in the silica gel G employed, and a precautionary statement has been inserted in the method. Paper and thin-layer chromatography of vanilla extracts were described by Synodinos, Kokkoti-Kotakis, and Kotakis (*630*), who listed the Rf values of some important components and common adulterants. A basis for comparison of authentic laboratory vanilla extracts with commercial extracts was established as to alcohol, vanillin content, lead number, resin, titratable acidity, and total amino acids, in a collaborative study described by Blomquist, Kovach, and Johnson (*61*). The essential oil content in vanilla extract was assayed by Mazur (*375*). Fitelson (*155*) adapted various methods for the analysis of extracts to the analysis of vanilla powders. Smith (*593*) proposed a two dimensional paper chromatographic procedure for the estimation of vanillic acid, *p*-hydroxybenzoic acid, and *p*-hydroxybenzaldehyde.

Based on a collaborative study of the ultraviolet absorption method for determining vanillin, Feeny (*148*) proposed a method for preparing a standard vanillin solution. This method was added to the official procedure. *o*-Vanillin was gravimetrically determined in vanillin products as the copper complex by Bogdanov (*63*). Fitelson (*157*) proposed an improvement over the official method for determining vanillin. The improvement was adopted, as was also a modified paper chromatographic procedure for determining vanillin and ethyl vanillin. Another method for the paper chromatographic estimation of vanillin and ethyl vanillin had been described earlier by Fitelson (*154*). Infrared spectroscopy was employed by Wolf and Kovacs (*711*) to analyze for the same components, and Rees (*508*) used column chromatography together with thin-layer chromatography and absorbance. A rapid colorimetric vanillin determination was developed by Fitelson (*156*). The method gave good agreement among five laboratories. Modi and Bose (*393*) devised simple spot tests for vanillin and other carbonyl and chelated hydroxy compounds. Smith (*594*) compared standard colorimetric and ultraviolet spectrophotometric methods for determining vanillin and proposed modifications which make it possible to determine both vanillin and *p* - hydroxybenzaldehyde. Stoll and Barnier (*618*) employed paper chromatography to separate hydroxymethylfurfural as well as vanillin and ethylvanillin. Bohnsack (*64*) identified vanillin by its addition complex with 1,3-butanediol, and Bohnsack and Seibert (*65*) isolated *p*-hydroxybenzyl alcohol and *p*-hydroxybenzyl methyl ether from vanilla pods and compared them with the synthesized products.

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Fertilizers

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THIS REVIEW covers the literature reported from December 1, 1964, to January 1, 1967, 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 authors' judgement, could be adapted easily to fertilizer analytical problems.

Some attention has been given toward developing methods for analyzing nitrogen, phosphorus, and potassium simultaneously. Kessen (*45*) and Varley (*90*) used the Technicon *Auto*Analyzer to simultaneously determine ammonium nitrogen, nitrate nitrogen, and phosphorus colorimetrically; and potassium flame photometrically. Constituents in fertilizers including urea and the phosphates, sulfates, chlorides, and nitrates of potassium, calcium, and ammonium and their solid solutions have been determined by x-ray diffraction using artificial spinel ($MgO \cdot Al_2O_3$) as an internal standard (*3*). Results agreed to within ±3% relative with those of chemical analyses for fertilizers containing 5 to 20% of total nitrogen ammonium nitrogen, total P_2O_5, soluble P_2O_5, total K_2O, and chloride. However, results for urea-containing fertilizers were unsatisfactory. In another study, it was pointed out that because of the uncertainties introduced by line broadening, interferences, and reactions occurring during physical preparation of the sample, it was difficult to estimate the accuracy of the x-ray diffraction method of analysis (*4*). Even with these uncertainties, this technique yielded useful information on the qualitative composition of minerals in mixed fertilizers.

OFFICIAL METHODS

The Association of Official Analytical Chemists (AOAC) in 1965 gave "official" status to a method for total nitrogen using chromium powder and HCl to reduce nitrates (2.049). The method is applicable to all fertilizers except those containing highly refractory organic matter and those containing both nitrate and organic matter. A single, precise, accurate method for total nitrogen in all fertilizers has not yet been developed. A flame photometric method for sodium using anion exchange resins to remove interfering anions was given official status (2.117). A spectrophotometric method for cobalt (2.012) and an atomic absorption method for the determination of copper, iron, magnesium, manganese, and zinc were also adopted (2.086). A method for determining slow-release nitrogen which involves adding boiling water to the sample, stirring for 30 minutes, filtering, and analyzing an aliquot by a Kjeldahl method was adopted. An oven method (2.014) for the determination of free water by heating at 50° C for 2 hours under a partial vacuum was collaboratively studied by the AOAC and given official status (*5, 6*).

SAMPLING

A bulk fertilizer sampling study was designed and initiated in 1965 under the direction of Dr. Charles W. Gehrke, the Associate Referee of the AOAC on sampling and sample preparation. Others cooperating in the program were the Bulk Fertilizer Committee of the Association of American Fertilizer Control Officials (AAFCO), the National Plant Food Institute (NPFI) Chemical Control Committee Task Force on Sampling Fertilizers, and industry. The report by Gehrke *et al.* (*26*) presents detailed results of the study on sampling bulk loads of granular, semi-granular, and blended fertilizers. The experiments were designed to obtain information on the various sampling instruments and sampling methods. Five different sampling tubes were studied. The AOAC slotted single and double tubes, a compartmented 524 grain probe, the modified Archer trier, and a Missouri interrupted-core compartmented double tube. It was shown that an accurate fertilizer sample can be secured by passing a stream sampling cup through the entire flow of material at equal timed-spaced intervals during the loading of a truck; this "stream" sample was found to be a more accurate indicator of the actual plant nutrient content than any of the trier samples. The stream sample was used as the "reference point".

It was conclusively found that the AOAC double or single tubes were not suitable for sampling bulk loads of fertilizers. Those tubes were biased and took $^2/_3$ of the sample by the top $^1/_3$ of the trier. The Missouri interrupted compartmented tube and the 524 grain probe did a good sampling job on semi-granular and granular fertilizers when used in accordance with the Baker-Gehrke sampling plan and if vertical cores were taken. The sampling plan (*26*) using these compartmented triers was adopted as official, first action, at the 80th Meeting of the AOAC, October 1966.

Smith, in a supporting study (*80*), concluded that the most representative trier sample was obtained by inserting the probe in a vertical position instead of at an angle.

Possible mechanisms of sampler bias on dry mixed fertilizers were investigated in an effort to develop better official sampling instruments and procedures (*7*). Three lots of dry mixed fertilizers with known physical and chemical composition were prepared in the laboratory. Twelve vertical and twelve horizontal cores, arranged in a Latin square sampling pattern were secured from each lot with four triers: the AOAC double tube trier, the double tube compartmented triers (524 grain probe and Missouri trier), and an experimental double half-tube trier, "Missouri D Tube," wherein the core was encompassed in place rather than being required to flow into a compartment as with conventional triers. Individual cores were analyzed physically and chemically. Only marginal significant differences were found between cores on the basis of tube opening size. The experimental double half-tube trier was less selective to particle shape or size than either the compartmented or AOAC triers. All triers produced more representative samples from vertical cores than from horizontal cores. Also, cores drawn at an angle of 60–75° from horizontal were not consistently different from vertical cores. Analytical data confirmed the sieve analysis results quite closely. Horizontal cores secured with the "Missouri D Tube" confirmed that the bias observed in horizontal cores was due to a downward drifting of small particles when the core area is disturbed by the insertion of the sampler. Cores secured with the sampler-retaining face upward contained an excess of fines, whereas cores secured with the sampler-retaining face to the side or downward more nearly resembled vertical cores in composition. The "Missouri D Tube" secured the most accurate sample and was the easiest sampler to use.

WATER

Studies were conducted on the moisture content of peat samples by a federal method using a 5- to 10-gram sample with drying at 70°C to a constant weight, and a proposed method using a 50-gram sample with drying at 105°C overnight (*84*). The Federal method gave significantly lower results in all cases, but both methods gave equal precision indicating that larger sample weights do not greatly improve the precision.

Johnston and Smith (*40*) proposed using the Karl Fischer reagent to determine moisture in granular fertilizers because of the risk of losing bound water with the usual vacuum desiccation or oven-drying techniques. The samples were ground under chloroform, extracted with solvent mixtures such as dry CH_3OH-$CHCl_3$; then aliquots were titrated with the Karl Fischer reagent. Proper selection of the extractant made it possible to reproduce the results of the various standard oven drying techniques.

A relative precision of ±1.5% was obtained when the Karl Fischer method was applied to N-P and N-P-K fertilizers using dry methanol as an extractant (*56*).

After extraction with a mixture of polar and nonpolar solvents, water was determined by measuring the capacitance of the solution with a high frequency titrator (*65*). The polar:nonpolar solvent ratio must be determined for each fertilizer, and it varies with the type of fertilizer analyzed.

A single water determination was reported requiring less than 7 minutes with a relative error of $<3\%$ (*39*). The method was based on measuring the capacitance of the solid sample. The capacitance was a linear function of the concentration of water for water contents from 2.5 to 6%. Optimum reproducibility was obtained when samples were compressed at 3.5 kg/sq cm.

A method for water based on the deceleration of fast neutrons by water in the sample was also reported (*63*). The slow neutrons were detected with a BF_3 proportional counter. An absolute error of less than 1% by weight was obtained for crude lime containing 25 to 35% water. The method was used to continuously monitor a commercial lime production process.

Satisfactory results for the water content of NH_4HCO_3 fertilizer were obtained by mixing the sample with CaC_2 and measuring the C_2H_2 formed (*53*).

NITROGEN

The AOAC continued studies in 1965 on methods for determining nitrogen in fertilizers. A collaborative study was made comparing the sulfuric–salicylic acid method (AOAC 2.045, 10th Ed.), the reduced iron method (AOAC 2.046), a modified reduced iron method (*28*), a chromium powder method (AOAC 2.048) using pacified chromium metal, and a Raney nickel catalyst powder method (*15*). The report showed that no one method was applicable to all types of fertilizers (*20*). The sulfuric–salicylic acid method was the easiest to perform but did not quantitatively recover nitrogen from liquid samples and samples with high chloride:nitrate ratios; however, good results were obtained when organic nitrogen was present in large amounts. The reduced iron method presented problems with foaming and bumping, was not precise, and did not have general applicability to all samples. The modified reduced iron, chromium powder, and Raney nickel catalyst powder methods gave essentially equal results. The chromium powder method was favored for its ease of manipulation and saving of time. However, the pacified chromium powder method did not quantitatively reduce nitrates in the presence of organic matter. In 1963 six different methods for determining nitrogen in fertilizers were subjected to Youden's ruggedness test (*14, 92*); these were a chromous solution reduction method, the Raney catalyst powder method, an improved reduced iron method, the chromium powder method, the sulfuric–salicylic acid method, and the reduced iron method. The reduced iron method failed the test; the other methods proved "rugged."

A comprehensive method for total nitrogen was developed in 1966 by Gehrke *et al.* (*29*), which has the capability of reducing nitrates either alone or in the presence of organic matter. Quantitative recovery of nitrogen was obtained from KNO_3 containing various organic substances and inorganic nitrogen sources including inorganic fertilizers, as well as the highly refractory organic compound nicotinic acid. The method was subjected to the Youden ruggedness test and was found to be "rugged" for all samples analyzed except nicotinic acid. The method is accurate and precise and applicable to all types of solid, liquid, and slurry fertilizers. This method offers a significant saving in analysis time.

More studies were conducted on the Raney catalyst powder method to shorten the time required to reduce nitrate (*15*). A catalyst powder containing 10% Co, 40% Ni, and 50% Al in 8N H_2SO_4 was found to reduce nitrates to NH_3 within 10 minutes. Neither chloride nor organic nitrogen interfered with the reduction. The method passed Youden's ruggedness test and results of the analysis of fertilizers for total nitrogen were in good agreement with data from AOAC methods.

A method for the determination of total nitrogen using sulfuric–salicylic acid to prevent the loss of nitrogen as NOCl was reported (*55*). In this method the fertilizer (80 to 100 mg N) was digested with H_2SO_4 containing salicylsalicylic acid in the presence of $Na_2S_2O_3$. Because of its low volatility, the sulfonitric acid (SO_2NO_2OH) formed prevents loss of nitrogen. The action of SO_2NO_2OH on salicylsalicylic acid forms a nitro compound which on digestion is reduced to an amino compound and finally converted to NH_3. The NH_3 reacts with H_2SO_4 to form $(NH_4)_2SO_4$. With less than 10% urea in the fertilizer, the absolute error was 0.4%. At higher amounts of urea, the absolute error did not exceed 2%. Time of digestion was 2 to 2.5 hours.

An improvement of the sulfuric–salicylic acid method for total nitrogen in fertilizers was reported by T'sai (*86*). The sample was combined with Zn-Fe powder and 10% H_2SO_4, boiled for 10 minutes, cooled, and then digested for 15 minutes with concentrated H_2SO_4. The digestate was cooled, then saturated $K_2Cr_2O_7$ solution was added and the digestate boiled for an additional 5 minutes. Subsequent distillation and titration gave the amount of nitrogen. Results were obtained in 16% less time than with the salicylic acid–$Na_2S_2O_3$ method (AOAC, 2.045) and in the range of 30 to 44% less time than with direct acid digestion.

A method for determining nitrogen in fertilizers containing ammonium, nitrate, and urea nitrogen was reported (*24*). Urea was decomposed by adding 1% urease solution and heating for 20 minutes at 50° C. Arnd's alloy and a solution of $MgCl_2$ were then added. Distillation of liberated NH_3 into 0.1N H_2SO_4 and back-titration with 0.1N NaOH gave the total amount of nitrogen from these three sources. The procedure required only 90 minutes and gave a standard deviation of ±0.15 mg nitrogen at a level of less than 90 mg of nitrogen.

Ion exchange resins have been used to determine nitrate–nitrogen and ammonium–nitrogen in nitrogenous fertilizer salts (*47*). Nitrate salts alone were analyzed by passing the sample solution through a cation exchange resin (MK-2 H^+ form), and titrating the effluent acid with 0.1N NaOH. Ammonium salts were similarly analyzed with an Amberlite (IRA-410 OH^- form) resin, the effluent being titrated with 0.1N HCl. Results obtained with samples containing NO_3^-, NH_4^+, or NO_3^- and NH_4^+ in combination agreed well with those from established methods.

A revised method of analysis for water-insoluble and slow-release nitrogen in fertilizers has been reported (*37*). Instead of analyzing the residue (AOAC 2.057, 10th Ed.) for the insoluble nitrogen, the filtrate was analyzed for the soluble nitrogen and another portion of the sample analyzed for total nitrogen. Results obtained were much more precise than those from the AOAC official method (2.057). Another method was developed for water insoluble nitrogen in fertilizers, based upon the biodegradability method used to determine the biological oxygen demand of wastes (*42*). By making the nitrogen content the limiting factor in the growth medium, the biodegradation of water-insoluble nitrogen sources can be studied. When the Warburg respirometer is used, the relationship between oxygen utilization and nitrogen content allows for a fairly accurate measurement of biodegradable nitrogen. An analysis takes about 2 weeks.

A method for determining nitrogen in inorganic nitrates by the Dumas method was reported by Ketchum (*46*). The sample was mixed with anthracene at 1 to 1.5 times its weight. The recovery of nitrogen was quantitative. When more than 15 mg of anthracene was used, it was necessary to check the blank for the formation of methane.

Conductometric titration has been used in the estimation of ammonium, and nitrate–nitrogen in ammonium sulfate–nitrate fertilizers (*75*). Total ammonium nitrogen was determined by conductometric titration with standard alkali. Ammonium nitrate was quantitatively extracted with acetone–water (189:11), and the nitrogen remaining, as $(NH_4)_2SO_4$, was determined by conductometric titration with standard alkali. The nitrogen present as NH_4NO_3 was calculated by difference.

Several methods have been reported for the determination of ammonium ions in fertilizers. The use of time–temperature–gravimetric titration and its application to ammonium fertilizers was reported (*48*). A constant drop rate was maintained which enabled the titration of small volumes. Also, the determination of ammonium ions in the presence of phosphate was accomplished by precipitating the phosphate as $CePO_4$, then titrating with $0.1N$ NaOH after the addition of formaldehyde (*17*). In another approach the determination of ammonium salts alone, in the presence of acids or ammonia, or in commercial fertilizers was done by treating the sample with tetraethyl ammonium hydroxide in acetone. The NH_4OH produced in the reaction and the free ammonia in the sample were determined by differential potentiometric titration (*49*). Alternatively, the unchanged tetraethyl ammonium hydroxide can be titrated with standard $HClO_4$ solution in methyl ethyl ketone. The relative error was approximately 0.5%. A rapid determination for ammonium nitrogen in ammonium hydrogen carbonate fertilizer was reported (*53*). Reaction of the NH_4HCO_3 with excess standard acid and titration of the excess acid with standard base after expelling the liberated CO_2 was used. If other acids or bases were present in the sample, the NH_4HCO_3 was first reacted with strong acid, the solution boiled to expel CO_2, then adjusted with NaOH to a methyl red end point. The sample was then reacted with HCHO and excess standard NaOH added, then back-titrated with standard H_2SO_4. The latter procedure was found to give a slight positive bias.

The use of $FeSO_4$ as a reductant for nitrate was reported by Vajda (*89*). The sample was reduced in a Parnas-Wagner apparatus with a solution of 20% $FeSO_4 \cdot 7H_2O$ plus 10% $CuSO_4 \cdot 5H_2O$ containing concentrated H_2SO_4. 30% NaOH was introduced into the mixture and the liberated NH_3 steam distilled into $0.1N$ H_2SO_4. The excess standard acid was back-titrated with $0.1N$ NaOH. Deviations were less than ±0.20% absolute.

Amperometric and indicator titration methods for nitrates in fertilizers have been reported (*44*). In the amperometric titration of nitrate, the sample was dissolved with heating in $0.1N$ LiOAc in glacial acetic acid and titrated with $0.2N$ $Pb(OAc)_2$ in glacial acetic acid at −1.0 volt in a polarographic cell. For the indicator titration method, the nitrate was similarly precipitated with $0.1N$ $Pb(OAc)_2$ in glacial acetic acid using diphenylamine as the indicator. Analysis time was 10 minutes and with a relative error of 1%. The reductimetric determination of nitrate by reaction with excess Fe^{+2} in strongly acidic media was improved by alterations in the order of reagent addition (*51*). The same investigator gave details of various potentiometric and automatic titrations of urea-containing fertilizers using $K_2Cr_2O_7$ or $TiCl_4$ (*51*). An ultraviolet spectrophotometric determination of nitrate ion was developed (*25*) based on the fact that the extinction *vs.* wavelength curve falls steeply for nitrate between 310 and 320 mμ, and is constant in this region for the other components. The amount of nitrate was calculated from a given equation. The relative error was less than 3% and the time for an analysis required only 15 to 20 minutes.

Two methods were reported for determining $Ca(NO_3)_2$ in calcium ammonium nitrate (*8*, *74*). One method (*74*) consists of extracting the $Ca(NO_3)_2$ into aqueous NH_3–methanol (3:97) solution and titrating the extract with EDTA using ZnO_2^{-2} as an indicator ion. A polarograph with a dropping mercury electrode and a standard calomel electrode was used. The other method (*8*) was based on the relative insolubility of $CaCO_3$ in aqueous NH_3–methanol solution (3:97) and the appreciable solubility of $Ca(NO_3)_2$ in the same solvent system. The methanol was removed by distillation, the residue dissolved in H_2O, and the Ca equivalent to $Ca(NO_3)_2$ in the sample determined by the $KMnO_4$ method. The standard deviation was ±0.01%.

Biuret in $0.1N$ NaOH exhibits maximum absorption at 225 and 260 mμ, but urea shows no absorption at this pH (*77*). In a buffer solution at pH 9.0, neither biuret nor urea show absorption at these wavelengths. Measurements were thus made at 225 and 260 mμ in both solutions to compensate for absorption by impurities, and the biuret concentration was calculated from an equation. Beer's law was obeyed up to 5% biuret when measurements were made in a 1-cm quartz cell. Also, estimations of biuret in urea have been made spectrophotometrically by a cobalt–biuret complex method (*76*). Biuret was reacted with $Co(NO_3)_2$ in the presence of alkali and a few drops of 30% H_2O_2 were added to develop a red-colored complex. The absorbance when measured at 500 mμ obeyed Beer's law. The relative error was ±8%.

Christian and Jung (*18*) reported a coulometric determination of total nitrogen. The samples were digested by official AOAC methods; then an aliquot was adjusted to pH 8.6, and the ammonia titrated with coulometrically-generated hypobromite. Results agreed with standard methods and were generally more precise. The method, while rapid for small numbers of samples, would require more man hours per sample for large numbers of samples than the AOAC official methods.

Bradstreet (*12*) published a comprehensive survey of the Kjeldahl method for organic nitrogen and its modifications as applied to natural products and organic materials. The book includes a bibliography of published papers covering the entire period from the inception of the method.

PHOSPHORUS

The use of a high concentration of molybdate was found to prevent citrate interference in the phosphomolybdovanadate spectrophotometric determination of phosphorus (*52*). For a solution containing 14 to 20 mg. of P_2O_5 in a volume of 250 ml, the reagent used contained 0.4% citric acid, 4.5 mmoles vanadium, and 0.11 moles molybdenum in $1N$ HNO_3. The wavelength giving the lowest reagent blank and the largest difference between the blank and the colored complex is 420 mμ. Calcium did not interfere at concentrations less than 0.4 gram per liter in the final solution. It was found that temperature variations had no effect on the color development if the standards and samples were prepared and read at the same temperature.

Dahlgren (*19*) examined existing wet ashing methods for destroying citrate and concluded that more precise ashing methods were needed to give reproducible amounts of mineral acid in the remaining solution after destruction of the citrate. Different ways of removing the acid, which interferes in the color development, were investigated. The preferred method employed partial neutralization of the acid after addition of the molybdovanadate solution.

The phosphomolybdovanadate spectrometric method for phosphorus has been automated by several investigators using the Technicon *Auto*Analyzer (*27*, *45*, *72*). Gehrke, Baumgartner, and Ussary (*27*) reported an automated method for "direct available P_2O_5." The method utilizes the *Auto*-Analyzer to determine phosphorus in the range of 2.0 to 4.5 mg P_2O_5 per 100 ml and at an analysis rate of 40 samples per hour. Citrate and organic coloring matter were destroyed and non-ortho phosphates were hydrolyzed by digesting the sample aliquot with a 10:4:2 mixture of HNO_3, $HClO_4$, and H_2SO_4. The effects of acidity and citrate concentration on the color reaction were determined. Acid concentration up to 1.4*N* had no effect, and it was found that all concentrations of citrate interfered with the color reaction. Heating to 90° C for 30 minutes or passing the reaction mixture through time delay coils of various lengths had no effect on the color development. The automated method was found to give results equally as accurate and precise as the AOAC official gravimetric quinolinium phosphomolybdate method ("Offic. Methods of Anal. of the Assoc. of Offic. Anal. Chemists," 10th Ed., Sec. 2.025b) and with a considerable saving in time of analysis.

Roberts (*73*) described a completely automatic "on-stream" system for quality control of phosphate rock. The sample is split continuously and reduced to a quantity suitable for analysis. This sample is further dried to an established moisture level, weighed, and analyzed, all within 16 minutes in a fully automated system.

A dry ashing technique was proposed by Brammell (*13*) for the destruction of citrate. Aliquots of the combined water and citrate soluble extracts were mixed with HNO_3, sucrose, and MgO, then the mixture was evaporated on a hot plate to hydrolyze non-ortho phosphates and to react them with the MgO to form heat stable compounds. The dry residue was then ashed at 550° C for 30 minutes.

It was reported (*85*) that interference by Fe and SiO_2 on the phosphomolybdovanadate color development was observed when phosphate in compost was determined. The color was developed in 0.8*N* H_2SO_4 and read at 420 mμ.

To improve the accuracy and precision of the AOAC official volumetric ammonium molybdophosphate method (2.026, 10th Ed.) HNO_3 was added to eliminate sulfate interference prior to the formation of the yellow precipitate (*10*); and after the precipitate was dissolved in alkali, HCHO was added to react with the ammonia. A mixed indicator (methyl green–phenolphthalein) was used which changes color at the equivalence point. This modification passed Youden's ruggedness test and gave results in excellent agreement with those obtained by the gravimetric quinolinium phosphomolybdate method.

Results from a collaborative study of methods for total phosphorus, comparing two modified volumetric procedures with the AOAC official quinolinium phosphomolybdate method, confirmed the accuracy and precision of the quinolinium method (*36*). The volumetric procedure (*33*), presented by the Phosphorus Task Force of the National Plant Food Institute Chemical Control Committee, gave accurate results but failed to give acceptable precision. The volumetric procedure proposed by Brabson (*10*) gave a slight negative bias.

An alkalimetric quinolinium phosphomolybdate method for phosphorus was collaborated on by the AOAC, but it was abandoned because of the difficulty of dissolving the precipitate with standard alkali (*35*). In a later study Duncan and Brabson (*23*) found that by using larger amounts of citric acid in the precipitating reagent larger crystals were formed which did not agglomerate during filtration and were readily soluble in alkali. The method was found to be rugged, and compared favorably in precision and accuracy with the official gravimetric quinolinum phosphomolybdate method (2.025b, 10th Ed.).

A method for phosphate was reported (*22*) in which the heteropoly acid H_3PO_4-$(MoO_3)_{12}$ is formed and then extracted with chloroform and butanol (4:1) from the excess molybdate reagent. The molybdate combined with the phosphate was then determined spectrophotometrically at 710 mμ as the green molybdenum(VI) complex with 2-amino-4-chlorobenzenethiol in chloroform. Amounts of phosphorus in the sample solution down to 0.008 ppm can be determined.

Phosphorus was determined indirectly with EDTA (*31*). The method involves precipitating phosphorus as $ZnNH_4PO_4$, then adding excess EDTA at pH 7 to 10 to complex the Zn in the $ZnNH_4PO_4$, and titrating the excess EDTA. The method is slow but precise and accurate.

A discussion of the EDTA titration of excess $Bi(NO_3)_3$ after filtration of precipitated $BiPO_4$ in 0.5*N* HNO_3 was given (*43*). Ions which interfere with this determination, except Fe(III), are not normally found in fertilizers. Fe(III) can be reduced with ascorbic acid.

Szekeres *et al.* (*82*) described a method for the sequential determination of calcium and phosphorus in the presence of Fe^{+3}, Al^{+3}, SO_4^{-2}, and F^-. A measured excess of EDTA was added to complex the Ca^{+2} and the excess EDTA then titrated with 0.1*M* $MgCl_2$ to a thymolphthalein complexan end point. Crystalline ammonium chloride and ethanol are then added to precipitate $MgNH_4PO_4 \cdot 6H_2O$ thus causing the blue color to be discharged. The titration of the liberated EDTA with $MgCl_2$ is then continued until the blue end point is again reached. Results agreed well with those obtained gravimetrically.

Determinations for total P_2O_5, water soluble P_2O_5, and citrate soluble P_2O_5 in superphosphate of lime were made by precipitating the phosphate as $Pb_3(PO_4)_2$, filtering, and converting the precipitate to $PbSO_4$ with boiling standard H_2SO_4, then titrating with standard alkali (*68*).

The phosphate content of superphosphate was determined with two titrations using two mixed indicators: methyl red and bromocresol green, and cresol red and phenolphthalein (*81*). In the first step, free acid was determined by titration with 0.1*N* NaOH to a light green color. Then Ca^{+2} was precipitated from a second sample with potassium oxalate and the phosphates thus released were titrated with 0.1*N* NaOH to the violet end point of the second mixed indicator.

Riedel (*72*) eliminated silicate interference in the gravimetric $MgNH_4PO_4$ determination by passing the sample solution through an ion exchange column before the precipitation step.

Both total phosphorus and water soluble phosphorus were determined with a d.c. arc spectrograph (*57*). The relative error was 4.0% for fertilizers containing 30% P_2O_5 and 2.6% for those containing 5% P_2O_5. The results agreed well with those obtained by colorimetric procedures.

The Netherlands alkaline ammonium citrate extraction method for the determination of available P_2O_5 was modified by Brabson (*11*) for routine use. The results of this extraction method correlate well with field tests on availability of P_2O_5 in the fertilizers.

Paper chromatography has been used for the separation of the various compounds of phosphorus in fertilizer (*79*, *91*). This technique was used to study the effects of the manufacturing process on the forms of phosphorus present.

As a basis for the qualitative determination of the presence of different forms of phosphorus in fertilizer materials differential thermal analysis curves were made of apatite [$Ca_5(PO_4)_3F$], phosphorite meal (fluoro- and hydroxy apatite with organic constituents), precipitated phosphate (Ca-

$HPO_4 \cdot 2H_2O + CaSO_4 \cdot 2H_2O$), plain superphosphate, and its granulated commerical form, Thomas slag, diamophos $(NH_4)_2HPO_4$, and a mixture of these compounds (*1*). Precipitated phosphate and superphosphates were easily observed by their characteristic endothermic effects.

Midkiff (*61*) described improved techniques for determining available phosphorus. The direct available gravimetric quinolinium phosphomolybdate method was modified so that the analysis time was reduced by one half. This was accomplished by using a multiple pipetter and other analytical glassware arranged in multiple units.

POTASSIUM

An automated flame photometric method for the determination of potassium in fertilizer grade potassium salts was developed by Ussary and Gehrke (*87*) at the Missouri Experiment Station Chemical Laboratories. This method gives results equally as accurate and precise as the AOAC official sodium tetraphenylborate method (2.083, 10th Ed.) and at an analysis rate of 40 samples per hour. The automated method was applied to mixed fertilizers and gave accurate and precise results without anion exchange cleanup (*88*). These investigators also applied the method to the analysis of potassium in the AOAC "direct available" P_2O_5 extract (2.037, 10th Ed.) (*88*). It was concluded that extraction of potassium by neutral ammonium citrate was quantitative and citrate did not interfere with the read-out. Thus, a single sample preparation and solution was used for the analysis of both phosphate and potassium.

In other studies, Gielfrich *et al.* (*32*) obtained accurate flame photometric results for potassium with a relative precision of 1%. Good agreement with chemical methods was obtained. Comparative studies were made between the titrametric sodium tetraphenylborate method and the flame photometric method for potassium by two different investigators (*16, 54*). Carmo (*16*) found that concentrations of interfering ions in fertilizer sample solutions were too low to affect the flame photometric determination of potassium when an air–butane flame was used. The tetraphenylborate method gave slightly higher results, but agreement was within 0.2% absolute with all samples of varying phosphate content. Magnitskii (*54*) compared the two methods for the analysis of hydroponic solutions and found them to give equal accuracy. However, the tetraphenylborate method gave slightly better precision.

The anion exchange resin Dowex 2 was used to eliminate sulfate and phosphate interference with the determination of potassium (*78*). After anion exchange cleanup the choice of analytical method was of secondary importance because of sampling error. Rapoport *et al.* (*71*), used formaldehyde and EDTA to eliminate interferences in the gravimetric sodium tetraphenylborate determination of potassium.

Potassium in the raw materials, the intermediates, and the products in potash fertilizer plants has been continuously and automatically analyzed by registration of the beta ray emission from ^{40}K (*9*).

SECONDARY AND MICRO-NUTRIENTS

A general atomic absorption method for the determination of Cu, Fe, Mg, Mn, and Zn in commercial fertilizers and raw materials was presented to the AOAC by McBride (*58*). After being subjected to ruggedness tests the method was studied collaboratively and found to be simple, accurate, and precise. The method was then re-examined by the AOAC and extended to include Ca and Na (*59*). However, the results for Ca and Na were not accurate. The method was adopted as an official method (2.086) for the determination of Cu, Fe, Mg, Mn, and Zn by the AOAC in 1965.

Fluorexon and metalphthalein were used as indicators in the complexometric determination of calcium and magnesium (*60*). Calcium and magnesium were titrated together using metalphthalein as indicator, and then calcium was determined separately with fluorexon after first precipitating magnesium as $Mg(OH)_2$. Iron and aluminum interferences were masked by adding triethanolamine. Magnesium was determined by difference.

A comparison of some of the methods for determining the chemical activity of lime fertilizers indicated that the determination in $5N$ NH_4Cl was the most adequate (*41*). Next in order of preference was an acetic acid extraction method.

Zinc and molybdenum were determined polarographically in trace element fertilizers (*70*). Zinc was determined in a medium of $0.5N$ $LiNO_3$, half wave of 0.97 ± 0.02 volt and molybdenum in a medium of $0.5N$ HNO_3, half wave of -0.17 ± 0.01 volt. Following a sample solution cleanup with a cation exchange resin the elements copper, cobalt, and zinc were eluted with HCl and determined polarographically with a relative error of 1 to 2% (*69*). Also, a method was proposed using polarography with photoregistration for determining copper in nitrogen–phosphorus fertilizers (*83*).

A method for the determination of molybdenum in molybdenized superphosphate makes it possible to separate molybdenum compounds into water soluble, oxalate soluble, and insoluble groups (*62*). Less than 20% of the molybdenum in ammoniated superphosphate was found to be water soluble.

Atomic absorption spectrophotometry provided a rapid, accurate method for the determination of zinc in fertilizers, soil samples, soil extracts, and plants (*2*). In the range of 0.3 to 8.0 ppm of zinc, a relative standard deviation of 2 to 3% was obtained; however, measurements with acceptable precision (relative standard deviation ~5%) were obtained on a solution containing 0.2 ppm zinc. The minimum detectable limit was 0.025 ppm zinc.

Hodgson and Lazar (*34*) determined cobalt by a spectrophotometric method using 2-nitroso-1-naphthol. The sample was digested with a ternary acid mixture; cobalt was extracted from an aliquot with an excess of 2-nitroso-1-naphthol into isoamyl acetate at pH 8.5. Excess 2-nitroso-1-naphthol and interfering substances were removed by successive washes with acid and base. A collaborative study indicated that while the precision was not as satisfactory as obtained for a major fertilizer constituent, agreement compared favorably with AOAC official methods for cobalt. The procedure was considered tedious, but less so than other colorimetric cobalt methods and less subject to interferences.

A dichromate method for iron was presented in 1965 to the AOAC for collaborative study (*38*). Ferric ion is reduced to ferrous with $SnCl_2$, and then titrated with dichromate solution using diphenylamine as indicator.

Boron was determined by precipitation as a complex borosaccharate, in a solution containing boric and saccharic acids. Barium chloranilate was then added and the released chloranilate ion measured spectrophotometrically (*66*). The method obeyed Beer's law up to about 10 ppm.

A method for determining B_2O_3 in boron double superphosphate and in boronitrophosphate was developed (*64*). The sample was dissolved in HCl. After cation exchange cleanup, to remove Ca^{+2}, Fe^{+2}, and NH_4^+, H_3BO_3 was determined by a sucrate method.

In another method boron was extracted as the crystal violet-BF_4 complex into benzene and measured photometrically. The method was applied to fertilizers, soils, and plant materials (*21*).

Pile and Williams (*67*), developed a phototurbidometric titration method for the rapid determination of sulfate in wet-process phosphoric acid. The titration and mechanical stirring were carried out in a photocolorimetric cell to the point where no increase in absorbance was detected after titrant addition. Impurities normally present in wet-process acids did not interfere.

The AOAC official flame photometric method for potassium in fertilizers (Sec. 2.077, 10th Ed.), which uses anion exchange to remove interfering anions, was applied to the determination of sodium (*30*). The precision and accuracy data from a collaborative study agreed well with those for the flame K_2O data reported in the Magruder check sample program.

A series of methods to qualitatively determine the elements in trace element enriched superphosphates has been proposed (*50*). Phosphate was identified in an aqueous extract of the sample as the yellow ammonium phosphomolybdate. Molybdenum was identified in a 10% HCl extract as orange-red $(NH_4)_2[MoO(CNS)_5]$; boron with curcumin, and manganese in the absence of molybdenum and boron as MnO_4.

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Food

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THIS REVIEW covers advances in food analysis since the 1965 review (*22P*) as reported in publications available to the authors through early October 1966. In reviewing the vast amount of literature on this subject, it has unfortunately been necessary to eliminate many useful but relatively minor changes in established procedures. Again, as previously, where parallel studies have been encountered, the authors have generally elected to cite the work reported in American journals while recognizing that equally fine work has been reported in other journals.

In the past two years a text on Food Standards in the United Kingdom (*1P*) has been published, and a review on Food has been published in the *Japan Analyst* (*23P*).

ADDITIVES

Many improved procedures for additives in foods have been proposed in the last two years. The use of gas chromatography and thin layer chromatography now provide simple and specific procedures for many of these components.

A review of antioxidant analytical techniques applicable to food products has been published by Stuckey (*54A*). Thin layer chromatography has been applied to the determination of BHA in edible oils by Amano *et al.* (*1A*); to the separation of a mixture of antioxidants using polyamide layers by Copius-Peereboom (*8A*); others have used silica gel to separate the antioxidants followed by thin layer separation (*14A*). Two-dimensional thin layer chromatography has been proposed by Sahasrabudhe (*45A*) as a means of separating the four most commonly used antioxidants, and Scheidt and Conroy (*48A*) propose a one-dimensional thin layer separation for the same four. An acetonitrile extraction and alumina column clean-up has been proposed by Schwien *et al.* (*50A*) as a means of preparing an antioxidant extract suitable for thin layer or gas chromatography. Sedlachek (*51A*) has suggested a mercury precipitation followed by complexometric titration for mercury as well as a paper chromatographic method for the phenolic antioxidants. Nishimoto and Uyeta (*36A*) have described a gas chromatographic procedure for antioxidants and preservatives. Colorimetric tests for gallic acid esters using ferrous salts have been discussed by Salazar Macian (*47A*). Procedures for testing for antioxidants in plastic packaging materials have been described by Woggon *et al.* (*61A*).

The detection of emulsifiers in materials used in alcohol free beverages have been reviewed and described by Rother (*43A*). A method for the column separation and subsequent determination of lactylated glycerides and polyglycerol esters in shortenings has been described by Sahasrabudhe *et al.* (*46A*). Small amounts of 1-monoglycerides in fats have been determined by oxidation to the corresponding fatty acid ester of hydroxyacetaldehyde (*55A*). Kroller (*25A*) has described a method for the separation and paper chromatographic determination of polyoxyethylene adducts in foodstuffs. A method for preventing the interferences of lecithin in the determination of polyoxyethylene glycols in food products has been described by Giordano *et al.* (*15A*). A procedure for the separation and identification of polyoxyethylene glycols and their esters in ice cream has been proposed by Monacelli and Doretti (*35A*). A turbidimetric method has been described by Ruch *et al.* (*44A*) for the direct determination of polyethylene oxide polymers in beer. Wetterau *et al.* (*60A*) have developed a separation and gas chromatographic detection for the determination of sorbitan monostearate in cake mixes.

Studies continue on means of determining and identifying preservatives in foods. Sodium benzoate has been determined by gas chromatography (*37A*). Benzoates and hydroxybenzoates have been determined by thin layer separation and ultraviolet measurement (*39A*). Schulman *et al.* (*49A*) have described a method for heptyl *p*-hydroxybenzoate in beer. Deniges reagent has been used to determine the 4-hydroxybenzoic acid esters in foods (*56A*), and the same compounds have been determined by gas chromatography (*26A*). The same technique has been applied by Goddijn *et al.* (*16A*) to the determination of benzoic acid, its derivatives, and sorbic acid, and by Groebel (*18A*) to the analysis of these compounds in foods. Walker *et al.* (*59A*) apply gas chromatography to the determination of propionic acid and sorbic acid in bread and flour after separation by steam distillation. Sorbic acid in wine is determined as the ethyl ester by gas chromatography (*12A*). Thin layer chromatography has been suggested by Cuzzoni (*10A*), Copius-Peereboom (*7A*), and Covello and Schettino (*9A*), as a means of rapid identification of preservatives. Luck and Courtial (*28A*) described a thin layer separation of benzoic and sorbic acids. Bromination is used to improve the separation of sorbic and benzoic acids on paper chromatograms (*38A*), and identification of preservatives on paper using short wave ultraviolet illumination has been suggested by Guthenberg and Beckman (*20A*). A method for sorbate in low-calorie salad dressing has been described by Ciaccio (*6A*), and an infrared method for sorbic acid has been proposed by Calloway and Schwartzman (*5A*). Chloracetic acid in wine has been determined photometrically by the formation of indigo (*4A*), and identification of monohaloacetates in wine by the formation of colored derivatives has been proposed by Maglitto and Gianotti (*29A*).

For the detection of artificial sweeteners Miskiewicz (*34A*) suggests paper chromatography for the detection of saccharin in the presence of dulcin, and Groebel (*19A*) has described a gas chromatographic method for saccharin after methylation with diazomethane. A colorimetric method for dulcin has been proposed by Ichibagase *et al.* (*24A*).

Methods for the analysis of sucrose fatty acid esters have been described by Armandola (*2A*), gas chromatographic analysis of their fatty acid composition by Tudisco and Turner (*57A*), and thin layer identification of the saturated long chain esters in these compounds by Linow and Ruttloff (*27A*). Enzymes have been used by Hankin and Wickroski (*21A*) for the determination of lactose in meat products, and the same authors (*22A*) suggest the use of amylases for the determination of maize syrup in prepared meat products. Mattioni (*33A*) has proposed a simple color test for the determination of caramel in beverages. A color test for sugar alcohols utilizing the reaction with a cyclic aldehyde in concentrated sulfuric acid has been described by Graham (*17A*). Glycerol in wine and must has been determined by a series of enzymatic conversion steps (*13A*). The ability of glycerol to form a copper complex has been used by McAloren and Reynolds (*30A, 31A*) as a means of both volumetric and colorimetric determination. Sorbitol has been determined in wine by paper chromatography (*32A*), and in chocolate as dibenzylidenesorbitol (*58A*).

Diethylpyrocarbonate is determined by gas chromatography of its decomposition product, diethyl carbonate (*40A, 41A*). Polarographic determination of nitrite has been suggested by Hartley and Bly (*23A*), and a colorimetric method using benzidine by Barakat and Sadek (*3A*). The control of interfering factors in the determination of sulfurous acid in products containing mustard oil has been described by Diemair and Bauer (*11A*). A simple screening procedure for traces of EDTA-type compounds in beer has been described by Stone (*53A*). Quantitative procedures for trace amounts of polyvinylpyrrolidone have been proposed by Shiraeff (*52A*). The gas chromatographic determination of higher alcohols in packing materials used in foods has been described by Reith *et al.* (*42A*).

ADULTERATION, CONTAMINATION, AND DECOMPOSITION

The investigation of indices for adulteration, contamination, and decomposition continues at a steadily increasing pace using a wide variety of techniques. The detection of mycotoxins, especially aflatoxin, has been of special interest to many investigators seeking to improve the sensitivity and specificity of available methods. Among the methods applicable to peanuts and peanut products is a rapid method utilizing a Florisil column clean-up and thin layer chromatography proposed by Heusinkveld *et al.* (*25B*); Sreenivasamurthy *et al.* (*68B*) proposed a paper chromatographic method. Robertson *et al.* (*60B*) have described a special extraction, and claim sensitivity of 2 ppb. A routine thin layer method has been described by Coomes *et al.* (*9B*). Chloroform extraction from moistened peanuts has been suggested by Lee (*40B*). A spectrophotometric method after thin layer chromatography for the four aflatoxins has been described by Nabney and Nesbitt (*51B*). A method for the classification of aflatoxin B_1 levels has been proposed by Coomes *et al.* (*8B*). The analysis of cottonseed and other products for aflatoxins has been described by Pons *et al.* (*54B*) and Engebrecht *et al.* (*17B*). Chen (*7B*) has described a method for aflatoxin in seed meals sensitive to 0.02 ppb. Strezleck (*69B*) has described a method for the separation of a fluorescent interference from aflatoxin B_1. Czaja (*12B*) has proposed a microscopic detection procedure for ergot and darnel in flour.

Among the procedure for antibiotics is one proposed by Alessandro (*1B*) who used both a colorimetric and a fluorescent procedure for chloramphenicol and tetracyclines in milk. Nisin has been determined by the use of special assay medium (*71B*). A sensitive method for penicillin has been proposed by Lemaitre (*42B*) which utilizes measurement of the rate of retardation of the acidification of milk.

In the investigation of oil mixtures methods for differentiating various olive oils continue to be of great interest. The selective hydrolysis of glycerides with pancreatic lipase has been used by Vela *et al.* (*73B*, *74B*) to detect esterified oils in olive oils. Infrared spectra at −30° have also been used to detect esterification (*5B*). Certain spectrophotometric indices have been proposed by Meduri *et al.* (*48B*) and by Jaforte (*31B*) to determine virgin olive oil in rectified olive oil. Eisner *et al.* (*16B*) have investigated the unsaponifiable matter of olive oils using column and gas chromatography, and have distinguished between pressed and solvent-extracted pomace oils. Thin layer chromatography has been proposed by Sliwiok (*66B*) as a method for detecting foreign vegetable oils in olive oil, and Valentinis (*72B*) has suggested a test depending upon the color developed with strong sulfuric acid. Gas chromatographic analysis has been used to detect adulteration of lard by Doro and Remoli (*15B*), Grieco (*22B*), Jacini *et al.* (*30B*), and Pascucci and Paolini (*53B*). The detection of animal oils in vegetable oil using gas chromatography of the sterols has been proposed by Recourt (*59B*). Gas chromatography of the unsaponifiable matter has been suggested by Guillaumin (*23B*) for the detection of foreign fats in butter; he also notes that gas chromatography of methyl esters and triglycerides may be helpful. Saponification and gas chromatography of the sterol acetates has been used by Cannon (*4B*) to detect foreign fats in dairy products. Adulteration of mustard oil has been detected by the number of spots obtained on a silica gel thin layer plate (*6B*). Fish oil in edible oil has been detected by Franzke (*18B*) after alkaline isomerization and spectrophotometric measurement. Rapid test tube chromatography has been suggested by Gorbach and Weber (*21B*) for the rapid detection of adulteration in pumpkin seed oil. Castor oil in other vegetable oils has been detected by the presence of triricinolein on a thin layer plate (*39B*). Triglyceride analysis using gas chromatography has been used by Leegwater (*41B*) to detect coconut oil or palm kernel oil interesterified with other fats. Thermal oxidative changes in frying fats have been studied by Rock and Roth (*61B*) who utilized the relationship of viscosity to the concentration of nonurea adducting fatty acids. The quinizarin reaction has been used by Jaforte and Piepoli (*32B*) to detect organic phthalates in oils.

The detection of polynuclear aromatic hydrocarbons has been studied intensely in the past few years. Thin layer chromatography has been used by Kohler *et al.* (*38B*). Genest and Smith (*19B*) have suggested extraction and thin layer detection for benz[*a*] pyrene in smoked foods. Howard *et al.* (*28B*) suggest a general method for these hydrocarbons in smoked foods and the detection of benz[*a*]pyrene in particular (*29B*). The latter compound has been detected in some smoked fish by Lijinsky and Shubik (*45B*). Chromatography on partially acetylated paper has been used to detect 3,4-benzopyrene in cereals (*2B*). Recovery studies of polynuclear hydrocarbons added to milk have been described by Howard and Teague (*27B*).

Freshness indices for foods include a description of an automated procedure for hypoxanthine (*36B*) and a study of hypoxanthine content as a means of judging the length of time fish has been held in storage (*67B*). Another index of decompostion in fish, the measurement of inosine-5-monophosphate, has been described by Jones and Murray (*35B*). A colorimetric procedure for the determination of trimethylamine nitrogen has been applied by Bethea and Hillig (*3B*) to distillates from fish to measure decomposition. Velankar (*75B*) has concluded that measurement of volatile reducing substances cannot be taken as an accurate spoilage index for fish stored in ice.

Residual solvents in solvent-extracted meals have been meaured by Lewis and Nelakantan (*44B*) using a special manometric apparatus. Residual hexane in meals has been determined by Mohan *et al.* (*49B*) by correlation of flash point *vs.* weight of solvent in the oil. Chlorohydrins in foods resulting from fumigation with ethylene and propylene oxide have been determined by Ragelis and Fisher (*58B*) using ether extraction and gas chromatography. The detection and examination of paraffin on raisins have been described by Werner *et al.* (*77B*). Thin layer chromatography has been used to detect adulterant mineral oil in vegetable oils (*47B*). Mineral oil in confectionery products has been detected by Motz (*50B*) using a semimicro method. Teague and Joe (*70B*) have described a method for the determination of unsaponifiables, including mineral oil, in milk.

An enzymatic ultraviolet method for the determination of uric acid in cereal foods has been described by Sen and Smith (*65B*). Studies of the solanum alkaloids indicate that they may be separated by thin layer chromatography (*64B*) and R_f values on silica gel are given (*52B*).

Peanuts have been detected in sesame products by measurement of the behenic acid in the extracted oil (*43B*). A spectrophotometric determination based on carotene content has been used by Roy and Bhalerao (*62B*) to detect buffalo's milk in cow's milk. Hole *et al.* (*26B*) have suggested histological procedures to identify offals in sausages. Czaja has suggested polarization-microscopic detection of maize flour in other flours (*14B*) and of latex vessels, particularly from coffee substitutes (*13B*).

Deterioration caused by alcoholic fermentation has been detected using special test paper (*20B*). Oxidative flavor deterioration studies by Privett and Romanus (*55B*) on egg powders have correlated UV spectrophotometric and organoleptic measurements. Procedures for measuring quality of milk chocolate and milk powders have been studied by Purr (*56B*) and several procedures were found which provide valuable measurements for judging quality. Bacterial or enzymic spoilage has been determined by Purr (*57B*) using a test paper for detection of esterases. Gibberellic acid in barley has been detected by Jones *et al.* (*33B*) by two-dimensional thin layer chromatography. The separation of gibberellic acid residue from rhubarb has been described by Kimura and Miller (*37B*). The effects of variations in procedure on the cryoscopy of milk have been discussed by Henningson (*24B*). Mercury in pink wheat has been determined by Jones and Schwartzman (*34B*) who used oxygen combustion and dithizone extraction.

Orange juice acids have been determined by Sanchez *et al.* (*63B*) as an aid to studies of adulteration, and Wucherpfennig and Franke (*78B*) suggest the thin layer determination of glycine as a means of determining adulterants in orange juice.

A paper chromatographic technique for screening volatile chemicals for their reactivity with foods has been described by Majumder *et al.* (*46B*). Residual vinyl cyanide and styrene monomers have been determined at the parts per million level by Crompton and Buckley (*11B*), and a polarographic method for traces of acrylonitrile monomer in liquid extractants has been described (*10B*). Plasticizers transferred to foods from plastic packaging materials have been determined by Wandel and Tengler (*76B*) using gas chromatography.

CARBOHYDRATES

Modern instrumental techniques have become more important in the analysis of the carbohydrate constituents of foods. These are discussed by Hall (*26C*) in Vol. 19 of the "Advances in Carbohydrate Chemistry." The official sugar analysis methods of Icumsa are collected in a text by DeWhalley (*16C*).

Increased use of gas chromatography for the determination of carbohydrates is shown by the number of papers published on this subject. Richey *et al.* (*42C*) have chromatographed the trimethylsilyl ethers of glucose and galactose. Martin (*36C*) has investigated the monosaccharides formed during fermentation by a similar technique. Alexander and Garbutt (*2C*) suggest the use of sorbitol as an internal standard for the gas chromatographic determination of glucose. Gas chromatography of the alditol acetates of monosaccharides has been suggested by Sawardeker *et al.* (*45C*) as a means of obtaining good quantitation. The trimethyl silyl derivatives of the methyl hexosides have been used to obtain good separations on gas chromatographic analysis (*51C*). Thin layer chromatography has been used for the separation of simple sugars on cellulose (*60C*), on silica gel (*37C*), and on silica gel impregnated with phosphate (*31C*). The latter system has also been used to separate sugars and sugar alcohols (*62C*). Column chromatography on extremely fine ion exchange resins is recommended by Arwidi and Samuelson (*3C*) for sugars, and sugar derivatives have been separated on anion exchange resins (*32C*). Carbon columns are used for the separation of glucose and fructose in refinery sirups (*53C*). Other analytical techniques for specific sugars include a method for fructose by enzymatic analysis (*63C*), lactulose by reaction with methylamine to form a violet reddish color (*1C*), and titrimetric determination of some monosaccharides by their reaction with basic permanganate (*4C*). Dialysis rates have been proposed by Siggia *et al.* (*50C*) for the analysis of mixtures of sugars and amino acids. The effects of heating saccharide substances have been studied by Lorant and Boros (*35C*) using a gravimetric thermal analysis procedure. Twenty-two compounds resulting from the thermal degradation of caramel have been identified by Sugisawa (*54C*). In an infrared study of glucose Segal (*47C*) has investigated the change of glucose from the β to the α form.

Chiba and Shimomura (*12C*) have accomplished the separation of oligosaccharides up to 6 glucose units on thin layer plates. Hydrolysis products of starch after enzyme hydrolysis have been analyzed by thin layer chromatography (*52C*). Molecular size distribution and separation of individual components of starches have been obtained by Ullmann (*59C*) using paper chromatography. Shibuya *et al.* (*48C*) report a photometric procedure for the carbonyl groups in dialdehyde starch using *p*-nitrophenylhydrazine. Microscopic evaluation, using potassium iodide–iodine has been used to determine amyloid in cell walls (*30C*). Oligogalacturonic acids from the enzymatic break-down of pectin have been detected by Koller and Neukom (*29C*) using thin layer chromatography. The quantitative aspects of the interaction of some hydrocolloids with cobalt salts has been investigated by Graham and Williams (*25C*).

A micro method for the determination of fructose, glucose, and sucrose in foods has been developed by Draper (*18C*) in the course of a study on browning in applesauce. Isotope dilution has been used to determine the true sucrose content of sugar beets and refinery products (*49C*). A study of the cell wall carbohydrates of the roasted coffee bean has been made by Thaler (*58C*). Gas chromatography of the trimethylsilyl derivatives has been used to determine the saccharide components of corn syrup through the tetra-saccharides (*9C*), and enzyme analysis has been used to measure the glucose in starch hydrolysates, corn sirups, and sugar solutions (*43C*). Dietetic foods have been analyzed for saccharide constituents using yeast fermentation, glucose oxidase, and invertase by Ruttloff *et al.* (*44C*). Paper chromatography has been proposed by Coleman *et al.* (*13C*) for the determination of glucose in egg solids. Carbon column chromatography has been used by Lees (*33C*) to separate maltotetrose from confectioners' glucose. Glucose oxidase, in which the contaminant invertase is inhibited by Tris buffer solution, has been used by White (*64C*), as a rapid photometric procedure for glucose in honey. Solubilization of the starch in meat products with dilute perchloric acid has been suggested by Glover *et al.* (*24C*) as a preliminary to the anthrone determination of the starch. Revised equations for the polarimetric determination of lactose in milk are suggested by Biggs and Szijarto (*5C*), and a simple colorimetric method for lactose in milk utilizing osazone formation is proposed by Wahba (*61C*). Glucose oxidase determination after invertase inversion is suggested as a method for sucrose in beet molasses by Taufel (*56C*). Column chromatography has been used by Binkley (*6C*) to isolate 1-kestose and 6-kestose from cane molasses, and Prey *et al.* (*41C*) have isolated raffinose from molasses using thin layer chromatography. Binkley (*7C*) has used paper chromatography for the same isolation. Planteose has been isolated from molasses using column chromatography (*8C*). Potter and Long have made several studies of the determination of sugars in plants suggesting the use of EDTA in the Munson-Walker sugar determination (*40C*), the use of ion exchange resin for clarification (*39C*), and the use of isopropyl alcohol for extraction (*38C*). Copper–EDTA mixture has been suggested by Tegge and Nierle (*57C*) as an improved method for determining reducing sugars in starch hydrolysates. Commerford *et al.* (*14C*) have described a macro paper–chromatographic method for the separation of glucose polymers up to 11 glucose units from corn–starch hydrolysates. Dextrins in wine have been detected by thin layer chromatography (*17C*). Gas chromatography of the trimethylsilyl ethers has been used to analyze the sugars of flavonoid glycosides (*28C*). Silica gel column chromatography has been used to separate and identify sucrose fatty acid esters (*34C*).

A clarifying agent including sulfuric acid, mercuric sulfate, and zinc sulfate has been suggested by Friedemann *et al.* (*20C*) for the removal of interfering substances in the determination of reducing sugars by the ferricyanide procedure. Studies of the Ofner reagent have been carried out by Friml *et al.* (*21C, 22C*). An application of the anthrone reaction to sugars after paper chromatographic separation has been described by Sunderwirth *et al.* (*55C*). The effect of toluene and similar compounds on the anthrone reaction has been studied by Devor *et al.* (*15C*). The interference of ammonium ions with the Somogyi-Nelson colorimetric procedure, and a means of preventing this interference has been described by Falanghe (*19C*). A review of volumetric methods for the determination of reducing sugars has been published by Hennart (*27C*). Studies of refractometric methods for sugar analysis have been made by Schmidt and Moller (*46C*), Glover, and Goulden (*23C*) and Charles (*11C*). Complete separation of mannitol from sorbitol has been obtained by Castagnola (*10C*) using thin layer chromatography. A simplified method for fibrous residue in breads has been described by Zentner (*65C*).

COLOR

Reflectance spectrophotometry has been used for the measurement of the color of translucent food samples (*19D*), for the determination of the color stability of heat-treated green peas (*17D*), as a means of measuring the myoglobin derivatives in meat samples (*33D*), and for estimating total meat pigment (*36D*).

Studies of the natural pigments in foods include an identification of the major anthocyanin pigments in Bing Cherries by Lynn and Luh (*20D*), and identification of pigments from black cranberries by Zapsals and Francis (*39D*). Date polyphenols and their relation to browning have been studied by Maier and Metzler (*22D*). A leucoanthocyan material was extracted from immature Elberta peaches as well as two related leucocyanidin compounds (*15D*). Morgan (*23D*) has identified 21 different carotenoid compounds in fresh pineapple. Two studies on tea include measurement of color in tea infusions including estimation of theoflavins, theorubigens, color, and brightness by Smith and White (*32D*), and the determination of the flavanols by paper chromatography (*24D*). Nutting, and Becker (*25D*) have made a study of methods using resin separation and two sets of equations for the determination of chlorophyll *a* and chlorophyll *b* in green vegetables. Methods for the rapid extraction of copper chlorophyllins in preserves, malvin in wine, and vegetable extracts in brandy have been proposed by Maglitto *et al.* (*21D*).

Studies of carotenes added or natural in citrus juices have been published by Benk (*3D*), using petroleum ether fractionation and thin layer chromatography, and in egg liquors of carotenoids foreign to eggs using alumina columns (*5D*). Benk and Brixius (*4D*) have also proposed a method for the determination of natural B carotene and added B carotene in vegetable oils. Capella *et al.* (*8D*) separate carotenoids in oils and feeding stuffs from the unsaponifiable matter using thin layer chromatography and assay spectrophotometrically. Carotenes, such as apocarotenal and canthoxanthin, used as colorings in food have been determined by Osadca *et al.* (*26D*) by solvent extraction, column chromatography, and ultraviolet measurement.

Annato and other food colors have been identified by thin layer chromatography by Ramamurthy and Bhalerao (*29D*), and the importance of using analytical grade reagents for this procedure has been stressed by Francis (*12D*).

Dyes have been separated from butter using alumina column chromatography (*38D*). Complex formation with quarternary ammonium salts has been suggested as a means of extraction of dyes from foods by Sohar and Sohar (*34D*). Artificial dyestuffs have been isolated from foods using acid activated alumina as the separation medium (*16D*). Bastianutti (*2D*) has described the separation and identification of methylene blue in bread-making flour and bread using Fehling solution B. Paper chromatographic R_f values of some nonpermitted synthetic colors have been tabulated by Pearson and Chaudhri (*28D*), as well as the R_f values of permitted water-soluble colors (*27D*). Ruiz (*30D*) has described the paper chromatography of triphenylmethane dyes, and Shelton and Gill (*31D*) have extended a single solvent paper chromatographic procedure to other permitted colors. More R_f values for Ponceau 6R and Ponceau Crystal as well as Chrysoin and Chrysoin SGX have been reported by Spalding (*35D*). Thin layer chromatography of some dyes on alumina and the measurement of their reflectance spectra has been discussed by Frei and Zeitlin (*13D*). Chromatography of fat-soluble food dyes on thin starch layers has been described by Davidek and Janicek (*10D*). Two solvent systems have been used by Dickes (*11D*) to separate 28 synthetic water-soluble colors by thin layer chromatography. Separations on thin layer plates of dyes authorized in Greece were found superior to paper chromatography by Synodinos *et al.* (*37D*). Electrophoresis of synthetic food dyes on cellulose acetate (*1D*) and on paper and thin films (*9D*) has been studied and good separations were obtained.

Paper chromatography of eosine has been found superior to column separation by Hanig and Koch (*14D*). The British Standards Institution has published standards for carmine (*6D*) and Red 6B (*7D*). Kroller (*18D*) has improved the spectrophotometric method for cadmium in dyestuffs.

ENZYMES

A simple gasometric apparatus has been described by Weeden (*15E*) suitable for the determination of yeast activity. Lipner *et al.* (*10E*) have studied the application of galvanic cells to the measurement of oxygen-consuming enzyme systems. Methods for the study of milk enzymes and the amounts of these enzymes found in cow's and human milk have been described by Wuthrich *et al.* (*16E, 17E*). The effectiveness of the falling-number method for evaluating α-amylase activity in cereals has been evaluated by Perten (*13E*). A modified method for the determination of both diastatic activity and α-amylase activity of malt on a single extract has been described by Bendelow (*1E*), and a method for the determination of limit-dextrinase activity in malted barley was reported by Greig (*3E*).

Guilbault and Kramer have continued their search for fluorometric enzyme activity methods, suggesting new substrates for dehydrogenase (*6E*), and cholinesterase (*4E*). Radioactivity, after action on a radioactive substrate has been used to measure esterase activity in cheddar cheese (*14E*). Peroxidatic activity has been measured spectrophotometrically using 4 methoxy-α-naphthol as substrate (*5E*). A dialysis method for the determination of phosphatase in dairy products has been described by Perlmutter and Lewis (*12E*).

McDonald and Chen (*11E*) found that the greatest sensitivity for the determination of proteinase activity is obtained by using the Lowry-Folin reagent for estimating proteolysis products. A review of methods for the determination of proteolytic enzymes has been compiled by Kucera (*9E*). Insoluble dye–protein complexes have been suggested as substrates for proteolytic enzymes providing a simple colorimetric means for the determination of enzyme activity (*7E*).

An enzyme which catalyzes the hydrolysis of pectins, pectates, and protopectins has been described by Gizis (*2E*) in a study of the pectic enzymes and substances in strawberries. Katz (*8E*) has described a potentiometric method for the determination of urease activity using a cationic sensitive glass electrode.

FATS, OILS, AND FATTY ACIDS

From the great number of papers published in the field of Fats and Oils since the last review, the authors have tried to include those articles which cover new or definitely improved techniques in fats and oils analysis. Minor modifications of previously published procedures have generally been excluded for the sake of brevity.

Among the review articles recently published are the papers from the American Oil Society's program on quantitative methodology in lipid research (*2F*), and the society's symposium on analysis of natural fat triglycerides (*3F*). Review articles on nuclear magnetic resonance spectrometry and its application to the fats and oil industry have been published by Harlan (*25F*). A literature survey compiled by Mangold (*48F*) covers thin layer chromatographic studies of lipids.

A new method for the extraction of fats from foods has been described by Winter (*93F*) using chloroform–methanol–water. Fats in chocolate as well as the liquid to solid ratios in this material have been determined by Oref (*63F*) using NMR. A refractometric method applicable to chocolate products has been described (*59F*). Fats have been extracted from dough mixtures and egg powders by Hadorn and Mostertman (*24F*) using a special apparatus. A special binary mixture of methanol–acetone was found by Parks and Hummel (*64F*) to yield the highest extract from fish meal. Nosel (*61F*) has suggested a rapid dielectric method for the determination of fat in fresh fish. Another refractometric method has been proposed by Rudischer (*76F*) as a rapid assay for fat in meat and meat products. Fat in milk has been measured by Walstra and Bosma (*92F*) using the hydroxamate method. The oil content of seeds has been determined by Conway, and Earle (*12F*) using wide line NMR. An improved method for the determination of the lipid content of starch using methanol–HCl has been suggested by Rogols (*74F*).

Lipid separations into gross fractions have been achieved by Carroll (*11F*) using acid-treated Florisil. Tipton *et al.* (*86F*) have used gel filtration to achieve preliminary separation of a lipid mixture. Wren and Szczepanowska (*96F*) suggest the use of an antioxidant to prevent peroxide formation in solvents used for chromatography of lipids. Gross distinction of lipid constituents on paper chromatograms has been obtained by Poxon (*70F*) using selected dyes which influence the shape of the chromatographic band. A special extraction for milk phospholipids has been described by Duthie and Patton (*15F*). Procedures for the isolation and analysis of phosphatides and sphingolipids have been discussed by Wagner and Wolff (*91F*). A method for sterols in cocoa fat has been described by Kroller (*38F*). Trimethylsilation followed by gas chromatography has been used by Rozanski (*75F*) to separate campesterol, β-sitosterol and stigmasterol.

Carbonyls have been determined by Linow *et al.* (*40F*) in the presence of hydroperoxides after boiling in anhydrous acetic acid. Studies of the oxygen uptake of fats have been described by Bishov and Henick (*6F*) using gas chromatographic measurement of the oxygen. Total and labile peroxide values have been determined by El Said *et al.* (*16F*) and the difference is said to represent the real stage of autoxidation of the fat. A modified Warburg apparatus for measuring oxygen consumption at constant pressure of oxygen is described by Marcuse *et al.* (*49F*). A study of the thiobarbituric value by Mihelic (*52F*) has indicated that this measurement is useful in controlling the freshness of fats. Moser *et al.* (*58F*) have described a fluorescent light test to measure the effect of luminous energy on flavor. A thin layer chromatographic procedure for the determination of oxidized glycerides has been described by Pokorny (*68F*). The measurement of *n*-pentane by gas chromatography has been used by Scholz and Ptak (*77F*) to detect rancidity in vegetable oils. Ultraviolet spectrophotometric measurements have been compared with peroxide values by Sedlacek (*79F*) and no relation has been found. Tarladgis *et al.* (*84F*) have studied the thiobarbituric acid test for rancidity and show that the acid–heat treatment is unnecessary. Pohle *et al.* (*67F*) have compared NMR and dilatometric determination of solids content of fats and shortenings. An improved method for the determination of unsaponifiables in butter oil has been described by Schwartz *et al.* (*78F*). A photometric procedure for iodine value of fats depending upon the formation of addition compounds with palladium chloride has been devised by Cuta and Stanek (*13F*).

Transesterification of fats and oils using 2,2-dimethoxypropane has been described by Mason and Waller (*50F*), and Simmonds and Zlatkis (*80F*) suggest the use of dimethylsulfoxide in this reaction to inhibit polymer formation. Peisker (*65F*) has described a rapid semimicro procedure for the preparation of methyl esters using chloroform–methanol–sulfuric acid. Rapid methylation has been claimed by Peterson *et al.* (*66F*) with the use of boron trichloride as methylating agent. A 10-minute conversion of fatty acids to methyl esters by Metcalfe *et al.* (*51F*) uses rapid saponification and the reaction with boron trifluoride. Special conditions for the gas chromatography of free fatty acids using a polar stationary phase on glass beads have been described by Nikelly (*60F*). Volatile fatty acids have been converted to their octyl ester and analyzed by gas chromatography (*14F*). Evidence has been obtained by Mikolajczak and Bagby (*53F*) for the migration of the double bond in methyl elaeostearate during gas chromatography. Ackman and Jangaard (*1F*) have identified the major unsaturated C_{16} acids in marine oils by using gas chromatographic separation factors found for C_{18} acids. Gerson *et al.* (*22F*) have prevented the loss of polyunsaturated acids in gas chromatography by using less-polar supports than ordinarily used for fatty acids. The determination of double bond positions in unsaturated fatty acids has been accomplished by Jones and Davison (*31F*) using oxidative cleavage, and gas chromatography. Fatty acid derivative analysis has been studied by Krishnan *et al.* (*37F*) and a method is described for the identification of functional groups based on logarithmic plots of retention time *vs.* carbon chain length. Critical pairs of fatty acids have been separated on paper by bromomethoxylation (*29F*). Catalytic hydrogenation on paper using palladium catalyst has been used by Kaufmann and Chowdhury (*34F*) before paper chromatographic analysis of fatty acids. Paper and thin layer partition chromatography of the *p*-phenylazophenacyl esters of C_1 to C_{10} fatty acids has been used by Vioque and Maza (*88F*) to separate these acids. A special separation using partition paper chromatography has been described by Sliwiok and Kwapniewski (*81F*) for the higher fatty acids. Critical pairs of higher fatty acids are separated by paper chromatography by Rankov and Rankov (*73F*) after obtaining the products from the reaction with iodine. Traces of fatty acids in milk fats have been determined by fractionation of their urea adducts (*100F*). Column chromatographic separation of the methyl esters of unsaturated C_{18} acids has been achieved by Wurster *et al.* (*97F*) using cation exchange resin containing silver ion. Nuclear magnetic resonance has been used by Johnston *et al.* (*30F*) to investigate the hydrogenation of linolenate. Proton magnetic resonance spectra of unsaturated fatty acids have been obtained by Purcell *et al.* (*71F*). A quantitative procedure for the β-olefinic group in fatty acid esters has been developed by Glass and Dutton (*23F*) using NMR. Spot tests for α-amino- and α-halogeno-fatty acids have been developed by Feigl and Yariv (*18F*).

Separation of isomeric long-chain polyhydroxy acids has been accomplished by Morris (*55F*) using paper chromatography. Thin layer chromatographic behavior of a number of oxygenated fatty acid derivatives has been described by Morris and Wharry (*56F*). Gas chromatography for hydroxy fatty acids has been described using methyl esters and acetoxy-acid methyl esters (*62F*). A review on methodology for hydroxy fatty acids has been published by Radin (*72F*). Gas chromatographic conditions for isomeric hydroxy and acetoxy stearates and oxostearates have been described by Tulloch (*87F*). Trimethylsilyl ethers of hydroxy fatty acids have been analyzed by gas chromatography (*94F*). Cyclopropenoid fatty acids have been determined by Bailey *et al.* (*4F*) by quantitation of the Halphen test. Fioriti *et al.* (*19F*) have described a new colorimetric method for epoxides using picric acid. A stepwise titration method using hydrogen bromide has been suggested by Harris *et al.* (*27F*) for the determination of cyclopropenoid and epoxy derivatives. Gas chromatography has been used by Herb *et al.* (*28F*) for the determination of epoxyoleic acid. Infrared examination of oil extract has been used by Magne *et al.* (*46F*) to determine cyclopropenoid fatty acids. An aqueous hydrochloric acid method for cyclopropenoid fatty acids has also been proposed (*47F*). Geometrical isomers of monoenoid fatty acids have been separated on talc by Carreau and Raulin (*10F*). Isolated trans isomers have been determined by infrared spectrometry and the errors in the method noted by Firestone and LaBouliere (*20F*). Argentation with a macro-reticular exchange resin has been used by Emken *et al.* (*17F*) to separate cis- and trans-fatty acids. Infrared measurement for trans isomers has been shown by Kaufmann and Mankel (*35F*) to be more accurate when applied to the methyl esters than to the glycerides, but if corrections are applied, the measurement may be made on the glycerides according to Luck and Kohn (*43F*). Gas chromatographic separation of cis-trans isomers has been reviewed by Litchfield *et al.* (*41F*). A gas chromatographic procedure using nitrile–silicone columns for cis-trans isomers has also been described (*42F*). The gas chromatographic behavior of fatty acids containing triple bonds on polar and nonpolar columns has been investigated by Zeman (*99F*).

Many investigators have studied techniques for the separation of glycerides. Thin layer chromatography has been used by Franzke and Jantz (*21F*) and Pokorny and Herodek (*69F*) for the separation of mono-, di- and triglycerides and fatty acids. Isomeric monoglycerides have been separated on silica gel impregnated with boric acid by Thomas *et al.* (*85F*); while Wood *et al.* (*95F*) have determined monoglycerides by gas chromatography of their corresponding trimethylsilyl ethers. Glyceride compositions of vegetable oils have been determined by Youngs and Subbaram (*98F*) by gas chromatography of the oxidized glycerides. An integrated system for the study of the triglyceride structure of natural fats has been described by McCarthy and Kuksis (*44F*), using three forms of chromatographic separations. Kaufmann and Wessels (*36F*), suggest the use of thin layer and paper chromatography and selective enzyme hydrolysis for the determination of triglyceride structure. The phase behavior of some triglyceride systems has been studied by Moran (*54F*) using microscopy, x-ray, and differential thermal analysis. Near-infrared spectroscopy has been used by Susi *et al.* (*82F*) to study hydrogen bonding, rotational isomerism, and the differentiation of diglycerides. Gas chromatography of the triglycerides from fish oils has been used by Harlow (*26F*) to detect molecules containing C_{20}–C_{24} fatty acids. Two-dimensional glass fiber paper chromatography has been used by Swartwout and Gross (*83F*) to partially separate triglycerides. Separation of oil triglycerides has been achieved by de Vries (*89F*) by column chromatography on silica impregnated with silver nitrate, and he has defined the fatty acid composition of the fractions by gas chromatography. Liquid-liquid column chromatography has been used by Black and Hammond (*7F*) to separate glycerides. Thin layer chromatography on silver nitrate treated silica gel has been used by Jurriens (*32F*) to separate triglycerides according to the number and location of the double bonds. Triglyceride separation on thin layer plates impregnated with paraffin has been described (*39F*). Distinct triglyceride fractions have been obtained by de Vries and Jurriens (*90F*) from oils by thin layer chromatography on silica gel–silver nitrate plates, and Blank *et al.* (*8F*) use a similar system combined with lipase hydrolysis to determine triglyceride structure. Barrett *et al.* (*5F*) have determined triglycerides in several oils using the silica gel–silver nitrate system.

Fatty alcohols have been analyzed by gas chromatography as the *p*-phenylazobenzoyl esters by Katz and Keeney (*33F*). Fatty amines have been analyzed by McCurdy (*45F*) as the trifluoroacetyl derivatives. The direct separation of fatty amides up to 24 carbon atoms was achieved by Morrissette and Link (*57F*) using a silica gel column to retain the amide. Subsequent detection of the nitrile with infrared spectrophotometry or thin layer chromatography is then applied (*9F*).

FLAVORS AND VOLATILE COMPOUNDS

As reported in the last review, gas chromatography (GC) continues to be the chief analytical tool in the flavor field, with more emphasis being placed recently on simultaneous separation and identification using gas chromatography coupled to spectrometric instruments. Rohwedder *et al.* (*80G*) described modifications to increase time of flight mass spectrometry sensitivity in a coupled GC application. Review articles have covered analytical progress in analyses of flavoring agents (*53G*), chemical and sensory aspects of odor identification (*109G*), and general gas chromatographic techniques with respect to food aroma research (*107G*). The correlation between quality and GC analysis of flavor extracts was discussed by Bayer (*8G*). Rhoades and Millar (*78G*) gave a GC method for comparative fruit flavor analyses, and Wahlroos (*103G*), Nawar (*70G*), and Jennings (*50G*) discussed a gas flushing concentration technique, direct headspace volatile analysis, and temperature and salt effects on headspace vapor equilibrium, respectively.

Derivative preparation continues to serve for secondary verification of identity. Soukup *et al.* (*90G*) prepared the 2,4-dinitrophenylhydrazone derivatives of various carbonyl compounds and separated them using GC, and Gadbois *et al.* (*32G*) reported a Girard-T reagent modification for concentrating carbonyls while Mehlitz *et al.* (*62G*) used thin layer chromatography to separate the 3,5-dinitrobenzoates of saturated and unsaturated alcohols. Methods for determining added flavoring materials have been reported such as ethyl butyrate in blended oils (*77G*), and benzaldehyde in flavors (*11G*), cordials (*10G*), and sirups (*92G*). Vanillin and related compounds have been determined in flavors using ultraviolet absorption (*26G*, *29G*), paper chromatography (*28G*, *89G*), and thin layer chromatography (*54G*). The detection of 5-hydroxymethyl-2-furaldehyde in vanilla caramels without interferences has been reported using paper and thin layer chromatographic techniques (*95G*). An enzymic procedure was used to determine low amounts of ethanol in beverages and aroma distillates (*97G*). Maltol was estimated in food materials by solvent extraction and GC using an internal standard (*102G*).

As in the past, many publications dealt with exhaustive identification of the individual components of flavor essences. Among the materials studied were white bread (*110G*), maple sirup (*27G*, *99G*), and wood smoke components (*46G*, *91G*). Apple juice (*34G*) and apple aroma (*57G*, *72G*) have been studied using GC. Andersson and Von Sydow (*4G*) identified the higher boiling compounds of black currants using, gas chromatography, mass spectrometry, and infrared. Winter *et al.* (*111G*) reported on the volatile sulfur and other compounds (*112G*) of the strawberry. Tomatoes have been examined for carbonyl compounds (*81G*) and volatile alcohols (*82G*) by Schormüller and Grosch. An investigation of bitter constituents in orange peel juice was reported by Swift (*96G*).

Schultz *et al.* (*84G*) identified juice volatiles using combined gas chromatography and mass spectrometry. Alcohols were identified in orange oil and aqueous essence by Hunter and Moshonas (*47G*) and Attaway *et al.* (*7G*). Ikeda and Spitler (*48G*), using column and gas chromatography, identified some esters and alcohols of lemon oil.

Terpene hydrocarbons have been analyzed using thin layer chromatography (*6G*) and terpene alcohols have been identified through their removal from a gas chromatogram by an injection esterification with boric acid (*40G*). Mitzner has reported the infrared spectra of some terpineols (*66G*) and Dhont and Dijkman (*20G*) have separated isomeric ionones and methylionones by multiple thin layer chromatography. Other fruits which have been examined with respect to their flavor and aroma composition are Concord grapes (*94G*). Bartlett pears (*41G–44G*) (*51G*), peaches (*52G, 85G*), and pineapple (*86G*). Coconut oil has been similarly analyzed (*3G*). The volatiles from off-flavor peanuts were partially identified using GC retention times by Pattee *et al.* (*73G*). Chive volatiles have been examined by Wahlroos and Virtanen (*104G*). The volatile carbonyls in stored unblanched frozen peas were identified using their 2,4-dinitrophenylhydrazone derivatives (*108G*). Potato chips have been studied with respect to their volatile carbonyl compounds (*15G, 67G*), the changes in volatiles during storage (*22G*), and 2,5-dimethyl-pyrazine content (*19G*). In potatoes, amino acids and sugars as flavor precursors (*24G*), alcohols and amines (*83G*), fat autoxidation and browning aldehydes (*14G*), and volatile sulfur compounds (*38G*) have all been reported. Graham has discussed the quantitative determination of piperine (*35G–37G*).

A procedure for the removal of volatile flavor constituents from cooked meat has been reported (*45G*). Wasserman and Gray (*105G*) fractionated the water soluble flavor precursors of beef, while Nonaka and Pippen (*71G*) and Minor *et al.* (*64G, 65G*) separated and identified flavor compounds in cooked chicken. Hexanal and valeraldehyde were found to decrease upon curing ham when the volatiles were analyzed by Cross and Ziegler (*17G*). Mendelsohn and Steinberg, (*63G*) using cryogenic gas chromatography, examined the volatiles from Haddock flesh and the relative change in these compounds on storage and cooking. An analysis of butter culture volatiles was reported by Lindsay and Day (*59G*) and a "mushroom compound" in butter fat and skim milk has been identified as oct-1-en-3-ol (*93G*).

Cheese aroma components have been separated as derivatives using paper chromatography (*76G*). Cheddar cheese flavor (*30G*), its hydrogen sulfide content (*58G*), and the aroma components of Blue cheese (*18G*) have been reported. Additional information about the volatile constituents of coffee has been supplied by Gianturco *et al.* (*33G*), Viani *et al.* (*100G*), and Weeren *et al.* (*106G*). The pyrazine in coffee aroma has been determined using polarography and oscillopolarography (*31G*). The aroma potential of cocoa beans was assessed using micro roasting of extracted precursors and subsequent gas chromatography (*79G*). Dietrich *et al.* (*21G*) extracted and examined cocoa bean flavor components and Boyd *et al.* (*9G*) studied monocarbonyl classes in beans and liquor. Kobayahsi, using gas chromatography, identified volatile organic acids in black tea essential oil (*56G*).

Beer flavor and flavor stability have been investigated by Harrison (*39G*), and Ahrenst-Larsen and Hansen (*1G, 2G*), and a bottle headspace sampling technique was reported by Jahnsen and Horn (*49G*). Hops have been analyzed for biacetyl and other components by polarography (*16G*) and for volatile constituents by combined gas chromatography–mass spectrometry (*12G, 13G*). The automatic analysis of beer alcohol content using Technicon equipment has been reported, requiring eight or nine minutes from sample introduction to data presentation (*5G*). Procedures have appeared for determining aromatic alcohols in beer (*61G*), and fusel oils (*23G, 68G, 74G*), the latter three involving gas chromatography. Some volatile constituents of Passion fruit wine were reported by Muller *et al.* (*69G*). The gas chromatographic determination of various higher alcohols in alcoholic beverages has been performed by Prillinger and Horwatitsch (*75G*) and Singer and Stiles (*87G, 88G*). Isomeric farnesols were separated from grape and other fruit brandies by thin layer chromatography (*98G*). Whiskey esters have been quantitatively determined using gas chromatography (*60G*) as has phenethyl alcohol in cider (*55G*). The determination of methanol in alcoholic beverages by various methods has been examined and discussed (*25G, 101G*).

IDENTITY

Many analytical studies of typical foods have been published recently; these data provide a basis for confirming the identity of a food or its adulteration or dilution with another food. Characteristic gas chromatograms of alcoholic beverages have been established by Bober and Haddaway (*5H*). The results of the analytical examinations of cognacs, rums, and whiskeys are reported (*24H*). A gas chromatographic study of rum constituents has been reported by Maurel (*44H*). Oenological indexes for the establishment of the authenticity of wines have been suggested by Ghimicescu *et al.* (*21H*). The constituents of apple aroma have been determined by Korytnyk and Metzler (*39H*) using gas chromatography. Czaja (*12H*) has suggested a microscopic method for distinguishing bean and pea meal. The betanin content of spray-dried beet powder may be used to judge the quality of this product (*64H*). The major free fatty acids in Blue cheese have been determined by Anderson and Day (*1H*), and in swiss chesse by Langler and Day (*41H*). A study of ewe's milk cheese has been described by Massimo (*43H*).

Several studies on the composition of fruits and fruit juices have been published. Osborn (*51H*) has presented data on many fruits, juices, jams, and jellys. The potassium, phosphorus, and nitrogen contents of citrus fruits from several localities have been determined by Money (*47H*), and by Hulme *et al.* (*30H*).. Carotenoid and sterol contents of lemon juice have been determined by Vandercook (*63H*). The measurement of betaine content has been suggested by Lewis (*42H*) as a means of evaluating the orange juice content of soft drinks. Hadorn (*28H*) has improved the method for the determination of theobromine in chocolate products and gives an average alkaloid content for fat-free cocoa. Coffee oligosaccharides have been studied by Glomaud *et al.* (*23H*). Determination of the anthocyanin pigments in Concord grape juice has been accomplished by Rice (*56H*) using paper chromatography. Averages and ranges of analytical data for American honey have been compiled by Schanderl and Bedford (*58H*). The composition of fruits and vegetables grown in the Middle East has been studied by Simaan *et al.* (*59H*). Analysis of the fats from pasta has been used by Franciosi and Giovannini (*19H*) to detect pasta without eggs or made either with eggs or frozen eggs. Studies of the fat from grain products have revealed improved methods for distinguishing hard and soft wheats. Guiducci and Morgantini (*27H*) have used the spectral curve from 7 to 10 microns for this identification, and Brogioni and Franconi (*6H*) also use infrared detection. Jaforte and Cavallaro (*35H*) use infrared measurement after column chromatographic fractionation, and Muntoni *et al.* (*48H*) have suggested gas chromatographic analysis of the fat and determination of the linoleic to oleic acid ratio. Franciosi and Giovannani (*20H*) suggest specific ratios of fatty acids determined by gas chromatography to distinguish hard and soft wheats and also the admixture of bran. The sitosteryl esters of semolina have been identified by Gilles and Youngs (*22H*). Olive oil mixed with lard in bread has been detected by gas chromatography (*50H*). Further identification of fats used in bread has been reported by Muntoni *et al.* (*49H*) using gas chromatography. Differences in the fatty acid content of the lipids of flour, shorts, and bran are reported by Burkwall and Glass (*7H*). Mineral contents of 10 hard wheat blends have been reported (*13H*).

Fatty acid ratios for butter fat have

been redetermined by Doro and Gabucci (*16H*). A study of cocoa butter and its substitutes, including the fat constants and paper chromatographic analysis of the fatty acid esters has been reported by Baroja *et al.* (*2H*). Ultraviolet spectra have been suggested by Fincke (*18H*) as a method of determining cocoa butter purity. Fatty acids in nut chocolate have been determined by Canuti (*9H*). Studies of coffee fat have been described by Calzolari and Cerma (*8H*) and by Carisano and Gariboldi (*10H*). The gas chromatographic determination of the fatty acids in pig fats has been accomplished by Jaarma (*34H*). Spanish lard has been analyzed by Romero (*57H*). Studies of olive oil using spectrofluorescence, (*37H*), ultraviolet examination (*46H*), and fatty acid composition (*33H*) have been published. Characteristics of safflower oil have been determined by Ibrahim *et al.* (*31H*) and Biffoli (*3H*). The effect of hydrogenation on the constituents of cottonseed oil has been described by Sreenivasan *et al.* (*61H*), and changes in some vegetable oils during processing have been investigated by Pallotta *et al.* (*52H*). Fatty acid contents of lipids from oil seeds and fruits have been determined by Grieco and Piepoli (*26H*); of vegetable oils, marine oils, and others by Iverson (*32H*); and of several food fats and oils by Hivon *et al.* (*29H*). Other analyses of fatty acids have been reported by Pettersson (*54H*) for vegetable oils, by Zeman (*66H*) for rape-seed oils, and by Kuemmel (*40H*) who found minor component fatty acids of vegetable oils. Glyceride composition of fats such as cocoa butter, peanut oil, palm oil, etc. has been reported by Jurriens and Kroesen (*36H*), and for 14 animal and vegetable fats by Subbaram and Youngs (*62H*). The results of the gas chromatography of the unsaponifiable matter from a number of oils has been given by Eisner *et al.* (*17H*).

Protein typing using electrophoresis has been suggested as a means of differentiating fish, pork, and beef by Payne (*53H*). Gas chromatography of the unsaponifiable fraction of the fat has been suggested by Cook, and Sturgeon (*11H*) as a means of identifying meats. Changes caused by treatment of milk were followed using an infrared analyzer (*25H*). The carotene content of the milk has been used to differentiate buffalo's milk and cows' milk by Singh *et al.* (*60H*). The structure of milk fat triglycerides has been determined by Dimick, and Patton (*15H*) and Blank, and Privett (*4H*).

A study of the chemical composition of green peas during ripening has been made by Racik (*55H*). The volatile aroma of fresh strawberries has been analyzed by Winter and Willhalm (*65H*). Lycopene content has been reported by Darbishire (*14H*) to be a poor means of determining the tomato content of a food. Mecca (*45H*) has published new iodine values suitable for differentiating fermentation and artificial vinegars.

INORGANIC IONS

The use of instrumental methods of analysis for metallic ions in foods continues to increase. Tuchscheerer (*45J*) described x-ray fluorescence methods. Tertian *et al.* (*43J*) determined arsenic by this technique. Interference by antimony in determining arsenic colorimetrically has been minimized (*18J*). Aluminum and cobalt were determined spectrophotometrically without ashing in beer (*36J*). Cobalt methods for beer are described by Segal and Lautenbach (*39J*). Calcium has been determined in milk by x-ray fluorescence (*2J*), visible colorimetry (*33J*), and constant potential amperometry (*38J*). The latter method also gives magnesium content. Alkali metals and calcium were measured by conductivity in molasses (*5J*). Interferences from manganese and aluminum were minimized in magnesium determinations in plant material by Chenery (*9J*). A copper determination in butter using atomic-absorption spectroscopy was described by Willis (*46J*). The sensitivity of the colorimetric dicupral method for copper in foods was increased by extraction of the complex (*34J*). Lead-210 in mill products was separated by complex formation and then counted (*40J*). Ion exchange was used to separate lead from other metals in food and other materials (*26J*). Atomic-absorption was used to estimate lead in chocolate and beer (*10J*) and fish flour (*42J*). Mercury was measured in wheat and tobacco by neutron activation analysis following ion exchange separation (*24J*). Nickel in hydrogenated fats was also determined by neutron activation analysis (*41J*). A sensitivity of 1 ppm for selenium in wheat using atomic-absorption was reported (*37J*). Tin and aluminum in beer have been determined by arc emission spectrography after precipitation (*3J*). Tin has been concentrated in beer by precipitation with tannin (*13J*) and determined colorimetrically in canned fruit juice and fruits with phenylfluorone (*23J*). Several procedures for the rapid determination of important radionuclides in foodstuffs and water are described by Bergner and Jägerhuber (*1J*). Plutonium-239 was counted after electrodeposition after various ashing procedures (*29J*). Strontium has been determined in food and bone by Goldman and Anderson (*16J*), and by Champion *et al.* (*7J*) using x-ray fluorescence. It has also been measured in foods by neutron activation analysis (*31J*) and flame photometry (*21J*). A variation on the method of Harley is claimed to be more rapid for strontium-90 (*11J*).

Neutron activation analysis has been used to determine bromine and iodine in sodium glutamate and flour (*8J*). The microdetermination of iodine utilizing its catalytic effect on cerium reduction is described for foods as well as for other materials (*32J*). Chlorine has been determined in bleached flour using x-ray spectrographic analysis (*15J*). Rapid potentiometric methods for determining salt in cured meat (*27J*), and cheese (*12J*) have been described. An extensive review by Hennart of the measurement of fluorine in organic materials has appeared (*19J*). The assay of hydrogen sulfide in beer has been examined by Jansen with respect to copper interference (*20J*). Nitrite and nitrate were determined in meat products by difference before and after reduction with metallic cadmium followed by azo dye formation (*28J*) and (*22J*). Nitrate has been determined by its interference with a rhenium complex (*4J*) and by solvent extraction with crystal violet (*47J*). A survey of the analysis of mixtures of mono and disodium orthophosphate is presented by Lóránt (*30J*). Phosphates in milk were separated by ion exchange by Hoff (*17J*). Ultraviolet measurement of the molybdophosphate complex was used to estimate inorganic phosphate in the presence of labile phosphate esters (*25J*). Condensed phosphates were separated on starch thin layers (*6J*), and on paper (*35J*). A new trapping system for sulfur dioxide analysis, permitting titration during analysis was described by Thrasher (*44J*). Carbon-14 has been determined in foodstuffs and oils by liquid scintillation counting after combustion and conversion to methyl benzoate (*14J*).

MOISTURE

The proceedings of the first International Symposium on Humidity and Moisture have been published (*1K*). Gas chromatography has been further applied for moisture determination in fruits and fruit products (*3K*). Synthetic porous polymer beads have been found to perform well as a GLC stationary phase for direct water analysis down to low levels in solvents by Hollis and Hayes (*7K*). The GC analysis of products formed by reaction of water with 2,2-dimethoxypropane provided the basis for a quantitative method by Martin (*9K*). Conditions for water determination in freeze-dried shrimp by oven drying were reported (*5K*) and a rapid infrared method for gelatin moisture was described (*8K*). An apparatus for rapid water determination in green malt has been tested and found satisfactory (*11K*). Bulk coffee bean moisture determination has been accomplished by neutron scattering (*12K*) and by neutron thermalization in lactose (*10K*). The Karl Fischer method was found suitable for molasses in a collaborative study (*6K*). Data on water activity for coffee, wheat, and peanuts is given by Ayerst (*2K*), and some interrelationships between storage stability and moisture sorption properties are given for dehydrated foods by Chandet (*4K*).

ORGANIC ACIDS

A review covering the analysis of propionic acid in foods by extraction, distillation, and chromatography has been prepared by Walker and Green (*39L*). Shortenings containing lactylated glycerides were analyzed for lactic acid (free and esterified) by Buswell (*6L*) and Pohle (*23L*). Lactic acid and several Krebs cycle acids were separated by thin layer cellulose chromatography and their spots were fixed with an aniline–ribose spray (*12L*). Schweiger (*32L*) also separated lactic and other organic acids on thin cellulose layers. Gas chromatography of esters of the tricarboxylic cycle acids has been reported by Kuksis and Vishwakarma (*16L*) and McKeown and Read (*17L*). Rohan and Stewart (*28L*) have found acetic to be the principal volatile acid and citric the major nonvolatile acid in cocoa beans. Several aromatic acids characteristic of fermented cocoa and other acids also present in unfermented cocoa were reported by Quesnel (*25L*).

Scarpati and Guiso (*30L*) showed that isochlorogenic acid of coffee is actually a mixture of three dicaffeoylquinic acids. The determination of caffeic and chlorogenic acids has been reported using paper chromatography and subsequent spectrophotometry of a color formed with triphenyltetrazolium chloride (*4L*), and by direct colorimetry at two wavelengths after color formation with nitrous acid in acetic acid (*1L*). Free and esterified quinic acid were measured before and after hydrolysis by oxidation of quinic acid to citric dialdehyde and color formation with thiobarbituric acid (*18L*). The presence of *p*-coumarylquinic acid has been demonstrated in wine by paper chromatography (*5L*). High results in citric acid determinations in coffee by the pentabromoacetone procedure were traced to interference by quinaldic and chlorogenic acids by Schormüller and Rubach (*31L*).

Paper chromatography and ultraviolet absorption were used to measure chlorogenic acid in potatoes (*38L*). In the determination of caffeic acid in potatoes, conversion of the cis-isomer to aesculetin occurs during acetic acid paper chromatography (*37L*), and a system for avoiding this conversion and measuring the caffeic acid was described (*9L*).

The organic nonvolatile acids of tea plants were separated on silica gel and reported by Sanderson and Selvendran (*29L*), and several volatile acids related to flavor were reported by Kobayahsi (*15L*). The ultraviolet spectra of several phenolic acids in methanol solution and the resultant bathochromic shifts on addition of aluminum chloride have been reported by Nakagawa *et al.* (*20L*). Pyruvic acid in wine was determined by direct spectrophotometry after enzymic reduction by Rankine (*26L*).

Further study of polarography as a method for determining fumaric acid in foods was recommended by Smith and Gajan (*34L*) and results of a collaborative assay were later reported (*33L*). Gradient elution chromatography with formic acid on Dowex 1-X10 was used to establish acid profiles in Concord and other grape products by Jorysch and Marcus (*14L*) and thin layer and gas chromatography of the acids in orange juice was used to detect adulteration (*24L*).

Because of side and incomplete reactions of various plant acids with diazomethane a mixture of thionyl chloride, methanol, and hydrogen chloride was recommended for methylation by Gee (*10L*). Organic acids have been separated in pear fruits by paper chromatography (*19L*), in beets by displacement chromatography on anion exchangers (*40L*), and in rice and other cereal seeds by ion exchange followed by silicic acid chromatography (*13L*). Paper chromatography and titration were used to study the 5-oxopyrrolidine-2-carboxylic acid content of beet molasses (*42L*).

Malic acid in must and wine was determined by titration after other acid interference was minimized (*8L*) and by chromotropic acid colorimetry (*27L*). A relatively specific fermentation procedure for malic acid was reported (*22L*). The analysis of lipulin and resin acids by ultraviolet measurement was performed by Askew (*2L*). Theiss and Chereches determined α-iso bitter acids in beer by ethanolic potentiometric titration with sodium ethoxide (*35L*), and Callesen *et al.* (*7L*) determined lead-titratable bitter acids in hop concentrate. Volatile acids were studied in beer by Conway dish diffusion and titration (*21L*), and in wine by distillation (*11L*). 2-Oxogluconic acid was determined in vinegar by paper chromatography and subsequent spectrophotometry (*41L*). Butter fat γ- and δ-oxo acids were detected by esterification and gas chromatography and also by conversion to lactones followed by gas chromatography (*36L*). Optical enzymic methods for determination of trace amounts of intermediate metabolites (such as oxo acids) were reviewed by Blouin (*3L*).

PROTEINS, AMINO ACIDS, AND NITROGEN

An intermediate level treatment of basic protein chemistry and reactions was published by Schultz and Angelmier (*37M*). New methods for protein separation and purification were discussed by Turba *et al.* (*48M*). A faster protein determination using an alkaline digestion was suggested by Kovacs (*23M*). Protein nitrogen factors for veal (*43M*) and liver (*42M*) were submitted by the Meat Products Subcommittee of the Society for Analytical Chemistry. The biuret reaction for protein determination in milk after the lactose interference is diminished was suggested by Schober *et al.* (*36M*) and a formol titration was suitable for protein in milk and ice cream (*12M*). Milk proteins have been separated by ascending boundary electrophoresis (*22M*), Sephadex G-100 filtration (*19M*), and diethylamino cellulose fractionation (*29M*). Protein fractions among other water-soluble constituents of bleached cake flour were separated by curtain electrophoresis (*44M*) and subsequent electrophoresis, dialysis, and paper chromatography (*45M*). Soft wheat proteins were separated by carboxymethylcellulose column fractionation and characterized with starch gel–urea electrophoresis (*15M*) and Wright *et al.* used Sephadex G-100 columns to fractionate proteins from baker's flour (*49M*). Other Sephadex gels were evaluated for wheat flour fractionation by Abbott and Johnson (*1M*). Polyacrylamide gel zone electrophoresis showed 15 to 17 apparent protein fractions from flour (*32M*). Fractionation of cereal flour proteins by continuous extraction was reported by Mosse *et al.* (*31M*). Protein chromatography on polyacrylamide gels has been discussed by Curtain (*9M*) and Fawcett and Morris (*13M*). Polarography has been used for continuous analysis of protein fractions eluting from a column (*21M*). Individual mononucleotides were separated by ion exchange chromatography by Macy and Bailey (*28M*). Gelatin has been detected in yogurt and curds by paper electrophoresis (*33M*). NMR has been used to determine the amino acids sequence of di- and tripeptides (*39M*). Analyses for protein-bound sulfhydryl groups (*52M*) and free and masked sulfhydryl in heated milk and milk powder (*27M*) were described. Various methods for determining sulfhydryl groups in meat protein were discussed by Hamm and Hoffmann (*18M*). Mercapto groups of albumins were determined with 2-hydroxyethyl 2,4-dinitrophenyl disulfide (*5M*).

Amino acids in coffee extracts, both free and after protein hydrolysis, were analyzed and reported by Thaler *et al.* (*46M*), and the free amino acid distribution in different cocoa bean varieties was examined by Rohan and Stewart (*34M*). Potato amino acids were separated and identified using ion exchange and paper chromatography (*14M*). An improved automatic analysis of amino acids has been reported by Thomas *et al.* (*47M*) and Benson and Patterson (*4M*) using a spherical resin. Thin layer silica gel chromatography of amino acid dinitropyridyl and nitropyrimidyl derivatives was studied by di Bello and Signor *et al.* (*11M*). Gas chromatography of volatile derivatives of amino acids has continued to receive attention. Hagen and Black described derivation formation and recommended columns (*17M*); Shlyapnikov recommended chromatographing the isopropyl or butyl esters of the *N*-acetyl derivatives on polar columns (*40M*); Blau and Darbre chromatographed the *N*-trifluoroacetyl derivatives of the pentyl esters (*6M*), and Lamkin and Gehrke worked with the *N*-trifluoroacetyl derivatives of the butyl esters (*25M*). Two-dimensional paper chromatography with 3-fluoro-4-nitropyridine *N*-oxide reagent was used by Skrabka-Blotnicka to separate the derivatives of casein hydrolyzate amino acids (*41M*). Tryptophan was determined in meat with *N*-bromo-succinimide (*7M*) and in foods and diets with *p*-dimethylaminobenzaldehyde (*26M*). Hydroxyproline is reported to give a more stable color in a modification of the Neuman and Logan method (*50M*) and another study (*38M*) reports elimination of tyrosine and tryptophan interference. Cysteine has been determined by an apparently specific reaction with noradrenochrome (*35M*) and by polarography (*51M*) along with methionine. A modification of the McCarthy and Sullivan method for methionine analysis was used for millet, casein, gluten, hemoglobin, brewers yeast, and ox-blood serum (*30M*). A microdetermination of choline in phospholipids was described by de Koning (*10M*) utilizing the molybdenum blue color after a sealed tube hydrolysis. Histidine, 1-methylhistidine, and 3-methylhistidine were separated on a thin layer of silica gel G in a rapid method for detecting whale extract in soup products (*8M*). Glycine has been determined in the presence of other amino acids after reaction with ethyl chloroformate followed by spectrophotometry at 420 mμ (*20M*).

Amino acid nitrogen content of worts and beer has been measured automatically by omitting a dialysis step (*2M*). Amino nitrogen analysis on cereal proteins by a Van Slyke procedure has been expedited by the use of a single-phase dimethyl sulfoxide system (*24M*). The determination of ammonium, amide, amino- and nitrate nitrogen in plant extracts by a modification of the Kjeldahl method of Varner *et al.* has been reported (*3M*). Trace amounts of nitrogen in vegetable oils can be determined after a Kjeldahl digestion with selenium, H_2SO_4, $CuSO_4$, and K_2SO_4 by using a modified Nessler reagent (*16M*).

VITAMINS

A summary report on a vitamin C methodology conference has been published (*9N*), and a review of chromatographic and extraction techniques for the aforementioned was given by De Ritter (*4N*). Ascorbic acid was determined, in the presence of ferrous and stannous salts, in canned fruit juices and drinks using EDTA chelation before titration by Pelletier and Morrison (*22N*). A new color reaction for ascorbic acid was shown by Sarma (*27N*).

High voltage paper electrophoresis (*15N*) and an improved paper chromatographic method (*23N*) have been used to separate ascorbic acid from other compounds. A method involving precipitation with $HgCl_2$ and subsequent titration of excess iodine with thiosulfate after oxidation of the HgCl precipitate was useful for colored fruit juice syrups and comparable in results to a potentiometric method (*14N*).

Ascorbyl palmitate was determined in stabilized fats using thin layer chromatography and spectrophotometry after colored derivative formation (*28N*). Ion exchange separation of vitamin B_6 components in grains and cereals was accomplished by Polansky *et al.* (*25N*), and pyridoxol was determined fluorimetrically as the cyanohydrin after oxidation to pyridoxal (*24N*). Vitamin B_{12} was separated chromatographically on Florisil from an orange juice beverage and eluted for spectrophotometric measurement (*26N*).

Naturally occurring vitamin A alcohols were separated and determined in foods by Hartel (*8N*). Suggestions for improving the assay of vitamin A were given by Deutsch *et al.* (*6N*) and a procedure for using a common saponification and extraction for vitamins A and D was reported (*5N*). Thin layer chromatography was used to determine tocopherols in fats without previous saponification (*10N*) and in the unsaponifiable matter from oils (*7N*).

Alumina column chromatography has been used to separate α-tocopherol from orange flavedo (*21N*). Gas chromatography has been used to determine vitamin D_2 after trimethylsilyl ether formation (*29N*) and to analyze for tocopherols and vitamin K (*2N*, *20N*). A rapid technique for the separation and identification of vitamins A, D, and E using thin layer chromatography was reported by Ludwig and Freimuth (*18N*). The advantages of using fluorescent dyes for detection of vitamin D after thin layer chromatography were reported by Chen (*3N*). Milk fat tocopherols were protected with ascorbic acid during saponification in a method by Krukovsky (*13N*). The polarographic behavior of some B, K, and E vitamins was reported by Knobloch (*11N*) and tocopherols in cottonseed and soya bean oils were determined. The measurement of free thiamine and its derivatives in foods by fluorescence was described (*19N*), and a specific microbiological assay for thiamine was detailed by Barton-Wright (*1N*). Riboflavine was determined in alcohol molasses residue using fluorimetry (*12N*).

A wick trough system for thin layer chromatography permitting horizontal, ascending, or descending chromatography was successfully used to separate large samples of feed extract unsaponifiables into vitamin A, xanthophyll, carotene, and extraneous material (*30N*). Vitamins K_1 and K_3 were separated from $K_{2(30)}$ and $K_{2(35)}$ by silicic acid chromatography (*16N*). The use of inorganic luminescent substances to show vitamin spots against a fluorescent background was described by Ludwig (*17N*).

MISCELLANEOUS

Allen (*1P*) has discussed food standards in the United Kingdom. A report by Grier (*8P*) summarizes the results of a survey of the problems, practices, and apparatus involved in preparing plant material for analysis. Van Soest (*24P*) presented a literature review on the use of crude fiber data for nutritional studies and described a new system utilizing detergents, the data from which are claimed to be more consistent with modern nutritional views. The English edition of a text on the use of thin layer chromatography on food constituents by Bollinger *et al.* (*3P*) has been published and includes chapters on vitamins, aliphatic lipids, amino acids and derivatives, nucleic acids and nucleotides, and sugars and derivatives. A publication dealing with malting, brewing, and allied processes has appeared (*16P*) containing a section on analytical methods.

Several additional papers dealing with the determination of caffeine and alkaloids have appeared. Borker and Sloman (*4P*) reported the results of a collaborative study on a modified Levine spectral-chromatographic procedure for caffeine in coffee and beverage sirup. Conacher *et al.* (*6P*) employed the reduction of 5-hydroxymethyl-2-furaldehyde with metabisulfite to remove its spectral interference in caffeine determination in coffee-chicory mixtures. Hadorn and Zurcher (*9P*) analyzed for caffeine in coffee by charring the other organic matter with sulfuric acid and subsequently using spectrophotometry after chloroform extraction and column cleanup. Jayaraman (*12P*) reported a method for estimating caffeine in beverages based on a color formed with a 2% malonic acid–acetic anhydride reagent. Kamp (*14P*) used ion exchange columns to separate caffeine, quinine, and strychnine in kola syrup. Kalberer (*13P*) investigated the leaves of *Coffea Arabica L.* to show caffeine decomposition and its breakdown products. The theobromine content of crude coffee was determined using thin layer chromatography by Lehmann and Martinod (*15P*). Raisch (*20P*) discussed the use of electrometric titrations on coffee infusions for following changes (in acids) during roasting. Solvent systems and procedures for the thin layer chromatography of alkaloids and anthocyanin pigments from plants were given by Paris *et al.* (*19P*). Chapman *et al.* (*5P*) used spectrophotometry to determine total alkaloids in cake and cocoa.

Minnaar *et al.* (*17P*) determined nicotine in flour by steam distillation and ultraviolet absorption. Cuzzoni *et al.* (*7P*) examined the purines of various meats to try to differentiate them but found no significant useful differences. The various volatile amines in beer have been reported by Hrdlicka *et al.* (*11P*). Sacher (*21P*) found *N*-behenyltryptamine in cocoa shell and gave some of its properties. Extraction, ion exchange, and colorimetry were the basis for an improved micro procedure for determining sulfonamides in meat, blood, and milk by Mooney and Pasarela (*18P*).

The content of egg in noodles has been estimated utilizing differences in the fatty acid composition of egg and flour by Armandola (*2P*). Hadorn and Zurcher (*10P*) substantiated the correctness of calculating egg content in dough based on magnesium sulfate–copper sulfate precipitable water-soluble protein differences. Van Stijgeren (*25P*) reviewed methods for determining egg yolk solids content by Lecithin phosphorus and recommended a procedure.

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Ferrous Metallurgy

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THIS REPORT reviews the advances in ferrous metallurgical analysis from October 1964 to December 1966. As in the past (*38*), it draws extensively from abstracts and is not intended to be a complete compilation of the papers published in the field.

The increased use of fast steelmaking, processes, vacuum deoxidation, and continuous casting techniques has greatly influenced the need for rapid and automated ferrous metallurgical analytical methods (*14*). In addition, stringent requirements on steel composition by steel users have forced the analytical chemist to search not only for rapid, but also for more accurate analytical methods. The majority of methods found in the literature still involve modifications of well established techniques and procedures rather than the introduction of new approaches.

Greatest progress was made in the application of atomic absorption spectrometry (*502*) and neutron activation techniques (*335, 357*) to the routine analysis of iron, iron ores, slags, and steels, and in the automation and computerization (*166*) of chemical and instrumental methods.

Present steel sampling and sample preparation techniques are generally too slow and inadequate for control of the new steelmaking processes and thus present the most apparent restriction to the rapid analytical methods. The realization of this problem was indicated by the relatively large number of papers published on sampling (*95, 428, 429, 480, 505, 511, 520*) and on attempts for the direct analysis of molten steel (*32, 135, 158–161, 344, 366, 567*).

Emission and x-ray spectrometric, atomic absorption spectrophotometric, and other instrumental techniques with various degrees of automation became the most important means for better process and quality control. Chemical routine methods, even the automated ones, decreased in importance except in the field of nonmetallic inclusion isolation where there is a new renaissance in the use of chemical approaches (*10, 293, 381*).

Schemes were given for the analyses of cast iron (*484*) and slags (*288, 417*).

Differential spectrophotometry (*522*), electrochemical methods (*380*), atomic absorption spectrophotometry (*502*), gases in metals analysis (*42, 214, 242, 265, 364*), and standard deviation of methods (*132*) in connection with steel analysis were reviewed.

ALUMINUM

Volumetric aluminum procedures involved variations of EDTA (*156, 257, 331*), bromatometric (*440*), and potentiometric (*134*) titrations. Modifications of the 8-hydroxyquinoline (*81, 112*), aluminon (*120, 448, 487*), eriochrome cyanin R (*220, 223*), solochrome cyanine (*411*), chrome azurol S (*69, 77, 421*), and alizarin red S calcium (*106*) spectrophotometric and of the 8-hydroxyquinoline spectrofluorometric (*200*) methods were described. Most procedures involved preliminary separations by mercury cathode (*109, 200, 440*), solvent extraction (*69, 112, 120, 200, 309, 411, 448*), precipitation (*200, 257*), or ion exchange (*309*).

Aluminum was polarographically determined in stainless steel (*443*), ferrotitanium (*199*), and ferro-niobium (*198*).

Neutron activation analysis (*22, 357*) and atomic absorption spectrometry employing a nitrous oxide–acetylene flame (*11*) were applied to the determination of low concentrations of aluminum in steel.

ANTIMONY

Antimony was determined spectrophotometrically as the methyl violet 6B complex after distillation separation (*554*), and also as the iodide complex (*245*), the Rhodamine B complex (*448*), or the antimony tetramethylenedithiocarbamate complex (*313*). Other methods involved the simultaneous polarographic determination of antimony, bismuth, and lead (after a sulfide separation) (*323*) and neutron activation analysis (*123*). The neutron activation method has little practical value because it involves 3.3 days' irradiation and chemical separations.

ARSENIC

Modifications of the molybdoarsenate spectrophotometric method were used for the determination of arsenic in cast iron and carbon steel (*315*) and in pig iron, steel, and ores (410). A two-step solvent extraction separation was necessary prior to the polarographic determination of arsenic in steel (519).

Wavelengths of 1806.2 and 1972.6 Å were found suitable for the direct spectrographic determination of arsenic in iron and steel (187).

Both nondestructive and destructive methods were discussed for the neutron activation analysis of arsenic in iron (357).

BERYLLIUM

A rotating disc spectrochemical technique was proposed for the determination of beryllium in steel (395).

BISMUTH

Bismuth was determined spectrophotometrically with thiourea (119) and xylenol orange (12), and polarographically (323, 443) after precipitation separations. Bismuth can be extracted with amyl acetate as the bismuth iodide complex and determined as such spectrophotometrically (336).

BORON

Boron contents greater than 0.1% in cast iron were determined by mannitol titration after ion exchange separation (101). The curcumin method was modified for the direct determination of boron in mild steels (217), and the methylene blue–dichloroethane extraction method was used to determine boron in steels containing niobium and tantalum (146). Pyrohydrolysis separation was employed prior to the constant current coulometric determination of boron (576).

A spectrographic method for the determination of 0.015 to 0.25% boron in stainless steel was developed (275). Trace amounts of boron in steel and iron were determined spectrographically using an aluminum counter electrode to suppress the iron spectrum (183), using a predispersion system to ensure complete separation of boron 2497.73 Å from Fe 2497.82 Å (539), using a carbon monoxide atmosphere to enhance the boron spectrum (298), and using a d.c. arc with the sample positive (276).

Thermal neutrons were also used for the determination of low boron contents in steel (165).

CADMIUM

Cadmium was determined in stainless steel by polarography (443) and atomic absorption spectrometry (568).

CALCIUM

No preliminary separation was required, but iron was masked with triethanolamine in the EDTA titration of calcium in iron (535), while precipitation separation of calcium was necessary prior to the spectrophotometric determination of micro amounts of calcium in steel (17).

CARBON

Direct oxidation at high temperatures remains the major method used for extracting carbon from steels. The emphasis has been on the different devices for measuring the evolved carbon monoxide and dioxide. Gas chromatographic instruments were extensively used (136, 272, 341), while titrimetric (60, 253, 254) and gravimetric (206) methods were still employed. Infrared (562), low pressure measurement (21), and flame ionization detection (516) were also described. Low temperature combustion and a modification of the Strohlein apparatus was described for the simultaneous determination of carbon and sulfur (444). In the calibration of instruments below 50 ppm carbon, gaseous carbon dioxide was recommended (372).

Emission spectrographic methods were investigated (191, 321), including one for determining carbon in molten metal (477). Newer techniques include activation analysis using low energy deuterons to determine carbon in steel (425), and liquidus arrest methods for cast irons (209, 512, 552) and steel (300).

For production control purposes, carbon was determined in molten metal indirectly with infrared analyzers by measuring the carbon dioxide evolved during the high temperature combustion of steel in oxygen (135, 567).

CHROMIUM

Ferrous sulfate–permanganate titration with a photometric detection of the end point was applied to the determination of chromium in steels containing vanadium and tungsten (345). Chromium was separated from other metals by ion exchange and then determined spectrophotometrically with chromatropic acid (435). In the presence of large amounts of vanadium, a modification of the diphenylcarbazide method was used (238). Other methods reported for the determination of chromium in steel include amperometric titration (155), paper chromatography (525), flame photometry (438), atomic absorption spectrophotometry (29), and neutron activation analysis (357).

COBALT

EDTA titration was used after separation of the cobalt from interferences by ion exchange (219). Nitroso-naphthol (404, 540), diiodobis (dimethylglyoxime) (80), and *N*-mercaptoacetyl-*p*-toluidine (85) were recommended for the spectrophotometric determination of cobalt in steel. The rapid approaches included atomic absorption (502) and flame photometry (438). The neutron activation determination of cobalt in steel is slow, both by the direct method (179, 357), and by the indirect method involving chemical separation (225, 357, 384).

COPPER

Carbamic acid derivatives continue to be the principal reagents for the determination of copper in steel. Spectrophotometric methods were proposed with dithizone (401), sodium tetramethylenedithiocarbamate (314), and sodium diethyldithiocarbamate (93). Dithizone solvent extraction separation was used prior to polarographic analysis (164), and diethyldithiocarbamate extraction before EDTA titration (197) or activation analysis (322).

Other techniques for copper determination were polarography (87, 305, 400), flame photometry (438), turbidimetry (115), atomic absorption spectrometry (502), anodic-stripping voltametry (192), and neutron activation analysis (357).

GASES IN METALS

The commercial application of vacuum steelmaking and continuous casting techniques along with the intensified demand for "cleaner steels" has resulted in an increased importance for the analysis of gases in metals. Several papers contained reviews with extensive bibliographies (42, 214, 242, 265, 364).

In the past two years, neutron activation has become an accepted method for oxygen determination (68, 82, 168, 169, 307, 339, 340, 375, 409, 422, 424, 550, 572). However, most papers still dealt with modifications of well established fusion (20, 153, 176, 212, 343, 399, 496, 499, 500, 536), carrier gas fusion (147, 390), and emission spectrometric (41) methods.

Various sampling techniques were reviewed, compared, and evaluated (94, 124, 213, 316, 325, 463, 473, 480, 520). The newer steel sampling methods included the use of a sampling mold which permitted the collection of gases evolved during solidification (154) and sampling under protective atmospheres (1, 420).

Hydrogen. Hermetically sealed mild steel tube standards containing known pressures of hydrogen (504) and primary steel standards (104) were developed for hydrogen in steel analysis.

Hot extraction under vacuum (2, 42, 103, 142, 148, 167, 176, 212, 348, 524) and carrier gas extraction (147, 154, 188, 189, 268) have remained the principal methods for the determination of hydrogen in steel.

The electrochemical–gas volumetric (474) and the electrochemical–coulometric (163) methods were shown to have little promise. However, when the evolved gases were analyzed both for hydrogen and nitrogen, good results were claimed (*96*) for hydrogen in steel.

Nitrogen. Several "wet" chemical and instrumental methods were described for nitrogen analysis in plain carbon and silicon steels (*350*).

Modifications of the Kjeldahl distillation separation indophenol-blue method (*7, 23*), and of the isobutyl alcohol–indophenol-blue solvent extraction were reported (*261, 396*). Electrochemical methods were based on the conductometric determination of nitrogen absorbed as ammonia in sulfuric acid (*180*) and on the analysis of the gases liberated during the anodic dissolution of the sample (*96*). This latter method is also suitable for the simultaneous determination of hydrogen and nitrogen.

Complete extraction of nitrogen from steels is still a great problem. Vacuum fusion and vacuum hot extraction techniques were critically evaluated and compared with other methods (*193*). Various oxidizing fusion techniques (*52*), an iron bath (*476*), a platinum bath (*149*), an addition of nickel–cerium alloy and emptying the crucible after each determination (*178*), and other small modifications (*20, 176, 177, 212, 302*) were recommended for the established vacuum fusion determination of nitrogen in steel. Oxidation fusion was also applied to the determination of nitrogen in slags (*98*).

Direct current carbon arc excitation was used prior to the gas chromatographic determination of nitrogen (*569*). Several modifications were made to improve carrier gas fusion method (*76, 147, 215, 270, 271, 297, 390*) and the isotopic dilution technique (*151, 152, 171, 419*).

Oxygen. New standards for oxygen in steel became available through the National Bureau of Standards (*371*) and the preparation of standards for oxygen determination in steel from aluminum isopropoxide and iron powders was reported (*105*).

Nephelometric methods were used when no analyzers were available for the determination of oxygen in steels (*237, 352*).

While neutron activation analysis became a practical approach for the determination of oxygen in steels (*40, 68, 82, 168, 169, 307, 339, 340, 375, 422, 424, 550, 572*) and metal powders (*409*), various vacuum fusion methods still play an important role (*176, 212, 302, 534*), and modifications of the carrier gas fusion techniques (*76, 147, 215, 235, 284, 297, 390, 553*) are commonly used for the routine determination of oxygen in steel.

In vacuum fusion (*20, 176, 318, 514*) and carrier gas fusion (*235*) techniques, and in neutron activation analysis, very rapid methods were developed requiring only one to two minutes for each oxygen determination. Unfortunately, the sampling and sample preparation techniques are much slower (*1, 94, 213, 316, 420, 463, 520*).

In connection with vacuum fusion techniques, the effects of aluminum and manganese (*303*), the sources of error with aluminum-killed steels (*297, 319*) and the mechanism of the reduction of oxide inclusions (*44*) were studied. The effect of a platinum bath was also critically evaluated (*149*).

Most carrier gas fusion methods were based on the gas chromatographic measurement of oxygen, but coulometric titration (*1, 190*) and conductometric (*464*) methods were also used for steels, and gravimetric methods were used for the macro determination of oxygen in metal oxides (*230, 236*) and iron powders (*128*). Besides conventional fusion extractions, arc-fusion techniques (*267, 268, 569*) were successfully applied to the extraction of oxygen from iron and steel samples.

Isotope dilution methods were also recommended for the rapid determination of oxygen in steels (*172, 337, 557*) and iron powders (*231*).

Since sampling techniques are too slow for modern steelmaking processes, methods are being developed for the direct determination of oxygen in molten steel by electromotive force measurements (*32, 158–161, 344, 366, 461*).

HAFNIUM

Variations of the arsenazo III spectrophotometric method were used for the determination of hafnium in iron and steel (*28, 262*) and the method was shown to be more interference-free and faster than the xylenol orange method (*28*).

LEAD

Prior to spectrophotometric measurement as the dithiozanate, lead was extracted with dibenzyldithiocarbamic acid–chloroform from nickel–chromium steels (*508*), and with dithiozone–carbon tetrachloride from steels and cast iron (*157*). Pyridylazoresorcinol was used (instead of dithizone) in the photometric determination of lead in steel (*113*).

Differential cathode-ray polarography was employed in the trace determination of lead in stainless steels (*443, 454*). Conventional polarographic techniques were used for the determination of lead without preliminary separation (*418, 467*) and after separation by ion exchange (*210, 398*), solvent extraction (*400*), or precipitation (*211, 323*).

Atomic absorption was employed to determine lead in steels after solvent extraction separation of the interferences (*111*). Lead was concentrated by coprecipitation with strontium sulfate and bismuth was added as the internal standard prior to spectrographic analysis (*116*).

MAGNESIUM

No separation was required in the EDTA titrimetric (*535*), in the atomic absorption spectrophotometric (*185*), and in the flame photometric determinations of magnesium in steel (*438*).

Magnesium was determined in nodular cast iron after irradiation with 12-Mev deuterons and chemical separation (*75*).

MANGANESE

Volumetric procedures were modified for the determination of manganese in steel and iron (*65*), in plain, stainless or tungsten steels (*50*), in stainless steels (*45*), and in ferromanganese (*67*). Manganese was spectrophotometrically determined as the MnO_4^- complex (*8, 377*) and with formaldoxime (*181*). In the amperometric titration of manganese, two indicator electrodes were used (*155*). Automatic potentiometric (*469*) and spectrophotometric (*471*) methods were also developed for manganese determination in steelmaking alloys (*469*) and steels (*471*).

Because atomic absorption spectrometry is relatively interference-free, it was widely applied to the determination of manganese in iron and steel (*39, 502*) and iron alloys and alloy steels (*521*).

Emission spectrographic methods were investigated for interferences due to alloying elements (*367*) and sulfur (*92*).

Nondestructive neutron activation analysis was successfully used for trace amounts of manganese in iron (*357*).

MOLYBDENUM

Various spectrophotometric procedures using thiocyanate continued to be the main methods for determining molybdenum in steel (*35, 57, 67, 118, 308, 346, 351, 358, 436*). Other reagents include mercaptoacetic acid (via solvent extraction) (*445*), *N*-2-mercaptopropionyl-*p*-phenetinidine (*86*), toluene-3,4-dithiol (*273*), and thioglycollic acid (*251*).

Molybdenum was determined in ferromolybdenum by direct titration (*412*) and amperometrically in alloy steels with 8-mercaptoquinoline (*51, 517, 518*). Polarography was used after the solvent extraction of molybdenum (*523*) or after the precipitation separation of interferences (*423*). Vanadium- and tungsten-bearing stainless steels were analyzed for molybdenum with a modification of the 8-hydroxyquinoline gravimetric method (*442*).

Acetylene–air (*391*) and acetylene–nitrous oxide (*286*) flames were employed for the atomic absorption determination of molybdenum in steels. Sensitive activation methods employing epithermal neutrons were also offered for determining molybdenum in steel (*74, 357*).

NICKEL

A modification of the dimethylgloxime method for determining nickel was used with steel containing cobalt and copper (*99*). After ion exchange separation (*219*), solvent extraction separation (*437*), precipitation separation of nickel with dimethylglyoxime (*201*), or of interferences (*73*), EDTA titration was used for the determination of nickel in iron and steel.

Atomic absorption (*283, 502*) and flame photometry (*438*) were successfully applied for the rapid determination of nickel in steel.

NIOBIUM AND TANTALUM

Separation of niobium and tantalum from interfering elements is usually necessary prior to determination and can be achieved by solvent extraction (*330*), ion exchange (*393*), or precipitation (*133, 462*).

A combination of gravimetric and spectrophotometric procedure was used to determine niobium in ferro-niobium (*198*). Various complexing agents were employed in the colorimetric determination of niobium in steels (*144, 246, 250, 382*), alloy steels (*61, 466*), and heat resisting alloys (*327, 460*). In stainless steel niobium was determined polarographically (*277*).

Emission spectrographic methods which were developed were a point-to-plane technique using a spark discharge in argon atmosphere (*274*) and a rotating-disc solution method (*241*). An x-ray spectrochemical procedure was also offered for niobium and tantalum in which the sample was converted to oxides and pelletized (*533*). Neutron activation analysis can be used for the determination of microquantities of niobium in steel after preconcentration steps involving extraction and precipitation (*555*). Various chemical methods for the determination of niobium and tantalum in steels and isolated carbides were investigated and evaluated (*46*).

The spectrophotometric methods for the determination of tantalum involved the use of pyrogallol (*393*) and malachite green (*246*).

NONMETALLIC COMPOUNDS

The number of books (*10, 293*) and articles (*381*) on the isolation, identification, and determination of nonmetallic compounds in steel increased with the rising demand for quantitative evaluation of "steel cleanliness" (*571*) and with the introduction of new steelmaking processes.

Most methods described for the isolation and analysis of nonmetallic inclusions are improvements of established methods. Mechanical isolation with an ultrasonic "jack hammer" (*173*) and chemical isolation by chlorination (*228, 279, 528*), bromination (*355*), selective dissolution techniques (*36, 234, 360, 387, 415, 446, 528*), and electrochemical approaches (*48, 233, 234, 260, 289, 290, 299, 406, 433, 575*) were used for the isolation of various nonmetallic inclusions and for the separation of austenite and martensite.

Flotation was employed for the removal of graphite from isolated carbide residues (*49*). To decompose carbide inclusions in isolated residues, $HIBr_2$ (*291*) and dilute HCL-$KClO_4$ (*386*) solutions were used, and to differentiate between the various carbides of chromium, H_2O_2 (*577*) was employed. Methods were also reported for the isolation of carbides of niobium (*360, 387*), titanium (*543*), and vanadium (*414*); nitrides of aluminum (*36, 407, 528, 548*), niobium (*360, 387*) and silicon (*575*); oxides (*228, 234, 260, 369, 415, 446*); and sulfides (*249, 370*).

Chemical microanalytical methods (*295, 296, 385, 580*) and spectrographic solution techniques (*175, 258*) were improved for the analysis of isolated residues.

Identification and determination of inclusions were accomplished by x-ray instrumentation (*16, 63, 479, 545*), an emission spectrograph with a micro setup (*304*), electron probe microanalyzers (*102, 107, 162, 222, 252, 294, 416, 451*), a laser micro probe (*368*), and differential thermal analysis–effluent gas analyzers (*26, 27*).

PHOSPHORUS

Phosphorus was determined in steel by titration with hydroxyquinoline (*338*) and as the phosphorous molybdenum blue complex after precipitation-separation (*498*), solvent extraction separation (*34, 130*), ion exchange separation (*556*), and distillation separation (*510*). The latter method was applied without separation to ferromolybdenum (*482*), to ferrochromium (*483*), to ferrosilicon (*402*), and to iron (*181*). Modifications of the molybdenum blue method extended the range of analysis to 0.0002% phosphorus in a 0.1-gram sample (*263, 546*) and was also automated for determining phosphorus in steel (*471*). Neutron activation analysis was successfully applied to the simultaneous determination of phosphorus and sulfur (*22, 59*).

RARE EARTHS

Cerium was determined after ion exchange separation by titration with EDTA (*5*), and colorimetrically after precipitation separation (*194*), ion exchange separation (*453*), and solvent extraction separation (*403*). Cerium was nephelometrically determined as the iodate (*456*). Spectrographic methods were used for the determination of yttrium (*240, 241, 388*), samarium, gadolinium, dysprosium, erbium, and scandium (*150*), and neutron activation methods for cerium (*356*) and scandium (*357*) in various steels and alloys.

SELENIUM

Selenium was determined in steels as the yellow complex of selenium(IV) with 3,3′-diaminobenzidine hydrochloride (*413*) and in iron by neutron activation analysis (*356*).

SILICON

Acid resisting high silicon iron was decomposed with a mixture of solid ammonium chloride, bromine, and hydrobromic acid (*64*). The K_2SiF_6 titration for silicon was applied to cast iron, alloy materials, and slags (*389, 495*), and the heat of formation of K_2SiF_6 was measured in the rapid thermometric determination of silicon in ferrosilicon (*547*).

Photometric techniques of silicon analysis in steels were reviewed (*317*), and precautions were outlined for the use of the molybdenum blue methods (*62*). A modification involved the preliminary diffusion separation of silicon as SiF_4 (*232*) and another, the complete automation of the molybdenum blue method (*471*). Several other modifications were also used for the determination of silicon in low alloy steels and irons (*89, 121, 181, 224, 247, 285, 478*), in ferromolybdenum (*328*), and in oxide inclusions and irons (*280*).

The silicon results obtained on various steels by neutron activation analysis were accurate (*22*).

Thermoelectric voltage measurements were used for the determination of silicon in cast iron (*145, 312, 320*).

SLAGS AND ORES

New schemes were recommended for the rapid analysis of iron and steel slugs (*288, 470*). The analyses for the oxides of manganese, phosphorus, aluminum, and of total iron were automated (*472*).

Sintering with a mixture of Na_2CO_3 and oxalic acid was used (instead of fusing) for the quantitative dissolution of iron ores in HCl (*426*).

Complexometric methods were further refined for the determination of aluminum (*450*), calcium (*332, 450*), iron (*560*), and magnesium (*332, 450*) in slags, and for arsenic (*122*), calcium (*84, 362*), iron (*84, 560*), and lead (*282*) in iron ores.

In slag analysis titrimetric procedures were employed for the determination of fluoride after ion exchange separation

(383) or after volatilization (as H_2SiF_6) (491); for SiO_2, after K_2SiF_6 precipitation separation (389); and for carbon after its volatilization as CO_2 (558). Electrometric methods included a polarographic method for copper (559), a potentiometric method for aluminum (134), and an amperometric method for vanadium (513). Oxidative fusion gave good results in the determination of nitrogen in slags (98), and methods were reviewed for the determination of hydrogen in slags (129).

In ores, photometry was used for iron (565), oscillography for cadmium (564) and nickel (563), and polarography for zinc (282).

The photometric methods for ore and slag analysis were reviewed (565).

Thermometric methods were used for rapid determination of basicity and FeO content of steel slags (392), and of SiO_2 in ores and slags (458).

In the atomic absorption spectrophotometric determination of calcium and magnesium in slags, the interferences were eliminated by the addition of strontium (185).

The emission spectrographic methods for the analysis of slags included various solution techniques (196, 202, 541), the use of rotating disc with adhesive (239), and pelletization (or briquetting) techniques (54, 55, 70, 88, 138, 140, 174, 447).

Pelletization techniques were also used for the spectrographic analysis of iron ores (292, 447).

The great number of x-ray fluorescent methods indicates the necessity for fast and reliable slag (125, 139, 306, 430, 431, 488, 506, 570) and iron ore (184, 218, 452, 455, 475, 485, 570) analyses.

Other techniques involved the use of electron probe microanalyzers for the analysis of ores and slags (127), radioisotope–x-ray fluorescence (566) for the determination of CaO, iron, and SiO_2 in sinters, a radiometric approach (204) for iron in iron ore, and neutron scattering techniques for moisture determination in iron ore and sinters (326).

SPECTROMETRY

Emission. Progress in this field continued at a fast rate even though atomic absorption and x-ray spectrometry are in many cases in direct competition. The role and modern trends of emission spectrometry were discussed (13, 256), and its use as a rapid control method was described (574).

The vacuum spectrometric technique was investigated as to source conditions (347), to the purity of argon (529, 537), to various gas mixtures (311), and to the effect of pressure (186). The development of vacuum spectrometry and the instruments available were discussed (137).

The effects of source parameters and various metallic counter electrodes were studied in argon atmosphere (527), and the effect of sample mass in various atmospheres was investigated (79).

As the instrumental methods improve, it is becoming more apparent that sampling is most important and efforts to ensure that the sample analyzed is representative of the whole is increasing. For increased speed of analysis by vacuum spectrometry, thin suction samples were used (374); and for sampling molten iron, it was concluded that the samples should be taken from the transfer ladle (83). Pressing of lathe turnings proved very successful (509), and a comparison was made between remelting and briquetting of chips (229).

A number of evaluations of the laser as a source were reported (6, 203, 457), and the plasma arc was studied for alloy analysis (394).

Spectrochemical methods for trace analysis which were proposed included preconcentration techniques such as solvent extraction (449), ion-exchange (33), and mercury–cathode electrolysis (551). Carrier distillation procedures were also used for trace analysis (248, 342).

New white cast iron standards were prepared (378), and an evaluation of various sampling techniques was made (269). Methods were proposed for the analysis of cast irons (24, 363, 365, 492, 494) and pig iron (126, 503).

Elimination or minimization of matrix or interelement effects is described in methods for the analysis of high alloy and stainless steels (9, 30, 72, 97, 281, 376, 542). Slag analyses by pelletizing (53, 70) and powder-dilution (481) were described. The direct analysis of inclusions in situ including possibly the determination of oxygen and nitrogen was proposed as a method (41).

The use of computers for photographic and direct reading instrument increased. One installation was described, including the mathematics used for calibrating, calculating, and correcting for matrix and interelement effects (18). A computer was used to give automatic programming of conditions for the analysis of pig irons (126).

Flame. A brief account of the uses of flame photometry in alloy analysis was given (561), and a review of the use of solvent extraction in flame photometry for metals analysis was presented (441).

Mass Spectrometry. The advantages of the solids–mass spectrometric method of analyzing iron and steels were described (108, 468). The simultaneous determination of carbon, oxygen, and nitrogen in iron with a limit of detection better than 0.5 ppm, was reported (216).

X-Ray and Electron Microprobe. The use of x-ray spectrographs as routine laboratory instruments has increased greatly (58, 114, 287, 486). A comparison of x-ray and optical emission analyses of alloy steels showed that the x-ray analysis is more reproducible (91). X-ray spectrographic methods were used for the analysis of cast iron (324), stainless steels (109, 208), alloy steels (9, 349, 526, 579), and ferrochrome (170). A special application of x-ray fluorescence is the determination of coating thickness of tinplate (56).

Methods of sampling and sample preparation of high alloy steels were discussed (221), and the analysis of steel turnings pressed into compact blocks was described (141).

Matrix and interelement effects in x-ray spectroscopy are constantly being investigated. Methods for overcoming these effects are the dissolution of samples (90), the fusion of samples (110, 507), and the mathematical treatment of data (3, 205, 226).

New techniques being investigated are the direct electron excitation of samples (405, 501, 538), a portable x-ray instrument using radio-isotope sources (266), and the use of energy dispersion rather then wavelength dispersion for the determination of major constituents in stainless steels (43).

The use of electron probe microanalysis has increased greatly and most of the uses are qualitative or semiquantitative (195, 243, 373, 379, 439, 578). Sensitivity limits of many elements were studied (47, 334).

SULFUR

A combustion–coulometric method extended the range of sulfur analysis in steels to 1 ppm (25). The use of air in place of oxygen suppressed the formation of the interfering ferric oxide (255). Coulometrically generated iodine from potassium iodide was used as the titrant, replacing the conventionally used iodine solution (333). Complexometric and radiometric methods were successfully applied to pig irons, cast irons, and steels (497). Neutron activation was used in the determination of sulfur in steels (22, 59).

TIN

Tin was determined in ferrotungsten alloys by titration with potassium iodate after its sulfide precipitation separation (66). Catechol violet was used for the spectrophotometric determination of tin after solvent extraction of tin iodide into toluene (408) or benzene (530) Other spectrophotometric methods were based on the color formation of tin with stilbazo (469) and 8-quinolinol phenylfluorone (434). Tin was determined polarographically in iron and steel after precipitation separations (361, 515) and in stainless steel by differential cathode ray polarography (443). The effects of various acids (353) and acid concentrations (*354*) in the separation and determination of tin in iron and steels were also studied.

TITANIUM

Titanium was extracted with 1,2-dichloroethane as the diantipyrinylmethane–catechol complex (*531*) and determined photometrically. *N*-furoylphenylhydroxylamine (*427*), dihydroxyphenylfluorone (*19*), sulfosalicylic acid after ion exchange separation (*490*), and diantipyrilmethane with differential spectrophotometry (*359*) were used for the spectrophotometric determination of titanium in steels and ores. Polarographic techniques were used directly (*329*), and after separation by precipitation with ammonia in the presence of EDTA (*301*).

Direct neutron activation analysis was used for the determination of trace amounts of titanium in iron (*357*).

THORIUM

Thorium was spectrophotometrically determined in iron and steel with arsenazo III after a mercury–cathode separation of iron (*278*).

TUNGSTEN

In the spectrophotometric determination of tungsten, ion exchange (*273*), and solvent extraction with thenoyltrifluoroacetone (*118*) were used for separation, and toluene-3,4-dithiol (*273*), KSCN (*118*), and tetraphenylarsonium chloride (*4*) were used as the color-forming agents. Other methods for the determination of tungsten in steel involved the use of radioactive isotopes (*78*) and the measurement of backscattering of beta particles (*182*).

URANIUM

Ion exchange was recommended for the separation of micro and milligram amounts of uranium from ferrous alloys (*310*). Arsenazo III was successfully applied to the photometric determination of uranium in iron and steel (*264*).

VANADIUM

Photometric titration (*397*) and spectrophotometric methods based on the reaction of vanadium with hydrogen peroxide and diphenylamine (*227*), with benzoylphenylhydroxylamine (*259*) or *p*-methoxybenzothio-oxamic acid (*493*) (in chloroform), with antipyrine (phenazone) derivatives (*432*) and with 2-naphthohydroxamic acid (*31*) were employed in the determination of vanadium in steels. A sharp end point was obtained in the amperometric titration of vanadium using two indicator electrodes (*155*). Potentiometric titration was applied to the determination of 0.01–0.3% vanadium in high chromium and tungsten steels (*100*). Thermal neutrons were used for the direct determination of vanadium in stainless steels (*207*) and iron (*357*).

ZINC

Atomic absorption with conventional apparatus was used to determine zinc in iron (*131*).

ZIRCONIUM

Successive cupferron and mandelic acid precipitation separations were used prior to the gravimetric determination zirconium (*67*). Zirconium was photometrically measured in cast iron with chlorosulfenol S (*117*), with alizarin red S in iron–aluminum alloys (*549*), with catechol violet in iron and stainless steels (*573*), and with arsenazo III in steel and cast iron (*465*). A new x-ray fluorescent method was developed for the determination of micro amounts of zirconium in steel employing a chemical concentration technique (*532*).

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Nonferrous Metallurgy

I. Light Metals

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THIS IS THE ELEVENTH review on nonferrous metallurgical analysis and covers the two-year period from September 1964 through August 1966 as documented by *Chemical Abstracts* and *Analytical Abstracts.* Also, the following journals were surveyed directly for the same period: ANALYTICAL CHEMISTRY, *Analytica Chemica Acta, The Analyst,* and *Talanta.*

This review has been limited to those analytical methods applied directly to products of the nonferrous metal industry. Many interesting methods potentially applicable to this field have not been included because of space considerations.

Significant progress has been made in atomic absorption analysis since the previous review (*205*). This advancement is reflected in increased publication and in expanded use of atomic absorption throughout the nonferrous industry. Inorganic gas chromotography, based on the formation and gas chromatographic determination of volatile metal chelates is emerging from infancy. The development and refinement of ion exchange separation of metals has been noteworthy, and the application of this technique should increase in the future. A slow, but obvious, trend toward automation of analysis in the nonferrous industry has been evident, with more incorporation of automatic analyzers, digital readout, and computers into analytical systems.

The determination of gaseous impurities in metals has been a subject of moderate interest. Korolev (*95*) used optical emission microanalysis to determine the distribution of gaseous impurities on the surface of titanium, and the distribution of alloying elements in the oxide layer and within the grains of the metal. Picklo (*147*) compared spectrographic, wet chemical, hot extraction, and vacuum melting procedures for determining oxygen, hydrogen, and nitrogen in aluminum and titanium. An 85-page review on the determination of oxygen in metal has been published by LePape (*107*).

Among several noteworthy reviews were those by Snell (*172*) on colorimetric analysis and by Fischer (*44*) on the determination of nonmetals in nonferrous metals.

An outstanding book, "Colorimetric Determinations of the Elements" by Charlot (*26*) has been published. Part I deals with the theory of colorimetry, fluorimetry, photometric titrations, solvent extraction, ion exchange, chromotography, and electrolytic separation. Part II describes specific methods for individual elements and cites extensive and pertinent references for the determination of individual elements in various materials.

The authors, in preparing this review, noted a trend [observed 34 years ago by Lundell (*110*)] that "... there is an increasing tendency to devote more and more time to determinations which deal with the final act of a chemical analysis, and less time to chemical analysis itself—in other words, to consider chemical analysis dealing with one or two variables instead of the dozen or more that are often involved ..."

It is suggested that future efforts in the nonferrous industry might be better directed toward developing selective methods which are applicable in the presence of other matrix constituents or investigating separations rather than increasing the catalog of methods for estimating an isolated metal.

Accompanying tables list methods referred to in the discussion along with other methods too numerous to mention individually. Table I shows the light metal materials for which compilations of procedures are available for determining miscellaneous alloying elements and impurities. In Table II, arrangement is according to the elements determined.

Aluminum. Tikhonov (*187*) reported a differential spectrophotometric determination of aluminum with chrome azurol S. (545 mμ). The color was stable at large aluminum concentrations and methods were suggested for magnesium and titanium. Florence (*49*) used a new reagent 5-sulfo-4'-diethylamino-2',2-dihydroxyazo-benzene (DDB) for the spectrophotometric determination of aluminum in beryllium. A 1:1 complex was formed and had a molar absorptivity of 41,000 at 540 mμ.

Turchinskii and Naiman (*192*) determined aluminum in magnesium by precipitating $Al_5(OH)_3(AsO_4)_4$ and determining the arsenate iodometrically.

Amos and Thomas (*3*) used atomic absorption to determine aluminum in aluminum bronze and other alloys. They employed the C_2H_2–N–O flame and report a sensitivity of 1.7 ppm Al. Wheat (*203*) also used atomic absorption to determine aluminum, iron, copper, and nickel in aluminum alloys and oxide. A double-beam spectrometer was used. Nikolaev and Aleskovskii (*134*) employed atomic absorption for the micro determination of aluminum in pure metals and alloys. The sample solution (0.005 to 0.01 ml) was applied to the tip of a carbon electrode and arced inside an argon-filled cell. Modulated radiation from a discharge tube containing a hollow aluminum cathode passes through the vapor and the transmission measured. Iron, nickel, cobalt, chromium, titanium and copper did not interfere even when present in 100,000-fold excess.

Kostitsyna and Skobets (*99*) reported that in a solution of Superchrome garnet Y (C. I. Mordant Red 5) buffered at pH 4.6, the presence of a small amount of aluminum results in a double polarographic wave. The first was due to the excess of dye. The second was due to the aluminum dye complex and was proportional to the aluminum present.

Antimony. Tumanov, Petukhova, and Grishin (*191*) suggested methods for determining aluminum and antimony in their binary alloy. Aluminum was determined chelometrically. Antimony was selectively extracted with 20% glycerol in ethyl alcohol and titrated with $KBrO_3$.

Beryllium. Hattori, Tsukahara, and Yamamoto (*66*) and Ishiguro, Niimi, and Shibata (*72*) determined micro amounts of beryllium in aluminum and magnesium alloys with Arsenazo I (580 mμ). Beer's law was obeyed in the range 0 to 0.6 ppm. Bugaeva and Mironenko (*18*) measured the extinction of beryllium sulphosalicylate at 320 mμ to determine beryllium in magnesium alloys.

Sudo and Ogawa (*177*) determined beryllium in magnesium–aluminum alloys down to 1 ppm spectrophotometrically using acetylacetone (295 mμ). Aluminum was removed from magnesium by solvent extraction of the acetylacetone complex with chloroform. Magnesium was masked with EDTA. Kamentseva and Stolyarova (*75*) extracted beryllium from ores with acetylacetone and then photometrically determined it with beryllon at 610–700 mμ.

Gottschalk and Dehmel (*59*) used a gravimetric approach for determining beryllium in alloys and oxide. Beryllium was precipitated as the Pirtea salt in the presence of EDTA and tartrate. Most metal ions did not interfere. Patkar and Varde (*142*) determined beryllium in beryl by homogeneous precipitation using urea. Moiseeva, Mashentseva, and Kuzenetsova (*118*) used 3-acetyl-2-hexanone.

Galkina and Markman (*54*) extracted small amounts of beryllium as the basic butyrate or valerate with chloroform. The beryllium was back-extracted with nitric acid. The final measurement was made fluorometrically using morin.

Two spectrographic techniques for studying the diffusion of beryllium in magnesium were presented by Erko and Krasnorutskii (*39*).

Bisby (*13*) described an apparatus employing ^{124}Sb as a γ-source and BF_3 as a neutron detector for determining beryllium in the laboratory and in the field.

Boron. Dianthrimide (615 mμ) was used by Kerin (*83*) for the spectrophotometric determination of boron in aluminum alloys.

Cadmium. The titrimetric determination of cadmium with antipyrinyl-bis-[*p*-(4-methylbenzylamino)-phenyl]-methanol was presented by Zhivopistsev and Chelnokova (*211*). The titration is performed in a two phase system (kerosene–water) and has been applied to magnesium and aluminum alloys.

Chang *et al.* (*25*) determined cadmium in lead and beryllium compounds using the anodic dissolution technique.

Calcium. The chelometric approach for determining calcium in nonferrous materials was still a favorite (see Table II).

Table I. Methods for Nonferrous Metallurgical Materials

Material	Constituents determined	Methods used	References
Al	Mn, Sm, Cu, As, Sb, Ga, Sc, Cr	A	(*212*)
	Fe, Co, Zn, Sc	A	(*85*)
	Sb, Sn, Sc, Hf, Cr, Co, Fe, Zn, In	A	(*86*)
	Dy, Ga, V, Ni, Mn, Cu, Sb, Zn, Sc, Hf, Cr, Co, Fe, Sn, In	A	(*87*)
	Cu, Sb, As, Ga, Sc, Fe, Zn, Cd	A	(*90*)
	Cu, Mn, Zn, Ga, Cd, Fe, Au	A	(*156*)
	Co, Fe, Sn, Zn, Cd, Cr	A	(*35*)
	Zn, Cd, Pb, Cu, Ca	P	(*108*)
	Cu, Pb, Cd, Zn	P	(*11*)
	Cd, Pb, Cu	P	(*33*)
	B, Cd, Li, P, O, H	S	(*140*)
	Cd, Mn, Ti, Ga, Zn, Gd, Sm	MS	(*46*)
Al products	Cu, K, Si, Cl, Mo, W	A	(*67*)
Al powder, sintered	Fe, Ni, Cu, Zn, Ga, V, Ti, Cr, Mn	X	(*200*)
Al alloys	Cu, Mn, Mg, Fe, Si, Pb, Zn	Col	(*31*)
	Si, Al, Ti, Mn, Zr, B, P	C, Col	(*70*)
	Ti, Cr, Ca, Mg, Mn, Zn	C, Col	(*128*)
	Fe, Cr, Mn, Zn, Mg, Si	X	(*15*)
	Cu, Zn, Mg, Ag	At	(*206*)
Al_2O_3	Si, Ti, P, Fe	Col	(*163*)
	Ca, Fe, Si, Ti, V, Cr, Zn	X	(*146*)
Al-silicates	Si, Fe, Ti, Al	C, Col	(*28*)
Bauxites	Si, Al, Mg, Fe, Mn, Ti, S, P, Ca, O	A	(*2*)
	Ca, Mg, Al, Fe, Si, Ti	S	(*176*)
	Si, Al, Fe, Ti, Ca, Zr	X	(*183*)
Be	Ba, Ni, Cu, Sb, Mo, Mn, Cd, Sn, Au, As	A	(*130*)
	In, Mn, Cl, Na, K, Cu, W, Co, Cr, Fe, Sc, Ta, Zn, Al, V, Si, C, O, N	A	(*38*)
	Ca, B, Mo and 15 other elements	S	(*21*)
	Fe, Ni, Cr, Cu, Si, Al, Mg, C, O, Na, Ca, K	MS	(*126*)
	Al, Co, Cr, Cu, Mn, Na, Si, Ta, W, Zn, O, C, N, F	A	(*36*)
BeO	Al, Fe, Ca, Co, Si, Mg, Mn, Cu, Na, Ni, Ag, Cr	S	(*145*)
	Si, Mg, Cr, Pb, Cu, Ca, Fe, Mn, Ni, Al	S	(*120*)
Mg	B, Cd, Mn, Si, Zn	S	(*131*)
	Al, Ca, Cu, Fe, Mn, Si, Cr, Ni, Sn, Sr, Ti, V, Zn	S	(*136*)
Magnesites	Mg, Ca, Al, Fe, Si	S	(*151*)
	Si, Al, Fe, Ca, Mn, Cr	S	(*190*)
Ti, Ti compounds	Zn, Cu, Mg, Mn, Ca, Si, Cr, Fe, Co, Ni, Al, Ag, Ga, In, Tl	S	(*73*)
Ti, TiO_2	27 elements	S	(*29*)
	Fe, Cr, Ni, Mn, Cu, Sn, Pb	S	(*114*)
Ti alloys	Si, Cu, Fe	S	(*137*)
	Sn, Zr, Nb, Al, Y	S	(*124*)
	Mo, Cr, Mn, Fe, Si	S	(*181*)
	Ti, Fe, Ca, Mn, Mg	C	(*92*)
TiO_2	Bi, Pb, Sn, Fe, Mn, Si, Cu, Mg, Al, Cr	S	(*74*)
	16 elements	S	(*123*)
	Al, Co, Cr, Cu, Fe, Mg, Mn, Nb, Ni, Pb, Sb, Si, Sn, V	S	(*100*)
	Al, V, W, Fe, Ca, Si, Mg, Mn, Cn, Ni, Nb, Sn, Pb, Ta, Cr	S	(*51*)

A	Activation	Col	Colorimetric	S	Spectrographic
At	Atomic absorption	MS	Mass spectrographic	X	X-ray
C	Chemical	P	Polarographic		

Table II. Methods for Elements in Nonferrous Metallurgical Materials

Constituent determined	Material	Reagent or method	References
Al	Alloys	Polarographic	(*99*)
	Al–Fe	Vacuum x-ray	(*55*)
	Al–Pu	Ionx, photometric-oxine	(*41*)
	Mg–Ti	Diff. spectrophotometric	(*187*)
	AlSb	Chelometric	(*191*)
	Alloys	Atomic absorption	(*3*)
	Pure metal alloys	Atomic absorption	(*134*)
	Be, BeO	Photometric	(*49*)
	Bauxite	Neutron activation	(*182*)
	Al alloys, Al_2O_3	Atomic absorption	(*203*)
	Al–Cu alloy	Chelometric	(*155*)
	Be	X-ray fluorescence	(*201*)
	Al alloy	Arsenatimetry	(*192*)
	Ti alloy	Chelometric	(*170*)
Al_2O_3	Al	Aqueous HCl sepn.	(*196*)
	Al	Pptn., microscopic exam.	(*96*)
	Al	Spectrographic	(*139*)
	Al powder, sintered	Gaseous HCl	(*164*)

(*Continued on page 104 R*)

Table II. Methods for Elements in Nonferrous Metallurgical Materials (*contd.*)

Constituent determined	Material	Reagent or method	References
B	Al alloy	Photometric, dianthrimide	(*83*)
Be	Al Al alloys	Photometric, acetylacetone	(*113*)
	Al alloys	Inox sepn., photometric	(*177*)
	Al–Mg–Be alloys	Photometric, arsenazo I	(*66, 72*)
	Be	X-ray activation	(*161*)
	Mg alloy	Photometric, salicylic acid	(*18*)
	BeO alloy	Gravimetric, $Co(NH_3)_6Cl_3$	(*59*)
	BeO	Homogeneous ppt., urea	(*142*)
	Be ores	Gravimetric, 3-acetyl-2-hexanone	(*118*)
	Be ores	Photometric, beryllon	(*75*)
	Be ores	Field apparatus, act. analysis	(*13*)
	Mg	Be differsion, spectrographic	(*39*)
	Be–Cu	Activation analysis	(*56*)
	Be Ore	Fluorometric, basic beryllium butyrate extn., morin	(*54*)
C	Al	X-ray activation	(*37*)
	Mg, Ti	High frequency conductometric	(*54*)
	$TiCl_4$	Conductometric, KI	(*157*)
	Ti	Combustion-conductometric	(*58*)
Ca	Mg	Chelometric	(*47*)
	Mg	Spectrographic	(*62*)
	AlF_3	Photometric, arsenazo	(*122*)
	Ti	Chelometric	(*186*)
	TiO_2	Chelometric, fluorescent ind.	(*111, 112*)
Cd	Mg–Al alloy	Titration after sepn. of $(CdI_4)^{2-}$	(*211*)
	BeO, $BeSO_4$	Voltammetry, hanging Hg drop	(*25*)
Cl	$TiCl_4$	Modified Volhard	(*148*)
	Ti	X-ray fluorescence	(*162*)
	Be	Spectrographic hollow cathode	(*97*)
Co	Co–Al alloy	Atomic absorption	(*203*)
Cu	Al	Amperometric titration dimercaptothiopyrone	(*51*)
	Al alloys	Spectrochemical	(*84*)
	Al alloys	Photometric, thiosemicarbazides	(*98*)
	Al alloys	Photometric, *N*-acetylanabasine	(*180*)
	Al–Zn alloys	Photometric, biquinolyl dicarboxylic acid	(*125*)
	Al alloys	Amperometric 8-mercaptoquinoline	(*195*)
	Al alloys	Atomic absorption	(*203*)
	Al alloys	Polarographic	(*82*)
	Al–Cu alloy	Chelometric	(*155*)
F	Al_2O_3	Photometric, xylenol orange	(*135*)
	Ti	Activation analysis	(*105*)
	Be	Spectrographic, hollow cathode	(*97*)
Fe	Al	Activation analysis	(*61*)
	Al, Mg alloys	Photometric, EDDHA	(*93*)
	(Al alloys)	Atomic absorption	(*8*)
	Ti, Mg	Differential photometric	(*185, 188*)
	TiO_2	Photometric, tributyl phosphate	(*138*)
	Bauxite	Photometric, 1-10 phenanthroline blue	(*202*)
	Al	Photometric, NH_4SCN	(*141*)
	Cryolite, AlF_3	Spectrographic	(*209*)
	Mg alloys	X-ray fluorescence	(*109*)
	Al alloys	Amperometric, 8-mercaptoquinoline	(*195*)
	Al alloys	Atomic absorption	(*203*)
	Al alloy	Redox.	(*192*)
Ga	Al	Photometric, rhodamine B	(*133, 141*)
	Al, bauxite	Photometric, crystal violet	(*32, 102, 173*)
	Aluminate liquor	Chelometric titration	(*174*)
	Al	Photometric, polarographic	(*133*)
	Red Mud	Chelometric	(*117*)
	Al	Photometric, 1-(2-pyridylazo)-2-naphthol	(*179*)
	Mg alloy	Flame photometric	(*57*)
	Mg alloy	Fluorometric	(*94*)
H	Al alloys	Vacuum extraction	(*132*)
	Al, Ti	Manometric	(*40, 45*)
	Al, Ti	Spectrographic	(*197*)
	Be	X-ray activation	(*37*)
		Hot extraction	(*60*)
In	Mg alloy	Flame photometric	(*78*)
	Mg alloy	Fluorometric	(*94*)
K	(See Na)		
Li	Be, BeO	Photometric, thoron	(*4*)
	BeO, MgO	Activation analysis	(*9*)
Mg	Mg alloy	Electrographic	(*165*)
	Al alloy	Photometric, polymethine barbituric acid	(*7*)

(*Continued on page 105 R*)

Moshkina and Kozlova (*122*) determined calcium in aluminum fluoride photometrically using Arsenazo (green filter). The fluoride was removed by fusion with potassium bisulfate and the aluminum content was allowed for by constructing a calibration curve with calcium-free aluminum fluoride.

Carbon. A rapid determination of traces of carbon in metals by using a high-frequency condenser electrode (submersible type) was described by Fujishima and Takeuchi (*53*). The method describes the use of an electrode consisting of a thin-walled glass tube lined with two bands of gold film. $Ba(OH)_2$ was used as the absorbent for evolved CO_2. Pribyl (*157*) used combustion and conventional conductivity to determine carbon in titanium tetrachloride.

Chlorine. Rudolph and Nadalin (*162*) determined microgram quantities of chloride in high purity titanium by x-ray spectrochemical analysis. The sample was dissolved in HF, the titanium(III) oxidized to titanium(IV) and AgCl precipitated. The silver was then measured with a Norelco bulk spectrograph.

Korovin (*97*) used a spectrographic approach to determine chlorine and fluorine in beryllium. The method is based on the use of molecular bands of beryllium halides produced by secondary chemical reactions in the high temperature operating conditions of a discharge in a hollow graphite electrode.

Copper. An amperometric titration of copper was reported by Arishkevich and Usatenko (*5*). The copper was titrated with a solution of dimercaptothipyrone, in a background of acetic acid, by electrolytic oxidation of the reagent on a Pt–miro–electrode at 0.8–0.9 V (*vs.* SCE). Large amounts of aluminum, nickel, and zinc did not interfere. Usatenko and Suprunovich (*195*) amperometrically determined copper, iron, and zinc in aluminum alloys by titrating with 8-mercapto-quinoline.

Several photometric methods were reported for determining copper in aluminum alloys. Koshkin and Agrest (*98*) studied thiosemicarbazides for determining 0.01 to 0.3% Cu. Talipov, Nigai, and Abramova (*180*) extracted the *N*-acetylanabasine–thiocyanate complex into chloroform and measured the extinction at 410 mμ. Ammonium fluoride was used to mask aluminum and iron. 2,2′-biquinolyl-4,4′-dicarboxylic acid was used to determine copper in a zinc–aluminum alloy. The extinction of the violet complex was measured at 550 to 570 mμ.

Fluorine. An indirect method for determining fluorine, based on the color produced by the reaction of ZrO^{2+} with xylenol orange, was reported by Novak and Arend (*135*). It was used for determining F in micro-crystals of alumina prepared from a lead fluoride melt. Pure alumina was used in the preparation of standards.

Gallium. Traces of iron and gallium in high purity aluminum were determined spectrophotometrically after extraction of their chloride complexes with ethylether (*133*, *141*). Rhodamine B (565 $m\mu$) was used to determine gallium and ammonium thiocyanide (480 $m\mu$) was used to determine iron. Sokolova and Bagdasarov (*173*) and Dragulescu and Costinescu (*32*) describe a photometric method for gallium in bauxite and aluminum materials using crystal violet. The water-insoluble gallium-chloride-crystal violet complex was extracted from a 6*N* HCl solution into an organic solvent and the extinction measured at 589–610 $m\mu$. Suzuki (*179*) used 1-(2-pyridlazo)-2-naphthol (550 $m\mu$) for the spectrophotometric determination of gallium in aluminum following an alcohol–chloride extraction.

Korenman, Sheyanova, and Starodubova (*94*) studied thiooxinato complexes for the fluorometric determination of gallium in aluminum and magnesium alloys.

Hydrogen. Nikiforov and Silant'eva (*132*) determined hydrogen in aluminum and its alloys by vacuum extracting the sample in the solid state. To suppress the sublimation of volatile components and eliminate the effect of surface-absorbed moisture, the sample was pretreated with a chromate solution, washed, dried, and calcined. Escoffier (*40*) and Fischer and Leicht (*45*) determined hydrogen in various metal samples using a manometric approach.

Indium. Kanaev (*78*) determined indium in magnesium by means of flame photometry. Calibration standards were prepared containing magnesium. Aluminum showed no interference.

Iron. Kono and Maekawa (*93*) have found that the reaction between ethylenediamine - *N,N'* - bis[(*O*-hydroxyphenyl) acetic acid] (EDDHA) and iron in aluminum was sensitive at pH 2–8. The red color was stable for eight hours and the absorption maximum occurred at 470–480 $m\mu$. A differential spectrophotometric determination of iron in titanium and magnesium products was reported by Tikhonov and Rychkova (*185*). They used 1-10 phenanthroline (516 $m\mu$).

Lead. Stross and Clark (*175*) reported on the polarographic determination of lead in aluminum alloys containing molybdenum and up to 5% of iron. Interference by molybdenum and iron was prevented by extracting them from 6*N* HCl with isobutylmethylketone. Microgram amounts of lead were determined voltammetrically by anodic dissolution using a hanging-mercury-drop electrode (*25*) (see Cd).

Table II. Methods for Elements in Nonferrous Metallurgical Materials (contd.)

Constituent determined	Material	Reagent or method	References
	Al alloy	Photometric, arsenazo I	(*65*)
	Al alloy	Atomic absorption	(*178, 184*)
	Al alloy	Chelometric	(*19, 171*)
	Al	X-ray fluorescence	(*50*)
	Al alloy	Arsentimetry	(*192*)
MgO	Mg–Ni	Residual resistance	(*30*)
	Mg slags	Photometric titration	(*101*)
Mn	Al	Plasma jet	(*168*)
Mo	Ti foil	X-ray fluorescence	(*14, 79*)
	Ti	Photometric, *o*-nitrophenyl fluorone	(*129*)
N	Al	Photometric, chloramine T	(*127*)
	Ti sponge	Kjeldahl	(*22*)
	Mg	Vacuum heating	(*160*)
	Ti	Dc arc, gas chromatographic	(*207*)
	Be	X-ray activation	(*37*)
Na, K	Al_2O_3	Flame photometric	(*194*)
	TiO_2	Flame photometric	(*52, 159*)
	Al	Polarographic	(*143*)
Ni	Al alloy, Al_2O_3	Atomic absorption	(*203*)
	Al	Activation analysis	(*88*)
	Mg alloy	X-ray fluorescence	(*109*)
O	Metals	Fast neutron activation	(*63*)
	Ti	Inert gas fusion	(*80, 198*)
	Be	Neutron activation	(*20*)
	Ti sponge	Titrate CO_2	(*22*)
	Ti	Mass spectrographic	(*27*)
	Al foil	Neutron activation	(*106*)
	Mg	Vacuum heating	(*160*)
	Ti	Dc arc, gas chromatographic	(*207*)
P	Bauxite	Redox. oxine, $KBr\text{-}KBrO_3$	(*104*)
Pb	Al alloy	Polarographic	(*175*)
	BeO	Voltammetry	(*25*)
Pd	Ti–Pd alloy	Photometric, thiourea	(*204*)
	Be	Activation analysis	(*210*)
Rare Earths	Al powder	Reversed phase chromatography, spectrographic	(*23, 24*)
	Al	X-ray fluorescence	(*16*)
	Al, Mg alloy	Polarographic	(*169*)
	Al-Lu	Chelometric	(*17*)
	Be–Cu alloy	Photometric, methyl thymol blue	(*76, 77*)
S	Be, BeO	Fluorometric, quinine sulfate	(*43*)
Sb	AlSb	Chelometric	(*191*)
Si	Ti, Al	Photometric, molybdenum blue	(*34, 69*)
	MgO	Spectrographic	(*208*)
	Al alloys	Differential photometric	(*115*)
	Al alloys	Acid–base, quinoline	(*64*)
	Be	X-ray fluorescence	(*201*)
	Cryolite, AlF_3	Spectrographic	(*209*)
	Bauxite	Neutron activation	(*182*)
Sn	Al	Photometric, phenylfluorone	(*152*)
	Ti alloy	Iodometric	(*170*)
Ti	Al	Photometric, salicylate-quinine	(*199*)
	Al	Photometric, molybdophosphate	(*166*)
	Al alloy	Photometric, dihydroxyphenylfluorone	(*6*)
	Al alloy	Photometric, phenylfurohydroxamic acid	(*149*)
	Al alloy	Photometric, diantipyrinylmethane	(*153*)
	Bauxite	Gas chromatographic	(*167*)
	Ti nitride	Photometric titration, chelometric	(*103*)
	Be	Photometric, dichlorochromotropic acid	(*10*)
	Bauxite	Gravimetric, salicylhydroxamic acid	(*150*)
U	Al	Photometric, H_2O_2	(*143*)
	Al	Neutron activation	(*91*)
	Be matrix	Spectrographic	(*121*)
V	Al_2O_3, bauxite	Photometric, *o*-dianisidine	(*89*)
	$TiCl_4$	Coulometric	(*158*)
	Al	Photometric, tungstophosphate	(*12*)
	Al	Activation analysis	(*88*)
Zn	Bauxite, Al_2O_3	Photometric, dithizone	(*119*)
	Al alloy	Photometric, xylenol orange	(*116*)
	Al–Cu alloy	Polarographic	(*48*)
	Al alloy	Ionx chelometric	(*71*)
	Mg	Photometric, 1-(2-thiazolylazo)-naphthol	(*81*)
	Al alloy	Amperometric	(*198*)
	Al, Mg alloy	Fluorometric	(*94*)
Zr	Al, Mg alloy	Photometric, quercetin	(*154*)
	Al–Fe alloy	Photometric, alizarin red S	(*193*)
	Al–Zr ore	Fluorometric, quercetin	(*190*)
	Al–Zr alloy	Chelometric	(*1*)

Lithium. A spectrophotometric method for determining trace quantities of lithium in beryllium metal and its oxides was described by Apple and White (*4*). Lithium was selectively separated from beryllium by extraction from *M* KOH into 0.1*M* dipivaloylmethane in ethyl ether. Beryllium was masked by fluoride. The lithium was back-extracted into 0.1*M* HCl and determined spectrophotometrically with thoron.

Magnesium. A rapid electrographic method for determining magnesium in magnesium–aluminum alloys was developed by Shiobara (*165*). The method consisted of stripping the alloy constituents into a filter paper moistened with a supporting electrolyte solution and then measuring the intensity of color developed on the paper. This is an extension of the classical electrographic technique for qualitative analysis of alloys.

Asmus and Klank (*7*) determined from 0.01 to 10% magnesium in aluminum alloys by the color change from red to blue of a polymethine barbituric acid derivative. Interfering ions were removed by extraction into chloroform with 8-quinolinol. Hattori, Tsukahara, and Yamatomo (*65*) determined magnesium in aluminum, zinc, copper and nickel base alloys with Arsenazo I. Triethanolamine was used as a masking agent for iron and aluminum. Chromium was removed as chromyl chloride. Divalent ions were masked with KCN and Be with sulfosalicylac acid.

Tietz (*184*) determined magnesium in aluminum alloys and cast iron by atomic absorption using a direct approach. Suzuki, Yanacisawa, and Takeuchi (*178*) also used atomic absorption spectrophotometry to determine magnesium in aluminum metal and alloys. These authors, however, concentrated the magnesium by extraction of the 8-hydroxyquinolate or 8-hydroxyquinaldate into isobutyl methyl ketone.

Burke (*19*) used an EDTA method for the determination of magnesium in aluminum-base alloys. The sample was dissolved in a mixture of NaCN, triethanolamine and NaOH. The precipitated $Mg(OH)_2$ was dissolved, an aliquot mixed with buffer, NaCN and indicator and then titrated with EDTA. Calcium if present in large amounts was separated with ammonium oxalate.

Ferran and Belin (*42*) determined magnesia in the presence of magnesium. The mixture was heated gently with a minimum volume of 5% aqueous CrO_3. The metallic residue was collected and dissolved in *N* HCl for the determination of magnesium either gravimetrically or volumetrically. The method is useful in the analysis of corrosion products of magnesium metal.

Manganese. Sirois (*168*) determined manganese in matrixes of aluminum, iron, copper, and zinc by plasma jet spectrochemistry.

Molybdenum. A photometric method for determining molybdenum impurities in refractory metals was reported by Nazarenko and Shustova (*129*). Molybdenum was extracted from 6*N* H_2SO_4 containing citric or tataric acid with diethyldithiocarbamic acid into chloroform and determined using (*O*-nitrophenyl) fluorone (584 mμ).

Nitrogen. Namiki, Kakita, and Goto (*127*) determined micro amounts of nitrogen spectrophotometrically after organic solvent extraction. The aqueous solution of the blue complex of NH_3, phenol and chloramine T was saturated with NaCl and extracted into isobutyl alcohol and measured at 655 mμ. Beer's law was obeyed for 0.1 to 60 μg of N in 10 ml of extract. The method has been applied to the analysis of aluminum, iron, and steel.

Rodyakin *et al.* (*160*) determined oxygen and nitrogen in magnesium metal. Oxygen was determined by the difference between the vapor pressures of MgO and magnesium metal at 1000°C. Heating under vacuum at >700°C removes the magnesium leaving a residue of MgO and Mg_3N_2. From a determination of magnesium and nitrogen in the residue, the contents of oxygen and nitrogen are calculated.

Winge and Fassel (*207*) simultaneously determined oxygen and nitrogen in titanium and other metals by a direct current C-arc, gas chromatographic technique. With the aid of a Pt flux the O and N were liberated from the metal as CO and N into a static helium atmosphere. An aliquot of the residual gas was then passed through a gas chromatograph for analysis.

Oxygen. Kashima and Yamazaki (*80*) determined traces of oxygen in titanium and silicon by inert gas fusion using a SiC–graphite crucible. A graphite crucible was placed in a silica tube. A portion of silica was placed in the graphite crucible and heated at 2000°C for 5 minutes in a stream of argon. After cooling, the sample was introduced and heated at 2000°C to melt the sample. The CO produced was determined gas chromatographically. Vasserman and Turovtseva (*198*) presented a review (41 references) of the determination of oxygen in metals and inorganic compounds by the method of reductive fusion in an inert-gas atmosphere.

A mass spectrometric approach for determining oxygen in titanium was used by Chupakhin *et al.* (*27*). A relationship between the TiO and oxygen in the sample was derived and used in the analysis of samples. Interference from surface titanium oxides was avoided by treatment with HF, and layers of chemisorbed oxygen were removed by subjecting the sample to an ion-source in a vacuum.

Palladium. A photometric determination for palladium in palladium–titanium alloys was described by Widtman (*204*). The sample was dissolved in H_2SO_4–HF and the titanium oxidized to Ti(IV) with nitric acid. The final determination was made with thiourea at 310 mμ. Bismuth and silver interfered.

Phosphorus. Lebedeva and Martinson (*104*) performed a volumetric determination of phosphorus as the 8-hydroxyquinoline complex with molybdophosphoric acid. The precipitate was collected, washed with 1% HCl or NaCl solution, and treated with ethanol–HCl (1:1) and boiled with 0.5 g of oxalic acid. The cooled solution was diluted with 2*N*-HCl and titrated with $KBr–KBrO_3$.

Potassium. (See Sodium).

Rare Earths. Cerrai *et al.* (*23*) separated rare earths from sintered aluminum powder and then determined them spectrographically. A reversed phase column chromatographic technique was used. The sample solution (0.2*M* HNO_3) was applied to a column of Kel-F powder treated with 0.5*M* trioctylphosphine oxide in cyclohexane. The rare earths were retained while the aluminum was not. After removal of the rare earths with 6*M* KNO_3, and further purification to remove titanium and vanadium, the determinations were made by the graphite spark technique. The same authors in a separate paper (*24*) extended their method for application to routine analysis.

Bonissoni and Paganelli (*16*) used x-ray fluorescence to determine 0.1 to 1% lanthanum in metallic aluminum. Skobets and Chernyi (*169*) determined the sum of the rare earths in aluminum and magnesium polarographically by the displacement of lead from a solid phase. An aliquot of the dissolved sample was treated in a polarographic cell with 10 ml of 0.5*N* NH_4Cl, 0.2 ml of 2% gelatin and 0.5 g of dry lead oxalate. The displaced lead was determined polarographically.

Brueck and Lauer (*17*) determined lutetium in a binary aluminum alloy by an EDTA titration. Aluminum was masked with sulfosalicylic acid. Quantitative results were obtained for 1–5% lutetium. Kaneko, Okada, and Goseki (*76*) determined yttrium or lanthanium in beryllium–copper alloys. The matrix was removed by mercury-cathode electrolysis. Remaining copper was masked with thiourea and beryllium with acetylacetone prior to the spectrophotometric determination with methylthymol blue. Kaneko and Goseki (*77*) also performed the same determination spectrographically after first converting the alloy to the oxides.

Silicon. Dymov and Koreneva (*34*) studied the use of $SnCl_2$, $Ti_2(SO_4)_3$,

$TiCl_3$, $NaHSO_3$, thiourea, ascorbic acid, and amalgams of Ti and Bi for reducing molybdosilicic acid in the colorimetric determination of silicon in titanium. The bulk of the titanium was removed by hydrolytic precipitation or by ion exchange with a cation exchange resin. The intensity of the blue color differed with each reducing agent, but the calibration curves were straight in each case. In the study, $SnCl_2$ gave the strongest color followed by Bi amalgam, ascorbic acid ≅ Thiourea ≅ $NaHSO_3$, $Ti_2(SO_4)_3$ = $TiCl_3$, and Ti amalgam, in that order. Householder and Russell (*69*) used the reduced molybdenum blue color to determine silicon in copper-, aluminum-, and iron-base alloys; fluoride-bearing ores; and other types of materials including phosphoric acid, titanium dioxide, and dolomite. They used the isopiestic distillation of H_2SiF_6 in a plastic Petri dish to separate the silicon. The concentration range studied was between 0.003 and 2.7% Si. The elimination of most interfering cations and anions was accomplished by their procedure. For the determination of greater than 16% Si in aluminum alloys, Matrosova and Zubkova (*115*) used the differential spectrophotometric approach. They used ammonium molybdate as the coloring reagent and ferrous sulfate as the reducing agent. Accuracy equaled that of a gravimetric determination.

A rapid titrimetric determination of silicon in aluminum alloys was described by Hasegawa (*64*). The quinoline complex of molybdosilicic acid was quantitatively precipitated at a pH less than one and redissolved in excess sodium hydroxide. The H_2MoO_4 reacts with the sodium hydroxide. Each equivalent of base corresponds to 1/24 of the molecular weight of silicon. P, As, and Ge interfere.

Sodium and Potassium. Fratkin, Moshkovich, and Filippova (*52*) determined sodium and potassium in titanium dioxide using a flame photometer. The sample was heated in a graphite tube at 400° C in a stream of HF to remove the titanium as TiF_4. The residue was evaporated with nitric acid and taken up in water for the final determination. Rezac and Dvorak (*159*) also used the flame photometric approach to measure sodium and potassium in titanium white. They used sulphosalicylic acid to mask titanium and after adding ammonium acetate passed the solution through an ion exchange resin (either H^+ or OH^- form depending on whether it was desired to remove the titanium or the sodium and potassium).

A polarographic approach was used by Pedanova (*143*) to determine alkali metals in aluminum solutions, pulverized ores, clinkers, and nephelites. Aluminum did not interfere but iron, calcium, and magnesium had to be removed. The sample was added to 25 ml of 3% Et_4NI and 5 drops of 0.2% methyl red and a polarogram obtained. $E_{1/2}Na = -2.11$ V and $E_{1/2K} = 2.13$ V (SCE).

Sulfur. A luminescent method was proposed by Fioletova (*43*) for determining small amounts of sulfur in beryllium, zirconium, niobium, and their oxides. Quinine sulfate produces a complex with sulfur having a yellow fluorescent color in the ultraviolet (365 mμ).

Tin. Pollock and Zopatti (*152*) determined tin in silicate rocks, aluminum, iron, nickel, and copper. They extracted the tin with 8-quinolinol and finished the determination spectrophotometrically using phenylfluorone.

Titanium. Volkova, Get'man, and Emtsova (*199*) determined 0.006 to 0.022% titanium in aluminum containing also silicon, iron, copper, and zinc. The procedure calls for taking a sample containing 6 to 20 mg of titanium adding NH_4SCN and $Na_2S_2O_3$ (to discharge color produced by iron); then adding 0.5*M* sodium salicylate and 5 ml of chloroform. After filtering the chloroform through a dry paper the extinction was measured at 400 to 420 mμ. Shkaravskii (*166*) recommends the determination of small amounts of titanium in metallic aluminum after extraction as molybdotitanophosphate. The heteropoly-complex is extracted into butanol and the extinction measured at 360 mμ.

Dihydroxyphenylfluorone was used by Asmus, Kurzmann, and Wollsdorf (*6*) to determine titanium in aluminum alloys. The extinction was measured at 546 mμ against a reagent blank. The method was used for samples containing 0.001 to 1.2% titanium. Pilipenko, Shpak, and Boiko (*149*) used the colorimetric reagent *N*-phenyl-2-furohydroxamic acid to determine μg quantities of titanium in steel, ores, and aluminum alloys. The sample (0.5 to 1 g containing 10 to 50 μg of titanium) was dissolved in HCl, an aliquot treated with 1% $SnCl_2$ and the reagent added. The complex was then extracted into chloroform and the extinction measured at 413 mμ.

Polyak (*153*) photometically determined titanium in aluminum and molybdenum alloys by means of diantipyrinylmethane. Without prior separation the extinction of the colored species was measured (no wavelength reported). The sensitivity in the range 0.1 to 1.0 mg of Ti was 0.001% to 0.03%. Basargin, Kukisheva, and Solov'eva (*10*) used 3,6-dichlorochromotropic acid to measure titanium in the presence of beryllium (10 μg Ti–0.1 g Be).

Lazarev, Lazareva, and Ukshe (*103*) determined titanium in titanium nitride by a photometric titration. In the presence of H_2O_2 and acid, Ti(IV) forms a Complexon III complex which can be titrated with $Bi(NO_3)_2$ solution. A green filter was used and the equivalence point determined graphically. Poddar *et al.* (*150*) proposed a method for the gravimetric estimation of titanium in bauxite, ilmenite, titaniferous magnetite, and ferrotitanium by using salicylhydroxamic acid. The method was stated to be simple and efficient and the results comparable to those obtained by standard methods.

Sievers, Wheeler, and Ross (*167*) reported the microanalysis of titanium by gas chromatography. Titanium oxide in bauxite was quantitatively converted to $TiCl_4$ which was determined by gas chromatography. The $TiCl_4$ is prepared in a sealed tube which is later broken in a helium carrier gas stream of a gas chromatograph. The instrument was temperature programmed and used a stainless steel column packed with 15 wt % Histowax on 60–80 mesh gas pack F (flux-calcined diatomaceous earth support coated with a perfluorocarbon polymer).

Uranium. Petit, McCall, and Kienberger (*144*) used the yellow $U–H_2O_2$ complex (410 mμ) to determine 100 to 500 ppm uranium in aluminum. The aluminum was dissolved in HCl and the uranium extracted into trioctylphosphine oxide–benzene. The uranium was then re-extracted into ammonium oxalate solution, ammonia added and the $U–H_2O_2$ complex formed. Moroshkina (*121*) determined uranium in materials containing high quantities of beryllium using a spectrographic approach. Arc excitation using copper electrodes was used and methods were presented for solutions and solids.

Vanadium. Klug and Metlenko (*89*) proposed a new photometric method for determining vanadium in aluminum products. Vanadium was separated from iron and chromium by extracting with 5% 8 hydroxyquinoline in chloroform or tributylphosphate or by cation exchange (V eluted with 0.1*N* H_2SO_4). Vanadium was then determined with *O*-dianisidine (440 mμ), *p*-$H_2NC_6H_4NMe_2HCl$ (512 mμ), or 3,3'-dimethylnaphthidine (560 mμ). This technique permits the determination of 0.01 μg V/ml.

A coulometric method for determining vanadium in titanium tetrachloride was proposed by Pyatnitskii and Erisov (*158*). VO_3 is titrated with electrolytically generated ferrous ion. The titration was carried out at constant current density and the endpoint determined amperometrically using platinum electrodes. Chromate and permanganate ions interfere and must be reduced to lower oxidation states. The determination of 0.001% or more of vanadium in a 5 g sample of titanium tetrachloride takes 5 to 10 minutes.

Zinc. A rapid photometric determination of zinc in aluminum alloys using xylenol orange was presented by Miyajima (*116*). The alloy was dissolved in NaOH, filtered and acidified with HCl to a pH of 0.3. Thiourea, ascorbic acid, and ammonium fluoride were used as masking agents. Kawase (*81*) used 1-(2-thiazolylazo) naphthol to determine zinc in magnesium. Zinc forms a blue or red-violet chelate which can be extracted with chloroform to give a solution with an absorption maximum near 600 mμ. The interference of 29 cations and 11 anions was examined.

Ishibashi and Komaki (*71*) determined zinc in aluminum alloys chelometrically after extraction by a liquid anion exchanger. Zinc was extracted with a 15 volume % kerosene solution of Amberlite LA-1 or LA-2 from $3N$ HCl triethanolamine and KCN were used as masking agents. Zinc was liberated with 4% HCHO and titrated with EDTA.

A polarographic method applicable to the determination of 0.01 to 0.33% zinc in copper–aluminum alloys was discussed by Fleury (*48*). The alloy contained 5–8% of iron, nickel, and manganese. Copper was removed by electrolysis, iron extracted as the chlorocomplex with isobutyl methyl ketone, and the zinc reduced at a dropping-mercury electrode in the presence of $2N$ NaOH and EDTA (Mask Ni). The polarogram was recorded between −1.3 and −1.8 V.

Zirconium. Polyak and Bashkirova (*154*) used quercetin to spectrophotometrically determine zirconium (0.2–0.9%) in magnesium and aluminum alloys. In acid solution zirconium forms a precipitate with quercetin which is soluble in ethanol or acetone. The resulting solution had an optimum wavelength for absorption of 420 to 440 mμ. The sensitivity is 0.005 μg of Zr per ml. Interference from iron and copper was avoided by use of hydroxylamine and thiourea. Alimarin, Golovina, and Tenyakova (*1*) used the fluorescent nature of quercetin as the basis of a method for determining zirconium in titanium- and iron-containing ores. An aqueous–alcohol solution of the zirconium complex fluoresces. Those of titanium and iron have a quenching effect on the zirconium complex. Iron, titanium, and zirconium are initially separated by a paper chromatographic technique. The method was tested on a sample containing ZrO_2, 6.7; TiO_2, 1.35; Fe_2O_3, 47.8; SiO_2, 11.58; and CaO, 6.6%. The amount of ZrO_2 found was 6.2–6.4%.

Tramm and Pevzner (*189*) reported the chelometric determination of zirconium and aluminum in binary alloys. EDTA was used in the classical manner. The U.K.A.E.A. (*193*) reported the use of alizarin red S (560 mμ) for the determination of from 0.1 to 0.45% zirconium in an iron-aluminum alloy.

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Nonferrous Metallurgy

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

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In this review as in the preceding one of this series (*26*), an attempt was made to present a cross section of the methods available for the determination of the elements under consideration. The papers for the most part describe actual analytical methods which have been or can be used for the determination of an element. In each section, also, a few papers are described which only discuss separation methods and in which either the actual determination is not described or only a reference is made to the use of a standard method. Many other papers in which a method of determination is described are included even though the separation is the main contribution of the paper. An effort was made to exclude all papers from this review that appeared in the earlier review. Unfortunately, because of the tremendous volume of chemical literature, a number of very worthwhile papers may have been overlooked. Also, the papers describing the research upon which many new analytical methods will no doubt be based, but, which in their present form do not describe a usable analytical method, were omitted. Analytical methods are summarized in Tables I to XVI.

ZIRCONIUM AND HAFNIUM

As in the previous review of this series, zirconium and hafnium will be considered together because most of the gravimetric, titrimetric, and spectrophotometric methods for zirconium work equally well for hafnium. At the end of this section are included a few methods published specifically for hafnium. The chemistry of zirconium and hafnium allows them to be determined by gravimetric, titrimetric, and spectrophotometric methods and their nuclear characteristics allow radiochemical and activation methods. They also lend themselves easily to spectrochemical and x-ray fluorescence methods.

Papers which cannot conveniently be classified by method of determination, include a review of many aspects of the inorganic chemistry of zirconium by Clearfield (*100*), a separation of zirconium and hafnium by solvent extraction of their thiocyanates by Fischer, Pohlmann, and Adam (*145*), a review of methods of solvent extraction by Green (*175*), studies of separations by reversed-phase partition chromatography by O'Laughlin and Banks (*377*) and Cerrai and Ghersini (*85*). Ion exchange separation studies including zirconium were published by Strelow, Rethemeyer, and Bothma (*521*), Kallmann (*229*), Hamaguchi *et al.* (*187*), Holloway and Nelson (*200*) and Danielsson (*106*).

As might be expected, the methods of gravimetric determination included applications of the familiar reagents and introduction of a few new reagents which react with zirconium, and generally all other tetravalent and many trivalent cations. A thermogravimetric study of the mandelates of zirconium and hafnium was reported by Adams and Holness (*1*). Norwitz (*375*) used mandelic acid for the determination of zirconium in zirconium metal. Hydroxypropylmandelate was used by Rowe, Gordon, and Jackson (*448*) for the precipitation of zirconium mandelate from homogeneous solution. Zirconium was precipitated by α-hydroxybenzylphosphonic acid by Ryabchikov and Dedkov (*453*) and by Yen, Yung, and Liu (*595*). Cupferron was used by Dosch and Conrad (*121*) for the determination of zirconium following a cation exchange separation and by Majumdar and Mitra (*312*) following an anion exchange separation. Pietsch and Ludwig (*401*) studied the conditions for the precipitation of zirconium with isomers of dimethylphenylarsonic acid and two trichlorophenylarsonic acids (*402*). Jain and Singh (*212, 214*) used 6,7-dihydroxy-4-phenylcoumarin and 7,8-dihydroxy-4-phenylcoumarin for the determination of zirconium in the presence of iron. Additional gravimetric reagents are 2,5-dihydroxy-1,4-benzoquinone used by Singhal and Ryan (*504*); *m*-nitrocinnamic acid used by Chang and Liu (*89*); iodobenzoic acid used by Chang (*88*); 2-hydroxy-3-naphthoic acid derivatives used by Agarwall and Mehrotra (*2*); an azopyrazole derivative used by Popa *et al.* (*414*); phenylacetic acid used by Pande and Kapoor (*386*); α-methoxy phenylacetic acid used by Liang and Wang (*292*); *N*-benzoylphenylhydroxylamine used by Lukyanov and Knyazeva (*305*); the potassium salt of 7-hydroxynaphthalene-1,3-disulfonic acid used by Popa, Baiulescu, and Moldoveanu (*415*); purpurogallin used by Dutt and Singh (*126*); 5,5′-thiodisalicylic acid used by Good and Srivastava (*170*); and sodium phosphate following a liquid ion exchange separation used by Green (*176*).

Papers describing spectrophotometric methods for zirconium are largely applications of previously published methods or use the method following a new separation method. Ishiwatari (*207*) determined zirconium along with niobium in the presence of uranium, thorium, and iron with xylenol orange. Dixon and Headridge (*117*) used xylenol orange following an anion exchange separation from titanium, niobium, tantalum, molybdenum, and tungsten. Elinson and Nezhnova (*132*) determined zirconium in niobium and niobium alloys with xylenol orange with no separation. Nee, Chu, and Liang (*359*) extracted zirconium with *N*-phenylbenzohydroxamic acid then determined it with xylenol orange. Arsenazo III was used by Mel'chakova, Trubetskaya, and Peshkova (*330*) for the determination of zirconium in the presence of hafnium. Budesinsky (*70*) made a study of the complex formed between zirconium and Arsenazo III and described the conditions of maximum sensitivity. Zirconium was determined by Arsenazo III after extractiod by TTA in work reported by Onishi and Toita (*379*). Arsenazo III was used by Pisarenko and Bondarenko (*405*) for the determination of zirconium in bronzes and copper-base alloys and by Savvin, Kadaner, and Ryabova (*468*) for its determination in steel and cast iron. Onishi and Nagai (*378*) made a detailed study of the zirconium–arsenazo complex at several pH values. Culkin and Riley (*101*) used quinalizarinsulfonic acid for the determination of zirconium in silicate rocks following TBP extraction and a cation exchange separation. Alizarin red S was used by Schutte (*475*) for the determination of zirconium in South African granite and by Stern (*518*) in copper–zirconium alloys. Khopkar and Dhara (*247*) extracted zirconium into mesityl oxide, back-extracted it into water, and determined it with alizarin. Wood and Jones (*586, 587*) used pyrocatechol violet following a TOPO extraction and Hibbits, Rosenberg, and Williams (*198*) used a cupferron precipitation, then a TOPO extraction followed by a pyrocatechol violet determination. Leeb and Hecht (*291*) used pyrocatechol violet for zirconium in steel and Uzumasa, Hayashi, and Ito (*562*) used it following a TTA extraction. Quercetin was used by Polyak and Bashkirova (*413*) for the determination of zirconium in magnesium and aluminum alloys and by Vanossi (*563*) following a general method of separating zirconium from other elements. Shakashiro and Freund (*480*) preceded the determination with 8-quinolinol with an anion exchange separation. Van Santen, Schlewitz, and Toy (*564*) described the conditions for the determination of zirconium using 8-quinolinol, Sugawara (*527*) separated zirconium from tungsten, molybdenum, and titanium by an anion exchange separation and TTA extraction prior to determination with thorin. Biswas, Seth, and Dey (*44*) described the conditions for the determination of zirconium with chrome azurol S. Picramine R and nitrosulfophenol S were compared by Savvin and Dedkov (*467*) as reagents for zirconium and naphthazarin and β-hydronaphthazarin were studied by Bottei and Quane (*53*). Dedkov *et al.* (*112*) determined zirconium in cast iron using chlorosulfophenol S. A reagent consisting of fluorotitanic acid and hydrogen peroxide was used by Fukamauchi *et al.* (*149*). Hahn, Joseph, and Salciccioli (*184*) used bromanilic acid; Popa and Costache (*419*) used 2,6,7-trihydroxy-9-methyl fluorone; Popa, Baiulescu, Paralescu, and Mircea (*417*) used nuclear fast red; Pan *et al.* (*385*) used pyrogallolsulfonephthalein, and Kekedy and Balogh (*244*) used gallein. Caletka (*82*) reviewed the aqueous chemistry of zirconium and mentioned complexes suitable for spectrophotometric determinations. Shigematsu (*490*) used morin as the reagent for the fluorometric determination of zirconium in sea water following a TTA extraction.

Papers describing titrimetric determinations of zirconium deal largely with EDTA titrations with xylenol orange being one of the more popular if not the most popular indicator. Pilkington and Wilson (*404*) studied the effect of polynuclear zirconium species on the direct titration with EDTA using xylenol orange. Pribil and Vesely (*425*) described the conditions for the direct titration of zirconium in 0.3 to 0.6*M* nitric acid and applied this method (*424*) to the determination of zirconium in the presence of thorium and titanium. EDTA with xylenol orange was used by Nazarchuk and Mekhanoshina (*354*) for the determination of zirconium in zirconium–nickel–aluminum alloys, by Tramm and Pevzner (*556*) for the analysis of zirconium–aluminum alloys, by Brecny and Kurcova (*61*) for the analysis of zircon and zirconia and by Ryabchikov and Bukhtiarov (*452*) for the determination of zirconium in molybdenum alloys following cation exchange separation. Back-titrations of the excess EDTA with bismuth to the xylenol orange end point was used by Fritz and Frazee (*148*) to determine both zirconium and hafnium after separation from each other by reversed-phase partition chromatography using the thiocyanates. Kuteinikov and Lysenko (*281*) also used a back-titration with bismuth to determine the sum of the zirconium and hafnium in a mixture as did Kozyreva, Kuteinikov, and Zharova (*275*) for the analysis of zirconium metal, carbide, and oxide. A mandelate separation followed by a back-titration with zinc to the xylenol orange end point was used by Su (*526*) for the determination of zirconium in siliceous materials. Other indicators used for the EDTA titration of zirconium are pyrocatechol violet by Baran and Tympl (*29*); 1-hydroxy- or 3-hydroxy-naphthoic acid by Sen and Chauhan (*477*); β-SNADNS-6 by Datta and Saha (*108*) and gallein by Sun (*529*). Hung and Chang (*204*) studied thirteen indicators for the titration of zirconium with EDTA. Pribil (*423*) studied various complexing agents including EDTA for the determination of zirconium, thorium, and titanium separately in mixtures. Mel'chakova, Stanislavskaya, and Peshkova (*329*) used a slightly different approach in performing a spectrophotometric titration of zirconium with arsenazo III as the titrant to determine zirconium in magnesium and aluminum alloys. Konkin and Zhikhareva (*263*) titrated with Trilon B to the xylenol orange end point. Verdi-Zade (*567*) and Verdi-Zade and Elbendov (*568*) precipitated zirconium periodate, filtered, and then dissolved the precipitate and titrated the periodate with thiosulfate. Shakhtakhtinskii and Sarkisyan (*483, 484*) precipitated zirconium arsenate, separated the precipitate and determined the arsenic iodimetrically.

Amperometric titrations have also been used for the determination of zirconium. Terenteva and Bernatskaya (*550*) used a rotating platinum electrode and titrated with EDTA. Popa *et al.* (*420, 421*) titrated with flavazine L and with disodium-2-(3-methyl-5-oxo-1-phenyl-2-pyrazolin-4-ylazo)-6,8-naphthalenedisulfonate. Sodium molybdate was used as a titrant for zirconium by Gupta (*180*) and neocupferron was used by Gallai *et al.* (*151*).

A few workers have reported polarographic methods for the determination of zirconium. Zakharov and Stromberg (*602*) found useful waves in both methanolic lithium chloride and aqueous potassium chloride. Sharipov and Songina (*486*) also reported a polarographic method for the determination of zirconium. Kasagi and Banks (*236*) used a sine-wave polarograph for the determination of zirconium in the concentration range of 0.05 to 0.4 mg/ml.

Brooks and Boswell (*68*) compared cathode and anode excitation in the direct current arc for a number of elements including zirconium. Lachin (*283*) proposed a spectrographic procedure which is reported applicable to all solid, liquid, and dissolved substances. Concentration by extraction of their 8-quinolinolates into chloroform or by anion exchange prior to spectrographic determination was proposed by Moroshkina and Smirnova (*341*). Ko (*254*) used an amine extraction before the spectrochemical determination of zirconium in plutonium. Huff (*203*) used spectrographic determinations in a study of the behavior of nineteen elements on a strong-base ion exchange resin and applied the method to the determination of impurities in tantalum. Brooks (*65*) used the powder–d.c. arc technique for the determination of zirconium and other impurities in niobium metal and oxide, and he (*66*) also used the fusion–d.c. arc for the same purpose. Spectrographic techniques were used by Pikhtin (*403*) for the determination of zirconium in silicon carbide; by Ivanova, Tsyvenkova, and Kovalenko (*209*) in nickel and its alloys; by Briere and Kurylenko (*64*) in zircons and by Berdyev, Golovkova, and Karakhanov (*37*) in geological samples.

X-ray fluorescence was used by Taylor (*549*) for the determination of microgram amounts of zirconium in uranium. Hakkila, Hurley, and Waterbury (*185*) determined 5 to 50% zirconium in mixed carbides with molybdenum and uranium by this technique. Erlank and Willis (*136*) used x-ray fluorescence for the determination of the zirconium–hafnium ratio in chondrites and Butler and Thompson (*78*) determined the zirconium–hafnium ratio in igneous rocks.

Neutron activation has also proved to be useful for the determination of zirconium and hafnium. Yule (*600*) included zirconium and hafnium in a study in which the thermal neutron activation analysis sensitivities for 118 reactor thermal neutron products were experimentally determined. Zirconium and hafnium were also included in an activation analysis review by Laverlochere (*288*) and Choy, Lukens, and Andersen (*94*) used an activation analysis technique in conjunction with a solvent extraction for the determination of small amounts of zirconium. Neutron activation was used for the determination of zirconium and hafnium in meteorites or other minerals by Merz and Schrage (*331*), Setser and Ehmann (*479*), Schmitt, Bingham, and Chodos (*473*) and Thompson (*551*); Stary and Ruzicka (*517*) used neutron activation and isotope dilution along with separations using substoichiometric amounts of reagents for the determination of zirconium and other metals.

Radiochemical methods were used in a number of papers on the determination of zirconium. Chromatographic separations preceded the radiochemical determination in papers by Welford and Chiotis (*574*), Welford, Choitis, and Morse (*575*) and Breccia and Spalletti (*60*). The determination of radionuclides including zirconium dissolved in water was the subject of papers by Wood and Richards (*588*), Chakravarti *et al.* (*86*) and Bizollon and Moret (*45*).

Table I. Spectrophotometric Methods

Element	Reagent: Material (References)
Zirconium and zirconium plus hafnium	Xylenol orange: Ti, Nb, Ta, Mo, W (*117*); Nb (*132, 207*); following extraction (*359*)
	Arsenazo III: Study of complexes (*70*); following TTA extraction (*379*); Hf (*330*); bronzes and Cu alloys (*405*); steel and cast iron (*468*)
	Arsenazo: Study of complexes (*378*)
	Quinalizarinsulfonic acid: Silicate rocks (*101*)
	Alizarin red S: Granite (*475*); Cu (*518*)
	Alizarin: Following extraction (*247*)
	Pyrocatechol violet: Following TOPO extraction (*198, 586, 587*); following TTA extraction (*562*); steel (*291*)
	Quercetin: Study of conditions (*563*); Mg or Al (*413*)
	8-Quinolinol: Mo (*480*); Study of conditions and interferences (*564*)
	Thorin: W (*527*)
	3″ - sulfo - 2″,6″ - dichloro - 3,3′ - dimethyl - 4 - hydroxyfuchon - 5,5′-dicarboxylic acid: Study of conditions and interferences (*44*)
	Picramine R and Nitrosulfophenol S: Study of conditions (*467*)
	Naphthazarin and β-hydronaphthazarin: Study of complexes (*53*)
	Chlorosulfophenol S: Fe (*112*)
	Fluotitanic acid–hydrogen peroxide: Study of conditions (*149*)
	2,5-Dibromo-3,6-dihydroxyquinone: Study of complex (*184*)
	2,6,7-Trihydroxy-9-methyl fluorone: Study of conditions (*419*)
	Nuclear fast red: Study of conditions (*417*)
	Pyrogallolsulfonephthalein: Study of complex (*385*)
	Gallein: Study of conditions and interferences (*244*)
	Review including reagents suitable for spectrophotometric determination (*82*)
Hafnium	Arsenazo III: Iron and steel (*232*)
	Alizarin red S: Study of complex and interferences (*459*)
	5-Dimethylamino-2-(2-thiazolylazo)phenol: Study of complex (*334*)
Vanadium	8-Quinolinol: Cr, Mo (*271*); W (*519*); water (*346*)
	7-Iodo-8-quinolinol: Study of conditions and interferences (*279*)
	Xylenol orange: Study of conditions and interferences (*72, 383*)
	Alizarin red S; Study of conditions and interferences (*531*)
	Diaminobenzidine: Water, biological materials, rocks (*87*); ethylene–propylene rubber solutions (*509*)
	4-(2-pyridylazo)-resorcinol: Study of conditions and interferences (*71, 155*)
	3-Hydroxy-2-methylpyran-4-one: Study of conditions and interferences (*222, 223*)
	5-Hydroxy-2-hydroxymethylpyran-4-one: Study of conditions and interferences (*462, 463*)
	Tungstovanadophosphate: Study of interferences (*131*); petroleum products (*439, 533*); P, Si, Mo, W (*91*)
	6-Hydroxy-1,7-phenanthroline: Study of conditions and interferences (*122*)
	Hydrogen peroxide: Ti (*493*); alunite (*482*); silicate rock (*520*)
	NaOH: LiCl–KCl eutectic (*447*)
	Oxalic acid: Study of conditions and interferences (*278*)
	Organic acids: Study of conditions (*337*)
	5-Dimethylamino-2-(2-thiazolylazo)-phenol: Study of complex (*334*)
	Pyridine–2,4,6-tricarboxylic acid: Study of conditions (*340*)
	o-Hydroxyacetophenone oxime: Study of conditions and interferences (*407*)
	Formaldoxime: Study of conditions and interferences (*321, 322*); air and urine (*216*)
	N-methylanabasine-α′-azo-α-naphthol: Study of conditions and interferences (*13*)
	Formazan: Mo, W (*525*)
	Hexamatoxylin: Petroleum (*308*)
	Acetylacetone: Study of interferences (*465*)
	N-(*o*-tolyl)-benzohydroxamic acid: Study of conditions and interferences (*310*)
	N-(*p*-chlorophenyl)-benzohydroxamic acid: Study of conditions and interferences (*311*)
	N-phenylbenzohydroxamic acid: Study of conditions and interferences (*554*)
	4-Methoxybenzothiohydroxamic acid: Study of conditions and interferences (*507*)
	2-Naphthohydroxamic acid: Study of conditions and interferences (*32*)
	1-Naphthylamine: Study of conditions and interferences (*8*)
	Diphenylaminesulfonate: Study of conditions (*353*)
	Ammonium sulfide: Steel, Fe (*607*)
	Tris-(2-hydroxyalkyl)-amines: Study of conditions (*188*)
	2-Thenoyltrifluoroacetone: Study of conditions and interferences (*109*)
	Rutin: Study of conditions and interferences (*115*)
	Glyoxal bis-(2-mercaptoanil): Study of conditions and interferences (*93*)
	Potassium chlorate–aniline: Re (*454*)
	Potassium chlorate–benzidine: Study of conditions and interferences (*552*)
	Potassium bromate–aniline: Water, Re (*290*)
	Ammonium persulfate–gallic acid: Water (*146*)
	Potassium chlorate–potassium bromide: Study of conditions (*49*)
	Potassium bromate–potassium bromide–ascorbic acid: Study of conditions (*50*)

(*Continued on page 113R*)

Osterberg, Pearcy, and Kujala (*382*) determined zirconium in tissue from a male fin whale. A number of papers consider the separation of zirconium-95 from niobium-95 or the determination of each without prior separation. The papers are listed here and are not repeated in the niobium section. Rudenko, Dziomko, and Kremenskaya (*450*) separated the isotopes by extraction with 8-hydroxyquinaldine and caproic acid. Shigematsu, Tabushi, and Matsui (*491*) used an acetylacetone extraction. Tri-isooctyl amine was used by Susic and Maksimovic (*532*). Jurriaanse and Moore (*224*) extracted with diisobutylcarbinal. Brezhneva *et al.* (*62*) extracted with tributylphosphate. Salicylhydroxamic acid was used by Qaim (*430*) and benzene solutions of fatty acids by Kyrs (*282*). Guillon, Colonomos, and Sauvagnac (*179*) and Kodochigov and Glazunov (*256*) determined the two elements without separation.

Spectrophotometric methods proposed for the determination of hafnium include alizarin red S by Sangal (*459*); arsenazo III for hafnium in steel by Kammori, Taguchi, and Komiya (*232*) and 5-dimethylamino-2-(2-thiazolylazo)-phenol by Minczewski and Kasiura (*334*). An amperometric titration with tartrazine or flavazine L was used by Popa, Baiulescu, and Moldoveanu (*416*). A neutron activation study by Girardi, Guzzi, and Pauly (*165*) included hafnium. Hafnium was determined in zirconium by means of neutron activation by Mignonsin and Albert (*333*), Girardi, Guzzi, and Pauly (*166*) and Grothe and Reinhardt (*178*). Szabo *et al.* (*538*) determined hafnium in rocks and Kiesl, Hecht, and Sorantin (*249*) determined it in aluminum. Hafnium was determined in zirconium by Waring (*572*) using a spectrochemical technique. X-ray fluorescence was used for the determination of hafnium in zirconium by Polonio (*411*) and Ramous (*433*).

VANADIUM

Although each section of this review is arranged by method of determination, it is felt that a few papers describing separation methods in which determination methods are not discussed at length should be mentioned. Danielsson (*106*) studied the adsorption of various metal ions on Dowex-1 from sulfate solution. Korkisch and Ahluwalia (*267*) utilized Dowex-1 and a nitric acid–methanol mixture for the separation of several elements, including vanadium, from uranium. Equilibrium distribution coefficients of a number of elements with Bio-Rad AG50W cation exchange resins in nitric and sulfuric acid solutions were studied by Strelow, Rethemeyer, and Bothma (*521*). Green (*175*) reviewed solvent extraction separations for a

Table I. Spectrophotometric Methods (*Continued*)

Element	Reagent: Material (References)
Niobium	Comparison of several reagents: Study of conditions (*287*)
	Thiocyanate: Study of extraction (*137, 335, 336*); alloys (*117*); Ta (*303, 372, 596*); U (*497*); steel (*120, 225, 347, 578*)
	Xylenol orange: Zr (*207*); steel (*457*)
	Hydrogen peroxide: Sn (*92*); Zr (*585*); Pb, Ba (*561*)
	Hydroquinone: Preparation of standard (*374*); steel (*63, 243*); 70 elements (*197*)
	Pyrogallol: Steel (*284*)
	Bromopyrogallol red: Study of conditions and interferences (*35, 36*)
	4-(2-thiazolylazo)resorcinol: Study of conditions and interferences (*396*)
	4-(2-pyridylazo)resorcinol + H_2O_2 + EDTA: Study of conditions (*286a*)
	5-Dimethylamino-2-(2-thiazolylazo)-phenol: Study of reactions (*334*)
	Arsenazo: Study of conditions (*269*)
	Eriochrome cyanine: Study of complexes (*22*)
	2,3′-Bipyridyl-pyrocatechol: Study of conditions (*18*)
	1,2′-Bipyridyl-pyrocatechol: Study of conditions and interferences (*544*)
	4-(4′-Biphenylylazo)catechol + thiocyanate: Study of conditions and interferences (*545*)
	2,2′-Bipyridyl-Fe(III): Study of reactions (*81*)
	Hematoxylin: Study of conditions (*23, 270*)
	Morin: Study of reactions (*23*)
	Salicylhydroxamic acid: Study of complexes (*22*)
	Tiron: Ta (*492*)
	N-phenylbenzohydroxamic acid–thiocyanate: Study of conditions and interferences (*364*)
	16 Derivatives of trihydroxyfluorone: Study of complexes (*357*)
	Sulfochlorophenol S: Zr (*466*)
	Lumogallion: Loparite concentrate (*11*)
	Gossypol: Study of conditions and interferences (*246*)
	4-Methoxybenzothiohydroxamic acid: Study of reactions (*507*)
	Basic solution: Steel (*52*)
Tantalum	Several reagents: Discussion and comparison (*23, 287, 314, 576*)
	Pyrogallol: Steel (*63, 347*); 70 elements (*197*)
	1,2-Bipyridyl + pyrocatechol: Study of conditions and interferences (*544*)
	4-(2-pyridylazo)-resorcinol: Alloys (*133*)
	4-(2-pyridylazo)-resorcinol + hydrogen peroxide + EDTA: Study of conditions (*286a*)
	Arsenazo: Study of complexes (*24*)
	Crystal violet: Study of conditions (*12*); alloys (*117*)
	Tiron: Nb (*492*)
	Salicylohydroxamic acid: Study of complexes (*10*)
	Rhodamine 6G11 and Butylrhodamine B: Study of conditions (*119*)
	Sodium molybdate: Study of reaction (*183*)
Chromium	*s*-Diphenylcarbazide: Study of reaction (*272, 541*); study of oxidation (*16*); refractory materials (*444*); ruby (*500*); rocks and minerals (*129*); steel, Fe (*474*); sea water (*96*); canned food (*339*); beer (*113*)
	1-Phenylthiosemicarbazide: Study of conditions and interferences (*259*)
	EDTA: Silicate rock (*520*); chrome alum (*601*); biological material (*589*)
	Isonicotinic acid hydrazide and 2,3,5-triphenyltetrazolium chloride: Study of conditions (*189*)
	o- and *m*-Toluidine: Metal soap (*316*)
	Chromotropic acid: Steel (*422*)
	Alizarin red S: Study of conditions (*458*)
	Chrome azurol S: Study of conditions and interferences (*315*)
	Pyridine–2,6-dicarboxylic acid: Study of conditions and interferences (*47*)
	Antipyrinylbis-[*p*-(4-methylbenzylamino)-phenyl]-methanol: Study of conditions and interferences (*5*)
	2-Hydroxy-1-(2-hydroxy-4-sulfo-1-naphthylazo)-naphthalein: Study of conditions and interferences (*557*)
	None: Ruby (*118*)
Molybdenum	Thiocyanate: Study of complex (*257, 580*); study of conditions (*319, 368*); study of interferences (*131, 371*); following precipitation separation (*367*); 70 elements (*197*); steel, Fe (*33, 54, 265, 300, 304*); F^- solutions (*525*); Cu ores and concentrates (*124*); Pb (*559*) ores (*369*); soil (*266*)
	Toluene-3,4-dithiol: Study of reaction (*4*); following ion exchange separation (*186, 527*); Cu (*611*); geochemical samples (*323*); biological materials (*43*).
	8-Quinolinol: Study of conditions (*38*); U (*344*).
	8-Mercaptoquinoline: Study of reaction (*3*)
	Mercaptoacetic acid–diphenylguanidine: Study of conditions (*76*)
	Thiomalic acid: Study of conditions (*325*); Fe (*326*)
	Thioglycollic acid: U (*219*)
	Hydrogen peroxide: Molybdotellurous acid (*481*)
	Molybdovanadophosphate: P, Si, V, W (*91*)
	Sodium sulfide: Study of complex (*594*)
	Morin: Study of conditions (*252*)
	Thiourea: Study of conditions (*348*)
	Hydroxylammonium sulfate: Study of conditions (*599*)
	Phenylhydrazine: Study of complex (*57*)

(*Continued on page 114R*)

Table I. Spectrophotometric Methods (*Continued*)

Element	Reagent: Material (References)
Molybdenum (continued)	Tiron-diphenylguanidine: Study of conditions (*77*)
	Pyrocatechol: Solid lubricants (*230*)
	Alizarin red S: Study of conditions and interferences (*505*)
	3,5,7-Trihydroxyflavone: Study of conditions (*237*)
	Tetrahydro-4-hydroxy-2-(α-piperidino)-isopropyl-furan-3-one: Study of conditions (*508*)
	3-Hydroxy-2-methylpyran-4-one: Study of interferences (*221*)
	o-Dihydroxycoumarins, derivatives of: Study of complexes (*211*)
	2,3,4-Trihydroxybenzenesulfonic acid: Study of conditions (*201*)
	Glyoxal bis-(2-mercaptoanil): Study of reactions (*93*)
	2-Amino-4-chlorobenzenethiol hydrochloride: Study of conditions (*250*)
	2,6,7-Trihydroxyfluorone, 13 derivatives of: Study of conditions (*499*)
	(*o*-Nitrophenyl)fluorone: Refractory metals (*356*)
	Diethyldithiocarbamate: Study of conditions (*498*)
	Nicotinohydroxamic acid: Study of conditions (*449*)
	4-Methoxybenzothiohydroxamic acid: Study of interferences (*507*)
	2′,3′,4′-Trihydroxyacetophenone: Study of interferences (*418*)
	2,2′-Bipyridyl: Study of conditions (*289*)
	Hydrogen peroxide–iodide–variamine blue: Study of conditions (*534*)
	Hydrogen peroxide–iodide–starch: Water (*15*)
	Hydrogen peroxide–dithio-oximide: Study of conditions (*387*)
	Perborate–iodide: Study of conditions (*579*)
	Methylene blue–hydrazine: Study of conditions (*48*)
Tungsten	Thiocyanate: Following precipitation separation (*370*); As, Sb (*488*); Ta (*6*); Mo (*487*) (*584*); V, Mo (*525*); steel (*110, 111, 302*); ores and concentrates (*400*)
	Toluene-3,4-dithiol: Cu (*611*); WSi_2 (*528*); soils (*55*)
	Hydroquinone: Steel (*243*); Ni (*536*)
	8-Quinolinol: Steel and ores (*519*)
	Hydrogen peroxide–methylsalicylate: Study of conditions and interferences (*438*)
	Morin–dimethylsulfoxide: Study of conditions (*205*)
	2,3,7-trihydroxy-6-fluorone, derivatives of: study of conditions and interferences (*412*)
	Dithio-oxamide–hydrogen peroxide: Steel, soil, rocks (*387*)
	Tungstovanadophosphate: P, Si, V, Mo (*91*)

Table II. Other Optical Methods

Element	Method-reagent: Material (References)
Zirconium	Fluorometric–morin: Sea water (*490*)
Vanadium	Flame photometric: Study of conditions (*345*)
	Atomic absorption spectrophotometric: Study of conditions (*582*)
Tantalum	Turbidimetric–thiosulfate–hydrogen peroxide: Study of interferences (*593*)
Chromium	Flame photometric: Steel, Fe (*427, 571*); feces (*102*)
	Atomic absorption spectrophotometric: Study of conditions (*139*); steel (*30*)
Molybdenum	Polarimetric–(+)-tartaric acid: Study of conditions (*273*)
	Atomic absorption spectrophotometric: Ferrous alloys (*343*)
Tungsten	Polarimetric–(+)-tartaric acid: Study of conditions (*210, 273, 350*)

number of elements including vanadium. O'Laughlin and Banks (*377*) included vanadium in a study of the reversed-phase partition chromatographic separation with organophosphorus compounds.

A great variety of methods have been used for the spectrophotometric determination of vanadium, some of which were proposed as new methods and some were common methods which were used to demonstrate the effectiveness of a separation. Kotsuji (*271*) determined vanadium with 8-quinolinol after separation on paper treated with hydroxamic acid, Mulikovskaya (*346*) concentrated vanadium from natural water by coprecipitation with ferric hydroxide then determined it with 8-quinolinol. Stolyarov (*519*) determined tungsten along with vanadium with 8-quinolinol. Kurmaiah, Satyanarayana, and Rao (*279*) used 7-iodo-8-quinolinol-5-sulfonic acid (ferron) for the extraction and determination of vanadium. Molybdenum, tungsten, and iron interfere. Otomo (*383*) determined vanadium(IV) using xylenol orange with ascorbic acid as the reducing agent and reported the effects of 24 cations, ten of which interfere. Budevski and Pribil (*72*) masked the majority of interfering elements with DCTA and determined vanadium(V) with xylenol orange. Vanadium in ethylene–propylene rubber solutions was determined with 3,3′-diaminobenzidine by Smith (*509*). Chan and Riley (*87*) used diaminobenzidine for the determination of vanadium in sea water, marine sediments, and marine plants. Budevski and Dzhonova (*71*) studied the interference of 30 elements in the determination of vanadium with 4-(2-pyridylazo)-resorcinol. Geary and Larsson (*155*) studied interference by chromate, molybdate, tungstate, and nitrate in the determination of vanadium with the same reagent. Jungnickel and Klinger (*222, 223*) determined vanadium with 3-hydroxy-2-methyl-pyran-4-one (maltol) by extraction of the complex into chloroform. Satyanarayana, Kurmaiah, and Rao (*461, 462*) used 5-hydroxy-2-hydroxymethyl-pyran-4-one (kojic acid) as a reagent for the determination of vanadium. Serious interferences are tungsten, oxalate, and EDTA. Tungstovanadophosphate was used by Elbeih, Elbakry, and Aly (*131*) for the determination of vanadium following a paper chromatographic separation, by Rayner (*439*) for the determination in fuel oil, and by Svajgl (*533*) for the determination in petroleum and depleted desulfuration catalysts. The use of formaldoxime was described by Marczenko (*321*) and by Marczenko and Stepien (*322*), and the conditions were studied. Jaraczewska and Jakubowski (*216*) applied the method to the determination of vanadium in air and urine. Separation from titanium on a cation exchange resin was used by Shishkov and Shishkova (*493*) prior to determination of the vanadium with hydrogen peroxide. Shakhtakhtinskii, Guseinzade, and Khalilov (*482*) used the hydrogen peroxide method for the determination of vanadium in alunite and Stolyarov and Agrest (*520*) used EDTA along with hydrogen peroxide for the determination of vanadium in silicate rocks. Root (*447*) determined the sum of vanadium(IV) and vanadium(V) by measuring the absorbance at 270 mμ in 5*M* sodium hydroxide. Mittal and Mehrotra (*337*) measured the absorbance at 410 mμ which results from the addition of either acetic, succinic, malonic, benzoic, or phthalic acid to an ammonium vanadate solution. Kurihara and Nozaki (*278*) used the ammonium oxalate complex for the determination of vanadium in the presence of copper, nickel, aluminum, chromium, and iron. Among the organic reagents used for the spectrophotometric determination of vanadium are pyridine-2,4,6-tricarboxylic acid used by Morimoto and Sato (*340*), *o*-hydroxyacetophenone oxime by Poddar (*407*); *N* - methylanabasine - α′ - azo - α-naphthol used by Amirkhanova, Podgornova, and Shchesterova (*13*) and formazan used by Studenskaya *et al.* (*525*). Chromium, iron, and tungsten are serious interferences in the method studied by Dougherty and Mellon (*122*) in which the complex with 6-hydroxy-1,7-phenanthroline is formed in a 40% propanediol solution and then extracted into benzene. Low concentrations of vanadium in petroleum were determined by MacMillan and Samuel (*308*) using hematoxylin. Satyanarayana and Rao (*464*) extracted vanadium into an acetylacetone–benzene mixture and measured the absorbance of the organic layer after the addition of butanol or pentanol. Several hydroxamic acids have been studied as reagents for the determination of vanadium. Majumdar and Das (*310*, *311*) used *N*-(*o*-tolyl)-benzohydroxamic acid and *N*-(*p*-chlorophenyl)-benzohydroxamic acid. Tomioka (*554*) used *N* - phenylbenzohydroxamic acid, Skorko-Trybula and Minczewski (*507*) used 4-methoxybenzothiohydroxamic acid and Bass and Yoe (*32*) used 2-naphthohydroxamic acid. Albert and Stoia (*8*) used 1-naphthylamine for the determination of vanadium in the range of 30 to 640 μg per ml. Iron and molybdenum must be removed. Namiki, Watanabe, and Kawamura (*353*) studied the reactions involved in the determination of vanadium with diphenylaminesulfonate and recommended conditions to prevent the interference from excess nitrite. The red-brown sulfovanadate ion formed by the reaction of ammonium sulfide with vanadium was used by Zindel and Zeiher (*607*) for the determination of vanadium in steel. Hartkamp (*188*) used the reaction between vanadium and tris-(2-hydroxyalkyl)-amines in the presence of a reducing agent for the determination of vanadium. De and Rahaman (*109*) measured the absorbance of the vanadium-TTA complex in the butanol phase following extraction. Additional organic reagents used for the determination of vanadium are 5-dimethylamino-2-(2-thiazolylazo)-phenol by Minczewski and Kasiura (*334*), rutin by Dev and Jain (*115*), alizarin red S by Sinha and Dey (*531*), and glyoxal bis-(2-mercaptoanil) by Ch'i, Chang, and Hsu (*93*).

The catalytic action of vanadium upon the reaction between benzidine and potassium chlorate was utilized by Tien and Chang (*552*) for the spectrophotometric determination of vanadium. Similarly, Ryabchikov, Lazarev, and Lazareva (*454*) used chlorate and aniline to determine vanadium in rhenium. Lazarev and Tronina (*290*) used bromate and aniline to determine vanadium in water and rhenium. The catalytic effect of vanadium upon the amount of oxidation of gallic acid by ammonium persulfate in phosphoric acid solution which takes place in exactly 60 minutes was used by Fishman and Skougstad (*146*) for the determination of vanadium in water. Bognar and Jellinek (*49*) used the time of appearance of free bromine in the reaction of chlorate with bromide which releases bromine which reacts with aniline as a basis of a method for the determination of vanadium through its catalytic action. The same authors also used the bromate–bromine reaction (*50*) and measured the rate of bromine release.

Most titrimetric methods for the determination of vanadium are based on the fact that vanadium(IV) can be easily and quantitatively oxidized to vanadium(V) which can in turn be easily and quantitatively reduced to vanadium (IV). A few methods make use of the vanadium–EDTA complex. Strusievici (*522*) determined both vanadium(IV) and (V) in mixtures by first titrating the vanadium(IV) with permanganate then the total with iron(II) ammonium sulfate. Mittal and Mehrotra (*338*) used iron(III), iron(II), and permanganate for the titration of each of the oxidation states of vanadium in mixtures. Price and Maurer (*426*) separated vanadium from molybdenum and phosphorus on a cation exchange column and then titrated the vanadium with permanganate in the analyses of molybdovanadophosphoric acid. In order to determine vanadium(V) in the presence of chromium(VI), Rao and Devaraj (*437*) titrated the total with iron(II) and the reduced vanadium with permanagate. Satyanarayana and Rao (*461*, *465*) extracted vanadium into acetylacetone, back-extracted with either sodium hydroxide or water, oxidized with permanganate and titrated with iron(II). Claassen and Bastings (*99*) studied the conditions which cause high results in one permanganate–iron(II) procedure and recommended changes to eliminate the difficulty. Namiki and Watanabe (*352*) determined the end point spectrophotometrically in the permanganate titration of vanadium. Cerium(IV) sulfate was used by Giuffre and Cassani (*167*) for the potentiometric titration of vanadium(II) and (III) in catalysts; by Dikshitulu and G. G. Rao (*116*) for the titration of vanadium plus molybdenum in mixtures to the rhodamine 6G end point. K. B. Rao (*435*) titrated the total vanadium plus molybdenum to the methyl orange end point and then titrated the vanadium(V) to the diphenylaminesulfonate end point with iron (II). Edge and Fowles (*130*) reduced vanadium to vanadium(II) with a lead–10% sodium alloy and then titrated with chromate, permanganate or cerium(IV). Rao, Rao, and Rao (*434*) titrated with potassium dichromate in 12 to 13.5M phosphoric acid and determined cerium plus vanadium and iron plus cerium plus vanadium in the same solution. Murty and Bapat (*349*) used an excess of ascorbic acid to reduce vanadium, then titrated the excess with potassium iodate. Iodine monochloride was used for the titration of vanadium following reduction with mercury by Cihalik and Pluhar (*97*). Hayashi (*191*) reduced vanadium with cadmium or lead amalgam in the presence of EDTA, then titrated with potassium ferricyanide. Epik and Perchik (*135*) reacted vanadium with potassium ferrocyanide, then oxidized potassium iodide with the ferricyanide and titrated the liberated iodine with thiosulfate. Interferences in the titration of vanadium with EDTA to the pyrocatechol end point as described by Bhattacharya (*41*) are molybdenum and lead. An EDTA titration using a zinc back-titration to the xylenol orange end point with and without the vanadium masked by peroxide was used by Filipov and Kirtcheva (*144*) for the determination of vanadium and aluminum in steel. Majumdar and Mitra (*312*) studied the elution behavior of the thiocyanate complexes of a number of elements including vanadium from both anion and cation columns and determined the vanadium by a back-titration of excess EDTA by zinc at pH 10. Martinez and Barral (*324*) back-titrated the excess EDTA with copper to the disappearance of fluorescence from a fluorescein indicator. Charreton (*91*) titrated vanadium with an excess of 1,2-diaminocyclohexane - *N*,*N*,*N*′,*N*′-tetraacetic acid and back-titrated the excess with magnesium. Konkin and Zhikhareva (*263*) determined vanadium in silicates and alloys by titration with Trilon B. Vanadium in alumina-processing products was determined by Klug and Metlenko (*253*) with a titration using methyl orange as the titrant. Bobtelsky and Gancz (*46*) determined vanadium using a heterometric titration with 5,7-dibromo-8-quinolinol.

Various workers have successfully used amperometric methods for the determination of vanadium. Filenko (*140*, *141*) determined vanadium along with manganese and chromium in steel by titrating amperometrically with iron(II). Studenskaya and Makogonova (*524*) determined vanadium in refractory alloys and slags by titration with iron(II) using two indicator electrodes. Songina and Savitskaya (*510*) titrated vanadium(V) with iron(II) and vanadium(IV) with permanganate using two platinum indicator electrodes. Gallai and Mar'yanovskaya (*152*) titrated vanadium(IV) with vanadium (II) in 5M sulfuric acid in the presence of phosphoric acid. Novak, Mal'tsev, and Bogovina (*376*) used an electrolytic dissolution and then a titration with iron(II) for the determination of vanadium present as the carbide in steel. Other reagents for the amperometric titration of vanadium are chloramine T used by Deshmukh and Eshwar (*114*); thallium nitrate used by Yoshimura, Uno, and Noguchi (*598*) and silver nitrate used by Saxena and Sharma (*471*).

Scrosati and Laitinen (*476*) studied the electroanalytical chemistry of vanadyl sulfate in molten lithium chloride–potassium chloride eutectic. Gilbert (*163*) used a pulse polarographic method for the determination of up to 50 parts per million nickel and vanadium in petroleum stocks. A polarographic method was used for the determination of vanadium and thallium in mixtures by Saxena and Mittal (*470*). Kennedy and Jensen (*245*) studied the catalytic effect of molybdenum upon the reduction of vanadium in the presence of EDTA. Brainina and Roizenblat (*58*) included the vanadate ion in a study of the polarography of anions. Baumann (*34*) found that the height of the second vanadium wave is increased by the presence of nitrite ion and EDTA may be used to suppress the interference of some cations.

A coulometric titration following concentration by coprecipitation with iron (III) hydroxide was used by Yoshimori *et al.* (*597*) for the determination of vanadium in common salt. Electrically generated iron(II) was used by Pyatnitskii and Erisov (*429*) for the coulometric titration of vanadium in titanium tetrachloride using an amperometric end point.

Plasma arc excitation was used by Heemstra and Foster (*195*) for the spectrographic dètermination of vanadium in petroleum fractions. Zotin (*613*) studied the intensities of homogeneous line pairs of vanadium–titanium and other elements in various mixtures excited with an a.c. arc on copper electrodes. The same author (*612*) also studied the mutual effects of silicon dioxide and certain elements including vanadium. Huff (*203*) used spectrographic analysis of column effluents in a study of the behavior of 19 elements, including vanadium, on a strong base ion exchange column at variable nitric and hydrofluoric acid concentrations. A concentration method involving a counter-current extraction with a moving aqueous phase was used by Brooks (*67*) in conjunction with spectrographic determination for the determination of vanadium in sea water. Applications of spectrographic methods were reported for the determination of vanadium in high polymers by Lachin (*283*); in silicon carbide by Pikhtin (*403*); in complex chromium–molybdenum–nickel alloys by Raff (*432*); in uranium tetrafluoride by Podobnik and Spenko (*409*); in high-purity hafnium by Carpenter and Lewis (*84*); in steel and cast iron by Nikitina and Ivanova (*366*) and Lounamaa (*301*); and in coke by Buncak (*73*). A quantometer was used by Buyanov, Ivanova, and Sukhova (*80*) for the determination of a number of elements, including vanadium, in heat-resistant alloys.

Table III. Titrimetric Methods

Element	Reagent: Material (References)
Zirconium and zirconium plus hafnium	EDTA: Study of conditions and complexes (*29, 404, 425, 529*); Th, Ti (*424*); Ni, Al (*354*); Al (*556*); zircon and ZrO_2 (*61*); Mo (*452*); Hf (*148*); high temperature materials (*281*); Zr + ZrC + ZrO_2 (*275*); siliceous material (*526*); Th, Al (*477*); following chromatographic separation (*108*); study of indicators (*204*); rare earths, Th, Ti (*423*)
	Arsenazo III: Mg, Al (*329*)
	Trilon B: Electrode slag (*263*)
	Periodate–thiosulfate: Study of interferences and conditions (*567, 568*)
	Arsenate–iodide–thiosulfate: Study of conditions (*483*); Fe, Al (*484*)
Vanadium	Iron II–permanganate: Mixed oxidation states of V (*338*); study of interferences (*461, 464*); Cr (*437*); steel, Fe (*99*), catalysts (*522*)
	Sodium nitrite–permanganate: Steel (*352*)
	Permanganate: Ammonium molybdovanadophosphate (*426*)
	Cerium(IV): Catalysts (*167*)
	Mercury–cerium(IV): Mo–V (*116*)
	Sodium–lead alloy–dichloromate or permanganate or cerium(IV): Study of conditions (*130*)
	Iron II–cerium(IV): Mo–V (*435*)
	Potassium dichromate: Ce, Fe (*434*); Mo, W, Si, P (*91*)
	Ascorbic acid–potassium iodate: Study of conditions (*349*)
	Iodine monochloride: Study of conditions (*97*)
	Cadmium or lead amalgam–ferricyanide: Mo (*191*)
	Ferrocyanide: Study of reactions (*135*)
	EDTA: Study of reactions (*41, 324*); study of separations (*312*); steel (*144*); silicates (*263*)
	Methyl orange: Al ores and concentrates (*253*)
	5,7-Dibromo-8-quinolinol: Study of conditions and interferences (*46*)
Niobium	8-Quinolinol: Nb metal, carbides and borides (*276*)
	Hydrogen peroxide–potassium permanganate: Study of conditions (*21*)
Niobium and Tantalum	Nitrilotriacetic acid–copper: Study of complexes (*286*)
Chromium	EDTA: Study of conditions (*196, 296, 324, 397*); following ion exchange (*312*); Al (*398, 399*); Cr ore (*511*); Al, Ca, Mg (*169*)
	Iron(II): Steel (*215*); silicates (*20*); Cr ores and refractories (*28*); rare earth alloys (*355*)
	Iron(II)–permanganate: V (*437*); steel (*299*); feces (*384*)
	Arsenic(III)–permanganate: Study of conditions (*410*)
	Permanganate: Chrome plating electrolyte (*380*); study of reduction method (*130*)
	Oxalate–cerium(IV): Study of conditions (*220*)
	Iodide: Ferrochrome (*19*)
	4-Phenylazo-1-naphthol: Study of conditions or interferences (*604*)
	Vanillin azine: Study of conditions (*327*)
Molybdenum	Cerium(IV): Study of indicators (*436*); V (*116, 435*); study of reduction method (*130*)
	Iron(III): Study of conditions (*455*)
	Potassium ferricyanides: Study of conditions (*190*); V (*191*)
	Iodine monochloride: Study of conditions (*98*)
	8-Mercaptoquinoline: Steel (*51*)
	EDTA: Study of conditions (*277, 295, 456, 590*); ferromolybdenum (*134, 241, 262*); catalysts (*560*)
	Trilon B: Ferromolybdenum and $MoSi_2$ (*365*)
	Antipyrinylbis-(4-dimethylaminophenyl)-methanol: Study of conditions and interferences (*605*)
Tungsten	Permanganate: Cr, Ta, Fe, Mo (*138*)
	Silver: Study of conditions (*496*)
	EDTA: Fe (*261*); study of conditions (*59*)
	Lead nitrate: Ferrotungsten (*239*)

Karttunen *et al.* (*235*) used tritium absorbed on zirconium as an excitation source for the generation of fluorescent x-rays in elements of atomic number 16 to 35. Dwiggins (*128*) used x-ray fluorescence for the determination of vanadium in petroleum and other organic material.

Mottola (*345*) studied the precision obtained in both a direct and an indirect flame photometric method for the determination of vanadium. Winefordner and Veillon (*582*) used atomic absorption spectrophotometry for the determination of vanadium in the organic phase following extraction into methyl isobutyl ketone.

Guthrie (*182*) reports the determination of vanadium and 26 to 50 other elements in the mass spectrographic analysis of erbium, cerium, and lutetium.

Vanadium was determined gravimetrically by Poddar (*408*) using *o*-hydroxyacetophenone.

Yule (*600*) reported an extensive study of activation analysis sensitivities of 118 reactor thermal neutron products which included vanadium. Neutron activation analysis was used for the determination of vanadium in aluminum by Kiesl and Hecht (*248*), in biological material by Livingston and Smith (*293*), and in zirconium by Mignonsin and Albert (*333*). Stary and Ruzicka (*517*) used neutron activation for the determination of vanadium following extraction by a substoichiometric amount of 8-quinolinol.

A "radio release" method for the determination of vanadium in water was used by Gillespie and Richter (*164*) in which vanadium displaced radioactive silver in a column, and the silver was then determined by usual radiochemical methods.

NIOBIUM AND TANTALUM

Because of their occurrence together in natural materials and because of their similar chemistry, niobium and tantalum are very frequently considered together in publications. Therefore, in order to eliminate a great many repetitions, they will be considered together in this review. A recent comprehensive book by Moshier (*342*) on the analytical chemistry of niobium and tantalum also includes a discussion of the properties of the two metals and their compounds and a list of 550 references. Patrovsky (*394*) reviewed the progress in the analytical chemistry of niobium and tantalum and included 130 references. Strelow, Rethemeyer, and Bothma (*521*) included niobium and tantalum in their study of the distribution coefficients of 49 cations between nitric and sulfuric acid solutions and a sulfonated polystyrene resin. Holloway and Nelson (*200*) described an anion exchange procedure for separating niobium from equal amounts of neptunium and larger amounts of zirconium using hydrofluoric–hydrochloric acid solutions. A separation of niobium from tantalum on an anion exchange column was worked out by Dragulescu, Kyri, and Oprescu (*123*). A chloroform solution of a liquid anion exchange resin was used by Green (*173*) for the extraction of niobium from a sulfate–oxalate solution. Green (*175*) also included four methods for niobium and three for tantalum in a review of methods of solvent extraction of various cations. A detailed study of the extraction of niobium and tantalum 8-quinonolates was made by Alimarin, Bilimovich, and Ts'ui (*9*). Trioctylamine was used by Nevzorov and Bychkov (*362*) for the extraction of niobium and tantalum from tartaric–oxalic acid solutions. According to Gibalo, Alimarin, and Davaadorzh (*160, 162*) niobium can be separated from tantalum, zirconium, titanium, and other elements by extraction into chloroform of the compound formed with tetramethylenedithiocarbamate. Niobium and tantalum were investigated along with various other cations by O'Laughlin and Banks (*377*) in a study of separations using reversed-phase partition chromatography with neutral organophosphorus compounds.

Table IV. Gravimetric Methods

Element	Reagent: Material (References)
Zirconium and hafnium	Mandelic acid: Thermogravimetric study (*1*); Zr metal (*375*)
	Hydroxypropyl mandelate: Homogeneous solution (*448*)
	α-Hydroxybenzylphosphonic acid: Study of conditions and interferences (*453, 595*)
	Cupferron: Ti, Pb (*121*); ion exchange study (*312*)
	Dimethylphenylarsonic acid, isomers of: Study of conditions (*401*)
	2,4,5- and 2,4,6-Trichlorophenylarsonic acid: Study of conditions (*402*)
	6,7-Dihydroxy-4-phenylcoumarin: Ti (*212*)
	7,8-Dihydroxy-4-phenylcoumarin: Fe (*214*)
	2,5-Dihydroxy-1,4-benzoquinone: Sc (*504*)
	m-Nitrocinnamic acid: Study of conditions and interferences (*89*)
	Iodobenzoic acid: Th (*88*)
	2-Hydroxy-3-naphthoic acid: Th (*2*)
	3 - Methyl - 5 - oxo - 1 - phenyl - 2 - pyrazolin - 4 - ylazo - 2′ - (6′,8′-naphthalenedisulfonic acid): Study of conditions and interferences (*414*)
	Phenylacetic acid: Study of conditions and interferences (*386*)
	α-Methoxyphenylacetic acid: Study of conditions and interferences (*292*)
	N-benzoyl-*N*-phenylhydroxylamine: Study of interferences (*305*)
	7-Hydroxynaphthalene-1,3-disulfonate: Study of conditions and interferences (*415*)
	Purpurogallin and derivatives: Study of conditions (*126*)
	5,5′-Thiodisalicylic acid: Study of conditions (*170*)
	Sodium phosphate: Ferrotitanium (*176*)
Vanadium	*o*-Hydroxyacetophenone oxime: Study of conditions and interferences (*408*)
Niobium	Boric acid–hydrolysis: Ti, Fe, Mn, Sn (*460*)
	Ammonium hydroxide–EDTA: W (*489*)
	Ammonium tetramethylenedithiocarbamate: Study of conditions (*161*)
	α-Hydroxybenzylphosphonic acid: Study of reactions (*453*)
	4-Phenyldaphnetin: Ta, Mo (*213*)
Niobium and tantalum	Cupferron: Ti, Zr (*469*)
	N-phenylbenzohydroxamic acid: Steel (*242*)
	N-benzoyl-*N*-(*o*-tolyl)hydroxylamine: Nb from Ta from Ta from Nb (*313*)
	Hydrogen peroxide–nitric acid or ammonium hydroxide: Study of reaction (*103, 104, 202*); tantalocolumbites (*105*)
Tantalum	Cupferron: Carbides (*542*)
	Benzenearsonic acid: Sn, Zr, W, Th, rare earths (*395*)
Chromium	Dimethylphenylarsonic acid, isomers of: Study of conditions (*401*)
	2,4,5- and 2,4,6-Trichlorophenylarsonic acid: Study of conditions (*402*)
	Deca-ammine-μ-peroxocobalt(III)–cobalt(II): Study of conditions (*441*)
Molybdenum	Calcium chloride: W (*360*)
	Purpurogallin; dibromopurpurogallin; 3′,4′-dihydroxy-α,β-benzotropolone: Study of conditions (*126*)
	1-Nitroso-2-naphthol: Study of interferences (*392*)
	2,2′-Pyridoin: Study of conditions (*40*)
	Sulfide: Study of conditions (*255*)
	Sodium selenide: Study of conditions (*543*)
	Acid dehydration: Molybdenum silicide (*231*)
	Electrolytic separation: Re (*154*)
Tungsten	8-Quinolinol: Study of conditions (*573*)
	N-Phenylbenzohydroxamic acid: Study of conditions and interferences (*226*)
	Cinchonine: Re (*610*)
	Cinchonine–tannic acid–antipyrene: Ores and concentrates (*400*)
	Tannin-β-naphthoquinoline: Nb (*489*)
	Calcium chloride: Mo (*360*)
	Acid dehydration: Study of coprecipitation (*156*)
	Hydrogen peroxide–acid dehydration: Nb, Ta (*105, 202*)
	Thermogravimetry of WO_3: (*83, 127, 307, 363*)

Much of the recent literature concerning niobium and tantalum deals with spectrophotometric methods. Most of these are applications of well known methods either to different material or in the utilization of new separation processes. Esson (*137*) used a radiotracer technique to study the thiocyanate–ethylacetate extraction determination of niobium and found recoveries as low as 50 to 60%. Minczewski and Rozycki (*335, 336*) studied the effect of either acetone, ethyl ether, or tributyl phosphate in the chloroform phase on the extraction and determination of niobium as the thiocyanate. Dixon and Headridge (*117*) used thiocyanate to determine niobium and crystal violet to determine tantalum in a study involving the anion exchange separation of these elements along with titanium, zirconium, molybdenum, and tungsten from solutions of hydrofluoric and hydrochloric acid. The determination of niobium and tantalum in chrome-nickel steel was performed by Munchow (*347*) by following an anion exchange separation with a thiocyanate determination of niobium and a pyrogallol determination of tantalum. Nishimura and Irokawa (*372*) determined niobium in tantalum and tantalum oxide by means of the thiocyanate method following an ion exchange separation. Yoshida and Kitamura (*596*) extracted the bulk of the tantalum with methyl isobutyl ketone before determining niobium in tantalum metal and oxide by the thiocyanate method. The extraction of the tantalum with methyl isobutyl ketone prior to the determination of niobium with thiocyanate was also used by Luke (*303*) for the determination of niobium in tantalum metal. Beryllium hydroxide was used as a carrier by Kageyama, Endo, and Tomori (*225*) for the precipitation of niobium as the hydroxide from an EDTA solution prior to the determination of niobium in steel with thiocyanate. Shrimal and Varde (*497*) separated niobium from uranium by dehydration with perchloric acid using a tantalum carrier prior to determination as the thiocyanate. Niobium was determined in steel with thiocyanate by Dorosh and Ivanova (*120*) following separation as the hydroxide and by White and Scholes (*578*) following separation as the phenylarsonate with zirconium as the carrier. Thiocyanate is by no means the only reagent used for the spectrophotometric determination of niobium. Ishiwatari (*207*) used xylenol orange for the determination of niobium and zirconium by first determining the total and then determining zirconium alone, by changing the order of addition of the reagents, and obtaining the niobium by difference. Xylenol orange was also used by Sakaki (*457*) for the determination of niobium in steel. Hydrogen peroxide was used by Cheng (*92*) for the determination of niobium in niobium stannide without separation; by Wood and Adams (*585*) for the determination of niobium in niobium–zirconium alloys; and by Uvarova and Sikora (*561*) for the determination of niobium in lead niobate and lead niobate–barium niobate mixtures. Nordling (*374*) recommends potassium niobate dissolved in oxalic acid, with the concentration determined by the gravimetric cupferron procedure, as a standard for the hydroquinone spectrophotometric method for niobium. Kawahata, Mochizuki, and Misaki (*243*) determined niobium, tungsten, and titanium simultaneously in heat-resistant steel by the hydroquinone method. Hibbits and Kallmann (*197*) studied the separation of microgram amounts of niobium and tantalum from each other and from ten milligrams each of 70 different elements by first precipitating with cupferron, then using an anion exchange procedure followed by a second precipitation with cupferron, after which the niobium is determined with hydroquinone and the tantalum with pyrogallol. Brhacek and Kurzova (*63*) determined niobium in steel with hydroquinone and tantalum with pyrogallol following a precipitation separation from an ammoniacal EDTA solution. Pyrogallol was used by Lapin and Prilutskaya (*284*) for the determination of niobium in steel after separation by precipitation with ammonium hydroxide. Belcher, Ramakrishna, and West (*35*) studied the conditions and interference in the determination of niobium with bromopyrogallol red in an EDTA–tartrate–cyanide solution. The same authors (*36*) later reported a further detailed study including molar absorptivities, composition of the complex formed, useful range, and interferences under various conditions. The color reactions of the peroxy complexes of niobium and tantalum with nine metallochromic indicators including pyrocatechol violet, PAN, bromopyrogallol red, 4-(2-pyridylazo)-resorcinol, and xylenol orange as a function of pH were described by Lassner and Puschel (*286*, *287*). Elinson and Rezova (*133*) described the determination of tantalum with 4-(2-pyridylazo)-resorcinol and reported the wavelengths of maximum absorbance and molar absorptivities under various conditions. Patrovsky (*396*) studied 4-(2-thiazolylazo)-resorcinol as a reagent for niobium. The complexes that niobium form with arsenazo I were investigated by Kornilova and Nazarchuk (*269*). Babko and Shtokalo (*24*) studied the relative stabilities of various tantalum complexes by determining the concentration of added ligand that was necessary to reduce the absorbance of the tantalum–arsenazo I complex to one half its original value. The niobium–eriochrome cyanine complex was used in the same way by the same authors (*22*) to study several niobium complexes. Asamov, Dzhiyanbaeva, and Talipov (*18*) determined niobium by measuring the absorbance of the organic phase after the extraction of the niobium–pyrocatechol violet–2,3′-bipyridyl complex into chloroform. Talipov, Dzhiyanbaeva, and Asamov (*544*, *545*) also used the extraction of the metal–pyrocatechol–1,2′-bipyridyl complex into chloroform for the determination of both niobium and tantalum and the extraction of the niobium–thiocyanate–4-(4′-biphenylylazo)-catechol complex into chloroform for the determination of niobium. A different approach to the use of 2,2′-bipyridyl was used by Bykovskaya (*81*) who reduced niobium bium to niobium(III), used this to reduce iron(III) to iron(II), and used the reagent for the determination of the iron(II). Kornilova and Nazarchuk (*270*) described the use of hematoxylin for the determination of niobium. Application of morin and hematoxylin for the determination of niobium and tantalum, and of pyrocatechol violet for the determination of tantalum in the presence of niobium was studied as a function of pH by Babko and Shtokalo (*23*). Other reagents used for the determination of niobium and tantalum were salicylhydroxamic acid by Alimarin and Borzenkova (*10*) and tiron by Shimizu and Hosohara (*492*). Additional reagents used for the determination of niobium are *N*-phenylbenzohydroxamic acid–thiocyanate by Ni and Liang (*364*); derivatives of trihydroxyfluorone by Nazarenko and Yagnyatins'ka (*357*); 5-dimethylamino-2-(2-thiazolylazo)phenol by Minczewski and Kasiura (*334*); sulfochlorophenol S by Savvin, Bortsova, and Malkina (*466*); malachite green–fluoride by West (*576*); lumogallion by Alimarin and Han (*11*); gossypol by Talipov and Khadeeva (*546*); 4-methoxybenzothiohydroxamic acid by Skorko-Trybula and Minczewski (*507*) and the niobate ion itself in basic solution at 235.4 mμ by Borlera (*52*). Reagents for the determination of tantalum include crystal violet, malachite green, and brilliant green used by Makarova and Alimarin (*314*); crystal violet–fluoride used by Alimarin and Makarova (*12*); rhodamine 6G 11 and butylrhodamine B used by Dorosh (*119*); and reduced 12-molybdotantalic acid used by Guyon (*183*). Yatsimirskii *et al.* (*593*) described a so-called catalytic method for tantalum consisting of a turbidimetric measurement of barium sulfate formed from the sulfate resulting from the oxidation of thiosulfate by hydrogen peroxide which is catalyzed by tantalum.

The gravimetric methods for the determination of tantalum consist largely of applications of previously described reagents and frequently in combination with a modified or improved separation method. Tada and Horiguchi (*542*) used cupferron for the determination of tantalum in carbides following an anion exchange separation. An anion exchange separation was also used by Sawada and Kato (*469*) preceding the determination of niobium and tantalum in titanium and zirconium alloys with cupferron. Munchow (*347*) also used an anion exchange separation and cupferron precipitation for the determination of niobium and tantalum in steel. Patrovsky (*395*) precipitated tantalum with benzene arsonic acid in the presence of hydrochloric, tartaric, and oxalic acids, and separated it from niobium and titanium. The use of α-hydroxybenzylphosphonic acid as a precipitant for niobium was described by Ryabchikov and Dedkov (*453*). Kawahata *et al.* (*242*) determined niobium and tantalum separately in stainless steel by precipitation with *N*-phenylbenzohydroxamic acid. *N*-benzoyl-*N*-(*o*-tolyl)hydroxylamine was used for the separation of niobium and tantalum from each other by Majumdar and Pal (*313*). Gibalo, Alimarin, and Davaadorzh (*161*) studied the precipitation of niobium with ammonium tetramethylenedithiocarbamate. Niobium was separated from tantalum and molybdenum and determined by Jain and Singh (*213*) using 4-phenyldaphnetin. Hoste, Adams, and Dams (*202*) used radiotracers to study the precipitation of niobium and tantalum by hydrolysis of their hydrogen peroxide complexes. Dams and Hoste (*103*) described a method for the determination of niobium and tantalum using the hydrolysis of these hydrogen peroxide complexes and attempted to apply the method to the separation of niobium and tantalum (*104*) and to the analysis of tantalocolumbites (*105*). Sheskolskaya (*489*) separated niobium from tantalum by precipitation of the hydroxide with ammonia from an EDTA solution. Pyrogallol was used by Sarma (*460*) as a masking agent for titanium, iron, manganese, and tin to prevent their precipitation along with niobium when the niobium was precipitated by hydrolysis.

A number of spectrographic methods have also been published during the period covered by this review. Huff (*203*) used the spark technique for the determination of niobium and tantalum in column effluents in a study of anion exchange separations using mixtures of nitric acid and hydrofluoric acid. Munchow (*347*) used hydrochloric acid and hydrofluoric acid with an anion exchange resin for the separation of niobium and tantalum prior to determination in chrome–nickel steel. Malinek (*317*) used a d.c. arc for the determination of niobium and tantalum in mixtures of oxides of the two elements following chemical separation from a tungsten–tin concentrate. Tantalum was determined in purified boron by Tarasevich and Zheleznova (*548*) following volatilization of the boron as the fluoride. Following the extraction of plutonium with trioctylamine, Ko (*254*) determined tantalum and niobium spectrographically. Brooks (*66*) used a pyrosulfate fusion followed by d.c. arc excitation for the determination of tantalum and other impurities in niobium. Tantalum was also determined in niobium by Balenko and Lifshits (*27*). Litomisky (*297*) used potassium carbonate and silica as a buffer to delay the distillation of niobium and tantalum and thereby increase the line intensities in the spectrographic determination of these elements in cassiterite–wolframite concentrates. Briere and Kurylenko (*64*) determined niobium in Madagascar zircons.

X-ray fluorescence is also a useful technique for the determination of niobium and tantalum. Bertin (*39*) used x-ray fluorescence with an intensity ratio *vs.* concentration ratio plot of the data for the determination of niobium and tin in niobium–tin compounds. Tantalum and niobium were determined in tantalite by Nishimura and Kawasaki (*373*), in niobiotantalite by Latorre and Polonio (*285*) and in minerals by Kakhana (*228*). Two methods involving the use of x-ray radiation directly are the determination by Toussaint and Vos (*555*) of tantalum in niobium by making the sample the target and measuring the x-ray radiation emitted, and the determination by Rekhkolainen (*443*), of niobium and tantalum in solutions by measuring x-ray absorption.

Yule (*600*) included niobium and tantalum in a determination of γ-photopeak yields for 118 reactor thermal neutron products and the estimation of detection limits from these data. Laverlochere (*288*) discussed neutron activation analysis and included 67 references. Neutron activation analysis was used by Verbeek (*566*) for the determination of niobium in steel following an extraction with methyl isobutyl ketone; by Dugain and Laverlochere (*125*) for tantalum in niobium with and without separation; by Simkova (*501*) for tantalum in iron and niobium; by Gleit *et al.* (*168*) for tantalum in filter media and high purity quartz; by Ricq, Capitant, and Troly (*445*) for niobium and tantalum in rocks and minerals following an extraction with methylisobutyl ketone; by Schlitz and Coquema (*472*) in rocks and minerals following a separation by paper chromatography; by Gebauhr and Martin (*157*) for tantalum in high purity silicon; by Szabo (*538*) for the determination of tantalum in rocks; and by Ricq *et al.* (*446*) for niobium and tantalum in rocks and minerals.

Table V. Electrometric Methods

Element	Method; Reagent: Material (References)
Zirconium and zirconium plus hafnium	Amperometric; EDTA: Organic compounds (*550*)
	Amperometric; Flavazine L: Study of conditions (*421*)
	Amperometric; Neocupferron: Study of conditions and interferences (*151*)
	Amperometric; Na_2MoO_4: Study of conditions (*180*)
	Amperometric; Disodium-2-(3-methyl-5-oxo-1-phenyl-2-pyrazolin-4-ylazo)-6,8-naphthalenedisulfonate: Study of conditions (*420*)
	Polarographic: Study of conditions and interferences (*486, 602*)
	a.c-polarographic: Study of conditions (*236*)
Hafnium	Amperometric; Tartrazine or flavazine L: Study of conditions (*416*)
Vanadium	Amperometric; Iron(II): Steel (*140, 141, 376*); refractory alloys (*524*)
	Amperometric; Iron(II)–permanganate: V(IV), V(V) (*510*)
	Amperometric; Vanadium(II): Study of conditions (*152*)
	Amperometric; Thallium(I): Study of conditions (*598*)
	Amperometric; Silver: Study of conditions (*471*)
	Amperometric; Chloramine T: Study of conditions (*114*)
	Polarographic: Study of conditions or reactions (*34, 58, 163, 245, 470*)
	Coulometric: NaCl (*597*); $TiCl_4$ (*429*)
	Electroanalytical study: LiCl–KCl eutectic (*476*)
Niobium	Amperometric; Pyrocatechol: Study of conditions (*260*)
	Polarographic: Steel (*227*), Pb-Ti-Zr ceramics (*171*)
	a.c.-polarographic: Study of conditions (*236*); steel (*240*)
Tantalum	Polarographic: Study of conditions and interferences (*69*)
Chromium	Amperometric; Fe(II): Steel (*140–142*); fluoride salts (*17*)
	Amperometric; H_2O_2: Study of conditions and interferences (*592*)
	Amperometric; Chloramine T: Study of conditions (*114*)
	Polarographic: Cement (*206*); glass (*274*); ruby (*332, 503*); study of reactions (*58, 502*)
	a.c.-polarographic: HNO_3 (*14*)
	Coulometric: Study of conditions (*95*); tinplate (*581*)
	Controlled potential coulometric: Study of conditions (*440*)
Molybdenum	Amperometric; $TlNO_3$: Study of conditions (*598*)
	Amperometric; V(II): Ferromolybdenum (*181*)
	Amperometric; EDTA: Study of conditions (*442*)
	Amperometric; 8-quinolinol: Study of conditions (*320*)
	Amperometric; 8-mercaptoquinoline: Steel (*530*)
	Amperometric; Bis-(4-dimethylaminophenyl)-methane: Study of conditions and interferences (*390*)
	Polarographic: Study of conditions and/or interferences: (*58, 478, 485, 570, 583*); In (*42*); Nb (*192, 193*); W (*258, 389, 514*); Steel (*240, 280, 393, 535*)
	Oscillopolarographic: Study of conditions (*328*)
Tungsten	Amperometric; Thallium(I) nitrate: Study of conditions (*598*)
	Amperometric; Lead nitrate: Steel (*51*)
	Amperometric; Lead acetate–EDTA: Minerals (*90*)
	Polarographic: Study of conditions (*58, 388, 515*); Mo (*258, 389, 523*); steel (*525*)
	a.c.-polarographic: Steel (*240*)
	Square-wave polarographic: Re (*233*)

Both a.c. and d.c. polarographic techniques were studied by Kajiyama and Kawahata (*227*) in the development of a method for the determination of niobium in steel following separation by dehydration with perchloric acid. A standard additions method was used by Goode, Herrington, and Jones (*171*) for the determination of niobium in lead–titanate–zirconate ceramics with a cathode ray polarograph. Kasagi and Banks (*236*) used an a.c. polarograph for the determination of niobium in aqueous solution and Kawahata *et al.* (*240*) separated niobium from the components of stainless steel by dehydration with perchloric acid and then determined it by an a.c. polarographic technique. Budarin, Rumyantseva, and Sherina (*69*) determined tantalum by means of the catalytic hydrogen peroxide current in an oxalate-sulfate solution. An amperometric titration using pyrocatechol violet as the titrant and EDTA to mask interferences was used by Komolova and Tserkovnitskaya (*260*) for the determination of niobium in steel.

Lassner (*286*) determined niobium titrimetrically by adding an excess of nitrilotriacetic acid or *N*-(hydroxyethyl)-ethylenediamine-*N*,*N'*,*N'*-triacetic acid and back-titrating with copper to the disappearance of the fluorescence of methylcalcein. Bykovskaya (*81*) reduced niobium to niobium(III) then oxidized it back to niobium(V) with iron(III) and then titrated the iron(II) formed with chromium(VI). Kozyreva, Kuteinikov, and Zharova (*276*) precipitated niobium with an excess of 8-quinolinol and titrated the excess bromatometrically. Babko, Lukianets, and Nabivanets (*21*) titrated the combined peroxide in the niobium peroxide complex with permanganate in order to determine niobium.

Guthrie (*182*) determined niobium and tantalum along with 24 to 48 other elements in erbium, cerium, and lutetium by a spark source mass spectrographic technique.

CHROMIUM

Because of the color of chromium, the stability of two of its oxidation states, the ease of changing oxidation states, and also its wide use in steels and high-temperature alloys, and the increasing interest in lasers, chromium has been determined by most of the common analytical techniques.

The occurrence and determination of chromium in air, foodstuffs, and biological tissue was reviewed by Gandolfo and Sampaolo (*153*) and 666 references were included. Strelow, Rethemeyer, and Bothma (*521*) included chromium in the determination of distribution coefficients of a number of cations between either nitric or sulfuric acid and a sulfonated polystyrene resin. Danielsson (*106*) studied the adsorption of 26 metal ions including chromium on an anion exchange resin from sulfuric acid solution. Korkisch and Hazan (*268*) studied the anion exchange behavior of a number of elements including chromium in a number of organic solvents containing hydrochloric acid. Koprda and Fojtik (*264*) found that while chromium and manganese cannot be separated using Dowex-2 and 10.5*M* hydrochloric acid at room temperature, the separation is quantitative at temperatures above 55° C. Green (*174*) listed four methods for chromium in a review of solvent extraction procedures for a number of elements. Neutral organophosphorus compounds were used by O'Laughlin and Banks (*377*) for the separation of a number of elements including chromium.

Chromium can be successfully determined by a number of spectrophotometric methods. Yablochkin (*589*) measured the absorbance of the chromium(III)–EDTA complex at 570 mμ. Zabiyako and Sharapova (*601*) used essentially the same method for the determination of chromium in chrome alum solutions. This method was also applied to the determination of chromium in silicate rock by Stolyarov and Agrest (*520*). Hashmi *et al.* (*189*) used an equimolar mixture of isonicotinic acid hydrazide and 2,3,5-triphenyltetrazolium chloride as a reagent for the determination of chromium following oxidation with silver(II) oxide. The complex formed by heating chromium (III) stearate with *o*- or *m*-toluidine was used by Malik and Ahmad (*316*) for the determination of chromium in metal soaps. A cation exchange separation was used by Popa, Radulescu-Grigore, and Basamac (*422*) prior to determination of chromium in steel as chromium(VI) with chromatropic acid. Sangal (*458*) studied the properties and analytical applications of the chromium–alizarin red S complex. Malat and Hrachovcova (*315*) made an extensive study of the chromium–chrome azurol S complex. The spectrophotometric determination of chromium using pyridine–2,6-dicarboxylic acid was studied by den Boef and Poeder (*47*). Komatsu and Takahashi (*259*) determined chromium by extracting the product formed by the oxidation of 1-phenylthiosemicarbazide with chromium(VI) into chloroform and measuring the absorbance at 372 mμ. Chromium was determined in steel by Aitova (*5*) using antipyrinylbis-[*p*-(4-methylbenzylamino)-phenyl]-methanol and extracting the complex into benzene. After separation of iron and aluminum, which are two of the most serious interferences, by extraction of their 8-quinolinolates into chloroform, chromium was determined by Uesugi and Katsube (*557*) using 2-hydroxy-1-(2-hydroxy-4-sulfo-1-naphthylazo)-naphthalein. Dodd, Wood, and Barns (*118*) used a nondestructive spectrophotometric technique for the determination of chromium in ruby in which the absorbance of single crystals was measured. Kovalenko and Petrashen (*272*) studied the complex that chromium(VI) forms with *s*-diphenylcarbazide. Szczepaniak (*541*) synthesized a *s*-diphenylcarbazide resin which he used to study the reaction between chromium(VI) and *s*-diphenylcarbazide. The *s*-diphenylcarbazide method was used by Appelbaum and Mashall (*16*) in a study of the oxidation of chromium(III) with silver(II) oxide. Applications of the *s*-diphenylcarbazide method include the determination of chromium in ores and refractories by Richards and Boyman (*444*); in ruby by Sil'nichenko and Gritsenko (*500*); in the presence of manganese in rocks and minerals by Easton (*129*); in steel and high-purity iron by Scholes and Smith (*474*); in rhenium and rhenium compounds by Ryabchikov, Lazarev, and Lazareva (*454*); in sea water by Chuechas and Riley (*96*); in canned food by Molodchenko and Lyubivaya (*339*); and in beer by Deschreider and Meaux (*113*).

Pavlov and Kuznetsov (*397*) recommended the addition of excess EDTA and a back-titration of the excess with zinc to the dithizone end point and found an error of 0.5% or less for the titrimetric determination of chromium. The same authors (*398, 399*) determined aluminum and chromium in mixtures by titrating the total in one aliquot and masking the aluminum with fluoride and titrating only the chromium in another. Sosin and Strzeszewska (*511*) used a back-titration of the excess EDTA with zinc for the determination of chromium in chromium ore. Golubovic and Ljustina (*169*) used a mixed indicator consisting of eriochrome black T, congo red, and eriochrome green B for the back-titration with zinc in the determination of chromium in the presence of aluminum, calcium, and magnesium. A back-titration with zinc to the eriochrome black T end point was used by Majumdar and Mitra (*312*) for the determination of chromium following anion exchange separations of several metals as the thiocyanate. A back-titration of the excess EDTA with copper to a fluorescent end point was used by Martinez and Barral (*324*). A determination of chromium(III) in the presence of chromium(VI) by Hentrich and Pfeifer (*196*) utilized the back-titration of EDTA with lead to the precipitation of lead chromate. Liteanu, Crisan, and Rencz (*296*) used thiocyanate and 8-quinolinol to detect the end point in the back-titration of EDTA with cobalt. The quantitative reduction of chromium(VI) with iron(II) was used by Janousek (*215*) for the determination of chromium and manganese in steel; by Babachev, Petrova, and Raeva (*20*) for the determination of chromium in silicates; by Balyuk and Zil'berg (*28*) for the determination of chromium in chromite ores and other refractories; by Nazarchuk *et al.* (*355*) for the determination of chromium in the presence of lanthanum; and by Rao and Devara (*437*) for the determination of chromium and vanadium in mixtures. Edge and Fowles (*130*) reduced chromium(III) to chromium(II), which was oxidized by iron(III) and the resulting iron(II) was then titrated with chromium(VI). For the determination of chromium in steel, Lounamaa (*299*) oxidized the chromium to chromium(VI), added an excess of iron(II), and titrated the excess with permanganate. Paar and Hawkins (*384*) also added excess iron but titrated the excess with dichromate for the determination of chromium in feces. Polak, den Boef, and de Galan (*410*) oxidized chromium to chromium(VI) by heating to 60° C. with an excess of permanganate and then titrated the excess permanganate with arsenic(III), detecting the end point photometrically. In order to determine chromium(III) in a chrome-plating electrolyte Onufrienok, Borodulina, and Gavryusheva (*380*) oxidized all of the chromium to chromium(VI), titrated the chromium(VI) to chromium(III) with standard hydrogen peroxide, then chromium(III) to chromium(VI) with potassium permanganate. The chromium(VI) was determined in the same way without the initial oxidation step, and the chromium (III) was then determined by difference. Jungnickel and Klinger (*220*) added excess oxalate to a chromium(III) solution forming the dioxalatochromate(III) ion and then titrated the excess oxalate with cerium(IV). Chromium in ferrochrome and other alloys was determined iodimetrically by Babachev (*19*). Chromium was determined in steel by Zhigalkina, Lev, and Cherkesov (*604*) using the titration of chromium(VI) in 50% sulfuric acid with 4-phenylazo-1-naphthol. The end point was determined conductometrically by Matyja (*327*) in the titration of chromium(III) with vanillin azine.

The publication of gravimetric methods for the determination of chromium is quite limited. Pietsch and Ludwig (*401*, *402*) studied the conditions for precipitation of chromium with isomers of dimethylphenylarsonic acid and trichlorophenylarsonic acid. A solution of deca-ammine-μ-peroxocobalt (III)–cobalt(II) chloride was used for the quantitative precipitation of chromium(VI) by Reibel' (*441*).

Various spectrographic methods have found considerable use in the determination of chromium, especially in steel along with other metals. Runge (*451*) used laser excitation for the determination of chromium in steel with a coefficient of variation of 5%. Titanium was volatilized as the tetrafluoride prior to the spectrographic determination of chromium in titanium in a procedure used by Fratkin and Shebunin (*147*). Huff (*203*) used spectrographic methods for the determination of the various elements in a study of the anion exchange behavior of 19 elements in solutions containing various concentrations of nitric and hydrofluoric acids. Procedures for the spectrographic determination of chromium in iron and steel were published by Ungureanu and Fodor (*558*), Zhelonkina *et al.* (*603*), Buyanov (*79*), Javurek (*218*), Nikitina and Ivanova (*366*), Lounamaa (*301*), and Baskov and Palladin (*31*). An arc or low voltage spark was used by Buyanov, Ivanova, and Sukhova (*80*) for the determination of chromium in heat-resistant alloys. Filimonov *et al.* (*143*) studied the factors affecting the accuracy of the determination of chromium in chromium bronze with a photoelectric spectrometer. Zirconium was used as an internal standard in the determination of chromium in aluminum alloys by Malusecki and Studencki (*318*). Muzgin *et al.* (*351*) determined chromium in vanadium pentoxide at the 0.001 to 0.1% level with an error of ±10 to 20%. Tarasevich and Moseli (*547*) atomized a solution of the sample into a spark discharge for the determination of chromium in titanium alloys. Carpenter and Lewis (*84*) described the determination of chromium and other impurities in high-purity hafnium. Brooks (*66*) used a pyrosulfate fusion prior to arc excitation in order to determine the impurities including chromium in niobium. Balenko and Lifshits (*27*) used a lithium carbonate buffer and niobium as the internal standard for the determination of impurities in niobium. Vasilevskaya *et al.* (*565*) and Pikhtin (*403*) determined chromium in silicon carbide. Plsko and Herkelova (*406*) determined chromium in silicates, using a lithium carbonate buffer and calcium oxide internal standard. Luppino (*306*) used molybdenum as an internal standard for the determination of various metallic impurities in electroplating solutions. Zhuravlev and Ryzhkova (*606*) used spark excitation of atomized solutions and photoelectric recording for the determination of eight elements including chromium. Lachin (*283*) determined chromium and many other elements in high polymers.

Flame photometry was used by Daly and Anstall (*102*) for the determination of chromium in feces. Waggoner (*571*) found that preliminary separation is not necessary for the determination of chromium and other constituents in stainless steel by flame photometry. Chromium was also determined in iron and steel by Prince, Coglianese, and Coless (*427*). Feldman and Purdy (*139*) determined chromium using atomic absorption spectroscopy by extracting chromium(VI) into methyl isobutyl ketone and aspirating the organic phase into an air–hydrogen flame. Barnes (*30*) used ammonium chloride to eliminate the suppression effect of iron when determining chromium in low alloy steels by atomic absorption spectroscopy.

X-ray fluorescence was used by Spielberg and Abowitz (*516*) for the determination of nickel and chromium in thin films. Three techniques were compared. Sparks and Britton (*513*) used x-ray fluorescence for determining the chromium content of thin oxide films. The chromium in ferrochrome was determined by Fukasawa and Takeuchi (*150*) with a standard deviation of 0.4%. A miniaturized apparatus using tritium absorbed on zirconium as an excitation source was used by Karttunen *et al.* (*235*).

The polarographic reduction of chromium(VI) in sodium or potassium hydroxide solution was used by Ishii, Hayashida, and Einaga (*206*) for the determination of chromium in cement; by Kowalski, Narebska, and Zarebski (*274*) for the determination of chromium in glass; and Miedzinski and Lukaszewski (*332*) and Singh (*503*) for the determination of chromium in ruby. Simoncini (*502*) used the polarographic reduction of chromium(III) for the determination of chromium. The conditions determining the sensitivity for the polarographic determination of certain anions in solution were studied by Brainina and Roizenblat (*58*). Anikina and Kuzub (*14*) determined chromium by oscillopolarography with 1*M* sodium hydroxide being used as the supporting electrolyte and the standard additions method being used to ascertain the concentration.

Chromium in mixed fluoride salts in the range of 1 to 50 μg was titrated with iron(II) using an amperometric end point by Apple and Zittel (*17*). Filenko (*140*) determined chromium along with manganese and vanadium in steel by using various oxidizing conditions and an amperometric titration with iron(II) to obtain the sum of the three, then the chromium plus vanadium, and then the vanadium alone. The same author also used an amperometric titration with two indicator electrodes for the determination of manganese and chromium (*142*) and manganese, chromium, and vanadium (*141*) in steel. A rotating platinum electrode was used with hydrogen peroxide as the titrant for the amperometric titration of chromium by Yatrudakis and Zhdanov (*592*). The method used by Deshmukh and Eshwar (*114*) consisted of the addition of excess reducing agent and titration of the excess with chloramine T at an applied potential of +300 mV.

A differential controlled–potential coulometric technique in which the sample and standard were electrolyzed simultaneously in identical cells was used by Rechnitz and Srinivasan (*440*) for the determination of chromium. Christian and Feldman (*95*) performed an ultramicrocoulometric titration in which they determined as little as 0.004 μg of chromium. Wiltshire, Nancarrow, and Ludvig (*581*) used a coulometric titration method for the determination of surface chromium in electrolytic tinplate.

Chromium was included in those elements for which the neutron activation analysis sensitivities were determined by Yule (*600*). Girardi, Guzzi, and Pauly (*165*) studied a neutron activation method including direct calculation of weight from nuclear constants for a number of elements and evaluated accuracy and precision. Neutron activation was used for the determination of chromium in aluminum by Kiesl, Hecht, and Sorantin (*249*) and by Zmiyewska and Kozminska (*609*); in biological materials by Bowen (*56*); and in high purity silicon by Gebauhr and Martin (*157*). Chromium was included in the work done by Stary and Ruzicka (*517*) on neutron activation determination following extraction with substoichiometric amounts of complexing agents.

Table VI. Spectrochemical

Element	Material (References)
Zirconium	Following ion exchange (*203*); comparison of methods (*68*); in solutions (*341*); Nb (*65, 66*); Ni (*209*); SiC (*403*); zircons (*64*); minerals and ores (*37*); rubber polymers (*283*); Pu (*254*)
Hafnium	Zircon (*572*)
Vanadium	Plasma excitation (*195*); study of electrode processes (*613*); following ion exchange (*203*); following solvent extraction (*67*); SiO_2 (*612*); SiC (*403*); Cr-Ni-Mo alloys (*432*); UF_4 (*409*); Hf (*84*); steel, Fe (*301, 366*); coke (*73*); heat-resistant alloys (*80*); rubber polymers (*283*); comparison of methods (*68*)
Niobium	Zircon (*64*)
Niobium and tantalum	Ores and concentrates (*297*); Nb_2O_5-Ta_2O_5 (*317*); following ion exchange (*203*)
Tantalum	Boron (*548*); Pu (*254*); Nb (*27, 66*)
Chromium	Laser excitation (*451*); following ion exchange (*203*); following removal of volatile fluorides (*147*); steel, Fe (*31, 79, 218, 301, 366, 588, 603*); Cr bronze (*143*); Al (*318*); V_2O_5 (*351*); Ti alloys (*547*); Nb (*27*) (*66*); Hf (*84*); heat-resistant alloys (*80*); SiC (*403, 565*); silicates (*406*); electroplating solutions (*306*); solutions (*606*); rubber polymers (*283*); comparison of methods (*68*)
Molybdenum	Following ion exchange (*203*); comparison of methods (*68*); Ti (*547*); Hf (*84*); Nb (*65, 66*); heat-resistant alloys (*80*); steel (*159, 301*); UF_4 (*409*); B (*548*); rocks (*208, 512*); ashes and ores (*537*); rubber polymers (*283*)
Tungsten	Following ion exchange (*203*); Hf (*84*); Nb (*65, 66*); heat-resistant alloys (*80*); B (*548*); Pu (*254*); rocks (*208, 512*); aerosols (*294*); rubber polymers (*283*)

Table VII. X-Ray Fluorescence

Element	Material (References)
Zirconium	Steel and U (*549*); U (*185*); rocks (*78, 136*)
Hafnium	Zr (*411, 433*)
Vanadium	With portable source (*235*); organic materials (*128*)
Niobium	Nb_3Sn (*39*)
Niobium and tantalum	Minerals and ores (*228, 285, 373*)
Tantalum	Nb (*555*)
Chromium	With a portable source (*235*); Ni-Cr films (*516*); Sn plate (*513*); ferrochrome (*150*)
Molybdenum	Comparison of methods (*293*); study of conditions (*569*); U (*185, 391*)
Tungsten	Ni (*25*)

Table VIII. Neutron Activation Analysis

Element	Material (References)
Zirconium	Determination of sensitivities (*600*); review (*288*); study of separations (*517*); study of conditions (*94*); meteorites (*331, 473, 479*); igneous rocks (*551*)
Hafnium	Study of method (*165*); Zr (*333*); ZrO_2 (*166, 178*); Al (*249*); rocks (*78, 538*)
Vanadium	Determination of sensitivities (*600*); study of separations (*517*); Zr (*333*); Al (*248*)
Niobium	Steel (*566*); nuclear materials (*288*)
Niobium and tantalum	Determination of sensitivities (*600*); rocks and minerals (*445, 446*)
Tantalum	Nb (*125*); Fe, Nb (*501*); Si (*157*); rocks (*472, 538*); organic filters (*168*)
Chromium	Determination of sensitivities (*600*); study of separations (*517*); study of method (*165*); Al (*249, 609*); Si (*157*); biological material (*56*)
Molybdenum	Determination of sensitivities (*600*); following extraction (*217, 517*); W (*177*); Si (*157*); soil (*591*); hair and wool (*194*)
Tungsten	Determination of sensitivities (*600*); steel (*381, 608*); Al (*158*); Si (*157*)

McMillan (*309*) preceded a radiochemical determination of samples from pressurized water loops with group separations using both a cation and an anion exchange resin. Chakravarti *et al.* (*86*) presented methods for the determination of radionuclides in Pacific waters. Hilton and Reed (*199*) determined activation products in irradiated steels by using γ-ray spectrometry and an anion exchange separation with a precision of 2%. Sipos (*506*) used β-ray reflection for the determination of chromium in chrome salt solutions and spent chrome liquors.

Other methods which have been used for the determination of chromium are a mass spectrographic method for chromium in erbium, cerium, and lutetium by Guthrie (*182*); a direct conductivity measurement of chrome-plating electrolyte by Neustroeva and Sudakova (*361*); a paper chromatographic method on paper impregnated with s-diphenylcarbazide resin in which the size of the chromium spot is measured and compared with standards by Szczepaniak (*539, 540*); and a gas chromatographic study of chromium acetylacetonate and trifluoroacetylacetonate in which the accuracy and precision of electron capture and hydrogen flame detectors were evaluated by Albert (*7*).

MOLYBDENUM

Like chromium, molybdenum is of interest as a constituent of both steel and high-temperature alloys and it may be determined by a great variety of methods, but a large majority of the papers are concerned with spectrophotometric methods. Busev (*75*) authored a book dealing with the analytical chemistry of molybdenum in which he discussed methods of separation and determination and included 1580 references.

Separation of molybdenum by the use of a cation exchange resin from nitric and sulfuric acid solutions was studied by Strelow, Rethemeyer, and Bothma (*521*). Adsorption on an anion resin from sulfuric acid was studied by Danielsson (*106*). Kallmann (*229*) found that molybdenum, niobium, and tantalum could be separated from each other and from tungsten by passing ethanol or methanol through the column before eluting the cation. Molybdenum was separated from tungsten by Darbinyan and Danielyan (*107*) using an anion exchange resin in 4 to 5*M* sodium hydroxide. The hydrogen peroxide complexes of titanium and molybdenum were separated by Shiskov and Shishkova (*494*) by absorbing the titanium complex on a cation exchange column which does not retain the molybdenum. In an extensive review of solvent extraction methods, Green (*175*) listed several methods for molybdenum including acetylacetone, thiocyanate, and toluene-3,4-dithiol. Prosad and Yatirajam (*428*) separated molybdenum from rhenium by extracting molybdenum(III) from hydrochloric acid solution into amylacetate. Organophosphorus compounds were studied by O'Laughlin and Banks (*377*) as stationary phases for the reversed-phase partition chromatographic separation of a number of elements including molybdenum. Of the many reagents suitable for the spectrophotometric determination of molybdenum, the thiocyanate method was by far the most frequently discussed during the past two or three years. Kolling (*257*) studied the complex in an effort to ascertain its formula and concluded that it is oxypentathiocyanatomolybdate(V). Wilson and McFarland (*580*) oxidized the excess tin(II) and added the thiocyanate form of "tricaprylmethylammonium" chloride to ensure stable theoretical absorptivities for the oxypentathiocyanatomolybdate(V). Malyutine, Dobkina, and Pisareva (*319*) used thiourea as a reducing agent rather than the more frequently used tin(II) chloride. Hibbits and Kallmann (*197*) used the thiocyanate method in a test of the suitability of methods in the presence of 70 other elements. Nishida (*368*) studied the thiocyanate method and found more satisfactory results were obtained when the tin(II) chloride concentration was reduced. The same author (*367, 371*) separated molybdenum by adsorption on metastannic acid prior to determination as the thiocyanate and studied the effect of diverse ions on the method. Elbeih, Elbakry, and Aly (*131*) separated molybdenum by paper chromatography prior to determination with thiocyanate. Applications of the thiocyanate method include the determination of molybdenum in steel following an extraction with thenoyltrifluoroacetone by De and Rahaman (*110*) and in steel or ferrous alloys by Korabel'nik (*265*); Boulin and Jaudon (*54*); Luke (*304*); Lounamaa (*300*); Bauer (*33*); and Studenskaya *et al.* (*525*). Additional applications include the determination in copper ores and concentrates by Duczyminska, Kunz, and Ostachowska (*124*), in lead by Ustimov and Gladyshev (*559*) in ores by Nishida (*369*), and in soil by Korenman and Glazunova (*266*). Another frequently used reagent for the determination of molybdenum is toluene-3,4-dithiol. The reaction of molybdenum with this reagent was studied by Agrinskaya and Petrashen' (*4*). It was used for the determination of molybdenum in tungsten following an anion exchange separation by Sugawara (*527*); following the separation from rhenium using an anion exchange procedure by Hamaguchi, Kawabuchi, and Kuroda (*186*); in copper by Zopatti and Pollock (*611*); in geochemical samples by Marshall (*323*); and in biological materials by Bingley (*43*).

Table IX. Radiochemical Analysis

Element	Material (References)
Zirconium	After separation from Nb (*224, 282, 430, 450, 491*); in the presence of Nb (*179, 256*); following chromatographic separation (*60, 575*); following solvent extraction (*62, 532*); air (*574*); water (*45, 86, 588*); whale (*382*)
Vanadium	Displacement of Ag-110 (*164*)
Chromium	Steel (*199, 309*); biological materials (*86*)
Molybdenum	Study of separation (*431*); iron meteorites (*577*)

Table X. Miscellaneous Methods

Element	Method: Material (References)
Vanadium	Mass spectrographic: Er, Ce, Lu (*182*)
Niobium	X-Ray absorption: Nb-Ta mixtures (*443*)
Niobium and tantalum	Mass spectrographic: Er, Ce, Lu (*182*)
Chromium	Mass spectrographic: Er, Ce, Lu (*182*) β-Ray reflection: Chrome salt solutions (*506*) Conductivity: Chrome-plating electrolyte (*361*) Paper chromatographic-measurement of spot: Study of interferences (*539*); steel (*540*) Gas chromatographic: Comparison of detectors (*7*)
Molybdenum	X-Ray absorption: Ores (*246*); Be (*234*) Mass spectrographic: Er, Ce, Lu (*182*)
Tungsten	β-Ray back-scattering: Steel (*74, 172*) Mass spectrographic: Er, Ce, Lu (*182*)

Table XI. Compilation of Methods and Reviews

Element	Review
Zirconium	Inorganic chemistry (*82, 100*)
Niobium and tantalum	Review of analytical chemistry (*342, 394*)
Chromium	Determination and toxicology (*153*)
Molybdenum	Review of separation and determination methods (*75*)

The stability constant of molybdenum-8-quinolinolate complex in 50% acetone was measured by Berge and Kreutzmann (*38*). Motojima *et al.* (*344*) used 8-quinolinol for the determination of molybdenum in uranium. The reaction of molybdenum with 8-mercaptoquinoline was studied by Agrinskaya and Petrashen' (*3*). Martinez and Mourino (*325, 326*) studied the conditions for the determination of molybdenum with thiomalic acid and applied the method to the determination of molybdenum in the presence of iron. Tellurium was separated by precipitation prior to the determination of molybdenum as the peroxide complex by Shakhova and Ku (*481*). Charreton *et al.* (*91*) determined molybdenum as the vanadophosphate when present in mixtures containing phosphorus, silicon, vanadium, molybdenum, and tungsten. Measurement of the absorbance of the compound formed between ammonium molybdate and sodium sulfide was used by Yatsimirskii and Zakharova (*594*) for the determination of molybdenum. Kiss (*251, 252*) separated molybdenum by extraction with dibutyl hydrogen phosphate and determined it using morin. The blue product formed by the reduction of molybdate with thiourea was used by Murty (*348*) for the determination of molybdenum. Molybdenum can also be reduced with hydroxylammonium sulfate to give a species which can be measured spectrophotometrically as was done by Yuasa (*599*). Jogdeo and Mahajan (*219*) separated molybdenum from uranium by extraction into amylacetate prior to determination with thiogylcollic acid. Bozsai (*57*) studied the conditions and proposed a reaction mechanism for the formation of the molybdenum–phenylhydrazine complex. Tiron was used by Busev and Rudzit (*77*) for the determinaton of 5 to 80 μg of molybdenum. The same authors (*76*) also used the diphenylguanidinium salt of the anionic molybdenum complex with mercaptoacetic acid for the determination of 10 to 200 μg of molybdenum. Kalnin (*230*) used pyrocatechol for molybdenum in solid lubricants, Sinha and Dey (*505*) used alizarin red S and Katyal and Singh (*237*) studied the molybdenum–galangin complex. Molybdenum in steel was determined by Skorokhod, Tishchenko, and Shedov (*508*) using tetrahydro-4-hydroxy-2-(α-piperidino)-isopropylfuran-3-one. Jungnickel and Klinger (*221*) determined molybdenum in the range 10 to 200 μg per ml using 3-hydroxy-2-methylpyran-4-one. Jain and Singh (*211*) studied the complexes that molybdenum forms with 4-methyl- and 4-phenyl-*o*-dihydroxycoumarin and determined the stability constants, optimum pH, and interferences. Horak and Okac (*201*) used 2,3,4-trihydroxybenzene sulfonic acid for the determination of molybdate in up to a 600-fold excess of tungstate. Ch'i, Chang, and Hsu (*93*) described the synthesis and analytical reactions of glyoxal bis-(2-mercaptoanil). Kirkbright and Yoe (*250*) were able to determine molybdenum in steel using 2-amino-4-chlorobenzene–thiolhydrochloride by masking the iron with EDTA. Shustova and Nazarenko (*499*) studied 13 derivatives of 2,6,7-trihydroxyfluorone and selected the 9-*o*-nitrophenyl derivative as the most sensitive and easiest to prepare. Nazarenko and Shustova (*356*) used (*o*-nitrophenyl)-fluorone for the determination of molybdenum in refractory metals following a diethyldithiocarbamic acid extraction and Shustova (*498*) used 2,6,7-trihydroxy-9-(2-nitrophenyl)-fluorone in the same way and also studied the molybdenum–diethyldithiocarbamic acid complex. The stability constants and interferences of the nicotinohydroxamic acid–molybdenum complex were studied by Rowland and Meloan (*449*) and Skorko-Trybula and Minczewski (*507*) studied 4-methoxybenzothiohydroxamic acid. Other reagents used for the determination of molybdenum are 2′,3′4′-trihydroxyacetophenone by Popa *et al.* (*418*) and 2,2′-bipyridyl by Lazarev (*289*). The change in the angle of rotation of the plane of polarization for a solution of (+)-tartaric acid caused by different concentrations of ammonium molybdate was used by Kovalenko and Zakharova (*273*).

The fact that certain oxidation-reduction reactions are catalyzed by molybdenum and that the reaction rate is proportional to the amount of molybdenum present has been used by some investigators for the determination of molybdenum. Svehla and Erdey (*534*) and Anokhina *et al.* (*15*) both used the catalysis of the reaction between hydrogen peroxide and iodide with Svehla and Erdey measuring the time of appearance of the violet color from the reaction between iodine and variamine blue and Anokhina measuring the absorbance of the reaction between starch and iodine after a certain period of time. Wilson (*579*) used the reaction between perborate and iodide and designed automatic apparatus for the comparison of two absorbance measurements made on the solution with a short intervening time. Bognar (*48*)used the catalysis of the reaction between methylene blue and hydrazine sulfate at 100°C and measured the absorbance at 3.2 and 45 minutes after mixing. The catalytic oxidation of dithio-oxamide by hydrogen peroxide was followed by Pantaler (*387*) by measuring the absorbance at one-minute intervals for ten minutes.

Gravimetric methods for the determination of molybdenum include a combination method by Nemirovskaya, Keremedzhidi, and Yashina (*360*) in which they precipitate calcium molybdate and calcium tungstate, then dissolve the precipitate, reprecipitate the tungsten as tungstic oxide from boiling nitric acid, and titrate the calcium with EDTA. The molybdenum is then determined by the difference between the precipitation and titration results. Dutt and Singh (*126*) studied the precipitation of molybdenum with purpurogallin and three related compounds and found them to be suitable for the determination of molybdenum. The interference of iron, vanadium, and titanium was prevented by masking with EDTA in the method for the precipitation of molybdenum with α-nitroso-β-naphthol used by Patil (*392*). The precipitation of molybdenum with 2,2′-pyridoin was studied by Bhat and Jain (*40*). Koch and Brockmann (*255*) used a weighed amount of magnesium oxide to prevent the loss of molybdenum through volatility while converting molybdenum to the oxide after precipitation as the sulfide. Taimni and Rakshpal (*543*) determined molybdenum by precipitation as the selenide. An electrolytic separation from rhenium was used by Gavrilko, Kovalenko, and Bagdasarov (*154*) followed by dissolution of the molybdenum, evaporation of the solution to dryness, and ignition to molybdenum trioxide for weighing. For the determination of molybdenum in molybdenum silicide, Kamenar and Herceg (*231*) dissolved the sample in hydrofluoric acid, volatilized the silica, evaporated the molybdenum to dryness with sulfuric acid, and then ignited and weighed the molybdenum trioxide.

Molybdenum can be determined by both oxidation-reduction and complexometric titration methods. Rao (*436*) titrated molybdenum(V) with cerium(IV) using methyl orange as the indicator in hydrochloric acid and methyl red in sulfuric acid. Dikshitulu and Rao (*116*) titrated molybdenum(V) with cerium(IV) in 4 to 8*M* sulfuric acid or in 1.5*M* sulfuric acid plus 10% phosphoric acid in the presence of vanadium(IV) which did not interfere. They then titrated the total in 0.75 to 1.25*M* sulfuric acid plus 2 to 3% phosphoric acid. Rao (*435*) used essentially the same procedure or titrated the vanadium(V) with iron(II) and Edge and Fowles (*130*) reduced molybdenum with a sodium–lead alloy to molybdenum(III), then added iron(III) and titrated the iron(II) formed with chromium(VI), cerium(IV), or manganese(VII). Sagi and Rao (*455*) titrated molybdenum(V) with iron(III) in a hydrochloric acid solution in the presence of citric or tartaric acid at 98 to 100°C. in a carbon dioxide atmosphere. Potassium ferricyanide was used by Hayashi (*190*) for the titration of molybdenum after reduction to molybdenum(III) by zinc amalgam in the presence of EDTA and tartrate. The same author (*191*) used this same reaction along with reduction with cadmium or lead amalgam in a stream of hydrogen for the determination of molybdenum and vanadium in mixtures. Cihalik and Sevcik (*98*) reduced molybdenum with mercury and then titrated it potentiometrically with iodine monochloride. Bogovina, Novak, and Maltsev (*51*) reduced molybdenum by the addition of excess iron(II) and then titrated potentiometrically with 8-mercaptoquinoline. Kula (*277*) studied the 1:1 and 2:1 complex formed between molybdenum(VI) and EDTA and suggested the possibility of titrimetric determination. Sajo (*456*) titrated molybdenum(VI) directly with EDTA using a diphenylcarbazone–methylene blue–vanadium(V) system for determining the end point. Yaguchi and Kajiwara (*590*) found a 2:1 molybdenum to EDTA complex when hydrazine sulfate was used as the reducing agent and a 1:1 complex when hydroxylamine was used. These workers determined molybdenum by back-titration of the EDTA with bismuth to the xylenol orange end point or with copper to the PAN end point. Endo and Higashimori (*134*) determined molybdenum in ferromolybdenum by using an excess of EDTA and back-titrating with lead nitrate. Konkin and Zhikhareva (*262*) determined molybdenum in permalloy and ferromolybdenum by titrating the excess EDTA with zinc or by precipitating lead molybdate with a known excess of lead and titrating the excess lead with EDTA. Iron was used by Liteanu, Crisan, and Gheorghe (*295*) for the back-titration of the excess EDTA. Kawahata *et al.* (*241*) used a system for the determination of molybdenum in ferromolybdenum in which he added an excess of EDTA, then an excess of copper molybdenum in which he added an excess of EDTA, then an excess of copper and titrated the excess copper with EDTA. Uvarova and Rik (*560*) used the same method for the determination of molybdenum in cobalt–molybdenum–aluminum catalysts. Other reagents used for the titration of molybdenum are Trilon B by Nikitina and Adrianova (*365*) and antipyrinylbis-(4-dimethylaminophenyl)-methanol by Zhivopistsev and Chelnokova (*605*).

Amperometric titrations have also been used for the determination of molybdenum. Thallium(I) nitrate as the titrant and a dropping mercury electrode were used by Yoshimura, Uno, and Noguchi (*598*). Molybdenum in ferromolybdenium was determined by Gusev and Nikolaeva (*181*) using vanadium(II) and a dropping mercury electrode in sulfuric acid as an electrolyte. A rotating platinum electrode with a potassium nitrate and hydrochloric acid mixture as a supporting electrolyte was used by Reishakhrit, Pustoshkina, and Tikhonova (*442*) for the amperometric titration of molybdenum with EDTA. Manok and Kovacs (*320*) used a dropping mercury electrode and an acetic acid–sodium acetate supporting electrolyte for the titration of molybdenum with 8-quinolinol. Suprunovich and Usatenko (*530*) used 8-mercaptoquinoline for the titration of molybdenum in steel. Bis-(4-dimethylaminophenyl)-methane was used by Papafil and Furnica (*390*). The liberation of iodine by the molybdenum-catalyzed hydrogen peroxide oxidation of iodide was followed in an amperometric cell using a rotating platinum electrode and an applied potential of 0.4 volt *vs.* a mercury iodide electrode by Sharipov and Songina (*485*).

A number of papers describing polarographic methods for the determination of molybdenum have recently appeared in the literature. Wittick and Rechnitz (*583*) studied the polarographic reduction of molybdenum in dilute hydrochloric acid and determined which species were the cause of the various waves. Sen and Chatterjee (*478*) studied the further reduction of reduced silicomolybdate and presented methods for the determination of molybdenum using just the first wave for concentrations from 5 to 5000 parts per million and the sum of the four waves for concentrations from 0.1 to 100 parts per million. A method for the determination of molybdenum based on the enhancement by tungsten of the molybdenum wave in perchlorate solution was proposed by Kolthoff and Hodara (*258*). Bikbulatova and Sinyakova (*42*) determined molybdenum in high-purity indium using a mixture of sulfuric acid and potassium nitrate as a supporting electrolyte. Violanda and Cooke (*570*) described methods for the separation and concentration of molybdenum when present as a trace impurity and also described the polarographic determination using nitrate as the supporting electrolyte. Molybdenum was determined in zirconium, titanium, and niobium alloys with sulfuric and hydrofluoric acids as a supporting electrolyte by Headridge and Hubbard (*193*). The same authors (*192*) also determined molybdenum in niobium by using citric acid as the supporting electrolyte. The use of pyrophosphate as the supporting electrolyte allowed the determination of both molybdenum and tungsten in mixtures by Pantani and Migliorini (*389*). Speranskaya and Kozlovskii (*514*) attribute the wave obtained with hydrochloric acid as the supporting electrolyte to the reduction of molybdenum(VI) to molybdenum(III) rather than to the reduction of mercury(II). Kurobe, Terada, and Tajima (*280*) applied the sulfuric–perchloric acid method to the determination of molybdenum in steel and Kawahata *et al.* (*240*) used a phosphoric–perchloric acid mixture. Sverak (*535*) used citric acid as the supporting electrolyte following extraction with α-benzoinoxime for the determination of molybdenum in steel. A citrate–phosphate–chloride electrolyte was used by Patriarche, Gerbaux, and Molle (*393*) for molybdenum in steel after separation of iron by precipitation as the hydroxide. Brainina and Roizenblat (*58*) studied the polarographic concentration of various anions including molybdate. Oscillopolarography with an *o*-diphenol such as pyrocatechol present in an ammonium nitrate–ammonium hydroxide solution was used for the determination of molybdenum by Matysik (*328*).

Spectrographic methods are also suitable for the determination of molybdenum. In his study of the behavior of 19 elements on a strong base anion exchange resin at various nitric and hydrofluoric acid concentrations, Huff (*203*) determined the elements in the column effluents by a spectrographic method. Brooks and Boswell (*68*) included molybdenum in their comparative study of cathode and anode excitation in the d.c. arc. Molybdenum was determined in titanium by Tarasevich and Moseli (*547*) who atomized the sample solution into the spark discharge. Carpenter and Lewis (*84*) included molybdenum in the list of elements which they determined in high-purity hafnium. Brooks (*65, 66*) determined impurities in niobium metal either by mixing the powdered sample with graphite or fusing it with potassium pyrosulfate before excitation. Molybdenum was determined in heat-resistant alloys by Buyanov, Ivanova, and Sukhova (*80*). Gerasimova (*159*) and Lounamaa (*301*) determined molybdenum in steel. Podobnik (*409*) used germanium as an internal standard and alumina to prevent the volatilization of uranium in the determination of molybdenum in uranium tetrafluoride. Tarasevich and Zheleznova (*548*) removed the boron by volatilization as the fluoride from solution in order to determine several impurities in purified boron. Molybdenum was determined in rocks by Spackova (*512*) who used a lithium fluoride buffer and by Ivanova (*208*); by Swaine (*537*) in coal ashes and ores and by Lachin (*283*) in rubber polymers.

In the determination of molybdenum in ferrous alloys by atomic absorption spectrophotometry, Mostyn and Cunningham (*343*) found that the addition of ammonium chloride would reduce the interference from manganese and iron but not from aluminum and nitrate combined.

A comparison of the direct-comparison and dilution x-ray fluorescence methods with the phenylhydrazine spectrophotometric method was made by Lingard and Willigman (*293*) who found that the direct-comparison method yielded lower results than the dilution or spectrophotometric methods for samples containing greater than 0.2% molybdenum and that the dilution x-ray fluorescence technique was much faster than the spectrophotometric method. Verkhovodov and Gorbatenko (*569*) used scattered radiation to compensate for variations in x-ray tube voltage and sample composition in the x-ray fluorescence method for molybdenum. Hakkila, Hurley, and Waterbury (*185*) used x-ray fluorescence for the determination of molybdenum in mixed carbides of uranium, zirconium, and molybdenum without prior chemical separation. Uranium and molybdenum in mixtures were determined by Parthey (*391*). The difference in absorption of radiations which have energies slightly less than and slightly greater than the K absorption edge energy of molybdenum was used by Khan and Abdulin (*246*) for the determination of molybdenum. X-ray absorption was used by Karev and Matyushenko (*234*) for the determination of molybdenum in beryllium.

A spark source mass spectrograph was used by Guthrie (*182*) for the determination of 26 to 50 impurity elements in erbium, cerium, and lutetium.

Yule (*600*) included molybdenum in his determination of detection limits for 118 reactor thermal neutron products. Jaskolska, Wodkiewicz, and Minczewski (*217*) combined solvent extraction separations with the neutron activation technique for the determination of trace impurities including molybdenum. Neutron activation was used by Grosse-Ruyken and Doege (*177*) for the determination of molybdenum in tungsten, by Gebauhr and Martin (*157*) for molybdenum in silicon, by Yamada (*591*) for molybdenum in soil, and by Healy and Bate (*194*) for molybdenum in hair and wool. Stary and Ruzicka (*517*) separated the elements being determined by extraction with substoichiometric amounts of reagent before determination by neutron activation. Wetherill (*577*) used isotope dilution for the determination of molybdenum in iron meterorites and Qaim and Butement (*431*) separated molybdenum from irradiated materials by the precipitation of molybdophosphate with nitron.

TUNGSTEN

From the number of papers appearing in the literature, it would seem that there is considerably less interest in the determination of tungsten than in the other elements included in this review.

Several papers will be included which describe methods of separating tungsten from other elements. Dixon and Headridge (*117*) included tungsten in a study of the anion exchange separation of several refractory metals in hydrofluoric–hydrochloric acid solution. Kawabuchi (*238*) separated tungsten from rhenium by anion exchange using ammonium thiocyanate followed by a mixture of sodium chloride and sodium hydroxide. Tungsten was separated from molybdenum, vanadium, and rhenium on an anion exchange column by Darbinyan and Danielyan (*107*) and from titanium on an anion exchange column by Shishkov and Shishkova (*495*). Several reagents for the solvent extraction for tungsten were included in a review of solvent extraction methods by Green (*175*). Nee, Chu, and Liang (*358*) studied the extraction of tungsten by *N*-phenylbenzohydroxamic acid in chloroform and listed the elements which do and do not extract under similar conditions. Titov (*553*) extracted tungsten into aniline from 1.5*M* hydrochloric acid, determined which elements were similarly extracted, and recommended ascorbic acid as a masking agent.

Several reagents have been used for the spectrophotometric determination of tungsten. Thiocyanate was used by Shcherbakov and Ivannikova (*488*) who compared titanium(III) chloride and tin(II) chloride as reducing agents. Nishida (*370*) separated tungsten by coprecipitation with metastannic acid and then determined it using thiocyanate. Akiyama and Kobayashi (*6*) used the thiocyanate method for the determination of tungsten in tantalum and tantalum oxide. Wohlmann (*584*) reduced the tungsten in a cadmium reductor in order to determine it in the presence of molybdenum by the thiocyanate method. Shcherbakov (*487*) separated tungsten from molybdenum by coprecipitation with iron(III) hydroxide prior to determination as the thiocyanate. An anion exchange separation was used by Studenskaya *et al.* (*525*) to separate tungsten from vanadium and molybdenum before determining it by the thiocyanate method. Tungsten in steel was determined using the thiocyanate method by De and Rahaman (*110*, *111*) following thenoyltrifluoroacetone extraction and tributyl phosphate extraction and by Luke (*302*) after separation of the other constituents by methyl isobutyl ketone and cupferron extractions. Peterson, Anderson, and Howcroft (*400*) applied the thiocyanate method to the determination of tungsten in ores and concentrates. Toluene-3,4-dithiol was used by Zopatti and Pollock (*611*) for the determination of tungsten in copper after removal of the copper by electrodeposition and extraction with neocuproin. The determination of free tungsten in tungsten silicide was performed by Sugawara (*528*) using toluene-3,4-dithiol following a hydrogen peroxide leach. Bowden (*55*) used toluene-3,4-dithiol in a field method for the determination of tungsten in soils and stream sediments. Kawahata, Mochizuki, and Misaki (*243*) used hydroquinone for the simultaneous determination of niobium and tungsten in steel by measuring the absorbance at two wavelengths. Sverak (*536*) separated tungsten from nickel by extraction with α-benzoinoxime and then determined it with hydroquinone after destruction of the organic material. Stolyarov (*519*) determined tungsten by measuring the absorbance of the organic phase after extraction of the tungsten-8-quinolinol complex into chloroform from an EDTA solution. The method used by Raspi and Ciantelli (*438*) was to measure the absorbance of a hydrochloric acid–acetic acid–hydrogen peroxide–methyl salicylate solution of tungsten. Ishibashi and Kohara (*205*) studied the conditions for the determination of tungsten with morin in an aqueous solution containing dimethyl sulfoxide. Poluektova and Nazarenko (*412*) compared 19 derivatives of 2,3,7-trihydroxy-6-fluorone and recommended the 9-(2′-hydroxyphenyl)- and 9-(9′-anthracenyl) derivatives for the determination of tungsten. Tungstate in the presence of molybdate was determined by Horak and Okac (*201*) using 2,3,4-trihydroxybenzenesulfonic acid. Charreton *et al.* (*91*) used tungstovanadophosphate for the determination of tungsten in the presence of vanadium and phosphorus. A catalytic method was used by Pantaler (*387*) for the determination of 0.01 to 0.1 μg of tungsten in which he followed the tungsten catalyzed oxidation of dithio-oxamide by hydrogen peroxide by measuring the absorbance at one minute intervals for ten minutes.

Kovalenko and Zakharova (*273*) used the effect of ammonium paratungstate on the angle of rotation of the plane of polarization for (+)-tartaric acid solutions for the determination of tungsten. The interferences in this method of determination were studied by Musil and Faber (*350*) who determined the maximum permissible amount of nitrate, sulfate, and fluoride. Jacobsohn and Azevedo (*210*) studied (−)-malic acid as well as (+)-tartaric acid.

Watanabe (*573*) studied the precipitation of tungsten with 8-quinolinol and found the order of adding the reagent and adjusting the pH to be important. The precipitate may be dried at 115° C and weighed or ignited to the oxide at 900° C in the gravimetric determination of tungsten with *N*-phenylbenzohydroxamic acid as described by Kaimal and Shome (*226*). Zolotukhin *et al.* (*610*) used cinchonine for the determination of tungsten without prior separation in tungsten–rhenium alloys with an error of 0.23%. Tungsten was determined in the filtrate from the precipitation of niobium in a method used by Sheskolskaya (*489*) who used tannin and β-naphthoquinoline for the precipitation. Bhat and Jain (*40*) studied the precipitation of tungsten from a solution of sodium tungstate with 2,2′-pyridoin. Gebauhr (*156*) used radiotracers to study the coprecipitation of various elements with tungstic acid in acid media and found little coprecipitation in 6*M* hydrochloric acid. Hoste, Adams, and Dams (*202*) used radioactive tungsten to study the precipitation of tungsten from homogeneous solution using a nitric acid–hydrogen peroxide media. Dams and Hoste (*105*) then applied the method to the determination of tungsten in tantalocolumbites. Duval (*127*) studied the thermal stability of a silicotungstate and recommended heating to greater than 470° C. Carey, Raby, and Banks (*83*) and Newkirk and Simons (*363*) studied the thermogravimetry of tungsten trioxide. The tungsten in alkali metal tungsten bronzes was determined by Lutz and Conroy (*307*) using the technique of volatilizing the tungsten trioxide by passing a stream of hydrochloric acid gas over the sample and weighing the remaining alkali chloride. In a combination method for the determination of tungsten and molybdenum by Nemirovskaya, Keremedzhidi, and Yashina (*360*), calcium molybdate and calcium tungstate were first precipitated, then the precipitate was dissolved, and the tungsten was precipitated from a nitric acid–hydrochloric acid solution and weighed as tungsten(VI) oxide after which the calcium was titrated with EDTA.

A titrimetric method for the determination of tungsten in alloys with chromium, tantalum, iron, and molybdenum was used by Fedorov (*138*) in which tungsten was reduced to a mixture of tungsten(III) and tungsten(IV) and then titrated potentiometrically with permanganate. Kawahata *et al.* (*239*) titrated tungsten dropwise with a solution of lead nitrate at pH 6 to 6.5 to the end point indicated by 4-(2-pyridylazo)-

resorcinol in order to determine tungsten in ferrotungsten. Konkin and Zhikhareva (*261*) precipitated lead tungstate with a measured excess of lead and then titrated the excess lead with EDTA to the xylenol orange end point in order to determine tungsten in ferrous alloys and ferrotungsten. Shivahare (*496*) titrated tungsten with silver and followed the titration potentiometrically using a silver electrode. A somewhat different approach to the titration of tungsten was used by Braun (*59*) who precipitated calcium tungstate, dissolved the precipitate, and titrated the calcium with EDTA using as a method of end point detection the increase in radioactivity of the solution caused by the dissolution of silver iodide, containing radioactive silver, by the excess EDTA.

Speranskaya and Mambeeva (*515*) studied the polarography of tungsten in hydrochloric acid solution with respect to electrode reactions and response to changing hydrochloric acid concentration. The polarographic behavior of tungsten in the presence of tartaric, citric, and oxalic acid was studied by Pantani (*388*) who found two cathodic waves in hydrochloric acid containing citric or tartaric acid and one wave in the presence of oxalic acid. Studenskaya *et al.* (*525*) separated tungsten from vanadium and molybdenum by anion exchange and then determined it polarographically with $10M$ hydrochloric acid as the supporting electrolyte. Studenskaya and Emasheva (*523*) also determined tungsten polarographically in a potassium citrate–hydrochloric acid solution after separation from other elements by anion exchange or by precipitation from hydrochloric acid solution. Pantani and Migliorini (*389*) determined tungsten and molybdenum individually when both were present using a sulfuric acid–sodium pyrophosphate supporting electrolyte by measuring the total height of the two molybdenum waves and separately the tungsten wave. The effect of tungsten on the molybdenum-catalyzed reduction wave of perchlorate was used by Kolthoff and Hodara (*258*) for the determination of tungsten by holding the molybdenum concentration constant. Brainina and Roizenblat (*58*) included tungsten along with other elements in a study of the sensitivity of the determination of anions. Square-wave polarography in $6M$ hydrochloric acid was used by Kaplan and Sorokovskaya (*233*) for the determination of tungsten in ammonium perrhenate following volatilization of the rhenium. Kawahata *et al.* (*240*) separated tungsten along with niobium from stainless steel by acid dehydration, then determined them by a.c. polarography in a hydrochloric acid solution containing ethylene glycol. Amperometric titration with thallium(I) nitrate using a tungsten-lead amalgam electrode was used by Yoshimura, Uno, and Noguchi (*598*) for the determination of tungsten. Chang, Tsao, and Shu (*90*) precipitated lead tungstate, dissolved the precipitate, and titrated the lead with EDTA to an amperometric end point in an ammonium hydroxide–ammonium chloride solution. Lead nitrate was used for the amperometric titration of tungsten using a rotating platinum electrode by Bogovina, Novak, and Maltsev (*51*) for the determination of tungsten in steel following a separation by hydrochloric acid dehydration.

Table XII. Separations

Element	Method; Reagent: Material (References)
Zirconium	Solvent extraction; Thiocyanate: Zr-Hf mixtures (*145*)
	Solvent extraction: Review (*175*)
	Reversed-phase partition chromatography; Organophosphorus compounds: Study of conditions (*377*)
	Reversed-phase partition chromatography; Tricaprylylmonomethylammonium chloride: U, Fe, Ni, Co (*85*)
	Ion exchange; Cation exchange resin, HNO_3-H_2SO_4: Study of 49 cations (*521*)
	Ion exchange; Anion exchange resin, HCl-HF: Zr, Ti, Mo, W (*229*)
	Ion exchange; Anion exchange resin, $(NH_4)_2SO_4$-H_2SO_4: Sc, Y, rare earths, Th, Zr, U (*187*)
	Ion exchange; Anion exchange resin, HCl-HF: Zr, Np, Nb (*200*)
	Ion exchange; Anion exchange resin, H_2SO_4: Study of 26 ions (*106*)
	Ion exchange; Anion exchange resin, HNO_3-HF: Study of 19 ions (*203*)
Vanadium	Solvent extraction: Review (*175*)
	Reversed-phase partition chromatography; Organophosphorus compounds: Study of conditions (*377*)
	Ion exchange; Cation exchange resin, H_2SO_4-HNO_3: Study of 49 cations (*521*)
	Ion exchange; Anion exchange resin, H_2SO_4: Study of 26 cations (*106*)
	Ion exchange; Anion exchange resin, Methanol-HNO_3: from U (*267*)
Niobium and Tantalum	Solvent extraction: Review (*175*)
	Solvent extraction; 8-Quinolinol: Study of conditions (*9*)
	Solvent extraction; Trioctylamine-$CHCl_3$: Fe, Mn, V, Al, Si, Ti (*362*)
	Solvent extraction; Tetramethylenedithiocarbamate-$CHCl_3$: Nb from Ta, Ti (162), from Ta, Ti, Zr, W, Be (*160*)
	Reversed-phase partition chromatography; organophosphorus compounds: Study of conditions (*337*)
	Ion exchange; Cation exchange resin, HNO_3-H_2SO_4: Study of 49 cations (*521*)
	Ion exchange; Anion exchange resin, HCl: Zr, Np (*200*)
	Ion exchange; Anion exchange resin, oxalic acid-HCl: Nb from Ta (*123*)
	Ion exchange; Anion exchange resin, oxalic acid-H_2SO_4: U (*173*)
Chromium	Solvent extraction: Review (*174*)
	Ion exchange; Cation exchange resin, HNO_3-H_2SO_4: Study of 49 cations (521)
	Ion exchange; Anion exchange resin, H_2SO_4: Study of 26 cations (*106*)
	Ion exchange; Anion exchange resin, HCl + organic solvent: U, Th, rare earths (*268*)
	Ion exchange; Anion exchange resin, HCl: Mn (*264*)
	Reversed-phase partition chromatography; organophosphorus compounds: Study of conditions (*377*)
Molybdenum	Solvent extraction: Review (*175*)
	Solvent extraction; Isoamyl acetate: Mo from Re (*428*)
	Solvent extraction; Dibutylhydrogen phosphate: Study of elements extracted (*251*)
	Reversed-phase partition chromatography; Organophosphorus compounds: Study of conditions (*377*)
	Ion exchange; Cation exchange resin, HNO_3-H_2SO_4: Study of 49 cations (*521*)
	Ion exchange; Anion exchange resin, H_2SO_4: Study of 26 cations (*106*)
	Ion exchange; Anion exchange resin, HCl or NaOH: Mo from W (*107*)
	Ion exchange; Anion exchange resin, HCl–ethanol: Mo from W, Ti, Zr (*229*)
	Ion exchange; Anion exchange resin, H_2O_2: Mo from Ti (*494*)
Tungsten	Solvent extraction: Review (*175*)
	Solvent extraction; *N*-Phenylbenzohydroxamic acid: Study of method (*358*)
	Solvent extraction: Aniline, Study of method (*553*)
	Ion exchange; Anion exchange resin, HCl-HF: Study of conditions (*117*)
	Ion exchange; Anion exchange resin, NaCl-NaOH: Re (*238*); Mo (*107*); Ti (*495*)

Spectrographic analysis of column effluents was used by Huff (*203*) in his study of 19 elements in a strong base ion exchange resin using mixed nitric and hydrofluoric acid solutions. Ko (*254*) determined tungsten in the aqueous phase spectrographically after extracting the matrix material, plutonium, into tri-*n*-octylamine in xylene. In order to determine tungsten in aerosols, Liplavk and Chernousova (*294*) collected the sample by passing 30 to 50 liters of air through a filter paper, ashed the paper, and mixed the ash with carbon powder before spectrographic excitation. Carpenter and Lewis (*84*) used a spectrographic method for the determination of tungsten in high-purity hafnium. Tungsten was determined in niobium metal and oxide by Brooks (*65, 66*) who used both the powder–d.c. arc technique in which he mixed the sample with an equal weight of silver chloride and the fusion–d.c. arc technique in which the sample is fused with potassium pyrosulfate. Buyanov, Ivanova, and Sukhova (*80*) used either an arc or low voltage spark for the determination of tungsten in heat-resistant alloys with a quantometer. The tungsten in high purity boron was determined by Tarasevich and Zheleznova (*548*) following removal of the boron by volatilization as the fluoride. Ivanova (*208*) studied the effect of adding polytetrafluoroethylene and copper chloride to the sample in order to increase the sensitivity in the determination of tungsten in granite-type rocks. Spackova (*512*) added lithium fluoride in order to determine tungsten in silicate and sulfide rocks. Lachin (*283*) determined tungsten in rubber making compounds.

Tungsten was determined in nickel in the range of 0.5 to 3% by Babusci (*25*) using x-ray fluorescence.

The proper sample treatment and conditions for the determination of tungsten in steel by β-ray back scattering were studied by Bunus, Popescu, and Stanciu (*74*) and Gorski and Lubecki (*172*).

Yule (*600*) included tungsten in his experimental determination of detection limits for some 118 reactor thermal neutron products. Zitnansky and Sebestian (*608*) used neutron activation following electrolytic dissolution for the determination of tungsten as combined tungsten carbide in steel. The same method was applied to chromium–nickel steel alloyed with titanium and tungsten by Opravil, Zitnansky, and Sebestian (*381*). Neutron activation was used by Gelli, Malvano, and Sacchetti (*158*) for the determination of tungsten in aluminum and Gebauhr and Martin (*157*) for the determination of tungsten in high-purity silicon.

Tungsten was determined in erbium, cerium, and lutetium metals by Guthrie (*182*) using a mass spectrographic method.

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Simultaneous Determination of Oxygen and Nitrogen in Metals by Carrier-Gas Fusion Extraction

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▶ A platinum–tin reaction medium has been applied successfully to the carrier-gas fusion determination of both the oxygen and nitrogen contents of 19 different base-metals. The composition of the reaction medium ranged from 80 (Pt):20 (Sn) at extraction temperatures in the region 1725–1950°C, to 95 (Pt):5 (Sn) for extraction temperatures greater than 1950°C. Quantitative accuracy was demonstrated by concordance of carrier-gas fusion results with values obtained by other analytical techniques such as Kjeldahl chemical, caustic fusion, isotope dilution, vacuum fusion, and hydrogen reduction; the theoretical oxygen contents of synthetic metal–oxygen standards; and values certified by the National Bureau of Standards or developed in cooperative ASTM analytical programs. A detailed description of the carrier-gas fusion, gas chromatographic analytical facility employed in this study is given. In this apparatus, a pyrolytic boron nitride thimble is employed to keep the operating blanks within tolerable limits.

OXYGEN AND NITROGEN impurities are commonly present in trace amounts in metals, occurring either as solid solutions in the interstices of the metal lattice, as oxide and nitride inclusions, or in some cases, as trapped molecular gases. The presence of oxygen and nitrogen as interstitial impurities normally results in profound effects on the physical, mechanical, and electrical properties of the host metal. For production control and metallurgical evaluation of such effects, accurate and precise analytical determinations on these impurity contents in metals and alloys are required.

The nitrogen impurity content is normally determined by classical chemical dissolution procedures (*24*, *52*), usually with acceptable accuracy and precision. In some cases, notably silicon steels and many of the recently developed corrosion-resistant alloys, the chemical technique frequently fails to provide quantitative results on the nitrogen content. Whether the low nitrogen recoveries are due to incomplete sample dissolution or to nonquantitative conversion of the nitrogen to ammonium salts has not been clearly established. In view of these uncertainties, it is appropriate to refine existing analytical techniques or develop new ones which may be generally applicable to the nitrogen determination.

The possibility of utilizing high-temperature fusion extraction techniques for determining the nitrogen content of metals along with the oxygen impurity has been previously considered by many investigators. The classical vacuum fusion (*56*) and the carrier-gas fusion (*47*, *49*) techniques both involve high-temperature fusion of the metal specimen in a graphite crucible, so that carbon reduction and thermal decomposition reactions may occur as follows:

$$M + \begin{bmatrix} M_xO_y \\ M_iN_j \\ M_mH_n \end{bmatrix} + C \rightarrow yCO + {}^1/_2 jN_2 + {}^1/_2 nH_2 + MC + M \quad (1)$$

There is now general agreement that quantitative formation and extraction of carbon monoxide from most refractory metals can be achieved if optimal environmental conditions are provided in the fusion crucible. Usually, molecular nitrogen is also evolved under the same experimental conditions. However, the validities of nitrogen results obtained in this way, especially for the refractory metals, have been clouded for many years by inadequate knowledge on the quantitative release and recovery of the molecular gas from the melt.

Table I. Summary of Prior Investigations on Fusion Extraction Determination of Nitrogen

Metal	Temperature (° C)	Fusion conditions	Reference
Ti	1800	Iron bath, vacuum fusion	*48*
Zircalloy	1900	Pt bath, Pt flux, vacuum fusion	*20*
Zr	1900	Pt bath, 15-mg samples, vacuum fusion	*10*
Zr, Steels, UN	1950	Pt bath, carrier-gas fusion	*25*
Steels	1850	Pt bath, vacuum fusion	*50*
Steels	1850	Pt bath, Pt flux, vacuum fusion	*14*
Nb	1900	Pt bath, Pt flux, vacuum fusion	*35*
Nb, Ta	2300	Pt bath, carrier-gas fusion	*16*
Mo	1950–2050	Pt bath, vacuum fusion	*42*
Steels	1900	No bath, single sample per crucible, carrier-gas fusion	*31, 33*
Steels	1900	No bath, carrier-gas fusion	*5*
Steels	1600	No bath, single sample per crucible, vacuum fusion	*17*

SIMULTANEOUS DETERMINATION OF OXYGEN AND NITROGEN BY FUSION EXTRACTION

For the quantitative determination of both nitrogen and oxygen by fusion extraction techniques, it is essential that:

1. A reaction medium is employed which accomplishes quantitative formation of carbon monoxide and quantitative dissociation of metal-nitrogen bonds.
2. The terminal or equilibrium solubility of carbon monoxide and nitrogen in the reaction medium is a negligible fraction of the total.
3. Quantitative transfer of the evolved gases to the analytical system is effected without significant bulk or dispersal gettering losses.
4. Requirements 1, 2, and 3 are achieved under tolerable blank conditions.

With reference to requirement 1, there is ample theoretical evidence in the literature (*2, 20, 48*) that even the most stable metal-nitrogen bonds should be decomposable at standard fusion extraction temperatures, especially those obtained under carrier-gas fusion conditions. There is also ample experimental evidence to support these conclusions. The summary in Table I shows some of the experimental conditions under which previous investigators have reported quantitative nitrogen recoveries. In spite of these studies, the authors of recent critical reviews on this subject have written as follows:

> "In general, vacuum fusion nitrogen values for metals other than steels amount to no more than numbers obtained incidental to oxygen determinations. . . No concentrated effort has been made to determine conditions required for quantitative recovery of nitrogen from refractory metals by vacuum fusion." (*40*)

> "Nitrogen values obtained by vacuum fusion are still, however, regarded with suspicion. This is particularly true for the reactive metals (Ti, Zr, Hf), and to a lesser extent, the refractory metals (V, Nb, Ta, Cr, Mo, W)." (*20*)

The views expressed by these authors accurately reflect the experiences of many analysts who have not found it possible to achieve quantitative nitrogen recoveries, even under the recommended environmental conditions. This suggests that one or more of the other requirements were not completely fulfilled.

With reference to requirement 2 listed above, Goward's (*20*) detailed theoretical treatment of vacuum fusion and extraction processes for the determination of nitrogen strongly suggests that the root of the problem is the difficulty of extracting nitrogen down to its equilibrium concentration in the melt. Goward's conclusions are supported by Ihida's observation (*27*) that 30 to 40 ppm of nitrogen remain unextracted from iron baths at 1850° C. According to Ihida's calculations, this residual nitrogen corresponds to an equilibrium nitrogen pressure of 7.6 mm at 1600° C, decreasing to 6.3 mm at 1900° C. Kraus' (*32*) theoretical deductions and experimentally determined mass transfer coefficients showed that the transport of dissolved gases from the interior of the melt to and through the melt-gas phase boundary indeed was the rate limiting step. Using a 20-minute extraction period as a criterion for a useful analytical method, Goward (*20*) also concluded that the transfer of nitrogen from the melt to the gas phase could be rate limiting. In harmony with these conclusions was the observation of Somiya *et al.* (*50*), that nitrogen recovery on steels was not quantitative unless the vacuum extraction time was extended considerably beyond that required for complete oxygen recovery. Because the thermodynamic environment (composition and temperature of fusion media), can influence the rate of gas transfer, it is not surprising that almost all reported instances of improved or quantitative recovery of nitrogen have involved trial and error modifications of bath compositions (*8, 14, 15, 17, 31, 33, 42, 44*), and temperatures (*14, 15, 19, 40, 43*), until maximal extraction was achieved. Thus, Peterson and Beerntsen (*44*) found that nitrogen in lanthanum could be quantitatively extracted with a nickel bath at 1600° C, while recovery with an iron bath was low even at 1850° C. Muramatsu (*42*) found that recovery of nitrogen in molybdenum was complete when a platinum bath was used, but was incomplete when nickel, nickel–tin, and platinum–nickel baths were employed. Lemm (*33*) demonstrated that quantitative recovery of nitrogen in steel was possible when a new, small graphite crucible was used for each sample extraction under a carrier-gas atmosphere. Koch and Lemm (*31*) later showed that nitrogen recovery progressively decreased when several steel samples were extracted in the same carrier-gas fusion crucible. However, recovery on a large number of alloyed and unalloyed specimens was quantitative when the crucible was changed after each extraction. Gerhardt, Kraus, and Frohberg (*17*) found that quantitative extraction of nitrogen from steels was very rapidly achieved when each sample was fluxed with a Ni-Ce-C alloy which contained approximately 1% cerium. According to Gerhardt *et al.*, the cerium caused precipitation of spheroidal graphite, which affected the viscosity of the melt considerably less than the normally precipitated flake graphite. An interesting feature of the furnace employed by Gerhardt *et al.* (*17*) was the "spinning crucible." After each sample extraction, the crucible was emptied by spinning it at a speed high enough to eject the melt by centrifugal force. The ejected melt was deposited on a graphite shield which surrounded the crucible. Fischer and Steinrücken (*15*) showed that nitrogen recovery on zirconium and titanium, and on their nitrides, was only 50% complete when a platinum reaction medium was used. They attributed the low results to an insufficient supply of carbon in the melt. Recovery was increased substantially when they employed a nickel bath, but results were still low and tended to further decrease with each successive sample extraction. This effect was attributed to the dissolution of an excessive amount of carbon in the nickel melt, which resulted in the rapid formation of graphite flakes at the melt surface. Fischer and Steinrücken (*15*) found that a composite platinum–nickel reaction medium provided the highest nitrogen recovery, although quantitativeness still was not demonstrated, as

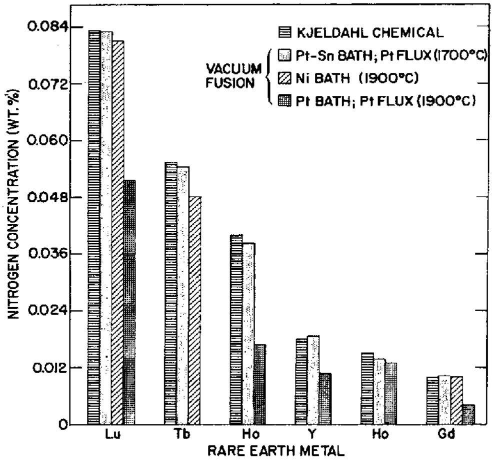


Figure 1. Studies on vacuum fusion reaction media for determination of nitrogen in lanthanide metals

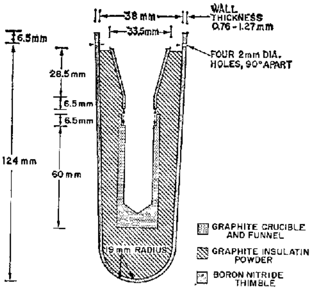


Figure 2. Carrier-gas fusion crucible assembly with the pyrolytic boron nitride thimble

recoveries amounted to 85–90%. Fassel and co-workers (*14*) showed that quantitative determinations of nitrogen in steel were not provided by the vacuum fusion, iron reaction medium, but that quantitative recovery was achieved by application of a platinum bath, platinum flux procedure.

The experiences of many investigators in the past are reflected in the graphical recovery data shown in Figure 1 for the nitrogen contents in several lanthanide elements. It is seen that a platinum–tin bath at a vacuum fusion extraction temperature of 1700° C provided essentially quantitative release of nitrogen, as evidenced by concordance of the results with Kjeldahl chemical data. In contrast, a platinum bath at 1900° C consistently yielded incomplete recovery. Thus, the presence of tin in the reaction medium contributed to more efficient extraction of the nitrogen contents of these metals at a lower fusion temperature than was required for a simple platinum bath. This is not the first observation on the beneficial effects of tin in the reaction medium. Previous investigators (*1, 3, 4, 6, 8, 11, 12, 18, 19, 21, 36, 40, 50, 55*) have shown that the presence of tin in various reaction media contributed to the more efficient and reproducible extraction of the oxygen content as well. The role played by the tin is not clear although a number of desirable functions have been attributed to it (*12*). There is considerable evidence that it is difficult to extract nitrogen down to its equilibrium level in many melts. Therefore, the increased fluidity of the platinum–tin melt (*21, 51*) and the sweeping (*1*) and stirring (*21*) action provided by the tin as part of its evaporates appear to be important functions.

A third requirement for quantitative analysis is that irreversible adsorption of the extracted gases through bulk (*34*) or dispersal gettering processes (*11, 53, 55*) be negligible. The rate of vaporization of certain metals and carbon from the fusion crucible may be the major contributing factor, and under vacuum fusion conditions appreciable amounts of metal and carbon are volatilized. Other things being equal, the amount of materials volatilized is directly related to the temperature of the crucible. The summary of data in Table I shows that extraction temperatures required for the vacuum fusion determination of nitrogen are in the 1600° C to 2000° C range. There is general agreement that gettering losses become serious beyond this temperature range. However, at these temperatures, even with the use of the platinum–tin reaction medium, we were unable to achieve quantitative recovery of nitrogen from several refractory metals. If kinetic factors play an important role in the extraction process, then an increase in operating temperature should improve extraction. The simplest expedient for operating at higher temperatures while keeping metal vaporization within tolerable limits is to increase the ambient pressure in the extracting system. This is precisely what is done in the carrier-gas fusion technique, which, as shown in Table I, has been successfully applied to several metal systems. The data to be presented later show that the combination of a platinum–tin reaction medium and extraction temperatures ranging from 1725° C to 2400° C under carrier-gas fusion conditions, has made it possible for us to determine simultaneously and quantitatively, the oxygen and nitrogen contents of 19 different metal systems.

An additional requirement for the quantitative determination of oxygen and nitrogen at the trace level is a low operating blank. High oxygen blanks are normally associated with the carrier-gas fusion technique when operating temperatures above approximately 2000° C are employed. After thorough degassing of the conventional crucible assembly, the principal source of this blank is the carbon monoxide formed by the high-temperature reaction of the graphite or carbon-black insulation with the quartz crucible–thimble, according to the reaction:

$$SiO_2 + 2C \rightarrow 2CO + Si \qquad (2)$$

This problem was solved by employing a pyrolytic boron nitride thimble in the manner previously described (*7*). A detailed drawing of the crucible–thimble assembly is shown in Figure 2. Since the publication of our earlier note, we found that extended periods at temperatures of 2200° C or above resulted in a gradual increase of the nitrogen blank when 15 to 25 grams of fusion melt were present in the crucible.

EXPERIMENTAL

The experimental approach used in this study was based on the determination of the oxygen and nitrogen contents of a large variety of base metals under various carrier-gas fusion conditions. To assess the quantitative accuracy of the results obtained, the nitrogen and oxygen values were compared, respectively, to those obtained by the Kjeldahl chemical technique and by standard vacuum fusion procedures. The nitrogen contents of the same samples were also determined by vacuum fusion procedures in order to evaluate their applicability to this determination. During the course of these studies, a consistent pattern of results developed. This pattern is illustrated in the bar graph recovery data shown in Figure 3. It is seen that platinum–tin reaction media at selected carrier-gas extraction temperatures consistently yielded maximal nitrogen recoveries which were in concordance with the Kjeldahl results, within experimental error. These intercomparative results are considered in more detail below.

Carrier-Gas and Vacuum Fusion Extraction Apparatus. A diagram of the carrier-gas extraction and analysis facility is shown in Figure 4. Pertinent experimental details and operating conditions are summarized in Table II. The helium carrier-gas which flushes through the furnace area is purified by means of a coiled column of 13× Molecular Sieve maintained at liquid nitrogen temperature. This purified helium enters the fusion-extraction zone by means of the gas manifold assembly connected to the bottom of the Vycor furnace jacket. Samples contained in the storage arms are introduced into the fusion crucible by means of a large ball valve. The impurity gases extracted from the sample are carried by the purified helium stream into the collection trap shown in Figure 5. At liquid nitrogen temperature, the 13× Molecular Sieve contained in the trap effectively removes the impurity gases from the carrier-gas stream. After completing the sample extraction and gas collection, the collection trap is switched from the furnace carrier-gas stream into the chromatograph helium stream, and the liquid nitrogen is removed from the

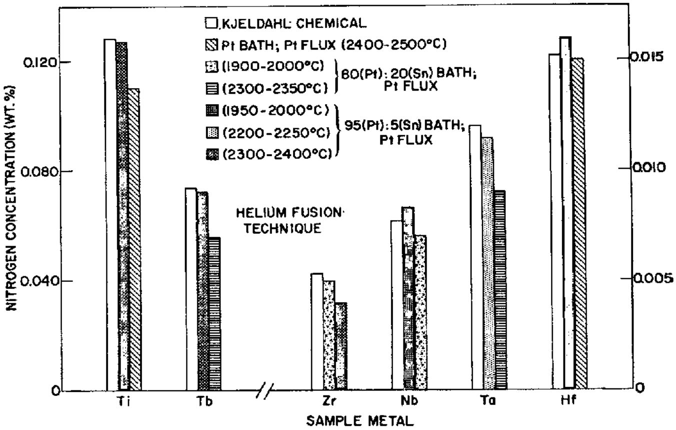
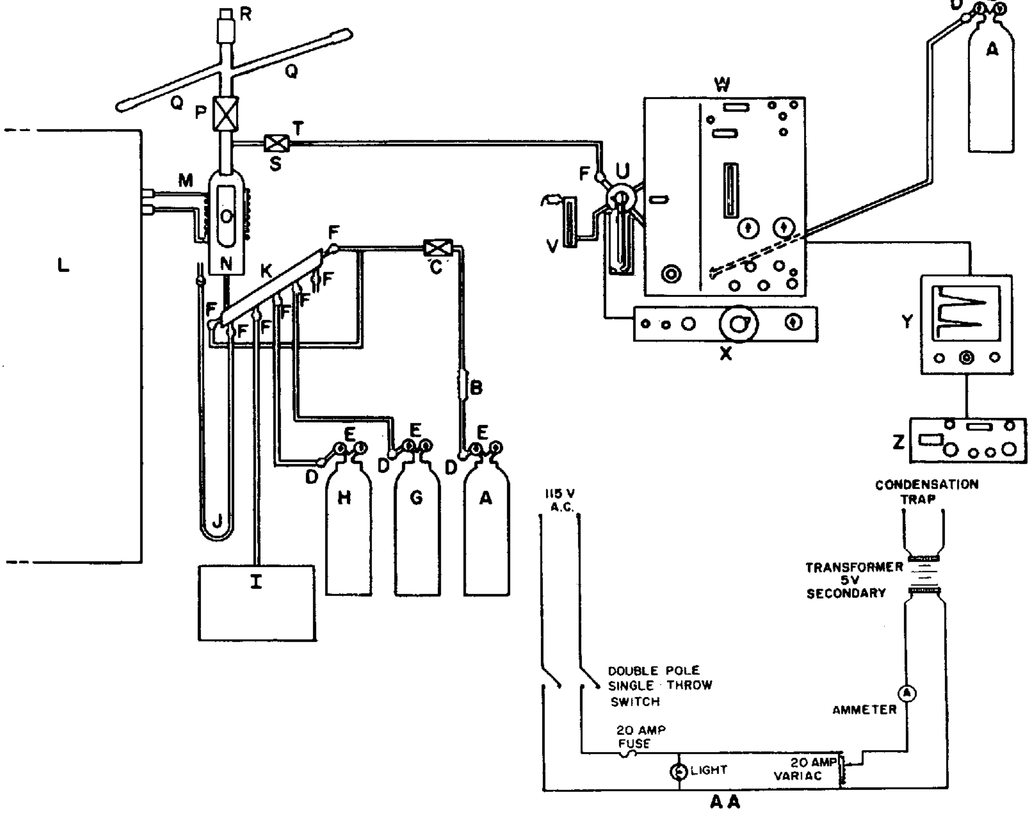


Figure 3. Studies on carrier-gas fusion reaction media for determination of nitrogen in metals



trap. The collection trap is then heated for 12–15 seconds by the circuitry detailed in Figure 4. Upon heating the trap, the collected impurities are immediately released and are flushed onto the chromatographic separation column. Just prior to elution of the nitrogen impurity from the separation column, the collection trap is switched back into the furnace carrier-gas stream. This action returns the recorder base line to zero from the deflection induced by pressure disturbances during the trap switching and heating procedures. The impurity gases are separated and detected by conventional gas chromatographic procedures. The areas under the recorded impurity peaks are automatically integrated and printed out.

Integrator response to nitrogen and carbon monoxide was calibrated directly in terms of counts μg^{-1} of impurity gas. The calibrations were achieved by introducing known amounts of nitrogen or carbon monoxide into the carrier-gas stream ahead of the furnace assembly. The calibrated volumes were measured in the gas manifold assembly. A typical calibration curve for the nitrogen impurity is shown in Figure 6. The calibration data summarized in Figure 6 were obtained at different crucible temperatures with the furnace assembly in various states of cleanliness. The location of calibration points on the 45° curve demonstrate that the various crucible temperatures and furnace conditions did not influence the calibrations in any systematic manner; the collection of the nitrogen impurity in the trap from the helium carrier stream was quantitative; and the blank corrections employed were accurate.

These calibrations are absolute and have a high degree of sensitivity under the experimental conditions used, amounting to approximately 85 counts μg^{-1} of nitrogen. The carbon monox-

◀

Figure 4. Carrier-gas extraction and analysis experimental facility

A = Helium supplies; Bureau of Mines, Grade A
B = Helium purification column
C = Hoke flow-control needle valve
D = Matheson shut-off valves
E = Matheson two-way pressure regulators
F = Hoke toggle valves
G = Calibration dry-nitrogen supply; Linde Air Products
H = Calibration carbon monoxide supply; Matheson, CP
I = Welch mechanical vacuum pump
J = Mercury manometer
K = Gas manifold
L = Induction generator
M = Work coil
N = Vycor furnace jacket
O = Crucible assembly
P = 1-inch Jamesbury ball valve; NRC Equipment Corp.
Q = Sample storage arms
R = Sighting prism and optical flat
S = ¼-inch Jamesbury ball valve; NRC Equipment Corp.
T = Glass-wool filter plug
U = Precision gas sampling valve-collection trap assembly
V = Flow meter
W = Gas chromatograph
X = Collection trap heater unit
Y = Recorder
Z = Printing integrator
AA = Electrical circuit for collection trap heater

Table II. Experimental Apparatus and Procedures for Simultaneous Determination of Oxygen and Nitrogen in Metals by the Carrier-Gas Fusion Technique

Heating facility	Lepel High Frequency Laboratories, Inc., high frequency induction generator, Model T-5N-1, with nominal power output of 5 kw and frequency of 400 ± 100 kc. Maximum power output coupling to work load achieved with maximum tank–coil setting and an 8-turn, 4-inch diameter work coil
Crucible assembly	Ultra Carbon Corp., graphite crucible C-625 and graphite funnel F-703, or equivalent, cut down to dimensions specified in Figure 2.
Furnace assembly	Design based on Guldner-Beach furnace (*22*). Crucible assembly components floated on Ultra Carbon Corp., UCP-2, −200-mesh graphite powder insulation, or equivalent, within specially fabricated "pyrolytic" boron nitride thimble (*7*) which is suspended by platinum wire hooks within air cooled Vycor jacket
Furnace carrier-gas	Purified helium, 275–300 cc min^{-1}
Helium purification	Furnace carrier-gas purified by passing it through a 12-foot coiled length of $^1/_4$-inch o.d. copper tubing packed with approximately 50 grams of 20–50 mesh 13× Molecular Sieve maintained at liquid nitrogen temperature. Sieve originally activated by heating *in vacuo* at 180–190° C for 24 hours
Crucible degassing and bath addition	Crucible temperature raised to 2450–2500° C over 1-hour period and degassed for 45 minutes. Temperature reduced to 1750° C and platinum–tin bath materials added slowly and intermittently to crucible. Crucible and bath then gradually brought to the desired operating temperature
Bath conditioning and degassing	Conditioning metal added to bath when operating temperature is reached. When operating temperature exceeds 1900° C, conditioner is added at 1900° C, after which desired temperature is gradually attained. Degassing of entire composition at operating temperature is then conducted for 30–45 minutes
Sample preparation	Careful filed and cleaned samples fluxed with 12-gauge platinum wire, platinum capsules formed from 1-mil foil, or both
Platinum flux blanks	Platinum wire: <2 ppm nitrogen; 5–8 ppm oxygen. Platinum foil: <2 ppm nitrogen; 10–15 ppm oxygen
Furnace blanks	Furnace blanks somewhat temperature dependent: 0.1–0.8 μg nitrogen min^{-1}; 0.2–0.6 μg oxygen min^{-1}.
Collection of evolved impurity gases	Achieved by passing furnace carrier-gas stream through the collection trap shown in Figure 5. The stainless steel trap is packed with approximately 2 grams of 20–50 mesh 13× Molecular Sieve which is maintained at liquid nitrogen temperature for the gas collection. Collected impurity gases released from the trap by 12- to 15-second heating period. Sieve originally activated by heating in an oven at 180–190° C for 24 hours
Gas chromatograph	Perkin-Elmer Corp., Vapor Fractometer, Model 154D, including a Precision Gas Sampling Valve, of which the collection trap is an integral part
Separation column	Perkin-Elmer Corp., synthetic zeolite column I. Column periodically reactivated *in situ* for 8–12 hours at 190° C, under a 100-cc min^{-1} flow of helium
Chromatograph carrier gas	Helium, 92 cc min^{-1}
Column and detector temperature	80° C
Detector voltage	8.0 volt
Recorder	Leeds & Northrup Co., Speedomax "H," AZAR
Recorder sensitivity	0–2 mv, full scale
Recorder response	1 sec, full scale
Recorder chart speed	$^1/_2$ inch min^{-1}
Integrator	Perkin-Elmer Corp., Printing Integrator, Model 194B.
Integrator calibration	Specified as integrator counts μg^{-1} of nitrogen or carbon monoxide, obtained by analysis of known quantities of nitrogen and carbon monoxide standard gases

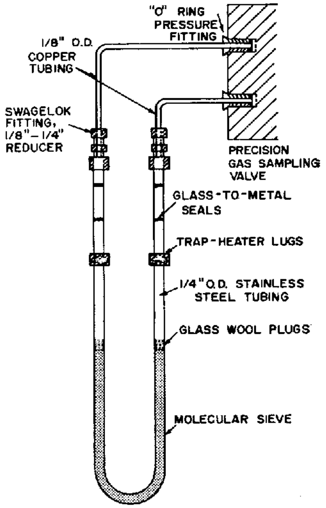


Figure 5. Gas collection trap for carrier-gas fusion analytical facility

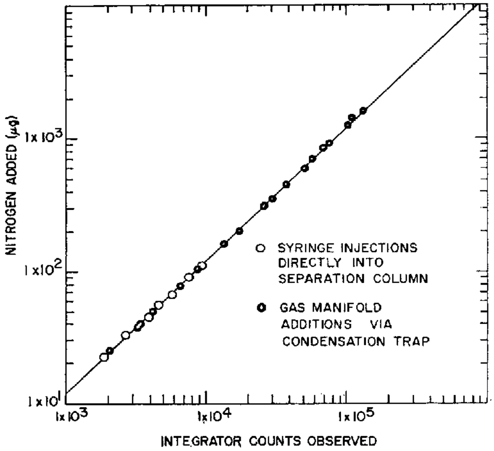


Figure 6. Calibration of integrator response to nitrogen impurity

ide calibrations are nearly coincident with the nitrogen curve because the calibrations are based on thermal conductivity changes, and the thermal conductivities of the two gases are nearly identical. Syringe injections of the calibration gases directly onto the separation column yield calibration values concordant with those obtained by the procedure described, and therefore may be used to evaluate the integrator response daily.

The results of a large set of experiments designed to delineate the platinum–tin bath compositions and extraction temperatures required for the simultaneous and quantitative extraction of both carbon monoxide and nitrogen led to the selection of the environmental conditions summarized in Table III. A higher ratio of platinum to tin was found to be desirable at crucible temperatures greater than 1950° C to reduce excessive vaporization of the tin. In addition, an 80(Pt):20(Sn) bath occasionally led to low nitrogen recoveries at extraction temperatures exceeding 2000° C.

Table III. Extraction Conditions for Simultaneous Determination of Oxygen and Nitrogen in Metals by Carrier-Gas Fusion Technique

Reaction medium composition[a,b] (wt. %)	Sample metal[c]	Fusion temp[d] (° C)	Platinum flux ratio	Maximum sample–metal concentration in final fusion melt (wt. %)
80(Pt):20(Sn)	Cu	1725–1775	3:1	10
80(Pt):20(Sn)	Cr	1775–1825	3:1	10
80(Pt):20(Sn)	Steel	1825–1875	3:1	25
80(Pt):20(Sn)	Mo	1850–1950	. . .[e]	20
80(Pt):20(Sn)	Zr	1850–1950	3:1[f]	10
95(Pt):5(Sn)	Nb	1950–2050	3:1	15
95(Pt):5(Sn)	V	2050–2150	3:1	15
95(Pt):5(Sn)	Ta	2150–2250	. . .[e]	20
95(Pt):5(Sn)	Sc, Y, Gd, Tb Dy, Ho, Er, Lu	2275–2375	3:1[g]	10
95(Pt):5(Sn)	Th	2300–2400	5:1[g]	10
95(Pt):5(Sn)	Hf	2300–2400	5:1[f]	10
95(Pt):5(Sn)	Ti	2300–2400	8:1[f]	10

[a] Not less than 15 grams total of the binary bath initially added to the crucible.
[b] Reaction medium preconditioned with 0.2–0.5 gram of sample–metal or steel.
[c] 0.05–1.0-gram samples extracted for a total period of 8 minutes.
[d] All temperature measurements obtained with optical pyrometer (Pyrometer Instrument Co., Bergenfield, N. J., Catalog No. 85) calibrated in accordance with instructions provided by the manufacturer.
[e] Platinum flux not required but normally used to help maintain more dilute sample–metal concentration in the melt.
[f] Samples encapsulated in Pt to prevent direct sample–crucible contact.
[g] Samples encapsulated in Pt to help minimize possible surface contamination by the atmosphere, and to facilitate sample penetration into the melt.

The vacuum fusion apparatus and its mode of operation were essentially the same as those previously described (*12*). The selection of environmental conditions for the oxygen and nitrogen determinations was based on previous published information or on personal experiences. These environmental conditions are summarized in Table IV.

The chemical dissolution procedures used for the nitrogen determinations were patterned after the classical (Kjeldahl) technique (*24*, *52*). The origins of other comparative data presented are appropriately identified in the table of analytical results.

RESULTS AND DISCUSSION

The analytical results shown in Table V are typical of those obtained under the experimental conditions detailed in this paper. These data represent only part of the comparative results collected during this study, but sufficient values are included to indicate the potentialities of both the carrier-gas and vacuum fusion techniques for the simultaneous determination of oxygen and nitrogen. Except for a few isolated samples, all of the data reported in Table V represent the average of two or more replicate determinations.

Overall, the carrier-gas fusion and chemical dissolution nitrogen values presented in Table V show a high degree of concordance. For many of the metal systems the vacuum fusion nitrogen values, too, show a high degree of concordance. This suggests that with further refinement, accurate nitrogen results may be achievable with this technique as well.

Although most of the intercomparative data shown in Table V are self-explanatory and require no additional comment, it is appropriate to discuss certain aspects of some of the results. With reference to the oxygen in copper determinations, we were unable to achieve the same degree of oxygen recovery as represented by the values in Table V with the vacuum fusion, iron bath (*48*) and carrier-gas fusion, dry crucible procedures (*46*). The platinum–tin bath reduced copper volatilization considerably, suggesting that significant gettering occurred for the other environmental conditions.

In the group of results for molybdenum, samples 4, 6, and 7, were milled specimens of the original rod samples 1, 3, and 5, respectively. It is evident that milling of the specimens consistently resulted in a 50–60 ppm oxygen content increase.

The determination of nitrogen in silicon steels has caused much concern in the past (*30, 43*). Recently a vacuum fusion procedure was described (*14*) which provided nitrogen results that were in analytical agreement with isotope dilution and caustic fusion values, and which appeared to be substantially more reliable for silicon steels than the chemical dissolution technique. The nitrogen data presented in Table V demonstrate that quantitative recovery of nitrogen was achieved on a variety of steel compositions, including a number of silicon steels, under both vacuum and carrier-gas fusion conditions. It is especially significant that quantitative recoveries were obtained by either furnace fusion technique, without the necessity of changing or emptying the fusion crucible after each sample extraction, which is required in the methods suggested by Gerhardt *et al.* (*17*) and of Koch and Lemm (*31*).

The vacuum fusion oxygen and nitrogen data for chromium in Table V were obtained under markedly different fusion conditions, as discussed under Remarks. At the lower temperature employed with the platinum–tin bath, it is likely that decomposition of the nitrides was incomplete. At the higher temperature employed with the simple platinum bath, nitrogen results were vastly improved, but oxygen results tended to be very erratic and unreliable, which may be attributed to uncontrollable vaporization of chromium from the crucible, with subsequent get-

Table IV. Reaction Media and Fusion Temperature Employed for Determination of Oxygen and Nitrogen in Metals by Vacuum Fusion Technique[a]

Reaction medium composition (wt. %)	Fusion temp[b] (° C)	Sample metal
80(Pt):20(Sn)	1625–1675	Cu
80(Pt):20(Sn)		Cr (O_2 only)
80(Pt):20(Sn)	1675–1725	Sc, Y, Gd, Tb, Dy, Ho, Er, Lu
Pt only	1850–1900	Steel
Pt only	1900–1950	Nb, V, Ta, Zr, Hf, Mo
Pt only	1950–2000	Th, Ti, Cr (N_2 only)
Fe only	1650–1700	Mo

[a] The apparatus and general experimental procedures were previously described in ANAL. CHEM. **38**, 421 (1966).

[b] Temperature measurements taken as described in Table III.

Table V. Analytical Results on Determination of Oxygen and Nitrogen in Metals

		Oxygen (wt. %)			Nitrogen (wt. %)					
Metal		Helium fusion	Vacuum fusion	Other results	Helium fusion	Chemical dissolution	Vacuum fusion	Other results	Remarks	
Copper	1	0.036	0.037	0.037[a]	...	...	...	...	Nitrogen content below determination level	
	2	0.046	0.045	0.047[a]	...	...	...	...		
	3	0.071	...	0.068[a]	...	...	...	...		
Molybdenum	1	0.0009	0.0007	0.0008	...	...	...	...	Vacuum fusion results on samples 2, 3, and 5 obtained with Fe bath, Fe addition procedure at ~1650° C; other vacuum fusion values obtained by Pt bath, Pt flux procedure, at ~1950° C. Average Cooperative Program values reported for samples 1 (*9*)[c] and 5 (*23*)[c]. Nitrogen comparative study not made because of high blanks encountered during lengthy chemical dissolution procedure	
	2	0.0011	0.0011	...	...	...	...	...		
	3	0.0022	0.0018	...	...	...	...	...		
	4	0.0058	0.0054	...	...	...	...	...		
	5	0.0076	0.0070	0.0069	...	...	...	...		
	6	0.0078	0.0077	...	...	...	...	...		
	7	0.012	0.013	...	...	...	...	...		
Steels	1	0.0059	0.0058	...	0.0056	0.0060	0.0055	...	Plain carbon	
	2	0.087	0.086	...	0.014	...	0.014	0.014[b]	Bessemer (NBS 129B)	
	3	0.029	0.028	...	0.0045	...	0.0039	.0046, 0.0050	3.3 Silicon (NBS 125A)	Isotope dilution and caustic fusion values from (*30, 41, 43*)[c] reported
	4	0.0017	0.0008	...	0.0051	0.0049	0.0045	.0045, 0.0050	3.4 Silicon (USS-2)	
	5	0.0019	0.0022	...	0.010	0.010	0.0092	.010, 0.010	2.6 Silicon (USS-3)	
	6	0.0066	...	...	0.0080	...	0.0076	0.0080	3.3 Silicon (1296-1)	
	7	0.0038	0.0037	...	0.010	...	0.011	0.011	3.3 Silicon (BPL-1)	
	8	0.0081	0.0082	...	0.0097	0.0097	0.0092	...	Low alloy (NBS 462)	
	9	0.028	0.027	...	0.023	0.024	0.024	0.024[b]	18 Cr:9 Ni (NBS 101D)	
	10	0.0061	0.0060	...	0.081	0.080	0.084	...	18 Cr:7 Ni:2 Mn:1 Nb	
	11	0.013	0.014	...	0.22	0.22	0.23	...	15 Cr:22 Ni:10Mn:2 Mo	
	12	0.019	0.018	...	0.12	0.13	0.12	...	21 Cr:20 Ni:19 Co:3Mo:1 Mn	
Vanadium	1	0.032	0.033	...	0.033	0.033	0.033	...	Previously demonstrated (*13*)[c] that vacuum fusion procedure provided quantitative oxygen recovery on a series of synthetic vanadium–oxygen standards	
	2	0.034	0.035	...	0.30	0.30	0.29	...		
	3	0.091	0.096	...	0.013	0.012	0.012	...		
	4	0.13	0.14	...	0.047	0.047	0.045	...		
	5	1.17	1.17	...	0.15	0.14	0.15	...		
Tantalum	1	0.0065	0.0069	...	0.0061	0.0053	0.0055	...		
	2	0.026	0.028	...	0.011	0.012	0.011	...		
	3	0.050	0.051	...	0.0023	0.0028	0.0023	...		

(*Continued on page 140R*)

Table V. Analytical Results on Determination of Oxygen and Nitrogen in Metals (*Continued*)

Metal		Oxygen (wt. %)			Nitrogen (wt. %)				Remarks
		Helium fusion	Vacuum fusion	Other results	Helium fusion	Chemical dissolution	Vacuum fusion	Other results	
Niobium	1	0.0055	0.0058	...	0.011	0.011	0.011	...	Average Cooperative Program oxygen value reported for sample 2 (*39*)[c]
	2	0.014	0.014	0.015	0.0083	0.0077	0.0070	...	
	3	0.021	0.020	...	0.080	0.080	0.080	...	
	4	0.070	0.071	...	0.033	0.032	0.031	...	
	5	0.072	0.072	...	0.042	0.044	0.045	...	
Chromium	1	0.0016	0.0016	...	...	...	...	...	Vacuum fusion nitrogen data with the Pt:Sn bath at $\sim$1650° C were consistently low; the vacuum fusion nitrogen data were obtained with the Pt bath, Pt flux procedure at $\sim$1950° C; oxygen results obtained under these latter conditions were not reproducible
	2	0.016	0.016	...	...	...	...	...	
	3	0.095	0.099	...	...	...	...	...	
	4	0.27	0.27	...	...	...	...	...	
	5	...	...	...	0.0085	0.0086	0.0074	...	
	6	...	...	...	0.026	0.028	0.025	...	
	7	...	...	...	0.093	0.093	0.094	...	
	8	...	...	...	0.23	0.23	0.22	...	
Zirconium	1	0.066	0.064	0.062	0.0009	0.0008	0.0012	...	Average Cooperative Program oxygen values reported for samples 1 (*29*)[c] and 5 (*45*)[c]
	2	0.079	0.077	...	0.0012	0.0015	0.0010	...	
	3	0.084	0.085	...	0.0010	0.0011	0.0008	...	
	4	0.095	0.095	...	0.0049	0.0053	0.0044	...	
	5	0.22	0.22	0.21	0.0048	0.0047	0.0041	...	
	6	0.41	0.42	...	0.011	0.012	0.012	...	
Thorium	1	0.023	0.021	0.020	0.0054	0.0054	0.0053	...	Theoretical oxygen contents reported for samples 1 and 2, which are synthetic thorium–oxygen standards (*12*)[c]
	2	0.12	0.12	0.12	0.0051	...	0.0048	...	
	3	0.032	0.033	...	0.011	0.012	0.009	...	
	4	0.033	0.033	...	0.15	0.14	0.14	...	
	5	0.12	0.11	...	0.016	0.016	0.013	...	
	6	0.13	0.13	...	0.0096	0.0092	0.0062	...	
	7	0.15	0.15	...	0.36	0.35	0.34	...	
Hafnium	1	0.023	0.024	...	0.0009	...	0.0006	...	Average Cooperative Program oxygen value reported for sample 4 (*28*)[c]
	2	0.028	0.034	...	0.014	0.013	0.012	...	
	3	0.031	0.037	...	0.016	0.015	0.014	...	
	4	0.032	0.032	0.028	0.0035	0.0041	0.0026	...	
	5	0.033	0.035	...	0.015	0.017	0.013	...	
Yttrium	1	0.20	0.20	0.20	0.0015	...	0.0007	...	Theoretical oxygen contents reported for samples 1 and 2, which are synthetic yttrium–oxygen standards (*12*)[c]
	2	0.48	0.48	0.48	0.0034	...	0.0008	...	
	3	0.037	0.036	...	0.0034	0.0034	0.0028	...	
	4	0.27	0.26	...	0.019	0.018	0.019	...	
	5	0.29	0.30	...	0.034	0.037	0.032	...	
Scandium	1	0.038	0.037	...	0.0023	0.0021	0.0016	...	
	2	0.070	0.071	...	0.0067	0.0078	0.0049	...	
	3	0.36	0.36	...	0.0040	0.0038	0.0043	...	
Gadolinium	1	0.081	0.081	...	0.0072	0.0063	0.0055	...	
	2	0.087	0.087	...	0.017	0.017	0.014	...	
Terbium	1	0.22	0.22	...	0.044	0.045	0.042	...	
	2	0.34	0.34	...	0.072	0.074	0.072	...	
	3	0.43	0.43	...	0.057	0.055	0.054	...	
Dysprosium	1	0.0042	0.0042	...	...	...	...	...	
	2	0.0071	0.0078	...	...	...	...	...	
	3	0.015	0.016	...	0.011	0.010	0.009	...	
	4	0.11	0.11	...	0.0033	0.0026	0.0027	...	
Holmium	1	0.17	0.17	...	0.044	0.040	0.037	...	
	2	0.23	0.21	...	0.052	0.051	0.046	...	
	3	0.24	0.24	...	0.0090	0.0090	0.0090	...	
Erbium	1	0.017	0.017	...	0.016	0.014	0.014	...	
	2	0.026	0.026	...	0.10	0.096	0.098	...	
	3	0.12	0.12	...	0.0025	0.0028	0.0024	...	
Lutetium	1	0.22	0.22	...	0.0027	0.0035	0.0031	...	
	2	0.23	0.23	...	0.084	0.083	0.083	...	
	3	0.46	0.43	...	0.0092	0.0094	0.0060	...	
	4	1.13	1.10	...	0.025	0.024	0.019	...	
Titanium	1	0.12	0.12	0.11 0.12	0.014	0.014	0.008	...	6 Al:4 V
	2	0.13	0.13	0.087 0.11	0.034	0.030	0.025	...	8 Mn
	3	0.15	0.13	0.14 0.14	0.0083	0.0093	0.0069	...	Unalloyed
	4	0.16	0.16	...	0.018	0.020	0.011	0.020	3.87 Al:4.02 Mn
	5	0.21	0.20	...	0.017	0.017	0.012	...	5.16 Al:1.3 Fe:1.4 Cr:1.4 Mo
	6	0.22	0.23	...	0.10	0.10	0.082	0.11	2.25 Cr:2.15 Fe
	7	0.23	0.22	...	0.037	0.041	0.027	0.040	3.02 Al:5.83 Mn
	8	0.30	0.31	...	0.13	0.13	0.11	0.13	4.32 Cr:1.56 Fe Average Cooperative Program oxygen values reported for samples 1–3, for both vacuum fusion (*37*)[c] and inert-gas fusion (*38*)[c] techniques; average Cooperative Program nitrogen values reported for samples 4 and 6–8 (*54*).[c]

[a] Hydrogen reduction values. [b] NBS certified values. [c] Numbers in parentheses refer to bibliography.

tering of carbon monoxide. Even at the higher fusion temperature, the vacuum fusion nitrogen results tended to be slightly lower than the chemical values.

For the group of metals zirconium, thorium, hafnium, yttrium, and scandium, and the lanthanide elements, there is excellent agreement among the helium fusion and chemical nitrogen results, as well as helium fusion and vacuum fusion oxygen values. The oxygen data confirm previous observations on the platinum–tin bath, vacuum fusion reaction medium (*12*). Although the same reaction medium consistently provided greater nitrogen recoveries from the rare earth metals than either simple platinum or nickel baths, the vacuum fusion nitrogen data in Table V are not always quantitative. There are only several instances of serious discrepancies, but there are a sufficient number of low results to indicate that a marginal situation still prevails for the vacuum fusion technique.

In the case of hafnium, agreement among the oxygen results was not demonstrated to the degree desired. In our experience, the fourth hafnium sample shown in Table V could not be successfully etched with the HF-HNO_3-H_2O solution recommended by the ASTM Task Force. Therefore no sample surface preparations were employed, which may account for the discrepancies noted in the oxygen results reported in Table V for this specimen. Apparent migration of the oxygen impurities away from the ends of arc-melted hafnium samples 2 and 3 is believed to account for the deviations noted between the vacuum fusion and helium fusion results on these two specimens.

Considering the usual inhomogeneities in the oxygen impurity distribution which is typical of titanium, the comparative oxygen results for this metal and its alloys show good analytical agreement. The one exception is sample 2 (8% Mn alloy), which normally would be expected to be troublesome, especially under a vacuum extraction environment. The effect of vaporization of manganese from the fusion crucible, with subsequent gettering of carbon monoxide evolved from the sample, immediately becomes apparent in the results reported by the Cooperative Task Forces (*26*, *37*, *38*). This problem appears to have been partially resolved in the second Cooperative Program for both the vacuum fusion (*37*) and the inert-gas fusion (*38*) techniques.

The quantitative extraction of nitrogen from titanium has been a serious problem for the gases-in-metals analyst. All literature reports on this determination appear to involve vacuum fusion procedures which invariably provide incomplete recoveries. The vacuum fusion nitrogen data summarized for titanium in Table V, substantiates this general experience of others. This propensity for vacuum fusion results to be low may be due to a combination of the high thermal stability of titanium nitride and the general restrictions imposed on the fusion temperatures. Significantly, the helium fusion nitrogen results on titanium metal and its alloys appear to be essentially quantitative when compared with the corresponding chemical values. More often than not, helium fusion results are slightly less than the chemical values, but never significantly so. Further indication of the accuracy of the carrier-gas fusion nitrogen determinations is afforded by the Cooperative Program chemical results (*54*), shown for four of the titanium alloys in Table V. Undoubtedly the high fusion temperature employed in the carrier-gas fusion procedure was at least partially responsible for this observed improvement in the nitrogen recovery.

Table VI. Precision Data on Simultaneous Determination of Oxygen and Nitrogen in Metals by Carrier-Gas Fusion Technique

	Oxygen			Nitrogen		
Metal	Wt. %	Std. dev.[a]	Rel. std. dev.[a]	Wt. %	Std. dev.[a]	Rel. std. dev.[a]
Dysprosium	0.0147	±0.00103	±7.1	0.0108	±0.00093	±8.6
Thorium	0.0256	±0.00138	±5.4	0.0632	±0.00349	±5.5
Hafnium	0.0300	±0.00264	±8.8	0.0156	±0.00145	±9.3
Tantalum	0.0497	±0.00198	±4.0	0.0023	±0.00018	±7.8
Zirconium	0.0842	±0.00277	±3.3	0.0010	±0.00015	±15.0

[a] "Guide for Measures of Precision and Accuracy," ANAL. CHEM. **37,** 1814 (1965).

Precision data on the simultaneous determination of oxygen and nitrogen by the carrier-gas fusion technique are presented for several base metals in Table VI. The precision data are based on ten determinations for each specimen. These data reflect not only the random errors in the determinations, but specimen inhomogeneities as well.

The precision data and the comparison analytical data in Table V amply document the general applicability of the carrier-gas fusion technique to the simultaneous, quantitative determination of oxygen and nitrogen contents in metals. The same applies to the vacuum fusion technique for some of the metals. In those instances where deviations are evident for nitrogen, the results are still encouraging. As indicated previously, the vacuum fusion environmental conditions were selected from previous published work and from our own experiences. Other than our previous work on the platinum–tin reaction media, no systematic effort was made to optimize the experimental conditions. With further refinements in the environmental and operating conditions, it may be possible to extend the scope of the vacuum fusion technique.

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Pesticide Residues

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Research in the field of methodology for residue analysis has received much attention during the two-year review period from November 1964 through October 1966. In preparing this review, no attempt has been made to include all articles dealing with residue analyses but, rather, the authors have tried to select those which, in their opinion, will be most useful to the worker in this field.

Much of the literature refers to the "multidetection" methods in which many chemicals can be determined during one analysis. The possible inclusion of so many compounds in one analysis is a tremendous step forward but it does increase the problem of interpreting the responses properly so that one has adequate assurance of the identity of the compounds reported. This problem is basically no different from interpreting the response of the older methods such as a color reaction of the "wet chemical" or "specific" methods; for example the "specific" colorimetric method for parathion also responds to paraoxon, methyl parathion, EPN, Chlorthion, etc. The GLC methods generally give separate response for each of these; thus the possible misinterpretation is more obvious. This problem has been given a great deal of study and considerable progress has been made as indicated in numerous places throughout the review.

Closely associated with the field of methodology, particularly with the multidetection methods, is that of the "nature of the terminal residues." A pesticide chemical may be altered by its environment after application to yield terminal residues sufficiently close to the parent chemical to appear as a response to the detection system of a multidetection method. Developments in this field should be closely watched and taken into consideration as aids in the interpretation of responses in many of the methods now available.

There is no single uniform system of nomenclature used in the literature for pesticide chemicals, so for many pesticides there is no single generally recognized name. Both the coined patent names and the common names vary with the country and worker; thus there may be a number of names to describe the same compound. On the other hand, some names have recognition by all workers in the field. Because of these problems and because the authors believe that the use of chemical nomenclature exclusively would make the review difficult to follow, we have arbitrarily adopted a mixed system. In Frear's "Pesticide Index" (*111*), probably the most comprehensive listing of pesticides, common names, trade and chemical names are listed and cross-indexed. We have tried to use the name which we believe will be most meaningful to the reader and, where possible, to use a name which may be found in Frear's Index. Common names which appear in the United States Food and Drug Administration tolerance regulations have been used where possible. Common names of other compounds have been used whenever they exist and are considered to be well known. For those compounds best known by a single trade name we have used the trade name in capitalized form. In those instances where the common or trade name may be confusing or is not well known, we have tried to include the chemical name as well. Thus the reader should, with the help of Frear's Index, be able to identify almost every compound referred to in this paper.

The literature on pesticide residues has maintained its rapid growth in the two years since the previous biannual review (*75*). Gunther (*133*) has continued his excellent series of "Residue Reviews" and 15 volumes have now been published. These volumes contain articles on many aspects of pesticide residues written by experts in the specific fields. Volume 1 of the Food and Drug Administration's "Pesticide Analytical Manual" (*19*), probably the most widely used manual of methods for pesticide residue analysis, has undergone continuous revision and updating. The methods in this volume have been studied in FDA laboratories and are known to be useful combinations of extraction, cleanup, and determinative steps which yield quite satisfactory qualitative and quantitative results for many compounds.

The U. S. Public Health Service has published a 2-volume "Guide to the Analysis of Pesticide Residues" (*55*) which includes methods for the analysis of water and soil as well as foods. However, this is primarily a compilation of methods recommended by a variety of laboratories and which have not necessarily been used or tested by the U. S. Public Health Service.

Several new periodicals of interest have appeared in the period covered by this review. The "Bulletin of Environmental Contamination and Toxicology" (*53*) is a bimonthly journal designed to provide a rapid publication in fields including pesticide residue methodology. The first issue was dated January-February 1966.

The U. S. Department of Agriculture began the publication of "Pesticides Documentation Bulletin" (*243*) on March 19, 1965. This biweekly is a ". . . computer produced permuted title index in three parts: Keyword Index, Bibliography, and Author Index." This is excellent as a general reference for those interested in many aspects of pesticides but is of little value to the residue analytical chemist as a specific reference because only those words which appear in the title of an article will appear as entries. There is no grouping of subjects such as "methods of analysis" or "chemical methods of analysis" unless those words appear in the titles of the original article.

More recent and potentially most valuable to the pesticide residue chemist is the "Health Aspects of Pesticides—Literature Bulletin" (*146*). This is an experimental monthly publication of the Office of Pesticides of the U. S. Public Health Service. The first issue, dated September 1966, contained 117 abstracts including 26 of recent articles dealing with analysis. In addition to author and subject indexes, the contents are also divided into sections dealing with pollution, toxicity and toxicological factors, analysis, etc.

This two-year period has also seen the beginnings of attempts to automate pesticide residue analysis. Gunther *et al.* (*136*) designed a system for the determination of total chlorine. They combined an automatic combustion apparatus with a continuous flow chloride ion detector. It was capable of handling as much as 2-gram equivalents of plant extractives and had a useful range of 0.01 to 500 ppm. The burning cycle of 7 minutes was automatic and the chloride ion measured and recorded. Ott and Gunther (*233*) reported an automated colorimetric phosphorus determination which had a sensitivity well below 0.1 μg phosphorus per milliliter of final solution. Samples were cleaned up and burned in a Schöniger flask and the solution was transferred to an *Auto*Analyzer which carried out the analysis and recorded the result. Several procedures were reported for the automatic determination of cholinesterase inhibition using the *Auto*Analyzer. Voss (*283*) described a procedure in which acetylthiocholine was used as the substrate. The liberated thiocholine acted on 5,5-dithiobis-2-nitrobenzoic acid producing a color which was measured at 420 mμ. Ott and Gunther (*231*, *232*) used acetylcholine as the substrate and measured the transmittance at 555 mμ as affected by change in color of phenol red.

All of the above procedures required prior extraction and cleanup before the sample solution could be placed in the *Auto*Analyzer. The first fully automated analysis was reported by Gunther and Ott (*137*) for the determination of biphenyl in citrus fruit rind. The sample was automatically homogenized in water and the biphenyl steam distilled. Oils and waxes were removed with H_2SO_4 and a cyclohexane solution of the biphenyl was passed through the cell of a recording UV spectrophotometer with readings taken at 246 mμ and recorded. The useful range was said to be from 1 to 150 ppm on a whole fruit basis with a reproducibility of about 3%.

GAS CHROMATOGRAPHY

Another area which received considerable attention was detectors for gas chromatography. As the need for detectors capable of selective specificity for compounds containing halogens, phosphorus, sulfur, and nitrogen became greater, several new approaches were taken. McCormack, Tong, and Cooke (*204*) developed a detector based on selective monitoring of the emission spectra of the eluted organic compound. Using argon as the carrier gas, the spectra are excited in the plasma of a 2450-Mc electrodeless discharge. By measuring the intensity of selected atomic lines and molecular bands, the system can be made quantitative and highly specific for the halogens, phosphorus, and sulfur. Bache and Lisk (*13*) used this emission spectrometer detector to analyze a number of foods for such organophosphorus pesticides as diazinon, dimethoate, disulfoton (Di-Syston), ethion, parathion, and ronnel by measuring the intensity of the 2535.65 Å. line. Recoveries of 72–115% were obtained at levels ranging from 0.03 to 0.60 ppm.

Brody and Chaney (*50*) developed a flame photometric detector for determining sulfur- and phosphorus-containing compounds. The flame emission spectra were generated in a hydrogen–air flame and narrow bandpass interference filters used for isolation of the phosphorus and sulfur emission at 526 mμ and 394 mμ, respectively. The sensitivity was said to be 0.25 ng for malathion or parathion and in the submicrogram range for sulfur-containing compounds.

Coulson (*77*) introduced an electrolytic conductivity detector for gas chromatography. The effluent from the GLC column was passed through a combustion tube where the compounds were oxidized to CO_2, SO_2–SO_3 and HCl. The gases were passed into a stream of deionized water and into an electrolytic conductivity cell where the conductivity was measured by a simple Wheatstone bridge. The detector was 10,000 times as sensitive for halogen or sulfur compounds as for carbon or nitrogen.

Coulson (*78*) later modified this system to determine nitrogen-containing compounds. The effluent from the GLC, instead of being oxidized, was reduced with hydrogen in the presence of a catalyst to change any nitrogen to NH_3. Any acids formed were removed with $Sr(OH)_2$ and the NH_3 was passed through to the electrolytic conductivity detector. The apparatus was used for the determination of such nitrogen-containing compounds as simazine, parathion, amitrole, etc.

Martin (*198*) reported a method for the determination of nitrogen-containing compounds which also reduced the effluent from the GLC column with hydrogen to produce NH_3. The NH_3 was then passed into a titration cell where it was automatically titrated with coulometrically generated hydrogen ion. However, with some compounds, some of the nitrogen atoms were converted to elemental nitrogen which would not be converted to NH_3. Guthion, for example, gave only 39% recovery. Although the method was developed for petroleum industry use, it was said to be applicable to pesticide analysis.

A number of papers were published

on the thermionic detector, all of which gave strong support to the validity of the detector. Beckman and Gauer (*26*) reviewed the literature and development of the sodium thermionic detector. They described the construction and operation of a detector based on Giuffrida's design. Hartmann (*145*) reported a thermionic detector for phosphorus in which cesium bromide was used as the alkali metal source. The cesium bromide plus a suitable filler was pressed under high pressure to form a ceramic-like pellet which was then shaped to serve as the tip of the burner. Coahran (*71*) described a modified detector in which a ceramic tube filled with granular anhydrous Na_2SO_4 was placed around the jet.

Giuffrida and Ives (*123*) used dual detectors in an investigation of cleanup procedures for organophosphorus pesticide residues. The effluent from the GLC column was passed through a stream splitter and into two detectors. The response of a regular flame ionization detector was indicative of the amount of plant extractives present and thus of the cleanup efficiency. The response of the thermionic detector showed the amount of pesticides recovered.

Giuffrida, Ives, and Bostwick (*124*) described and explained the operating parameters for electron capture and the thermionic detectors. Specific details were given on how to adjust each detector and GLC system for most suitable operation for residue analyses. This paper should be required reading for residue analysts using gas chromatography.

Karmen (*173*) described a stacked flame ionization detector for phosphorus and chlorine. He indicated that the detector worked because phosphorus and halides increased the vapor pressure of the alkali metal and thus made more of it available for ionization. Abel, Lanneau, and Stevens (*5*) reported a modified stacked flame detector, claiming a controlled specificity for phosphates and halides in the order of 100,000–200,000 to 1 over other organic species.

Burchfield *et al.* (*54*) discussed various types of GLC detectors, pointing out the advantages of each. Burchfield and Wheeler (*57*) described the use of the microcoulometric detector in both the oxidative and reductive modes. Burchfield *et al.* (*56*) also reported the use of the microcoulometric detector for the determination of phosphorus, sulfur, and chlorine. The effluent from the GLC was carried through a reducing oven with H_2, forming PH_3, H_2S, and HCl. All three products were measured by a microcoulometric titration cell with silver electrodes. By inserting subtraction columns before the cell, HCl (by silica gel) or both H_2S and HCl (by Al_2O_3) could be removed, thus giving the system a high specificity.

The electron capture detector continues in wide use but only a few papers suggested modifications. Yauger *et al.* (*287*) reported the use of Ni^{63} as the radioactivity source, the big advantage being that such a detector can be operated at temperatures up to 300° C. Abbott and de Faubert Maunder (*3*) described a simple electron capture detector that could be constructed from a standard 75-ohm co-axial cable plug and a strip of tritiated copper foil at a total cost of material of less than $10.

Gas chromatography has become so accepted in pesticide residue analysis that its use in procedures is now taken for granted much like the analytical balance or a spectrophotometer. However, several papers have appeared which treat gas chromatography as a general topic. Gudzinowicz (*125*) compiled a vast amount of data on the use of electron capture gas chromatography in pesticide residue analysis. He listed the R_f and sensitivities for a large number of the pesticides on a variety of columns. Burke and Holswade (*58*) tested 17 liquid phases in the search for a GLC column which would elute the common pesticides in a different order than the widely used DC-200 column. They recommended a column prepared by mixing equal portions of Gas Chrom Q coated with 15% QF-1 and Gas Chrom Q coated with 10% DC-200. They listed retention times and response data on the column for over 85 pesticide chemicals using both the electron capture and the microcoulometric detector.

Berck (*29*) listed retention times, both absolute and relative to *n*-pentane, for 34 fumigants on a column packed with 10% SE-30 on Diatoport-S. Kanazawa (*171*) evaluated and compared columns with two liquid phases, 5% Dow Silicone 11 and 2% polyethylene glycol, for the separation of chlorinated and phosphorus pesticides and herbicides. Linear ranges, sensitivities, and separation efficiencies are reported. Gaul (*114*) compared five methods of measuring GLC peaks and discussed the problems with toxaphene, chlordane, and BHC. She suggested ways of measuring the peak areas when the pesticides were separate and in mixture. It was also pointed out that in determining BHC the analyst should bear in mind that the electron capture response to the β-isomer is about 50% of the response to the other isomers.

Giuffrida (*121*) described a GLC system for the collection of fractions for infrared analysis. The fractions were collected individually directly on KBr and then pressed into micro disks. About 10 mg of KBr was used and good spectra were obtained with as little as 3 μg of pesticide.

CHLORINATED PESTICIDES

General Procedures. Chlorinated pesticides continue to be the most widely used group and it is natural that methodology for these compounds received a great deal of attention. Beynon and Elgar (*36*) prepared an excellent review of work published up to May 1965. They list 324 references and cover all aspects of residue analysis from the collection and storage of samples through extraction and cleanup to the numerous means of quantitation and identification.

Mumma and coworkers (*218*) investigated the effectiveness of a commonly used extraction procedure in removing pesticide residues which had been picked up by growing crops. Using crops grown in soil containing dieldrin, they found that the widely used hexane–isopropanol (2:1) extraction procedure removed only about 64% of the dieldrin present. When this was followed by a 12-hourextraction with a 1:1 mixture of chloroform and methanol complete extraction of the dieldrin was obtained.

In the past two years, several collaborative studies were made of widely used analytical procedures. Johnson (*167*) reported on a study of the Mills' procedure involving the determination of heptachlor epoxide and dieldrin in evaporated milk and in butterfat. The results for 20 laboratories showed a standard deviation of ±0.039 ppm for heptachlor epoxide at the 0.29-ppm level and a standard deviation of ±0.052 ppm for dieldrin at the 0.26-ppm level. Several collaborative studies of the Mills, Onley, Gaither procedure were also reported. Krause (*187*) studied the recovery of aldrin, DDE, and methoxychlor from potatoes. Gaul (*115*) investigated the recovery of lindane, heptachlor, and TDE from endive and cauliflower, and Davidson (*87*) reported on the determination of BHC, *p,p'*-DDT and endrin in apricots and strawberries. Each study demonstrated the validity of the procedure.

A large number of articles describe general procedures for the determination of chlorinated pesticide residues. Many of these are modifications of previously reported methods. Gunther and Barkley (*134*) modified a microcoulometeric gas chromatograph so that, when desired, the GLC column could be bypassed with the sample going directly to the combustion furnace. This permitted easy determination of "total chlorides." Advantages of the arrangement include a more accurate measurement of toxaphene since the entire residue registered as one peak.

Robertson and Tyo (*246*) determined chlorinated pesticides in oysters using a continuous perforated basket centrifuge for extraction of sample with acetonitrile. After partitioning of the residues into petroleum ether, the determination was made by electron capture GLC. Recoveries for heptachlor, heptachlor epoxide, DDE, and DDT ranged from 97 to 115% at the 0.16-ppm level. Kadis and Jonasson (*170*) used a modification of the method of Langlois *et al.* [*Milk and Food Technol.* **27,** 202 (1964)] to determine chlorinated pesticides in blood. The sample was ground with Florisil, transferred to a Florisil column, and eluted with 30% methylene chloride in petroleum ether. After evaporation and solution in hexane, analysis was by electron capture GLC. Jain and coworkers (*161*) used a simplified procedure to determine 23 pesticides including chlorinated, organophosphorus, and a nitro compound in blood. The sample was extracted with an acetone-ether mixture (1:1), evaporated, taken up in hexane, and injected into an electron capture GLC. There was no interference from the blood but the sensitivity was limited by the size of sample that could be chromatographed (equivalent to 1 mg blood).

Radomski and Fiserova-Bergerova (*245*) described the determination of chlorinated pesticides in tissues using electron capture GLC. They blended the sample with petroleum ether, added anhydrous Na_2SO_4, made to volume with petroleum ether, and injected an aliquot into the GLC without any cleanup. Sensitivities were reported in the range from 0.001 to 0.06 ppm. Hamence and coworkers (*142*) analyzed animal tissue by extracting with acetone, partitioning the residues into petroleum ether, extracting with acetonitrile, and again partitioning into petroleum ether. Final cleanup was on an alumina column. Determination was by electron capture GLC. To confirm identity and separate compounds with similar retention times, aliquots were reacted with HBr, alcoholic KOH, and chlorine, and gas chromatography was repeated. Data are given for 12 compounds.

Stanley and LeFavoure (*263*) used a perchloric–acetic acid mixture to digest samples of animal tissues. The fat and pesticides were extracted with *n*-hexane and cleaned up on a sulfuric acid–Celite column before determination by electron capture GLC. Aldrin, dieldrin, and endrin are destroyed by the procedure. Parker *et al.* (*238*) combined portions of previously reported methods for the determination of chlorinated pesticide residues in animal and human tissues. Frozen samples were blended with Dry Ice to a powder and extracted with hexane. Acetonitrile extraction and a column containing Florisil, Celite, attapulgus clay, and charcoal were used for cleanup before determination by electron capture GLC. Onley and Bertuzzi (*229*) reported a rapid procedure for the analysis of fish, meat, and fat by electron capture GLC. The method combined the use of a mixture of acetone, methyl Cellosolve, and formamide to extract the pesticide residues with the use of calcium stearate to coagulate and hold fatty constituents. Recoveries ranged from 76–108% at levels of 0.002–1.0 ppm. Kotula and Moats (*183*) used TLC to analyze eggs or poultry fat samples in less than 2 hours. Extraction was with ethyl ether with cleanup on a carbon–Celite 545 column. As an alternative, fat could be dissolved in petroleum ether and cleaned up on a Florisil column. In each case, suction was used to speed up the elution from the column. Eight chlorinated compounds were determined with a sensitivity of about 0.1 ppm. Sawyer (*254*) used acetone to extract chlorinated pesticides from eggs. After partitioning into petroleum ether, the residues were cleaned up on a Florisil column for determination by microcoulometric or electron capture GLC. In addition to being fast, it was claimed that this procedure eliminated interferences sometimes found in other procedures. Cummings and coworkers (*83*) combined features of the method of Stemp *et al.* [*Poultry Sci.* **43,** 273 (1964)] with those of Mills *et al.* [*JAOAC* **46,** 186 (1963)] for the analysis of eggs. The sample was ground with Florisil and anhydrous Na_2SO_4 and the mixture transferred to the top of a Florisil column. The pesticides were eluted in two fractions and concentrated for analysis by electron capture GLC. The sensitivity was reported as 0.001 ppm, and recoveries for lindane, heptachlor epoxide, DDT, dieldrin, and endrin ranged from 78 to 109%.

Moats (*214*) used TLC to determine chlorinated pesticides in dairy products with a sensitivity of about 0.125 ppm on a fat basis. Stemp and Liska (*265*) reported a simplified and shortened procedure for the analysis of milk. A 10-ml sample of milk was mixed with deactivated Florisil, slurried with 20% CH_2Cl_2 in petroleum ether, and decanted through a column of deactivated Florisil. The eluate was evaporated; the residue was taken up in hexane and injected into an electron capture GLC. Recoveries were over 90% at levels of 0.1 to 10 ppm whole milk basis. It was stated that 40–50 samples could be cleaned up by one technician in a day.

Giuffrida *et al.* (*122*) described a procedure for milk, fats, and oils. Milk was extracted with acetone and the residues were partitioned into petroleum ether. Fats and oils were dissolved in petroleum ether. The samples were transferred to a column of deactivated Florisil, and after removal of solvent pesticides were eluted with acetonitrile containing 10% H_2O. After partitioning into petroleum ether, the extracts were further cleaned up on an activated Florisil column and determined by electron capture GLC. Tolbert (*276*) used a column of sand, magnesium oxide, and Celite 545 to replace the Florisil column in the analysis of oils by electron capture GLC. Saha (*249*) determined aldrin, heptachlor, endrin, and dieldrin in wheat using electron capture GLC. The ground samples were extracted with acetonitrile in a Soxhlet; residues were partitioned into petroleum ether and cleaned up on a magnesia–Celite column.

Several procedures have been reported for the determination of chlorinated pesticide residues in water. Lamar and coworkers (*190*) extracted large (up to 4 liters) samples of water with hexane and used electron capture GLC for the determinative step. Smith and Eichelberger (*260*) described a thin layer chromatographic cleanup of the carbon chloroform extract (of water) resulting in a solution suitable for electron capture GLC. Lerenard and Simon (*194*) used an automatic liquid–liquid extractor which they found capable of extracting 80–90% of lindane and dieldrin from water at concentrations of 1 ppb. Sanderson and Ceresia (*253*) reported on a continuous liquid–liquid extraction apparatus. With a sample flow rate of 1 liter/hour, recoveries of about 90% at the 1-ppb level were obtained. Teasley and Cox (*271*) compared extraction procedures for removing endrin and DDT from soils. They reported that the Immerex extraction method was the best. The procedure involved a 16-hour extraction with *n*-hexane–acetone (9 + 1) in an Immerex tester, an apparatus designed for the analysis of bituminous paving mixture which uses an extraction basket for the sample container.

Samuel (*252*) reported a screening procedure for chlorinated and thiophosphorus pesticides in dairy products, fruits, vegetables, and animal tissue. After sample extraction, a combination of 1 or more of 3 cleanup procedures prepared the sample for final analysis by electron capture or microcoulometric GLC. Recoveries of 75–100% were reported at levels of 0.05–2 ppm. Water soluble organothiophosphorus compounds do not come through the procedure.

Considerable use has been made of thin layer chromatography in the analysis for chlorinated pesticides. Matherne and Bathalter (*200*) described a cleanup procedure making use of 8- × 8-inch plates with channels 10 mm wide by 2 mm deep which were filled with Al_2O_3–G coating. Sample extracts were spotted on individual channels and the plates developed twice with two different solvents. This separated the pesticide residues from the plant extracts and after elution from the scraped off adsorbent, the residues were in suitable form for electron capture GLC.

Kovacs (*186*) used $3^1/_2 \times 4$-inch microplates for TLC and reported that as many as 26 chlorinated pesticides could be resolved in 5–10 minutes and identified. The lower limit of detection for many of the commonly used chlorinated pesticides was 0.005 μg. Crabtree (*79*) used microscope slides coated with Al_2O_3 and developed in hexane in $3^1/_2$ minutes for rapid confirmation of identity. Beckman and Winterlin (*27*) described what they called "thin-strip thin layer chromatography." They used a tool to scrape coated 8×8-inch plates in such a manner that individual TLC strips or channels 4 mm wide were formed. As many as 20 channels could be used on one plate. The advantages claimed were that, since the spots could not spread, sensitivity was increased and that it was easier to remove separated spots for GLC and IR. Engst *et al.* (*99*) used silica acid gel plates and reported the detection of 6 chlorinated pesticides with a sensitivity of 0.05 μg. Abbott and coworkers (*4*) studied the effect of temperature on R_f values. R_f values for 16 chlorinated pesticides in 16 solvent/adsorbent systems were given. Ballschmiter and Toelg (*16*) investigated fluorescence indicators for TLC. Twenty-four substances were studied. Fluorescence or quenching of spots at levels of 0.02–5 μg were noted with six reagents. Adamovic (*7*), investigating spray reagents for TLC, reported that the chlorinated pesticides under ultraviolet light reacted with aromatic amines to form characteristically colored spots even without zinc chloride or iodine. A total of 18 aromatic amines were tested with the 6 most promising showing sensitivities down to 0.5 μg.

There have been several papers on chlorinated pesticides which are of general interest to the residue chemist. Gunther, Hylin, and Spenger (*135*) investigated the nature of the organic chlorine interferences in the total halogen methods for organic chlorine pesticides. Using Cl^{36} tracer, they have tentatively identified the interferences as quaternary chloride salts of lecithins. Burke *et al.* (*59*) studied the losses of pesticides in various methods of concentrating solutions down to volumes of 0.5 ml or less. They found that large losses occurred when the solutions were evaporated by a stream of air. Losses increased as the residual volume approached dryness and the percentage losses were greater when smaller amounts of pesticides were present. They found that by using a micro Snyder column, solutions could be rapidly concentrated to 0.1–0.3 ml on the steam bath without loss of pesticide. Moats and Kotula (*215*) speeded up the elution from cleanup columns by using suction. They reported that elution rates of 250 ml/min from Florisil columns and 100 ml/min from carbon–Celite columns gave good recoveries without adversely affecting the cleanup.

Mumma and Kantner (*217*) made use of the mass spectrometer for more positive identification of pesticides. They determined the mass spectra of several chlorinated pesticides and found that each gave easily recognizable molecular ion peaks and characteristic ion fragments. Their procedure was to collect the GLC peaks in medicine droppers containing GLC column packing material. The pesticide was washed out, concentrated, and injected into the mass spectrometer. The procedure has been run on dieldrin, DDT, and DDE from wheat and alfalfa. The sensitivity was 0.1 ppm, and 0.1 μg has given a good mass spectrum. Payne and Cox (*239*) used infrared for the identification of chlorinated pesticide residues in sludge, soils, industrial effluents, and fish and other aquatic fauna. Column and thin layer chromatography were used for the cleanup and separation of the individual pesticides. Minyard and Jackson (*213*) attempted to make identification by electron capture GLC more certain through the use of flash heater inserts packed with various salts to modify the pesticides. A number of salts were investigated, and the authors suggested the possible use of several modifiers in parallel ahead of a single column and detector. Sequential injection of an extract into the various modifiers would produce normal and modified chromatograms which were characteristic of the pesticide. Lee and coworkers (*193*) encountered a contaminant which had the same R_f on a silicone elastomer/Celite GLC column as aldrin, and on a column of Apiezon L had the same R_f as β-BHC. By means of infrared they identified the contaminant as dibutyl phthalate which they thought came from plastic containers and plastic-based paints used in the laboratory.

Specific Procedures. Shuman and Cieri (*257*) reported a method for determining residues of chlorbenside including its sulfoxide and sulfone oxidation products. Samples were extracted according to the Mills, Onley, Gaither procedure and all forms of chlorbenside residue converted to the sulfone by oxidation with chromic–acetic acid solution. After cleanup on an Al_2O_3 column, determination was made by electron capture GLC. Thruston (*275*) compared the electron capture gas chromatogram of a chlordane standard with that of a weathered chlordane residue found on squash. He noted that in the weathered residue, the first 4 major peaks of chlordane were small or had disappeared, while the last 3 peaks were not changed significantly. Gajan and Link (*113*) used oscilloscopic polarography for the determination of DDT. They reported that with an electrolyte of 0.1*M* tetramethyl ammonium bromide in 50% aqueous acetone–ethanol, only DDT and those analogs such as methoxychlor containing the trichloroethane group gave responses in the −0.3 to −1.7-volt range. The regular wave showed a peak indicating the total *o,p′*- and *p,p′*-DDT whereas the derivative showed two peaks whose ratios was equal to the ratio of the two isomers.

Several papers dealt with the determination of DDT. Dingle (*90*) modified Davidow's sulfuric acid cleanup for fat [*JAOAC* **33**, 130 (1950)] and obtained solutions suitable for PC, GLC, IR, or colorimetric determination. Recoveries were said to be better than 98%. Stempkovskaya and Vekshtein (*266*) reported a modification of the Schecter-Haller technique using KNO_3 or NH_4NO_3 with H_2SO_4 in place of fuming HNO_3. They also described a stable artificial color standard consisting of a solution of $CuCl_2$, crystal violet, and $K_2Cr_2O_7$ which was said to correspond to the color produced by 100 μg of DDT. Crosby and Archer (*80*) determined the DDT group in milk, blood, and tissue as their dehydrohalogenated compounds after treatment with KOH. Extraction was with pentane and determination by electron capture GLC. The first foot of the GLC column was packed with calcium carbide to remove traces of water and ethanol. Beckman and coworkers (*24*), after showing that the pesticides were present only in the yolk of eggs, determined DDT and DDE by extracting the yolks with acetone. The extract was evaporated; the residue was taken up in hexane and cleaned up on a Florisil column before determination by electron capture GLC. Recoveries at 0.05–1.0-ppm levels averaged 94%. The time required for analysis was less than 1 hour per sample. Schuntner and Schnitzerling (*256*) used gradient elution from a cooled, water-jacketed, silicic acid column to separate DDT and its metabolites into individual components. Compounds separated included *o,p′*- and *p,p′*-DDT, DDE, *p,p′*-DDD, Kelthane, *p,p′*-dichlorobenzophenone, and *p,p′*-DDA.

Hansen (*143*) reported that the colorimetric method of Jones and Riddick [ANAL. CHEM. **23**, 349 (1951)] for the determination of Dilan could be made about 10 times more sensitive by decreasing the total volume of the final solution while keeping the same ratios of reagents.

Harrison (*144*) was able to determine endrin in wildlife in the presence of large amounts (100–500-fold) of DDE and dieldrin by making a preliminary separation on TLC before using GLC. Although recoveries were slightly better on silica gel plates, alumina gave better separation of endrin from dieldrin. Engel *et al.* (*97*) determined heptachlor and heptachlor epoxide in alfalfa hay by blending the sample with water and ethanol and then extracting with hexane. They used the procedures of Samuel (*252*) for cleanup. Ott *et al.* (*235*) used thin layer chromatography and oscillopolarography to determine *p,p'*-Kelthane and *p,p'*-dichlorobenzophenone. They suggested that in analyzing crop extracts, TLC be used for cleanup before polarographing the sample. Mestres and Chave (*209*) determined lindane in flour by extracting with acetonitrile, cleaning the extract on a Na_2SO_4 and Florisil column, and then using electron capture GLC. Recovery of 0.25 ng of lindane was 95 ± 3%. To determine toxaphene in milk, fat, blood, and hay, Archer and Crosby (*12*) used KOH to dehydrohalogenate the toxaphene before injection into the electron capture GLC. Advantages claimed for the procedure were very rapid and effective cleanup, a higher and more compact peak which eluted before the DDT group, and a two-fold increase in sensitivity. Recoveries ranged from 74–95% at 0.1 and 0.5-ppm levels. Faucheux (*103*) investigated the use of diphenylamine–$ZnCl_2$ as a chromogenic reagent for toxaphene, DDT, and chlordane on alumina TLC plates. Characteristic colors were obtained from these pesticides. Five μg each of toxaphene and chlordane could be detected in mixture. DDT and TDE could be estimated semiquantitatively when all forms were present. Color reactions of 34 pesticides at the 20-μg level were reported.

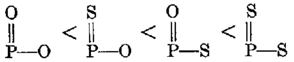
ORGANOPHOSPHORUS PESTICIDES

General Procedures. Storherr and coworkers (*268*) reported a procedure they used for the determination of five organophosphorus pesticides (malathion, parathion, methyl parathion, diazinon, and carbophenothion) in a number of vegetables and fruits. The sample was blended with acetonitrile and filtered. The extract was concentrated under vacuum to remove the acetonitrile and was then extracted with ethyl acetate. After cleanup on a column of carbon and Celite, the pesticides were determined by GLC using the thermionic detector. Other aliquots were examined by the colorimetric *p*-nitrobenzyl pyridine method (*118*), a total phosphorus procedure (*117*), and by paper chromatography. Watts and Storherr (*285*) described a rapid extraction procedure in which the crop sample was extracted by blending with ethyl acetate. Storherr and Watts (*269*) developed a rapid cleanup procedure for organophosphate pesticides which also shows promise of being useful as a general cleanup method for many other types of compounds. The procedure, called sweep codistillation, makes use of a heated short glass column packed with glass wool. An ethyl acetate extract of the sample was injected into the tube, and pesticides and solvent were swept through into a receiver by a stream of N_2. Repeated injections of small amounts of ethyl acetate resulted in nearly complete recovery of the pesticides while crop material remained in the tube. The effluent was clean enough for gas chromatography using the thermionic detector. Recoveries from five crops fortified with a mixture of carbophenothion (Trithion), diazinon, malathion, parathion, and methyl parathion ranged from 89 to 101%, and only 20 minutes was required to clean up each sample.

Getz and Watts (*118*) reported a colorimetric procedure which had a sensitivity of 2 μg of organophosphorus compound. A cleaned up residue was heated with 4-(*p*-nitrobenzyl) pyridine and cyclohexylamine for 2 minutes, diluted, and the absorbance was read at 520 mμ. The procedure worked for all 24 organophosphorus pesticides tested. Getz (*117*) described a procedure in which the organophosphorus compounds were quantitatively converted to orthophosphates by ammonium persulfate and then measured as molybdenum blue with an absorbance at 660 mμ. Residues were separated on paper chromatograms, the spots were cut out, and the determination was run directly with the piece of paper. Sensitivity was 0.1 μg P, equivalent to about 1 μg pesticide. Irudayasamy and Natarajan (*159*) used paper chromatography for the determination of thiophosphorus pesticides. After development, the dried chromatogram was exposed to bromine vapors, air dried, and sprayed with a solution of Congo red. The pesticides appeared as blue spots on a red background and were stable for 10 days if protected from light. The test was sensitive to 0.5 μg of pesticide. Zadrozinska (*290*) used paper chromatography in the determination of organophosphorus pesticides in various food crops. He extracted the sample with carbon tetrachloride and after separation by paper chromatography used enzymatic and fluorescein methods for making the spots visible. Bates (*20*) also used paper chromatography but extracted the food samples with acetone, cooled the extracts to −80°C. and filtered them to remove fats and waxes and used a MgO column for further cleanup. The pesticides were separated and identified by 2-dimensional paper chromatography. For quantitiation, the spots were cut out and phosphorus was determined by the molybdenum blue reaction after wet digestion or Schöniger flask combustion. R_f's are listed for 20 compounds.

Thin layer chromatography was used by a number of workers. Kovacs (*185*) separated thiophosphorus compounds on Al_2O_3–G plates and located the pesticides with a tetrabromophenolphthalein–$AgNO_3$ spray. R_f's were given for 19 compounds and 11 of these were detectable at the 0.05-μg level. Barney (*18*) investigated previously reported chromogenic reagents for organophosphorus compounds and developed 2 new tests. The developed plates were sprayed with HI solution, heated, sprayed with ammonium persulfate solution, heated again, and sprayed with ammonium molybdate followed by buffered benzidine solution. The procedure determined all compounds tested except for one phosphonium compound. Omitting the ammonium persulfate resulted in a test which detected organophosphates but only some of the organophosphonic acids. The lower limit of detection was less than 1 μg. Watts (*284*) adapted the *p*-nitrobenzyl pyridine reagent (*118*) for use as a chromogenic spray in paper and thin layer chromatography. Twenty organophosphorus pesticides tested gave distinct and persistent spots. The test was sensitive to about 0.5 μg for both the thio and nonthio organophosphorus pesticides. Klisenko (*180*) used Al_2O_3 plates and 3 chromogenic sprays for detecting organophosphorus residues. Zadrozinska (*291*) used silica gel and talc adsorbents with 16 mobile phases. El-Refai and Hopkins (*96*) described the use of plates coated with cellulose powder containing 10% $CaSO_4$ as binder, for the separation of organophosphorus pesticides and their oxons. Two solvent systems were used, each including an immobile solvent phase. Four spray reagents were used including one based on cholinesterase inhibition directly on the plate which was sensitive to 0.001–2 ng of the various pesticides. The authors discussed the choice of systems for specific separations and listed R_f values. Melchiorri *et al.* (*206*) used Silica Gel GF 254 plates for the separation and identification of 13 organophosphorus pesticides in vegetable oils. Various solvent systems are described. Salamé (*250*) studied the chromatography of 10 organophosphorus compounds on silica gel using 16 solvent systems and reported R_f values. He used two chromogenic reagents, one (Br_2, $FeCl_3$ and sulfosalicylic acid) had a sensitivity of about 5 μg and the other (palladium chloride) a sensitivity of about 2 μg. Stanley (*262*) used 3 × 1-inch microscope slides coated with silica gel-G. He listed R_f values for 31 organophosphorus compounds in 6 solvent systems and described 7 spray reagents.

A number of workers have used GLC for the separation and identification of organophosphorus pesticides. Horiguchi *et al.* (*155*) separated 9 compounds on three different GLC columns using an electron capture detector. Kanazawa and coworkers (*172*) were able to separate any combination of 19 organophosphorus pesticides by the use of three GLC columns, although no one column gave complete separation of all 19 compounds. Hrivnak and Pastorek (*157*) reported the successful separation of 11 *O,O*-dialkyl-*O*-(4-nitrophenyl)thiophosphates describing the columns and operating conditions used. Nelson (*223*) used microcoulometric GLC for the determination of 16 thiophosphates in 25 crops at levels as low as 0.1 ppm. Samples were extracted by the Mills, Onley, Gaither procedure [*JAOAC* **46,** 181 (1963)] and the residues partitioned into petroleum ether before the gas chromatography. Recoveries of over 70% were obtained for all compounds except Guthion (16%), demeton (46%), and dimethoate (0%). Later modifications (*224*) increased the recovery of dimethoate and Guthion to 70–98%. Cook and coworkers (*74*) studied the electron capture response of 7 organophosphorus pesticides in an attempt to correlate structure to response. They found that in general the electron affinity changed in the manner:



$$\overset{\displaystyle O}{\overset{\|}{P}}\text{—O} < \overset{\displaystyle S}{\overset{\|}{P}}\text{—O} < \overset{\displaystyle O}{\overset{\|}{P}}\text{—S} < \overset{\displaystyle S}{\overset{\|}{P}}\text{—S}$$

and that the methoxy group bonded to the central phosphorus atom resulted in lower electron affinity than did the ethoxy group.

McCaulley (*203*) used a combination of GLC with infrared for the determination of organophosphorus residues from fruits and vegetables. The sample was extracted by blending with acetonitrile, water salted out and the acetonitrile extract evaporated. The residue was subjected to distillation under a vacuum of about 0.5 micron with the pesticide residues being collected on a cold finger cooled with liquid nitrogen. The residues were rinsed off, adjusted to volume, and injected into a GLC. For identification, the peaks of interest were collected and their infrared spectra obtained. Hermann (*151*) used frustrated multiple internal reflection (FMIR) infrared for the identification of trace amounts of organophosphorus pesticides eluted from column, paper, and thin layer chromatograms.

Nangniot (*221*) determined 22 phosphoric acid ester pesticides by oscillographic polarography. Operating conditions for each are listed.

Damico (*85*) determined the mass spectra of 23 organophosphorus pesticides. In addition to giving the spectra, rearrangement and fragmentation patterns are discussed.

Specific Procedures. Blinn and Pasarela (*41*) used a colorimetric procedure to determine Abate, (*O,O,O′,O′* - tetramethyl - *O,O′* - thiodi - *p* - phenylene phosphorothioate) in water, mud, and oysters. After extraction and cleanup, the Abate was hydrolyzed to 4,4′-thiodiphenol and reacted with 4-aminoantipyrine under oxidizing conditions. The color formed was extracted into butanol and read at 510 mμ. Recoveries from water at 0.025–0.045 ppm levels ranged from 79 to 88% and from mud and oysters at 0.1–0.66 ppm levels, 60 to 82%.

Katague and Anderson (*174*) used electron capture GLC for the determination of Bay 37289 (*O*-ethyl-*O*-2,4,5 - trichlorophenyl - ethylphosphonothioate), its oxygen analog, and 2,4,5-trichlorophenol, in a number of crops including alfalfa, beans, carrots, and potatoes. After extraction of the sample with acetone/benzene, the 2,4,5-trichlorophenol was removed with 0.1*N* NaOH for separate determination. Bay 37289 and its oxygen analog were then hydrolyzed to 2,4,5-trichlorophenol, acetylated, and injected into the GLC. The sensitivity of the method was about 0.1 ppm with recoveries ranging from 75 to 104%.

Several procedures have been reported for the determination of Bidrin (dimethyl phosphate of 3-hydroxy-*N,N*-dimethyl-*cis*-crotonamide). Sun *et al.* (*270*) described a fly bioassay said to be specific for Bidrin in the presence of 1 or more of 46 insecticides. It was sensitive to 0.05 ppm. Stevens and Van Middelem (*267*) used electron capture GLC to determine Bidrin in cabbage with a sensitivity of 0.01 ppm. After extraction and cleanup, the Bidrin was reacted with NaOH and iodine–KI solution to form iodoform which was extracted and injected into the GLC. The method, which is specific for methyl vinyl phosphates will thus also detect Phosdrin and phosphamidon. Murphey *et al.* (*219*) described a procedure in which Bidrin was hydrolyzed with NaOH and the resulting dimethylamine distilled and determined colorimetrically as dimethyl dithiocarbamate following addition of Cu^{+2} and CS_2. Recoveries from alfalfa, lettuce, orange peel, string beans, etc. at levels of 0.2–10 ppm ranged from 80 to 108%. The color reaction is specific for dialkylamines. Thus, *N,N*-dimethylcarbamates such as Dimetilan isolan, and Pyrolan would interfere and could be determined by this reaction. Lau (*192*) used cholinesterase inhibition for the determination of both Bidrin and the closely related Azodrin (dimethyl phosphate of 3-hydroxy-*N*-methyl-*cis*-crotonamide) in crops with a sensitivity of 0.1 ppm. The two compounds could be separated from each other and from other insecticides through procedures that are described.

The insecticide, diethyl-1-(2,4-dichlorophenyl)-2-chlorovinyl phosphate, or compound 4072, is known in England as chlorfenvinphos or by the trade name Birlane. Claborn and Ivey (*70*) described a procedure for its determination in milk and tissue in which compound 4072 is hydrolyzed to 2,2′,4′-trichloroacetophenone and determined as such by electron capture GLC. Beynon and coworkers (*34*) reported the analysis for compound 4072 in soil and crops. After extraction of the sample and cleanup on a Florisil column, the insecticide was determined either by cholinesterase inhibition or by electron capture GLC. Compound 4072 consists of 6% cis isomer and 90% trans. When these isomers were gas chromatographed as the intact compounds, they had different retention times. Sensitivity of the two determinative procedures was about equal, 0.01 ppm. Robinson *et al.* (*247*) determined compound 4072 in sheep fat, liver, and other tissues. The parent compound and its metabolite, trichloracetophenone, were separated from chlorinated pesticides and from each other on a column of unactivated Florisil. Each was then determined by electron capture GLC. The sensitivity for compound 4072 was 0.003 ppm and for the trichloroacetophenone 0.001 ppm. Bazzi and Fabbrini (*22*) determined Cidial (ethyl mercaptophenylacetate, *O,O*-dimethyl phosphorodithioate) in oil by extracting a hexane solution of the oil with acetonitrile and after cleanup determining phosphorus as molybdenum blue.

Irudayasamy and Natarajan (*160*) reported a colorimetric method for the determination of carbophenothion (Trithion). The pesticide was hydrolyzed with alkali to *p*-chlorothiophenol which was then reacted with diazotized *o*-dianisidine to give a yellow color with maximum absorbance at 375 mμ.

Boone (*43*) used microcoulometric GLC to determine DDVP and naled (Dibrom) in apples, cabbages, and carrots. A silicic acid column was used for cleanup. Buechler *et al.* (*51*) modified and improved the resorcinol method for the determination of DDVP.

El-Refai and Giuffrida (*95*) used GLC with the thermionic detector to determine DDVP and trichlorfon (Dipterex) in water and in formulations. They also studied the hydrolysis rates of each pesticide and the rate of conversion of trichlorfon to DDVP. Anderson and coworkers (*10*) reported a method for the determination of trichlorfon and its metabolites, chloral hydrate, and trichloroethanol, in plant and animal tissue using electron capture GLC. After extraction and cleanup, trichlorfon and chloral hydrate were injected into the GLC with trichlorfon breaking down and both compounds registering as chloral. To determine the trichlorethanol, a separate aliquot was acetylated to form trichloroethylacetate and then chromatographed. Mustafa *et al.* (*220*) used a colorimetric procedure for determining trichlorfon. The sample was spotted on a filter paper impregnated with 3,5-dintrobenzoic acid and heated at 70°C. for 2 minutes. Trichlorfon gave a blue spot, which was measured in a densitometer with a 550 mμ filter. The reaction, based on the cleavage of the P — C bond and reaction of the phosphite esters with the 3,5-dinitrobenzoic acid, distinguished between trichlorfon and DDVP, which did not react. Szyszko (*298*) reported an oscillopolarographic method for trichlorfon in which lindane and DDT did not interfere. However, maneb and zineb did interfere. Szyszko (*297*) also reported an oscillopolarographic method for demeton-*S*-methyl in foods where maneb and zineb did not interfere. Giang and Schechter (*119*) described a method for the determination of demeton and its metabolites in fruits and vegetables. After extraction and cleanup, the parent compounds and metabolites were all converted to the sulfones by oxidation with *m*-chloroperbenzoic acid. After additional cleanup on a cellulose column, the residue was dissolved in CS$_2$ and read in a 5-mm infrared cavity cell using 5× scale expansion. The absorption at 7.55 microns was used for calculation. Recoveries at 0.6 ppm levels ranged from 76 to 102%.

Gilmore and Cortes (*120*) used dual band TLC as cleanup for the determination of diazinon. By means of a divider in the applicator, the plates were coated with a mixture of Darco G60 and Solka-Floc on the lower 4 cm and with silica gel H on the remaining 16 cm. The crude extract along with a standard were applied to the charcoal–cellulose band, and after development, the sample spots, located by comparison with the standard R_f's, were removed for analysis. Fifty grams of spinach was purified by the above procedure and recoveries as followed by S^{35} labeled diazinon, averaged 95%. Abbott and coworkers (*2*) reported the use of multiband chromatoplates for the TLC determination of dimethoate. They prepared plates having 3 bands of different adsorbents and spotted cleaned up sample extracts. Development separated the dimethoate from the remaining plant materials and most other organophosphorus compounds. The dimethoate spots were made visible by spraying with Brilliant green and exposure to bromine vapor. The square root of the spot areas plotted against the log. of amount of dimethoate gave a straight line. Recoveries from fruits and vegetables at $0.1-0.5$ ppm levels ranged from 80 to 108%. George *et al.* (*116*) described a colorimetric method for dimethoate in plants and milk. After extraction and cleanup, the residue was treated with methanolic NaOH and 1-chloro-2,4-dinitrobenzene to form a color which was read at 505 mμ. Although the oxygen analog would react, it did not come through the cleanup. The authors tested 33 insecticides, 3 herbicides, and 1 fungicide and found that they did not interfere with the analysis. Smart (*259*) compared three colorimetric procedures for the determination of dimethoate in fruits and vegetables, and reported that the procedure of Chilwell and Beecham worked best. Bache and Lisk (*15*) reported the use of GLC with the emission spectra detector (*204*) for the determination of dimethoate and phorate in soil.

Blinn and Boyd (*40*) reported a colorimetric as well as a thin layer procedure for the determination of the dithiolane insecticides, 2-diethoxyphosphinothioylimino-1,3-dithiolane, and its oxygen analog. After extraction and cleanup, the insecticides could be determined on TLC plates made with equal parts of silica gel-G and silica gel-HF. Under ultraviolet light, the compounds appeared as dark areas on a fluorescent background. In the colorimetric procedure, the cleaned up residue was treated first with acid and then with alkali to form thiocyanate which was converted to cyanogen bromide and reacted with benzidine in pyridine to form an intense red solution with an absorption maximum at 530 mμ.

Adams and Anderson (*8*) reported a spectrophotofluorometric procedure for the determination of Guthion [*O,O*-dimethyl - *S* - 4 - oxo - 1,2,3 - benzotriazin-3(4*H*)-ylmethyl phosphorodithioate] in milk and meat. After extraction and cleanup by liquid–liquid partitioning and the use of an alumina column, the pesticide was hydrolyzed to anthranilic acid, and divided into 2 equal aliquots; standard hydrolyzed Guthion was added to one. The fluorescence of both solutions was measured at 400 mμ using an activation wavelength of 340 mμ. The oxygen analog was measured as well as the parent compound. Sensitivity of the procedure was reported as 0.005 ppm in milk, 0.02 ppm in tissue, and 0.03 ppm in fat.

Anderson and coworkers (*11*) used a somewhat similar procedure for the determination of the anthelmintic, *N*-hydroxynaphthalimide diethyl phosphate. Based on the procedure of P. A. Giang [*J. Agr. Food Chem.* **9**, 42 (1961)] for the sulfur analog, Bayer 22,408, the fluorescence was measured at 480 mμ using an activation wavelength of 372 mμ.

Szyszko (*296*) used oscillographic polarography for the determination of Guthion. A most characteristic curve was obtained using a pH 4.0 acetate buffer as electrolyte.

Bowman and Beroza (*44*) reported two procedures for the determination of Imidan [*O,O*-dimethyl-*S*-phthalimidomethyl phosphorodithioate] in milk and corn plants. After extraction and cleanup, electron capture GLC was used for the determination step using a column which was preconditioned by injection of Imidan just prior to use. A colorimetric method, based on the hydrolytic cleavage to liberate formaldehyde which was then reacted with chromotropic acid, was also described although it was not so good as the GLC procedure.

Gutenmann and coworkers (*138*) also used electron capture GLC for the determination of Imidan. They reported that Imidan, its oxygen analog, folpet, and phthalic acid all had the same retention time. It was therefore believed that all broke down to phthalic anhydride on GLC.

Gudzinowicz (*126*) described some of the GLC properties of fenthion (*O,O*-dimethyl - *O* - [4 - (methylthio)-*m*-tolyl]-phosphorothioate) also known as Lebaycid. He used both electron capture and flame ionization detectors and reported that as little as 22 ng was easily detected.

Koivistoinen *et al.* (*182*) studied procedures for the extraction of malathion from fruits using a colorimetric procedure for the determinative step. They reported that for samples analyzed 2–3 days after pesticide application, tumbling the unmacerated fruit with benzene gave the highest values. However, for samples with longer periods between application and analysis, procedures which called for blending of sample with polar or mixed solvents gave higher values. Mestres and Chave (*210*) described a procedure for the determination of malathion in flour which involved extraction with acetonitrile and petroleum ether and Florisil column cleanup. Determination was by GLC using paired thermionic and flame ionization detectors. Sensitivity was reported as 0.1 ppm.

A number of workers reported procedures for the determination of parathion. Lodi (*195*) used electron capture GLC for its determination in wine and biological materials. With wine, a preliminary cleanup by paper or thin layer chromatography was needed. Ott *et al.* (*234*) described a rapid thin layer procedure having a sensitivity of 0.5 ppm by which they were able to obtain qualitative and semiquantitative results on canned peaches in one hour. Szyszko (*295*) used oscillopolarography in which zineb did not interfere but maneb did. Beckman and coworkers (*25*) analyzed for parathion in cole crops, using a Florisil column to remove crop interferences and chlorinated pesticides before the final determination by either electron capture GLC or the Averill-Norris colorimetric method.

Moye and Winefordner (*216*) reported a rapid method for the determination of *p*-nitrophenol in urine, using phosphorimetry. The method could determine as little as 0.01 μg in 5 ml of urine in 40 minutes with an average recovery of 88%. Skuric (*258*) described a fluorometric method for the determination of methyl paraoxon based on the oxidation of indole in the presence of methyl paraoxon.

Winnett and Katz (*286*) described a colorimetric procedure for phorate (Thimet) in vegetables in which the cleaned up phorate residue was hydrolyzed with HBr and the released H_2S determined as methylene blue. Claborn and Ivey (*69*) determined ronnel in milk and in animal tissues, using electron capture GLC after cleanup on a Florisil column. As little as 0.001 ppm could be determined in milk and 0.005 ppm in tissues. Sulfotepp was measured by oscillopolarography by Szyszko (*299*).

Sumithion [*O,O* - dimethyl - *O* - (3 - methyl - 4 - nitrophenyl)phosphorothioate] has been determined colorimetrically by alkaline hydrolysis to sodium 3-methyl-4-nitrophenolate with the absorbance at 400 mμ being measured for quantitation. Kovac and Sohler (*184*) used this procedure following extraction and cleanup on Al_2O_3 thin layer plates to determine Sumithion in fruits and vegetables at levels as low as 0.1 ppm, while Franz and Kovac (*110*) reported a similar determination in milk. Oi and Umeda (*227*) used infrared for the simultaneous determination of Sumithion and methyl parathion in spinach and lettuce at about 1 ppm. The absorption peak at 10.3 microns was used to measure the Sumithion and the peak at 10.8 microns for methyl parathion. Coahran (*72*) reported the use of GLC with the thermionic detector for the determination of Zinophos (*O,O*-diethyl-*O*-2-pyrazinyl phosphorothioate) in soil following overnight extraction in a Soxhlet.

CARBAMATES

General Procedure. Eberle and Gunther (*94*) conducted an extensive investigation of 5 carbamates—carbaryl, Dimetilan, isolan, Pyrolan and Zectran. They studied the effect of natural sunlight and ultraviolet light on these compounds and presented useful basic information concerning their analytical behavior with GLC, TLC, oscillographic polarography, and fluorescence spectrometry. Henkel (*149*) described the TLC behavior of herbicidal carbamates and presented methods for their determination in soil, water, and potato extract. Adsorbents, solvent systems, and spray reagents were discussed. In a later publication, Henkel (*150*) reported on 3 thin layer chromatographic systems and 5 chromogenic color development systems for a number of *N*-methyl and *N,N*-dimethyl carbamates. Limits of detections for these carbamates ranged from 0.05 to 0.15 μg. Hylin (*158*) used thin layer chromatography to determine the dithiocarbamates on leaves. R_f values were given for ziram, thiram, zineb, maneb, and others. Sensitivity was approximately 2.5 μg.

Zielinski and Fishbein (*293*) presented data on the GLC behavior of *N*-substituted carbamates on 3 different columns while Fishbein and Zielinski (*107*) described the GLC behavior of the trimethylsilyl derivatives of a number of carbamates and ureas. Damico and Benson (*86*) developed and tabulated the mass spectra of 14 carbamate pesticides. The significant fragmentation ions were noted and ions were postulated for 8 *N*-methylcarbamate rearrangements. Chen and Benson (*63*) reported the infrared spectra of 32 carbamate pesticides and model compounds. The characteristic absorption frequencies and associated structures were tabulated in a summary and presented in a correlation chart. Broderick *et al.* (*49*) reported that methyl anthranilate, because of its absorption bands at 2.86 and 2.95 microns, interfered in the infrared determination of methylcarbamates. They described a method for removing methyl anthranilate in the analysis of Concord grapes for carbamate residues.

Specific Procedures. Johnson and Stansbury (*162*) reviewed the various methods for determination of carbaryl in a variety of products and in water, and described extraction and cleanup procedures.

Gutenmann and Lisk (*139*) used electron capture GLC for the determination of carbaryl in various crops. After extraction and cleanup, the carbaryl was hydrolyzed to α-naphthol, which was then brominated with I and Br_2 in glacial acetic acid. The brominated residue was taken up in benzene and injected into the GLC, which actually determined brominated 1-naphthyl acetate. Van Middelem and coworkers (*278*) reported a somewhat similar technique in which the α-naphthol formed by hydrolysis was brominated with bromine and the electron capture GLC determination made of brominated α-naphthol. Results were reported for levels as low as 0.1 ppm. Benson and Finocchiaro (*28*) modified the official AOAC colorimetric method for carbaryl [Johnson, D. P., *JAOAC* **47,** 283 (1964)] to eliminate the need for special equipment and to shorten the time of analysis. Johnson and Stansbury (*164*) modified the official AOAC method to determine carbaryl in bees, using a Florisil column for additional cleanup.

Gajan *et al.* (*112*) reported an oscillographic polarographic procedure whereby carbaryl could be determined in the presence of α-naphthol. Using a modified cleanup, recoveries of carbaryl from fortified crops averaged 95% at levels from 0.2 to 10.0 ppm. Among a number of pesticides tested, only *o*-phenylphenol interfered. Engst and coworkers (*100*) formed nitro derivatives of carbaryl by treatment with HNO_3. These derivatives were then determined quantitatively by both d.c. and pulsed polarography with a sensitivity of 0.5 and 0.005 ppm of carbaryl, respectively.

Finocchiaro and Benson (*105*) used thin layer chromatography for the determination of carbaryl. After the samples were spotted and the plates developed, the carbaryl was hydrolyzed by spraying with KOH and then coupled with *p*-nitrobenzenediazonium fluoborate to produce blue spots. The procedure was sensitive to about 0.05 ppm and distinguished carbaryl from α-naphthol, which had a lower R_f. Dingle (*91*) determined carbaryl in cattle dipping solutions by simple dilution with ethanol and measuring the absorbance at 280 mμ. Correction for α-naphthol was based on its absorbance at 324 mμ.

Johnson and Stansbury reported similar colorimetric procedures for the determination of Temik, 2-methyl-2(methylthio) propionaldehyde *O*-(methylcarbamoyl) oxime (*165*) and for Tranid, 3-exo-chloro-6-endocyano-2 - norbornanone - *O* - (methylcarbamoyl)-oxime (*166*). The oxime carbamates were hydrolyzed with NaOH to form the oxime which was then hydrolyzed with HCl to release hydroxylamine. The hydroxylamine was oxidized with iodine to nitrous acid which diazotized sulfanilic acid. The latter was coupled with 1-naphthylamine to form a color which was read at 530 mμ. The sensitivity of these methods was reported to be about 0.03 ppm. Niessen and Frehse (*225*) described a colorimetric procedure for the determination of Bayer 39,007 (Baygon, Unden) (*o*-isopropoxyphenyl methylcarbamate) in leafy vegetables. After extraction and cleanup, the pesticide was saponified, neutralized, and treated with triethanolamine, aminoantipyrine, and $K_3Fe(CN)_6$ to form a color read at 490 mμ.

CHOLINESTERASE INHIBITION

Enzyme inhibition continues to be a useful tool in pesticide residue work. Its lack of specificity, objectionable as it may be in many uses, actually enhances its value as a screening tool, since dangerous amounts of inhibitor may be detected no matter of what nature. Beynon and Stoydin (*37*) reported such a rapid screening test for cholinesterase inhibition making use of agar-agar plates. As little as 0.001 μg of DDVP and other inhibitors could be detected.

Ortloff and Franz (*230*) conducted the test for detection of organophosphorus pesticides on TLC plates, using either 2-azobenzene-1-naphthylacetate (yielding white spots on a red background) or indoxyl acetate (white spots on blue) as substrate. Ackermann (*6*) used silica gel TLC plates for the semiquantitative estimation of organophosphorus and carbamate pesticides. Beam and Hankinson (*23*) reported a procedure for the determination of organophosphorus compounds and carbaryl in milk based on cholinesterase inhibition.

Several workers described the automation of cholinesterase inhibition determinations using the Technicon *Auto*Analyzer. Among these are Voss (*283*) and Ott and Gunther (*231*) whose procedures required prior extraction and cleanup. In a later publication, Ott and Gunther (*232*) used the spots scraped off a TLC plate as input sample for the *Auto*Analyzer.

Guilbault and Kramer (*128*) reported 2 new fluorogenic substrates, resorufin butyrate and indoxyl acetate. Both are nonfluorescent compounds which are hydrolyzed by cholinesterase to highly fluorescent materials. As little as 0.0003 units/ml of horse serum cholinesterase could be determined. However, in addition to cholinesterase, such enzymes as acylase, acid phosphatase, and chymotrypsin also hydrolyzed the substrates to varying degrees. Bauman *et al.* (*21*) reported an immobilized enzyme system which could be used for continuous monitoring of substrate concentration and thus for the detection of cholinesterase inhibitors. A urethane foam pad was impregnated with starch-immobilized cholinesterase and a solution of the substrate, butyrylthiocholine, passed through it. Any inhibition acting on the enzyme reduced the hydrolysis to easily oxidized thiocholine iodide. This caused a change in current flowing through 2 platinum electrodes placed on opposite sides of the pad and thus signaled the presence of an inhibitor. Guilbault and Kramer (*131*) used a similar immobilized enzyme pad in a continuous fluorometric system for determining anticholinesterase compounds in air and water. The substrates, the acetyl and butyl esters of 1- and 2-naphthol, which do not fluoresce, were continuously passed through the pad and were hydrolyzed to the fluorescent phenols. Upon inhibition, the fluorescence dropped.

HERBICIDES

Faust and Hunter (*104*) have reviewed the chemical methods for the detection of aquatic herbicides including diquat, paraquat, and the phenoxy alkyl acids. They discussed various cleanup and determinative procedures. Henkel (*148*) reported on the TLC behavior of the phenoxyalkyl acid herbicides. Adsorbents and pretreatment, liquid phases, R_f's and reagents for detection were discussed. Hosogai and Kawashiro (*156*) separated 16 herbicides in mixtures by TLC, using various nonpolar and polar solvents. Johnson (*168*) described a colorimetric method for the determination of *N*-1-naphthylphthalamic acid in cabbage, asparagus, and alfalfa meal. The sample was heated with zinc and NaOH and the released 1-naphthylamine steam distilled. After cleanup, the 1-naphthylamine was coupled with diazotized sulfanilic acid and the absorbance read at 535 mμ.

Olney and coworkers (*228*) used a modified procedure for the electron capture GLC determination of amiben in vegetables. The sample was digested with alkali to release amiben from any complexes. After extraction and cleanup, it was methylated and further cleaned up on a Florisil column before being injected into the GLC. Sensitivity of 0.01 ppm was reported.

Hilton and Uyehara (*152*) modified the colorimetric procedure of Storherr and Burke [*JAOAC* 44, 196 (1961)] to determine amitrole in sugar cane. Recoveries ranged from 71 to 125% at levels of 0.0025 to 0.5 ppm. Pease (*240*) used temperature programmed, microcoulometric GLC for the determination of bromacil in tissue, plants, and soil. Recoveries averaged 98% at levels of 0.04 to 5.6 ppm.

A number of methods have been reported for the determination of 2,4-D and other chlorophenoxy alkyl acid herbicides. Hagin and Linscott (*141*) described a procedure for the determination of 2,4-D and 2,4-DB in forage plants which made use of quick freezing and blanching of the plant material before extraction. Determination was by electron capture GLC after esterification with diazomethane.

Meagher (*205*) reported a procedure for 2,4-D and 2,4,5-T in citrus. The peel was extracted by blending with hot acetone, and the bound, the free acid, and the ester forms were separated, and each was hydrolyzed to the free acid. The free acids were esterified with 2-butoxyethanol saturated with HCl gas and cleaned up on a Florisil column before determination by electron capture GLC. Chromatographing the compounds as their butoxyethyl esters had the advantage that the long retention times separated the peaks from interferences present near the solvent front. Recoveries ranged from 89 to 93% at 0.0002–0.4 ppm levels. Crosby and Bowers (*81*) reported a method for the determination of 2,4-D in milk and other high protein samples where the 2,4-D may be bound to the sample. They refluxed the sample with NaOH and methanol to release the 2,4-D which was methylated for electron capture GLC determination. Yip (*288*) used programmed temperature microcoulometric GLC to determining a number of the chlorinated herbicides in oils. Recoveries ranged from 87 to 113% at 0.02–0.08 ppm levels. Yip and Ney (*289*) determined free 2,4-D and its esters in milk and forage. After extraction, cleanup, and methylation, determination was made by both microcoulometric GLC and paper chromatography.

Flanagan *et al.* (*108*) reported a paper chromatographic procedure for dalapon, using $AgNO_3$ and phenoxyethanol as chromogenic reagent. Smith and coworkers (*261*) described a method for dicamba in milk and a number of crops, using electron capture GLC after methylation. Meulemans and Upton (*211*) determined dichlobenil and its metabolite 2,6-dichlorobenzoic acid in fruits, soil, water, and fish. The two were separated and determined by electron capture GLC after cleanup. The dichlobenil was chromatographed as such but the metabolite was first methylated. Beynon and coworkers (*35*) reported an electron capture GLC method for the determination of dichlobenil and Chlorthiamid (2,6-dichlorothiobenzamide) in crops, soils, and water. Several extraction and cleanup procedures and 3 GLC columns are described. Recoveries ranged from 80 to 100% at levels of 0.03–5.0 ppm. Boyack *et al.* (*48*) used GLC with a flame ionization detector to determine diphenamid in vegetables and peanuts, with a sensitivity of 0.05 ppm.

Engelhardt and McKinley (*98*) studied the polarographic behavior of diquat. Using previously published extraction and cleanup procedures, they were able to determine diquat polarographically at levels as low as 0.01 ppm with recoveries of 84–97%.

Calderbank and Yuen (*61*) described an improved ultraviolet method for diquat in potatoes. After extraction and cleanup on a cation exchange column, the diquat was reduced to a free radical with sodium dithionite and its absorbance read at 379 mμ. Earlier, they had reported a similar method for paraquat (*60*). Katz (*175*) reported both colorimetric and TLC procedures for five substituted urea herbicides in water. After extraction with chloroform, diuron, monuron, linuron, neburon, and fenuron were hydrolyzed, diazotized, and coupled with *N*-(1-naphthyl)ethylenediamine dihydrochloride to form magenta dyes which were extracted with *n*-butanol and read at

555 $m\mu$. TLC with ninhydrin spray reagent was used for identification of the specific herbicide.

Gutenmann and Lisk (*140*) used electron capture GLC to determine DNOC, DNOSBP, ioxynil, and bromoxynil in milk, apples, and grains. They noted that reacting the phenolic pesticides with diazomethane to form the methyl ethers eliminated trailing on the GLC. Boggs (*42*) also reported the superior chromatographic behavior of the methyl ethers of the dinitrophenols. Bache and Lisk (*14*) reported a similar GLC procedure for ioxynil but used the emission spectrometric detector of McCormack, Tong, and Cooke (*204*) to measure the atomic iodine line at 2062 Å. Ford and coworkers (*109*) described a colorimetric procedure for the determination of norea (Herban) in vegetables, grains, and oil seeds with a sensitivity of 0.1 ppm. The herbicide was hydrolyzed by caustic to dimethyl amine and the primary bicyclic amine which were both steam distilled. After reaction with 1-fluoro-2,4-dinitrobenzene, the complex with the bicyclic amine was separated out on an alumina column and the absorbance in alkaline dimethylformamide read at 443 $m\mu$. Koivistoinen and Karinpää (*181*) reported a modified procedure for IPC and CIPC on fruits and vegetables. Samples were extracted by tumbling with benzene and the herbicides hydrolyzed. The amines were steam distilled, diazotized, and coupled with *N*-(1-naphthyl)ethylenediamine; the absorbance was read at 555 $m\mu$. Recoveries from spinach, cabbage, tomatoes, and strawberries ranged from 86 to 113% at 0.5–200 ppm levels.

Pease (*242*) described a gas chromatographic method for the determination of the herbicide 3-cyclohexyl-5,6-trimethyleneuracil in sugar beets and soil. Using the flame ionization detector, crop blanks ran as high as 0.04 ppm. Merkle *et al.* (*207*) used electron capture GLC after methylation to determine picloram (4-amino-3,5,6-trichloropicolinic acid) in soil. They noted that the acidity of the extracting solvent (acidified acetone) was very important. It had to be acid enough to convert the picloram to the free acid but not so acid as to convert the amino group to a quaternary salt.

Kerr and Olney (*176*) determined trifluralin in vegetables by electron capture GLC and prometryne by hydrolysis to hydroxypropazine which was measured spectrophotometrically. Drescher (*92*) described 2 methods for determining pyrazon. In one procedure which can be used for detection on paper or thin layer chromatograms, the pyrazon was diazotized, losing its chlorine atom, and was then coupled with 2-naphthol to form a dye. In the second procedure, pyrazon was treated with NaOH-methanol to split off aniline which was steam distilled, diazotized and coupled with *N*-(1-naphthyl)ethylenediamine. The absorbance, measured at 530 $m\mu$, permitted detection as low as 0.05 ppm.

Several workers have reported methods for the determination of the *s*-triazines. Mattson *et al.* (*201*) described a procedure for the determination of both chloro and methylmercaptyl *s*-triazines, using microcoulometric GLC with the chlorine and sulfur cells, respectively. A sensitivity of 0.05 ppm was attainable for most crops and recoveries ranged from 75 to 107%. Abbott and coworkers (*1*) used thin layer chromatography to determine 8 *s*-triazines in soil and water. Using silica gel G as the adsorbent the developed chromatograms were sprayed with an 0.5% solution of Brilliant green and exposed to Br_2 fumes. The triazines appeared as deep green spots on an off white background and were immediately marked in outline. For quantitation, the square root of the area of the spots plotted against the log of weight triazine gave a straight line. Manner (*197*) also used TLC to detect 8 *s*-triazines on silica gel GF254. Ninety-one mobile solvent systems and R_f's for each are listed. Plates were examined under ultraviolet light (254 $m\mu$) with the *s*-triazines appearing as dark brown spots on a yellowish green, fluorescing background. The spots could be eluted for additional determinations. Radke *et al.* (*244*) evaluated the pyridine–alkali colorimetric method for the determination of atrazine. They showed that the color intensity increased with acidity of the system and that 20° ± 2° C was a suitable temperature for color development. Chiba and Morley (*66*) reported a microcoulometric GLC method for trichloroacetic acid in wheat sensitive to 0.1 ppm. Compounds such as Kelthane, which could break down to give $CHCl_3$, interfered.

FUNGICIDES

Gunther and Ott (*137*) described a fully automated procedure for the determination of biphenyl in citrus fruit rind. The sample was automatically homogenized and steam distilled; waxes and oils were removed from distillate and the biphenyl in cyclohexane was fed through a cell of a recording ultraviolet spectrophotometer. Chioffi (*67*) used TLC on silica gel to determine biphenyl and *o*-phenylphenol in lemons. Norman and coworkers (*226*) used TLC for cleanup in the determination of biphenyl in citrus fruit and wrappers. Sample extracts were spotted on Eastman silica gel chromagrams and, after development, the spots were located under ultraviolet light. The spots were then cut out, extracted with ethanol, and the absorbance of the biphenyl was measured at 248 $m\mu$. Sensitivity was reported as 5 ppm in citrus fruit and 5 mg/wrapper. McCarthy and Winefordner (*202*) combined a TLC cleanup with phosphorimetric determination for biphenyl in oranges. For the phosphorimetry they used an excitation wavelength of 275 $m\mu$ and emission of 470 $m\mu$. Vogel and Deshusses (*281*) reported a GLC procedure for biphenyl in citrus fruit and wrappers. The biphenyl was distilled and absorbed in cyclohexane, which was injected into a GLC with an ionization detector. Sensitivity was reported as 0.5 ppm. Viel (*279*) reported a colorimetric method for the determination of captan and folpet in grapes and strawberries. After extraction and cleanup, the dried residue was treated with pyridine and then with KOH and the absorbance read at 435 $m\mu$. Fishbein *et al.* (*106*) used thin layer chromatography on silica gel for the determination of captan and Captax (2-mercaptobenzothiazole). As chromogenic reagents, they used resorcinol in glacial acetic acid for captan and cupric chloride–hydroxylamine for Captex. Cheng and Kilgore (*64*) described an electron capture GLC method for the determination of Botran (2,6-dichloro-4-nitroaniline) in stored fruits. A sensitivity of 0.01 ppm was attained by tumbling the macerated sample with benzene, drying the benzene with Na_2SO_4, filtering, and injecting into the GLC. Vogel and Deshusses (*280*) reported a polarographic procedure for 2,6-dichloro-4-nitroaniline which had an accuracy of ±3% at levels of 2–7 ppm.

Hoffman and coworkers (*154*) reported both a colorimetric and thin layer method for the determination of dichlone in tobacco. For the colorimetric determination, the residues were extracted by blending with benzene, cleaned up on a Florisil column, evaporated, dissolved in absolute alcohol, triethylamine added, and absorbance read at 640 $m\mu$. In the TLC method, the developed plates were sprayed with diethylamine and the spots compared with standards. Miller (*212*) investigated 4 colorimetric methods for dichlone and combined parts of 2 for collaborative study. The sample was stripped with benzene and cleaned up on a Florisil column, and color was developed with dimethylamine for reading at 495 $m\mu$. Ten collaborators analyzed samples containing 0.5–4.0 ppm dichlone and obtained recoveries with an overall range of 78–112%. Sensitivity of the method was 0.25 ppm.

Thornton and Anderson (*273*) used electron capture GLC for the determination of Chemagro 2635, a mixture of 1,2,4-trichloro-3,5-dinitrobenzene and 1,2,3-trichloro-4,6-dinitrobenzene. The sensitivity of the method was 0.1 ppm and recoveries from cucumbers, potatoes, spinach, cottonseed, etc. were over 85%. Lyalikov and Solonar (*196*) described the polarographic determination of hexachlorobutadiene and stated that other chlororganic compounds did not interfere.

Cullen and Stanovick (*82*) used electron capture GLC for the determination of korax, 1-chloro-2-nitropropane, in vegetables. The sample was blended with benzene and methanol and after washing and drying the benzene solution was injected into the GLC. Recoveries averaged 80–102% at 0.005–0.1 ppm levels. Voloshchenko and Klisenko (*282*) described a colorimetric method for the determination of Mylone, (3,5-dimethyl - 1,3,5,2*H* - tetrahydrothiadiazine-2-thione). The compound was hydrolyzed with acid to release CS_2 which was reacted with diethylamine and cupric acetate to form copper dithiocarbamate. The sensitivity of the method was reported as 0.02 ppm and recoveries from vegetables ranged from 93 to 120%. Cotta-Ramusino and Stacchini (*76*) reported a spectrofluorometric method for the determination of *o*-phenylphenol on citrus fruit. The extract was diluted with 0.1*N* NaOH and the fluorescence measured at 425 mμ, using an excitation wavelength of 325 mμ.

MISCELLANEOUS PESTICIDES

Kroeller (*188*) used the colorimetric method for arsenic in food based on the reaction of AsH_3 with silver diethyldithiocarbamate after wet digestion and preliminary separation by distillation from HCl. Kirchmann and Roderbourg (*179*) used radioactivation for the determination of arsenic in plant matter. After irradiation the arsenic was separated by wet ashing and precipitation as As_2S_5 before measurement of As^{76}. The limit of detection was 2×10^{-8} gram. Banderis (*17*) reported a colorimetric method for the determination of chlorates in plants and soil. It was based on the reaction of chlorates with HCl to release chlorine. The chlorine was reacted with *o*-tolidine to form a yellow color which was read at 448 mμ.

Several methods have been reported for the determination of cyanide. Kroeller (*189*) used a specially designed still to distill cyanide from foods under nitrogen. The distilled HCN was converted to cyanobromide which reacted with pyridine–benzidine to form a red color which was measured. Guilbault and Kramer (*129*) reported a fluorometric method in which the cyanide was reacted with quinone monoxime benzene sulfonate ester in dimethylsulfoxide to give a green fluorescence. With an excitation wavelength of 440 mμ and emission of 500 mμ as little as 0.5 μg of cyanide was easily detected and no other ions were found to interfere. These authors (*130*) investigated this reaction and those of various other quinone derivatives with cyanide, studying the effect of substituents, solvents, pH, and interferences. They found that the fluorescence produced with *p*-benzoquinone was proportional to the cyanide concentration over the range of 0.2–50 μg. They later reported (*132*) an ultra-sensitive specific qualitative test for cyanide, using *p*-nitrobenzaldehyde and *o*-dinitrobenzene to form a highly colored blue complex by which as little as 3 nanograms total cyanide could be detected.

Steller *et al.* (*264*) described a colorimetric method for cyanimide on cottonseeds. The seeds were extracted by tumbling with water followed by cleanup with activated charcoal. The cyanimide was then reacted with a solution of trisodium pentacyanoammine ferroate to give a red color which was read at 530 mμ. The sensitivity of the method was 0.03 ppm and recoveries at levels of 0.03–0.20 ppm averaged about 85%.

Cottonseed has been analyzed for DEF (*S,S,S*-tributyl phosphorotrithioate), using gas chromatography after Florisil column cleanup. Thomas and Harris (*272*) used the microcoulometric detector while Thornton and Anderson (*274*) used electron capture detection in their procedure. Bielorai and Alumot (*38*) reported a procedure for the determination of ethylene dibromide in foods and feeds, using GLC with a flame ionization detector. Benzene was added to the sample and distilled. The distilled benzene was dried and then injected into the GLC. Results by this method were in good agreement with the chemical titrimetric method at 15–1500 ppm levels. Kimura and Miller (*178*) reported a thin layer chromatographic procedure for the determination of gibberellic acid in rhubarb having a sensitivity of 3 ppb. The gibberellic acid spots were located on the acidified silica gel plate by their fluorescence under ultraviolet light. Zielinski and Fishbein (*292*) reported that they could gas chromatograph maleic hydrazide after reacting it in pyridine with hexamethyldisilazane in the presence of trimethylchlorosilane. Hoffman *et al.* (*153*) discussed possible interferences in the colorimetric method for maleic hydrazide and described a Norit-A cleanup to eliminate interferences. Lane (*191*) conducted a collaborative study of the colorimetric method for maleic hydrazide [J. R. Lane, *JAOAC* **46**, 211 (1963)]. Five collaborators obtained average recoveries of 70–92% from samples of cranberries, potatoes, onions, etc. fortified at 1.3- to 85-ppm levels.

A cold vapor atomic absorption apparatus was designed by Schachter (*255*) to measure submicrogram quantities of mercury in the vapor phase at room temperature. Using this apparatus, Pappas and Rosenberg developed procedures for the determination of mercury in wheat (*236*) and in fish and eggs (*237*) at levels as low as 0.01 ppm. Epps (*101*) used the colorimetric dithizone method for determining mercury in rice following digestion with nitric and perchloric acids. An excellent and thorough study of the dithizone method for mercury in foods was recently reported (*169*). Each step in the procedure was evaluated and the resulting method studied collaboratively. Recoveries at 0.1 ppm were excellent and the sensitivity was thought to be 0.05 ppm (dried sample). Neutron activation has also been used for the determination of mercury. Kim and Silverman (*177*) used it for the analysis of wheat and tobacco, making a chemical separation after irradiation before measuring activity of ^{197}Hg. Tomizawa and coworkers (*277*) used neutron activation to determine mercury in rice. Again, a chemical separation was made after irradiation but these workers measured ^{203}Hg.

Hearth *et al.* (*147*) reported an oscillopolarographic method for the determination of Morestan (6-methyl-2,3-quinoxalinedithiol cyclic carbonate) in orange rind. The hexane stripping solution was concentrated and cleaned up on silica gel TLC plates. The spots were located by their fluorescence under ultraviolet light, scraped off, and eluted with ethanol for the polarographic determination. Martin and Schwartzman (*199*) reported that the ultraviolet spectrophotometric method for nicotine, at times, could not distinguish between crop interference from mustard greens and nicotine; they described a TLC procedure which did make the distinction.

Narahu and coworkers (*222*) used the gas chromatograph with a thermal conductivity detector to determine pentachlorophenol in soy sauce. They chromatographed the PCP as the phenol, using dehydroacetic acid as an internal standard. Cheng and Kilgore (*65*) in determining pentachlorophenol and its sodium salt in fruits, first methylated these compounds with diazomethane before using electron capture GLC for the determinative step. Akisada (*9*) described a colorimetric method for pentachlorophenol and tetrachlorophenol in urine and in air. The phenols were distilled off from the acidified urine while the air was passed through an absorbing solution containing a borate buffer at pH 7.13. They were then reacted with 4-aminoantipyrine and $K_3Fe(CN)_6$ and the colors extracted into xylene. The absorbance was measured at 470 mμ for tetrachlorophenol and at 570 mμ for pentachlorophenol. Zielinski and Fishbein (*294*) gas chromatographed piperonyl butoxide and a number of 3,4-methylenedioxyphenyl derivatives, both as the compounds themselves and as the methyl and trimethylsilyl derivatives of these compounds. Mestres and Barrios (*208*) used gas chromatography to determine propylene oxide and propylene glycol in fruit. By means of a system in which 1–20 mg samples were introduced directly into the injection chamber, they demonstrated that propylene oxide was rapidly absorbed by prunes in which it was hydrolyzed to propylene glycol.

Delfel (*88*) described the use of HI as a color reagent for the detection of rotenone on paper chromatograms. Rotenone gave a characteristic blue color with the reagent while elliptone gave a pink or violet color. None of the other materials present in crude extracts of *Derris elliptica* or *Tephrosia vogelii* gave any color with the reagent. Delfel (*89*) also studied the TLC behavior of rotenone and related compounds and described a number of solvent systems and chromogenic agents to give desired separations. Johnson and Stansbury (*163*) reported a colorimetric method for the defoliant, sodium cis-3-chloroacrylate (Prep), in cottonseeds. The sample was acidified and the free acid extracted by blending with 1-butanol. After cleanup, it was reacted with pyridine and NaOH to produce a colored solution which was passed through an alumina column and then read at 395 mμ. Toxaphene, chlordane, DDT, and TDE did not interfere. Christian and coworkers (*68*) described a polarographic method for selenium in biological materials while Cummings *et al.* (*84*) used a colorimetric procedure measuring the absorbance of a complex of selenium with 3,3′-diaminobenzidine. Pease (*241*) determined sulfamates in apples and pears by removing all the sulfates and then reducing the sulfamates to H_2S which was reacted with *p*-dimethylaminoaniline to form methylene blue. Bowman and Beroza (*47*) reported a gas chromatographic procedure for the determination of tepa, apholate, hempa, and several other chemosterilants. Using the flame photometric detector of Brody and Chaney (*50*) they could detect as little as 0.1 ng of the sterilants. Bullard (*52*) used GLC with flame ionization detector to determine tetramine (tetramethylenedisulfotetramine), a systemic toxicant used to keep animals from feeding on seed and young seedlings. Recoveries from a variety of foliages consistently averaged above 80%. Billy and coworkers (*39*) reported a spectrophotometric procedure for the determination of the lampricide, 3-trifluoromethyl-4-nitrophenol (TFM), in water and in fish tissue. After cleanup by liquid–liquid partitioning and ion exchange column the determination was made by measuring the absorbance at 395 mμ.

MISCELLANY

Duggan (*93*) described the procedures used by the Food and Drug Administration to validate multiple residue methods on the varieties of foods. To illustrate the magnitude of the problem, he pointed out that with the 12 major food classes and the 50 chemicals most commonly found, there were 1.35×10^{16} possible different combinations that the residue chemist could encounter.

In exploring methods for the determination of organo-metallic fungicides on crops, Gudzinowicz and Luciano (*127*) showed that atomic absorption could be used to determine zinc, manganese, and iron. However, the amounts of these metals found in untreated plants were so high that their measurement did not seem a promising means of detecting fungicide residues.

Beroza and Bowman (*30*) introduced the concept of *p*-values—based on the distribution ratios between 2 immiscible solvents—as the basis for identification of pesticide residues and other compounds. In its simplest form, a solution of the residue was analyzed by GLC and then after shaking with an equal volume of immiscible solvent it was again analyzed by GLC. The ratio of the 2nd result to the first is the *p*-value. The authors refined the procedure by the use of a 5-plate Craig counter current distribution apparatus (*31*); they listed *p*-values for 131 pesticides in 6 binary systems (*45*); they designed an apparatus for rapid extraction (*32*) and a device as well as an equation for obtaining *p*-values using nonequilibrated solvents (*46*). They (*33*) also studied the extraction of added pesticides from milk with hexane–diethyl ether with and without prior mixing of sample with ethanol. They found that without ethanol, the extraction efficiencies paralleled the polarities as judged by *p*-values.

Farrow *et al.* (*102*) reported a cleanup procedure for both chlorinated and organophosphate pesticides based on vacuum sublimation. The dried sample extract was subjected to vacuum sublimation at 85°C. for 15 minutes and the pesticide residues were collected on a cold finger cooled with Dry Ice–acetone. The residues were rinsed from the cold finger, made to volume, and injected into the electron capture GLC. The procedure was tested on 35 pesticides in spinach and recoveries for 25 of these exceeded 80%. Most of the others were recovered in the 60–80% range except for a few low values from waxy plant extractives.

Rybakov (*248*) reviewed the use of polarography and discussed methods for the analysis of pesticides containing sulfur, phosphorus, chlorine, and nitrogen.

Coffin (*73*) reviewed the use of paper chromatography in pesticide residue analysis, discussing its advantages and disadvantages as well as various detection systems.

Salo and Salminen (*251*) tabulated TLC data for 29 common pesticides under a number of solvent systems.

Chen (*62*) described a micro technique for infrared by which good spectra could be obtained from as little as 1 μg of pesticide. The procedure, which has been used to identify pesticides separated by GLC, consisted of incorporating the sample into 4 mg of KBr in a micropellet formed in a 2-mm diameter hole in folded aluminum foil. It was pointed out that it was essential to compare sample spectra with standard spectra obtained in the same manner since weak bands are missing at microgram levels.

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Petroleum

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Introduction

THIS IS THE EIGHTH in a series of Reviews of Analytical Chemistry in the Petroleum Industry (*11A, 12A, 22–26A*) sponsored by the Petroleum Division of the American Chemical Society. It covers essentially the years of 1964 and 1965, or rather the papers abstracted in the ACS *Chemical Abstracts* (Sections 2 and 27), in the *American Petroleum Institute Refining Literature Abstracts*, in the *Journal of the Institute of Petroleum* and in the *Analytical Abstracts* (*London*) from the period July 1964 to July 1966. Because of the time interval between original publication and abstracting, some papers published prior to 1964, but not referenced in the earlier reviews, have been included. Generally, those published in 1966 and abstracted, have been set aside for the next issue in this series of Reviews.

Several persons (E. T. Scafe, Mobil Oil Corp., J. F. Hickerson, Humble Oil and Refining Co., and the writer) were involved in the searching of the several abstract journals mentioned above for a collection of abstracts of appropriate papers. These abstracts were further intensely screened (by R. L. LeTourneau, Chevron Research Corp. and the writer) and organized by various subjects that seemed to possess a community of interest. These were further screened by the sixteen Reviewers of the twelve subject classifications which follow. The success of this review is due to the generous assistance of these dedicated people.

In organizing the papers into subject classifications, it was the basic pattern to accommodate papers dealing with a class of products. Since many analyses by a given technique or by competitive techniques would be scattered throughout the Review, classifications by component or by property measured were set up to simplify location of the more closely related material. Thus, it became necessary to decide under which catagory a given paper belonged. Some readers will undoubtedly prefer that we should have classified many papers differently and in these cases we ask their tolerance.

While nearly all of the papers included in this review concern a specific subject, there are a few which deal with reviews of analysis of material types, or of specific analytical processes. On the side of products, there is the three-volume work on bituminous materials including their analysis by Hoiberg (*14A, 15A*). Advances in general gas analysis have been reviewed by Pavlenko (*37A*) and in fuel gas analysis by Raschke (*38A*). Il'ina (*18A*) describes the use of spectrographic analysis for measurement of impurities in fuels and lubricants. A detailed description of the analysis of catalytic feed streams, petroleum products and catalysts for the more common elements in the parts per million and parts per billion range appeared in a book by Milner (*34A*).

An interesting historical paper by Kurtz (*20A*) reviewed the development of hydrocarbon analysis during the past 100 years. Dawson (*7A*) discussed the role of hydrocarbon analysis in refinery quality control operations. Application of gas chromatography in this area is discussed by Dietz (*8A*), Mayor (*33A*), Martin (*31A*), and Andrezijak and Gilewiz (*1A*). Aspects of linear elution adsorption chromatography for extraneous material was reviewed by Snyder (*40A*).

Applications of neuclear magnetic resonance in analytical chemistry has been described by Flockhart and Pink (*10A*), Flanagan *et al.* (*9A*), Chamberlain (*5A*). Zimmerman (*42A*), Oelert and Luther (*35A*), Mair (*30A*) and Louis (*28A*). The large number of review papers indicate the rapidly growing use of this newer technic.

Another analytical process that is rapidly expanding its usefulness is neutron activation analysis; papers by Braier and Mott (*3A*), Iddings (*17A*), Hull (*16A*), and Guinn *et al.* (*13A*) have indicated its usefulness. The utility of x-ray fluorescence analysis has been described by Louis (*27A*) and by Okamoto (*36A*). Some applications of the mass spectrometer have been described by Reed (*39A*). The use of an electron probe microanalysis in a petroleum research laboratory has been reviewed by Bird (*2A*); he deals with analysis of surface layers of lubricants on metals and specimen sections of bearing metals.

Several papers from abroad indicate the growing use of modern methods there; such reviews as those of Ternoysksya (*41A*), Keil (*19A*), and Luther and Louis *et al.* (*29A*) are indicative.

The role of statistics in analysis was paid attention by Calder (*4A*). The ranking of laboratories and evaluating of analytical test methods used in cooperative test programs, as is very commonly done in petroleum analytical laboratories, was discussed by Lashof (*21A*). Most interesting is a new slant on the construction of specifications for petroleum products as described by Cranston and Gammon (*6A*).

Crude Oil

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A RAPID METHOD for the analytical batch distillation of petroleum crudes on a packed column was reported by Schwartz (*13B*). This proposed distillation technique was shown to be faster than the Vigreux column method, with good agreement between the two. The time advantage is obtained because a smaller sample is fractionated at a lower reflux ratio.

Gaylor, Jones, Landerl, and Hughes (*5B*) have developed a gas chromatography method for the simultaneous determination of crude oil boiling range distribution and hydrocarbon-type distribution. The exact location of a peak is achieved by assigning a major group number and a partial relative retention time. A qualitative characterization of major peaks and shoulders was done by using a time-of-flight mass spectrometer attached to the detector vent of the gas chromatography unit. The boiling range distribution obtained for paraffinic crudes was close to the distillation curves from an efficient fractionating column. Typical analyses of heavy naphtha and crude oil are given.

Infrared spectroscopy was used by Leutner (*9B*) to make approximate estimations of certain structural groups in crude oils from the Vienna Basin. By this method, a view of the hydrocarbon composition of an oil is obtained quickly. Karbasian (*7B*) reviewed the procedure for the evaluation of crude oils at Abadan, Iran. A detailed analysis of a crude oil from the Ahwaz Field is shown. Kerenyi (*8B*) has continued his studies of Hungarian domestic and imported petroleum. In this paper he describes a process for the laboratory investigation of crude oil by which the chemical composition and economic value can be accurately estimated. This involves distillation; fraction investigation with graphical evaluation of the results.

Schenck and Eisma (*12B*) developed a gas chromatography method to determine *n*-alkanes in crude oils and rock extracts. A gas chromatography column was placed in series with a column filled with molecular sieves. Two chromatograms were obtained, with and without *n*-alkanes, thus permitting their determination and distribution. A unique sampling analysis method for crude oil directly from the well was developed by Giraud (*6B*). The analysis is done by gas chromatography: specific peak areas for *n*-paraffins were established. Nestler, Triems, and Heinze (*10B*) have also reported a method for the determination of the paraffin content of crude oils. The crude is dissolved in hexane and filtered over fuller's earth. After removing the hexane, the waxes are crystallized and separated by precipitation from an acetone–toluene solution. Reproducibility is about 0.5 weight per cent for paraffin contents of 10 to 20 weight per cent.

Neutral resins in crude oil or wax were determined by a procedure developed by Tronov and Khamzina (*15B*). Asphaltenes are first separated and the sample is then split into two portions. One is treated with silica gel and then extracted with petroleum ether. The ether extract is combined with the unextracted portion, the optical density measured, and the resin content calculated by a formula derived by the authors.

Tumasyan and Babalyan (*16B*) measured the adsorption of asphaltenes from crude oil and synthetic mixtures of asphaltenes in kerosine-benzene solutions on sand and quartz. They observed the largest asphaltene adsorption from the synthetic mixture using sand as the filtration media.

A small angle x-ray scattering study of the colloidal nature of petroleum by Dwiggins (*2B*), showed that colloids derived from seven crude oils had different average Guinier radii of gyration, and some polydispersity. Comparison of these results with those of other studies suggests the colloids seen by x-ray scattering are the same as the asphaltic-rich colloids seen in ultracentrifuging. The small angle x-ray method shows promise for further study of colloids in petroleum.

Forney, Gulbrandsen, and Borup (*4B*) determined the salt content of crude oil by measuring the conductance of a solution of the crude oil in a mixture of xylene, methanol, and butanol. The apparatus was calibrated using a mixture of NaCl, $CaCl_2$, and $MgCl_2$; 7 to 2 to 1, respectively. Results were in good agreement with those obtained using a conventional extraction method. Apparatus was also developed to analyze a plant stream at 3- to 60-minute intervals, with a single analysis requiring about two minutes. Farley and Leonard (*3B*) developed a procedure for the determination of the salt content of heavy crude oils and asphalts. The crude or asphalt is dissolved in phenol–chloroform and extracted with a 0.1% solution of sodium or ammonium nitrate in water. The aqueous extract is centrifuged, and an aliquot titrated for chloride with silver ion. The method is fast and accurate. Crude oil as light as 27° API and asphalt as hard as 10 penetration have been analyzed successfully.

Trace amounts of vanadium in hydrocarbons were determined by Roberts (*11B*) using electron spin resonance. The samples were solidified to avoid inherent errors when samples are run in the liquid state.

Ultrasonic atomization of oil samples diluted 1:1 with alcohol gave improved flame photometric sensitivity when compared with other methods in the detection of alkali and alkaline earth metals. Augsten (*1B*) also reported a significant reduction in analysis time. The results suggest this technique can be adapted to the quantitative determination of sodium crude oils.

Sugihara and Garvey (*14B*) obtained higher recoveries of etioporphyrin I and mesoporphyrin IX when they substituted formic acid for acetic acid using the hydrogen bromide demetalation method for vanadyl porphyrins. This modified method has been used in the analysis of asphaltenes and crude oil.

Fuels

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VERNET AND KNIAZEFF (*110C*) describe methods of determining viscosity, thermal conductivity, and latent heat of liquefied natural gas at $\geqq -175°$ C and $\leqq$ 1067 psi, but no experimental results are given. An apparatus constructed with practically inert materials is being used for measuring viscosity of a corrosive natural gas by Lazarre, Martin, *et al.* (*62C*). Sens and Sallaberry (*97C*) discuss the theory of density measurement of natural gas. They have studied both a static and a dynamic densimeter. Gebert, Lugt, and Herbst (*34C*) have worked up a simple gravimetric technique for density measurement. They have compared results with those obtained on six different instruments of four types. Prahacs and Gravel (*85C*) arranged a positive-displacement gas metering device in series with an orifice flowmeter to serve as a rapid, simple molecular weight monitor and on-stream gas analyzer for a process stream. The compressibility indexes of natural gases of various compositions are reported by Herning and Wolowski (*46C*) and compared with U. S. published data. Makogon (*65C*) discusses the moisture capacity of natural gas. A table of water contents from −40° to 80° C and 1 to 500 atmospheres is given for natural gas with a density of 0.6 gram/cubic meter. A correction curve is provided for gases of other densities. Olund (*78C*) describes the application of the Rayleigh equation for calculating variability in composition of both residual and vaporized odorant added to natural gas. Andreen, Kniebes, and Tarman (*1C*) have developed a two-stage gas chromatograph for determining odorant sulfur compounds in natural gas. Twelve C_2 to C_4 mercaptans and sulfides were separated from interfering hydrocarbons and from each other. Mitrofanov and Makarov (*70C*) modified the MI-1305 mass spectrometer to allow the determination of helium and argon in natural gases with an accuracy of ±0.75 and $\pm0.05\%$, respectively. A gas chromatographic method is used for the simultaneous component analysis and BTU value of natural gas by Thompson and Cavenah (*106C*). Woelk (*116C*) has derived a formula for the calculation of calorific value of town gases, natural gases, etc. Experimental studies by Koecker (*58C*) indicate that errors in the determination of combustion heats of hydrocarbons and liquid and solid fuels are caused by incomplete combustion. These errors can be significantly reduced by an increase in the free combustion surface of the compound and by an increase in oxygen pressure from 30 to 45 atmospheres. Gas chromatography is used for comprehensive analysis of fuel gas by Blakemore and Hillman (*9C*). Razumovskaya and Kurenkov (*87C*) determine the composition of gas from butylenes dehydrogenation by means of gas chromatography.

Gas chromatography continues to be a popular method for the analysis of combustible gases. Aranda and Flaquer (*2C*) discuss the Janak method and compare it with the Orsat method. Roberts and Ward (*90C*) present a new gas chromatographic method with the density balance as the detector for permanent gases. The density balance detector has become commercially available only recently. For hydrocarbons, the argon ionization detector is used. Hofmann (*49C*) describes rapid test methods for checking the quality of LPG shipments when laboratory facilities are not available. A sturdy densitometer has been developed for use at an unloading station or LPG plant reports the same author (*48C*). The determination of density and pressure of liquefied (C_1 to C_5) hydrocarbon mixtures from chromatographic analytical data is reported by Thomas and Zander (*105C*). Gas chromatographic data are also used to calculate vapor pressure of LPG at elevated temperatures by Hammerich and Schmitz (*43C*). Baxter (*6C*) discusses knock rating of gaseous fuels. The standardized apparatus and technique used in the proposed ASTM Motor Method for LPG are described. A rapid color test for mercaptan odorant in LPG has been designed by Peurifoy, O'Neal, and Dvoretzky (*82C*). This technique can be used without laboratory facilities.

Solbrig, Saffert, and Schuberth (*101C*) make a quantitative analysis of flue gases by programmed gas chromatography. Rueb (*93C*) describes instruments which automatically determine carbon dioxide and carbon monoxide plus hydrogen in stack gases, thus permitting direct control of the combustion process. Rate of conductivity buildup and peak rate temperature are better indicators of the corrosiveness of flue gas than is the actual dew point report Clark and Childs (*22C*). *Engineer* (*26C*) describes a portable apparatus for measuring carbon in flue gas. Weber (*114C*) describes a new device for the continuous measurement of the solids content of flue gases.

Freund and Zalai (*32C*) present a summary of the testing and properties of motor fuels. Aviation turbine fuels can differ in their ability to cause sulfide corrosion of silver. Thompson, Hills, Brown, and Lewis (*107C*) have devised a simple laboratory test for determining silver corrosion and correlated it with full-scale laboratory pump rig tests under similated operating conditions and with practical aircraft operating experiences. The Coordinating Research Council (*23C*) has made a study of the test for assessing the water separation characteristics of fuels containing surfactants. The *Journal of the Institute of Petroleum* (*54C*) describes a method of fuel sampling for the determination of particulate matter. This method has been proposed by IP to ensure a common basis for discussion of contaminant levels. Ismailov (*52C*) evaluates the filterability of airplane gasoline by means of a special apparatus. Plogsties and Eckardt (*84C*) have established an empirical equation for predicting the luminometer number from a single measurement. They also have correlated luminometer number with chemical structure and with carbon deposition. Laboratory expressions for motor fuel volatility and their significance in terms of performance are the subjects of a paper prepared by Morrison, Ebersole, and Elder (*74C*). Morrison, Ebersole, and Tooke (*75C*) have modified the Reid vapor pressure test so that it predicts precisely the vapor lock tendencies of gasolines and other hot fuel handling difficulties. The change in pressure of a mixture of vapor(s) and air is determined by Neumann (*77C*) over the surface of the tested liquid fuel in a closed vessel immersed in a heated bath. Shikhov and Sitnikov (*98C*) describe an apparatus which may be used for measuring the density of electrostatic charges on the surface of flammable liquids, on the carbon-coated walls of vessels, pipes, or tanks, and on the portions of electrical conduits insulated from the ground. A portable instrument for measuring electrical conductivity of aviation fuel at airfields is described by Foster and Marsh (*31C*). Using this electrostatic monitor, Marsh and Hawks (*67C*) have collected measurements of the generation of static electricity during the routine fueling of aircraft. These data show that, although significant amounts of static electricity are frequently generated during fueling, it seems to be a negligible hazard.

Finnigan and Pfeifer (*30C*) have made a review of current methods for the evaluation of fuel and lubricant properties. A computer is used by Walker (*112C*) to calculate the effect of varying the compression ratio, combustion period, and spark angle on CFR engines. The development of methods for displaying knock signals from a car gasoline engine accelerated under Modified Uniontown conditions is described by Brockhaus (*13C*). The *Journal of the Institute of Petroleum* (*53C*) reports the results of an experimental study on the effect of engine operating condition tolerances on the accuracy of Motor, extended Motor, and Research Knock ratings. Robinson and Wagner (*91C*) have modified the Coordinating Research Council, Inc.'s vapor-lock procedure which employs full-throttle acceleration. They found that part-throttle acceleration provides a more realistic comparison with actual driving conditions. Road octane equations can be established by the regression analysis of the results of CFR bench engine experiments, report Brockhaus and Fischer (*14C*). Bauer and Callat (*5C*) describe the progress in the development of the distribution octane number (DON). The petroleum industry can realize savings through improvements in the precision of laboratory knock test methods and improvements in the ability of the methods to predict road octane number. Morris and Hoffman (*73C*) list some of the means for improving the precision and significance of these methods.

Mechanical Engineering (*69C*) reports the development of an engine test rig for conducting comprehensive performance trials and analyses on diesel fuel or gasoline. The ignition tendency (cetane number) of diesel fuels has been studied at low air intake temperatures by Wolf (*117C*). Futterer (*33C*) has conducted experiments on the scattering of cetane number measurements on commercial diesel fuels. Bird and Small (*8C*) have examined the carbonaceous residues and ash in the gas stream from the combustion of residual fuel. A technique has been devised by Monaghan (*72C*) to assess the effect of the initial size of liquid fuel drops on their rate of combustion. Kuchta, Bartkowiak, and Zabetakis (*60C*) give data on autoignition temperatures of JP-6 in oxygen–nitrogen atmospheres under constant volume and pressure.

A number of references on storage stability appear in the literature. Drabkina, Zyryanov, and Orechkin (*25C*) propose color change as a quality characteristic of kerosine which would permit an estimation of its stability during storage. Schwartz and Ward (*94C*) have studied the effects of fuel insoluble gums on the storage stability of distillate-type fuels and of soluble gums on gasoline stability. From these data, a general equation has been obtained which can be used to express fuel deterioration as per cent reaction of a fuel component or as the amount of gum formed. Roels (*92C*) has tried the accelerated aging of motor fuel in the presence of copper as a modification of the IP 40 and ASTM D 525 methods. An extensive experimental study including radioactive tracer measurements and accelerated aging by ultraviolet irradiation has been carried out by Schwartz, Whisman, *et al.* (*95C*). This study shows that gum formation results from chemical reactions in the presence of oxygen and not from extensive polymerization; the presence of sulfur compounds is essential to the formation of gum. A simple, rapid test for existent gum has been developed by Hills (*47C*). The results obtained with this test correlate well with the fouling of fuel inlet systems in the field. Haltner (*41C*) has used a tritium tracer technique to study the effect of 70 compounds on gasoline storage stability. This study reveals that sulfur and nitrogen compounds are the most reactive components in the gum-forming process. The thermal stability of supersonic jet engine fuel is measured by means of a miniature, single-tube heat exchanger. This exchanger has been developed by Burggraf and Shayeson (*16C*). Mardanov, Markhaseva, and Bizyaeva (*66C*) determine the thermal stability of fuel fractions by irradiation with ultraviolet light.

J. R. Ritchie (*89C*) reports on a six-year laboratory and field study on the storage stability of six distillate diesel fuels. The tests for soluble and insoluble gum, water retention, Thornton injector needle lacquering, and the U. S. Navy filter clogging were of little or no use in evaluating the stability characteristics of these fuels; but the 16-hour at 210° F (ESSO) and the four-week at 120° F (BP) stability tests predicted reasonably well the storage behavior of these fuels. A method for the investigation of the oxidation kinetics and thermoxidative stability of diesel fuels is described by Losikov, Rubinshtein, and Sobolev (*63C*).

Brink and Scholtz (*12C*) have made a general survey of the FIA procedure with emphasis on the effect of water in the silica gel. The *Journal of the Institute of Petroleum* (*55C*) reports a study of the precision of the FIA method on products distilling below 315° C. FIA combined with aniline point determination is a rapid analytical method for light petroleum distillates or products of catalytic reforming, report Kurchatkina, Orekhova, and Skripnik (*61C*). Liquid chromatography with luminescent indicators is used by Panchenkov, Zhorov, Venkatachalam, and Gurevich (*80C*) to determine the groups in hydrocarbon mixtures. Zhorov, Panchenkov, *et al.* (*121C*) introduce a new indicator—1,3-diphenylbutadiene—for the determination of aromatic hydrocarbons in the gasoline-kerosine range. The indicator spreads uniformly throughout the zone of aromatic hydrocarbons in silica gel chromatography, giving a bright bluish-purple color upon irradiation with ultraviolet light. The indicator is used together with Sudan III dye in benzene solution. Chromatographic analysis, using this new indicator (1,3-diphenylbutadiene), combined with the bromine number method is employed by Zhorov and Panchenkov (*120C*) to determine hydrocarbon types in petroleum products containing alkenylaromatic hydrocarbons. Washall, Mameniskis, and Melpolder (*113C*) determine saturates in heavy petroleum oils by treating the sample with a mixture of fuming sulfuric acid and nitric acid, absorbing the treated olefins, aromatics, and nonhydrocarbons on a bauxite–silica gel column, and recovering the saturates by solvent elution. Integrated analysis by FIA, bromine number, gas chromatography, and infrared spectroscopy is illustrated for a C_8 to C_{18} olefin fraction by Hachenberg and Gutberlet (*39C*). Ioffe and Batalin (*50C*) describe refractometric methods for the group analysis of gasolines. The molar absorptivity of the compound formed by the reaction of cyclohexene with sulfuric acid is measured by Makarov (*64C*) in the determination of small amounts on cyclohexane. Karzhev *et al.* (*57C*) report the separation of *p*-xylene from a mixture of C_8 hydrocarbons by the formation of a complex with antimony trichloride. Complex formation with $Ni(SCN)_2$-4-methylpyridine is used by Casu and Casellato (*18C*) to separate terphenyl isomers. Tetramethyluric acid has been found by Mold, Walker, and Veasey (*71C*), to be a satisfactory and selective complexing agent for the separation of several polycyclic aromatic hydrocarbons of similar structure. Vasilescu and Focsa (*109C*) suggest urea adduction for estimating the *n*-paraffin content of gas oils to be used in microbiological processes. This same technique has been used by Orszag and Bathory (*79C*) for the determination of *n*-paraffins in commercial oils and petroleums. Tago, Nambu, and Noguchi (*104C*) compare a photometric procedure with a gas chromatographic procedure for the determination of dicyclopentadiene in petroleum products. Baranova, Moskvin, and Kurochkina (*3C*) describe applications of colorimetric methods for the determination of cyclopentadiene in isoprene. An ultraviolet spectrophotometric analysis of hydrocarbon oil streams has been patented by Gulf Research and Development Co. (*37C*).

The ozone titration for the determination of unsaturation in olefins has been modified by Guenther, Sosnovsky, and Brunier (*36C*). This modification eliminates the problems of ozone control and end point detection without sacrificing accuracy. Belcher and Fleet (*7C*) have determined olefinic double bonds on 40 to 80 μg of organic material by their reaction with the nucleophilic reagent morpholine to form a tertiary amine. Phenols, C:C unsaturated compounds, and ethers are determined by photometric titrimetry with pyridinium bromide perbromide by Williams, Krudener, and McFarland (*115C*).

U.K. Atomic Energy Authority (*108C*) reports an acid equilibrium procedure for the determination of tributyl phosphate in active and nonactive mixtures with kerosine. Two procedures for the determination of Santolene C in jet fuel are described by Blok, Dahmen, and Verjaal (*10C*). The additive is extracted and then determined by either a potentiometric microtitration or spectrophotometic measurement of a methylene blue complex. Spitler (*102C*) reports on a carburetor icing field test, held at Vancouver, involving 129 cars and about 15,000 runs. An accurate laboratory method for determining the tendency of gasolines toward carburetor icing has been developed by Hammerich and Schildwaechter (*42C*). The advantages of a laboratory method over the use of a multicylinder engine are discussed. The analysis of anti-icing additives in jet fuel by infrared spectrophotometry is described by Hasegawa, Kajikawa, Kawaguchi, and Nishijima (*44C*). In a method developed by Yates, Rai, and Klass (*118C*), fuel detergency in carburetors is evaluated by electrical charge measurement during engine operation. Feldman (*29C*) combines solvent elution and infrared spectrophotometry to determine the amount of ester polymer additives in distillate fuel oils.

A large number of abstracts pertain to the determination of lead in gasolines. Five of the references cited make use of the chelatometric titration. Cipriano (*21C*) extracts the lead as directed in ASTM D 526, then titrates with ethylenediaminetetraacetic acid (EDTA) using Eriochrome black T indicator. Escolar and Castro (*27C*) treat the sample with a solution of chlorine in carbon tetrachloride, extract lead with water, then titrate chelatometrically. Koyama, Taguchi, and Eguchi (*59C*) have devised a procedure in which TEL is decomposed with a solution of bromine in carbon tetrachloride, the lead extracted with nitric acid, and titrated with EDTA. Riquelme (*88C*) extracts the lead from the gasoline by refluxing with concentrated hydrochloric acid, adds nitrilotriacetic acid to chelate the lead, and titrates the excess acid with sodium hydroxide. Zall (*119C*) employs electrolysis in two stages to remove the lead from the gasoline, then determines the lead content by one of three procedures: by weighing the anode; by stripping the anode and titrating with EDTA; or by stripping the anode and determining lead photometrically with diphenylcarbazone. A cooperative testing program was carried out by laboratories in six member countries of ISO on ASTM D 526-61, the Gost 63-52 modified by the Russians, and Gost 63-52 further modified by the Americans. The results are discussed by Siniramed and Renzanigo (*100C*). Farkas and Fodor (*28C*) determine TEL in gasoline by means of absorption of tritium bremsstrahlung. Dagnall and West (*24C*) have examined the effects of extraneous ions, solvents, and flame height on the determination of lead by atomic absorption spectroscopy. Three sources have used x-ray fluorescence spectroscopy for the determination of lead. Szeiman (*103C*) determines small amounts of lead in 3 to 4 minutes. Gunn (*38C*) uses an iron rod as an internal intensity reference to compensate for the effects of differences in absorption between samples. Hasegawa, Kajikawa, and Okamoto (*45C*) take advantage of the constant mole ratio between lead and halogens in commercial gasoline to determine both lead and bromine by measuring the intensity of lead alone. The possibility of determining tetramethyllead and tetraethyllead with coulometric bromination and mercurization at constant current with amperometric end point is reported by Pilloni and Plazzogna (*83C*). A gas chromatographic method for the analysis of lead alkyls has been developed by Barrall and Ballinger (*4C*). Two electron-affinity detectors were studied; one having parallel plates and the other, circular plates.

Pedinelli and Randi (*81C*) have investigated the Shell method for the rapid determination of halogens in gasoline. This method is dependent upon the reaction of the halogen compound with sodium amalgam. Stabilized plasma arc excitation is used by Vigler and Failoni (*111C*) to determine boron in gasoline. Ishii and Kambara (*51C*) determine free sulfur in gasoline by means of the alternating current polarograph. The alternating current polarograph is used also for the determination of manganese in gasoline by Musha and Ishii (*76C*). Shtifman, Kuryakova, Lastovich, and Lerner (*99C*) propose a polarographic method for the determination of manganese in gasoline and in engine deposits containing cyclopentadienylmanganese tricarbonyl. Kapitaniak (*56C*) determines the sodium content in ashed liquid fuels by flame photometry. Mashireva, Orlova, and Sorokina (*68C*) describe a spectrographic procedure for the determination of sodium in petroleum products without preliminary ashing. The method is also applicable to additives, to oils containing additives, to engine filter deposits, and to deposits on catalysts. Boettner and Dallos (*11C*) have investigated the comparative sensitivity of the electron capture detector to chlorinated and lead substituted compounds. The Dumas determination for nitrogen has been combined with gas chromatography by Hachenberg and Gutberlet (*40C*). The semimicro method is used because it yields enough nitrogen for the chromatographic determination of impurities.

Schwoegler, Putscher, *et al.* (*96C*) have developed a kit for the rapid analysis of 5 to 400 ppm sodium, calcium, magnesium, and vanadium and for sulfur above 0.01% in residual fuel oils. The sulfur is determined by oxygen bomb combustion followed by flame photometric or colorimetric analysis. Combustion of fuel oil in a calorimetric bomb is used in the determination of sodium and vanadium by Rayner (*86C*). Brogan (*15C*) also uses bomb combustion to prepare a fuel oil sample for the determination of vanadium. The bomb method is more rapid than the wet oxidation method. Neutron activation analysis for the determination of sodium in heavy fuel oils is described by Caddock and Deterding (*17C*). Chertkov, Marinchenko, and Zrelov (*20C*) describe a procedure for the analysis of microimpurities and deposits in fuels of the medium boiling range.

Geniesse (*35C*) describes the operation of a stream analyzer used to measure the temperature at which a haze or cloud forms in a liquid as it is cooled. The analyzer was developed to determine the moisture haze point of furnace oil. *Chemical Week* (*19C*) reports the development of a "Microb Monitor" for detection of microorganisms in distillate fuel oils.

Mineral Oils and Grease

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IN THE PERIOD covered by this review a large number of papers have appeared which deal with methods of evaluating oils for various types of service. These papers cover a wide scope of procedures including chemical and physical examinations, laboratory mechanical tests, and engine tests. Groszek (*49D*) used heat of adsorption measurements to study interactions between solute and solid surfaces pertinent to detergency, antiwear action, and metal corrosion. Kuenzli (*83D*) used a variety of oxidation tests and chemical analyses to evaluate new transformer oils. The quality of lubricants was determined by Zaslavskii *et al.* (*154D*) using a rapid analytical method involving radiation supplied from an external source and back scattered from the sample, or radiation derived from labeled carbon black introduced into the sample. A technique for evaluating the dispersant activity of high detergent additives was developed by Milowsky *et al.* (*98D*) in which the degree of dispersion is determined from sedimentation velocities. Brunner (*22D*) presented a critical review of conventional physical and chemical laboratory tests used in evaluating motor oils. Vamos and Kantor (*144D*) described a new technique for evaluating dewaxed and deasphalted oils by determining viscosity index, pour point, and distillation yields. The chemical and mechanical characteristics of a deep-dewaxed mineral oil hydraulic fluid were determined in two pump stand tests by Hopkins *et al.* (*58D*). Kahsnitz (*70D*) discussed the analysis of used oils for evaluating engine fouling, corrosion tendency, and other lubricating oil properties. An anonymous article in *Petroleum Management* (*108D*) discussed a portable test kit for in-service evaluation of oils. Zaslavskii *et al.* (*155D*) presented a brief review of test methods for oil additives and the properties they measure. Kishi *et al.* (*78D*) used the panel coking test to evaluate lubricating oil additives. Barcroft (*11D*) used a hot wire method to study reactions between EP additives and metal surfaces. Goto *et al.* (*44D*) used factorial statistical analyses to determine relative sensitivity of test methods for film-type rust preventive oils. Pino and Furby (*111D*) discussed the recent trends in hydraulic fluid test requirements with respect to particulate contamination, safety factors, thermal stability, hydrolytic stability, and compatibility with seals and the extent to which these reflect actual use.

Schlossberg (*123D*), in a survey on corrosion theory and accelerated testing procedures, emphasized the fallibility of humidity and salt spray cabinets. Lindley (*86D*) developed laboratory engine tests for corrosion testing which has given results that correlate with wintertime short-trip field tests. Spengler and Weber (*130D*) reported results of various corrosion tests for gear oils which concluded that none tested were capable of differentiating oils.

Schneider (*124D*) discussed the use of metallographic studies and electron micrographs to supplement conventional friction test results. A pendulum apparatus using various pairs of frictional materials was developed by Tillwich (*139D*) for measuring lubrication value of clock oils. Steele and Steele (*133D*) measured coefficients of friction with a system comprising a wire, under tension, sliding over a rotating cylinder. Vojnovic, Somers, and Roberts (*148D*) predicted rolling characteristics from coefficient of friction and load bearing capacity as determined by the Amsler and Shell testing machines. "Simulation" type lubricant test rigs were investigated by Tourret (*140D*) using ten different oils and a variety of combinations of rubbing metals in the Timken, SAE, and Four-Ball machines. It was found the results did not correlate with each other, even in comparing scuffing and wear tests. Schurmann (*125D*) discussed the advantages of the Four-Ball Apparatus with emphasis on the standardized size, composition, hardness, and surface finish of the steel balls used. Robertson (*119D*) studied wear and load-carrying capacity in the pin and disk machine and found the wear law of Archard *et al.* was not one of steady wear but rather one of zero wear and that wear should probably be thought of as transient. An apparatus for studying ball spinning friction, which simulates spinning friction in a ball-race contact, has been developed by Miller, Parker, and Zaretsky (*97D*). Grosse (*48D*) described a foil bearing machine, based on that of Blok, designed for testing lubricants in the roll-neck bearings of rolling mills. Givens and Talley (*43D*) developed an apparatus for kinetic friction measurements, known as the Tapered Spindle Top, which gives a "line" rather than single point contact between bearing surfaces. Gaensheimer (*39D*) studied the "friction rust" behavior of lubricants on the Fafnir, Molykote, and Lohmeier test machines. Crook (*28D*) used a four, and a two-disk machine to measure friction and effective viscosity. A theoretical study of contact temperatures in rolling/sliding surfaces was reported by Cameron *et al.* (*24D*). Crystallized glass ceramic balls were reported, in an anonymous article (*4D*), to offer many advantages in rolling contact fatigue tests. Barton and Powell (*12D*) reported a technique using a small automotive axle assembly to measure protection against moisture corrosion by gear oils. Munro and Seager (*99D*) discussed the determination of tooth loading in the IAE Gear rig, and two articles from the Institute of Petroleum (*61D*, *63D*) discussed variables and correlations of IAE Gear tests. Beane (*14D*) used the WADD high temperature gear machine to evaluate lubricants. The Four-Ball machine was used by Griffin and Swann (*47D*) to study effects of water contamination of hypoid gear oils, and an Institute of Petroleum article (*62D*) reported a study of the correlation between the Four-Ball machine and other tests. Bell (*15D*) published a survey of functions, requirements, and tests for hypoid gear oils. Hypoid oil tests, specifications, and applications were discussed by the Institute of Petroleum Axle Lubricants Performance Test Sub-Panel (*64D*). Naruse (*101D*), Assmann (*5D*), Bailey (*8D*), Fowle (*35D*), and Bartz *et al.* (*13D*) reported on the use of the FZG gear test for evaluating lubricants. A review of test techniques for evaluating gear lurbicants by the Coordinating Research Council Inc. (*27D*) pointed out that CRC-L-37 and L-42 tests measured the range of EP characteristics required for most American automobiles.

Borden *et al.* (*20D*) reported the results of three years testing of automatic transmission fluids in taxicab fleets in New York City. Various types of full scale engine tests were used by Heitmann (*54D*) for the study of long oil drain intervals and by Davies (*29D*) for the study of sludge formation. Pichler and Futterer (*110D*) described a one-cylinder, four-cycle engine test for use in the development of high-detergency motor oils. The use of the Petter AVI engine to evaluate oils was described in an article in *J. Japan Soc.. Lubrication Engrs.* (*66D*). Bencze *et al.* (*16D*) described laboratory and commercial test methods for two-stroke engine oils. Bench-scale engine tests for evaluating oil quality for diesel engine lubrication were described by Dichter and Maczel (*30D*), Baker and Davies (*9D*), and duJeu (*34D*). Hall *et al.* (*52D*) described two new engine test procedures for evaluating ashless dispersants and antioxidants in aircraft piston engine oils.

A number of papers have appeared describing the testing of electrical insulating oils. Adler and Cosgrove (*1D*) reported the results of a 10-year, elevated-temperature aging test of five inhibited and one straight mineral oil. Goddard and Simmons (*45D*) discussed factors affecting dielectric loss and d.c. conductivity measurements. Kaegler (*69D*) described an automatic bridge balance for use in power factor measurements of insulating oils. Lipshtein and Shtern (*88D*) described an aging test for determining tendency of oils to increase loss tangent. Kaplan (*71D*) reported studies on the electric strength of transformer oil at small interelectrode gaps. Metzler (*96D*) reported the results of the 1962 round robin tests by the Swiss Insulating Oil Committee on the dielectric loss factor. Recommended procedures for the test were presented. A new criterion of the gassing tendencies of insulating oils was reported by Hornsby *et al.* (*59D*). It was suggested that a better assessment of gassing is to determine the range of stress and temperature beyond which the oil evolves gas. Schroeder and Voss (*126D*) used microwave absorption measurements to evaluate aged transformer oils. Lipshtein and Shakhnovich (*87D*) used an electric field to measure transformer oil oxidation stability. Ivanov and Lipshtein (*65D*) published criticisms of conclusions drawn based on examination of oils by various procedures. Dichter and Ogrodowska (*31D*) studied various oxidation tests and concluded an accelerated IEC test was preferable.

A full-scale oxidation test for oils for European automatic transmissions was described by Partington and Haston (*103D*). Bratke (*21D*) and Keil *et al.* (*75D*) described laboratory methods for measuring oxidation stability of oils at elevated temperatures. Barcelo and Otero (*10D*) used a spectrophotometric method to follow oil oxidation. Differential thermal analysis was used by Krawetz and Tovrog (*82D*) to estimate the relative thermal stability of lubricants. Improvements in temperature control and oxygen dosage facilities were modifications of ASTM method D 943-54 described by Matejovsky and Spicka (*95D*). An acceptance test for the oxidation stability of insulating oil was developed by Wilson (*150D*) in which four sets of temperature, copper-oil ratio, oxygen rate, and test duration are considered. Strigel and Winkelnkemper (*137D*) have proposed a method for determining oil aging based on measurement of dielectric constant at various a.c. frequencies up to 10^6 cycles/second.

The use of infrared spectroscopy for determining viscosity-index improvers in oils was reported by Tanaka and Ono (*138D*). Gardner (*40D*) used this technique for identifying 16 lubricating oil additives, and also determined oil oxidation. Savinov and Klimenko (*121D*), and Keil and Rentrop (*74D*) used infrared in the investigation of the oxidation stability of oils. Frassa, Siegfriedt, and Houston (*36D*) used differential infrared analysis to determine chemical changes and contamination in used crankcase oils. An anonymous paper in *Oil and Gas Journal* (*102D*) describes the use of infrared to measure oil deterioration in large bore engines on gas pipelines.

A radiochemical procedure for determining the antiwear properties of oils was described by Pavelescu and Iliuc (*105D*). In this apparatus a radioactive tracer is placed on one of the rubbing surfaces and radioactivity is monitored in circulating oil. Pfeifer (*109D*) discussed radiotracer methods of evaluating engine wear. Anderson and Black (*3D*) reported a method for detecting cross contamination of lubricants using nonlicensed amounts of a radioisotope. King *et al.* (*77D*) used neutron activation to determine soluble copper in studies of the oxidation of electrical oils.

Several new laboratory rotational viscometers for extreme temperatures and pressures and for production control were described by Haake (*50D*). Partington and Haston (*104D*) used a Ferranti-Shirley viscometer to determine the frictional characteristics of automatic transmission fluids. A report by the Coordinating Research Council Inc. (*26D*) showed that the Ferranti-Shirley, the Forced-Ball and probably other laboratory viscometers can closely predict low temperature cranking characteristics of engine oils. A modified Ferranti viscometer was used by Billington and Huxley (*18D*) to measure the flow properties of thixotropic fluids. Measurements with a cone-plate viscometer were used by Prizer and Stringer (*114D*) to determine those oil components which contributed to engine oil viscosity at 0° F. Turian (*142D*) reported investigations of the effects of viscous heating on the results obtained by the cone-and-plate viscometer. Stewart, Lion, and Meyer (*134D*) described the development of a reciprocating viscometer for predicting the low temperature cranking characteristics of engine oils. Rein and Cordell (*115D*) developed an equation for predicting engine cold cranking performance of motor oils. A new CRC low temperature engine method to relate engine cranking performance to viscosity was reported by Lowther and Shea (*90D*). The American Society for Testing and Materials published a combined condensation of four Society of Automotive Engineers papers describing methods of determining low temperature cranking characteristics of oils (*2D*).

Evaporation losses were investigated by Wolf (*152D*) using a test strip, and by Martynov and Kuchinskaya (*92D*) using an apparatus similar to that of Bolynets. Skarpa and Kerleta (*129D*) determined adhesiveness from the temperature of sliding from a vertical steel plate. Keil *et al.* (*72D*) measured gas retention by an oil, after aspiration, to determine foaming properties. Kadmer (*68D*) summarized data from various sources to disprove that oils can be characterized by the pressure required to atomize them. Waby (*149D*) measured the fractional change of permitivity to determine per cent aeration of oil.

Improvements in potentiometric acid–base titrations were reported by Keil *et al.* (*73D*) using bismuth or antimony electrodes and by Wolf (*151D*) who modified ASTM D-664 by using a stronger base and more water in the solvent. Popovych (*112D*) showed the correlation between the apparent pH in ASTM solvent and the pH in water. Luneva (*91D*) determined oil additives by potentiometric titrations. Adsorption chromatography was used by Vamos and Simon (*143D*) to determine methacrylate type additives, and by Madejska and Madejski (*94D*) to determine resins in used oils. Carbonates in oil additives were determined by Ladyzhnikova and Druzhinina (*84D*) who released carbon dioxide and measured it volumetrically. In the method of Rimmer (*118D*) the liberated carbon dioxide is absorbed in organic solvents and determined by titration with sodium methoxide. A photometric determination of tar in oil using optical density at 350–450 mμ was developed by Lizogub and Groshev (*89D*). Rey and Sutter (*116D*) determined total sulfur before and after contact with mercury to obtain corrosive sulfur. Shombert (*127D*) reported the results if interlaboratory testing of ASTM D-1275-58. Kotakis and Kokkoti-Kotakis (*80D*) used a spot test with a reagent containing potassium thiocyanate to detect detergent additives. Rokhlenko (*120D*) placed an inverted U-tube in oil lines to determine air. Solid contaminants were determined with a photometer by Hoffmann and Radmacher (*56D*) and with a centrifuge by Kranold *et al.* (*81D*).

A few papers appeared on the testing of synthetic turbine oils. Hepplewhite and Oberright (*55D*) described a thin film oxidation test, and Buschfort and Hundere (*23D*) a hot surface deposits test. Baber *et al.* (*6D*) reported the results and correlations of several oils in a variety of chemical and mechanical tests. Hopkins and Wilson (*57D*) used the Four-Ball machine to determine wear properties. Haxby *et al.* (*53D*) evaluated the precision of the Ryder gear test and Benzing (*17D*) reported experiences with the IAE machine. Bade (*7D*) reported the development of an IAE gear machine, MK 2, of high temperature, high speed testing. Service evaluation of synthetic oils was done by Reynolds (*117D*) using physical, laboratory bearing, and stationary engine tests.

Peterson (*107D*) investigated modifications of ASTM method D-128 for determining soap content of greases. Except for aluminum soap greases, the original method was most reproducible. Presting *et al.* (*113D*) used a combination of ion exchange and silica gel chromatography to analyze greases. Gas chromatography was used by Carter and Baumann (*25D*) for controlling fatty components of grease. Kempe (*76D*) prepared samples for electron microscopy by freeze drying, permitting study of three-dimensional structure. Gardner (*41D*) used infrared for the qualitative study of grease composition. Phase diagrams and thermal changes in grease were obtained from differential thermal analysis by Trzebowski (*141D*) and from differential thermal analysis and thermogravimetric analysis by Vamos and Flora (*146D*).

The stability of greases toward oxidation was investigated by Sinitsyn *et al.* (*128D*) using a thin film on various metal plates. Dreher *et al.* (*33D*) studied ASTM method D-942-50 and found oxygen consumption could be greater than indicated by pressure changes. Storage stability was the subject of an investigation by Friede and Sangster (*37D*) who compared various established tests. In a study of grease stability, Vamos and Fehervari (*145D*) found that penetration values after 10,000 and 100,000 double strikes was an index of grease quality.

Dickinson (*32D*) reported on the significance of the ASTM dropping point test. Boeck *et al.* (*19D*) carried out a series of experiments to evaluate the reproducibility of the Boeck consistometer. Gottner (*46D*) discussed the problem involved in measuring the pumpability of grease. High temperature viscosity of grease was measured with a modified Brookfield viscometer by Woods *et al.* (*153D*) and with a viscometer consisting of two coaxial conical cylinders by Hoshino (*60D*). Pavlov and Vinogradov (*106D*) investigated the behavior of grease at constant shear rates and shear stresses. Streckeisen (*136D*) used a gear pump with a thermostat as a grease worker in the ASTM D-217 penetration test. Kadmer (*67D*) discussed the problems encountered with various tests used to evaluate greases. Haeussler (*51D*) evaluated laboratory test methods and found them inadequate for complete evaluation of greases. Field tests should supplement laboratory tests.

The mechanical stabilities of lithium greases obtained using the ASTM Grease Worker, Shell Roller Tester, and Wheel Bearing Tester are compared in an anonymous article in *Nafla* (*100D*). Schilling (*122D*) reported a survey covering the problems in grease testing. Stallings (*132D*) and Stock (*135D*) successfully used the Four-Ball tester to evaluate grease. McCarthy (*93D*) reported a high-speed, high temperature test rig for ball bearings built as an aid in the development and evaluation of grease for stringent applications. Spicer (*131D*) reported a survey of various rigs for grease testing and factors affecting their repeatability. Langstroem *et al.* (*85D*) discussed the testing of grease for rolling bearings, particularly on the SKF Emcor Machine. Gilbert *et al.* (*42D*) described a test rig for evaluating torque stability and noise prevention in service station grease. Vekser and Rodzaevskaya (*147D*) determined syneresis of grease by a centrifugal method. Froeschmann (*38D*) reported the results of comparative tests of the corrosion protective behavior of grease in different corrosion test machines. Knott *et al.* (*72D*) described a water spray resistance tester comprised of a temperature controlled water bath, a gear pump, spray head, mount for the test panel, and a shield.

Wax

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THE PERFORMANCE of wax laminations and heat seals was examined by Moyer and Zmitrovis (*21F*) and the factors leading to seal strength or failure were discussed. Finck (*3F*) described new measurements of hardness of waxes using a modified Haake consistometer. He developed new values of "absolute hardness" by correcting for the contact area with wax and by extending the testing time. Spengler and Wilderotter (*25F*) measured the penetration of spheres and cones into various waxes as a function of temperature and established a correlation by means of which the plastic state of waxes could be described. Fabian, Mozes, and Vamos (*2F*) proposed rheological parameters for characterizing the consistency of a petrolatum below the dropping point. Factors affecting the breakdown and regeneration of the quasi-plastic structure were discussed. A color testing method based on the Pulfrich photometer for wax stocks used to make cosmetic and pharmaceutical products was described by Fischer and Keil (*4F*). Krupskii and coworkers (*13F*) measured the thermal conductivity of paraffin wax at low temperatures. Differential thermal analysis was used by Lange and Jochinke (*14F*) to characterize waxes and wax mixtures including synthetic products.

Work has continued on testing of waxes for carcinogenic polycyclic aromatic hydrocarbons. Howard and Haenni (*10F*) used paper chromatography to separate polynuclear hydrocarbons extracted from wax by dimethyl sulfoxide. Paper chromatography was repeated until extracts from the cut-out bands gave significant spectral peaks. The same authors (*11F*) used column chromatography with Magnesia–Celite to separate compounds from a dimethyl sulfoxide–phosphoric acid extract from a solution of wax in an aliphatic solvent. Helberg (*7F*) continued his work with a paper chromatographic procedure. Lijinsky and coworkers (*15F*) used gas chromatography with an electron-capture detector to detect polycyclic aromatics in an extract from wax. Jaforte and Cavallero (*12F*) extracted wax with methanol and cooled the filtrate to precipitate paraffinic hydrocarbons prior to spectroscopic analysis of the extract. Hofmann (*9F*) summarized specifications and testing methods required in Germany and the U.S.A. for liquid petrolatum, hard paraffin wax, and microcrystalline waxes used in foods, drugs, and/or food packaging.

Three papers dealt with the measurement of oil content of wax. Lizogub and coworkers (*17F*) used an optical analyzer employing modulated radiation from a hydrogen lamp and a photocell. The same authors (*1F*) in a later paper described the AMP 2 analyzer (presumably the same instrument) for oil content determination. Triems and Heinze (*26F*) determined oil in microcrystalline wax using an acetone–benzene–toluene mixture, cooling, and washing the precipitate with fresh cold solvent.

Tronov and Khamzina (*27F*) determined neutral resins in samples of petroleum and industrial paraffin. After removing asphaltenes, optical density of the filtrate was compared to a control from which the resins had been removed with silica gel.

A nomograph for the estimation of paraffin wax composition was developed by Melpolder, Turner, and Wilbur (*20F*). It is based on the relationship of melting point, refractive index, and viscosity to chemical composition of the wax.

Raouf, Triems, and Heinze (*24F*) determined the composition of crude waxes obtained by fractional dewaxing of a neutral oil by n-d-M analysis, urea adduction, and column chromatography to determine optimum dewaxing (deoiling) temperature.

Three papers dealt with gas chromatography. Hildebrand *et al.* (*8F*) used high temperature gas chromatography to study chain length distribution of *n*-alkanes in lignite wax. The concentration of odd numbered was greater than that of even numbered *n*-alkanes. Ludwig (*19F*) separated *n*-alkane homologs in the urea adductible fractions of various waxes by gas–liquid chromatography reporting data up to a carbon number of 67. Van der Wiel (*28F*) used molecular sieves to separate normal and branched paraffins and determined carbon number distribution of the two fractions by gas chromatography.

Guseva and Leifman (*6F*) followed the crystal structure of narrow fractions of paraffins, obtained by urea adduction, as a function of temperature by means of refractive index measurements in the liquid and solid states. Ludwig (*18F*) analyzed films of waxes in the solid and molten states by means of infrared spectra and differentiated microcrystalline from paraffin waxes by changes in intensity of CH_2-chain rocking absorptions at 13.7 and 13.9 microns and other measurements.

Polyakova and coworkers (*23F*) reported on the mass spectrometric analysis of paraffin waxes. By modifying earlier methods to fit the instrumentation available, to correct for differences in relative ionization sensitivities, they could determine normal, iso-, and cycloparaffins and molecular weight distribution of the normal paraffins in waxes of predominantly normal paraffin type. Gabrielli and Puggelli (*5F*) used a monolayer method to determine the molecular weight of a paraffin. Measurements of the monomolecular film (expanded or condensed) on various solutions yielded values in agreement with cryoscopic data.

Lindeman and Nicksic (*16F*) studied the phase behavior of oil in commercial waxes by means of nuclear magnetic resonance. The effects of molecular weight distribution of the wax and other factors were discussed. Under selected conditions, oil contents correlated with the ASTM D721 method but nuclear magnetic resonance values were consistently lower.

Nyul, Juhos, and Furedi (*22F*) reported on analyses of petrolatum by adduct formation. The semiparaffins form no adduct with urea or thiourea, but can be separated by solvent dewaxing and make the major contribution to product properties. The *T*-paraffins form adducts with thiourea but not with urea. Analyses of high quality U.S. petrolatums were given.

Asphalt

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HOIBERG (*36G–38G*) is editing a three-volume treatise concerning itself with asphalts, coal tars, and pitches with Vol. I, Vol. II, Part 1, and Vol. III completed.

Redfield *et al.* (*73G*) described a proposed ring and ball test for asphalts, tars, and pitches softening between 35° and 150° C; Schmidt and Santucci (*78G*) described a falling-plunger viscometer for determining asphalt viscosity at low temperatures; and Griffith (*29G*) reported recent progress in the development and specification of fundamental viscosity measurements to replace empirical tests for asphalt cements. Kofalt (*48G*) reported that a single viscosity specification for a specific penetration grade cannot cover asphalts from all sources and Mapstone (*61G*) presented tables and nomograph for determining the asphalt penetration with a penetrometer weighing 50–250 grams from a known penetration with a 100-gram penetrometer and for determining the penetration index from the penetration of an asphalt at any two temperatures.

Schultheis and Woehlisch (*79G*) described a partially automated apparatus for determining the Fraass breaking point; Portnyagin and Mikhailov (*71G*) used a viscometer in which the sample is forced through a plain slot at vibrational frequencies of 50–166.7 cycles/second, and superimposed viscosity curves obtained on maximum and minimum viscosity measured in a coaxial cylinder viscometer. Majidzadeh (*60G*) measured the effect of rate extension, temperature, consistency, size of specimen, and specimen shape and arrangement on tensile properties of asphalts in thin films and Veverka (*86G*) described a method for determining firmness under tension of semi-solid road asphalts at −20° C.

Hrapia and Spur (*41G*) proposed a chromatographic separation followed by a fractional precipitation of paraffins for the determination of the paraffin wax content of bitumen in lieu of pyrolytic distillation and Knotnerus and Krom (*50G*) discussed the composition of wax isolated from bitumen. Altgelt (*1G–3G*) attempted to fractionate asphalts and asphaltenes using gel-permeation chromatography and Grinberg and Shved (*30G*) reported a gravimetric microchromatographic method for the analysis of bitumens based on the adsorption of various components on four adsorbents from a suitable solvent. Podolan and Bahidsky (*69G*) separated asphalt into seven fractions using silica gel and activated alumina columns and Davis, Petersen, and Haines (*24G*) used asphalt as the liquid substrate in a gas–liquid chromatographic analysis with the behavior of the volatile solutes being used to characterize the asphalt phase. Hrapia (*40G*) separated a Romashinko asphalt into 300 fractions and Boyd and Montgomery (*13G*) characterized an Athabasca bitumen using structural-group analysis. Millson and Montgomery (*64G*) showed that many important relationships in structural analysis can be demonstrated by considering the average hydrocarbon in a bitumen or other material as equivalent to two hydrogen atoms and a set of diradical structural units.

Il'ina and Teplitskaya (*43G*) described a luminescence standard for petroleum and bitumens; Botneva and Rasnitsyna (*12G*) followed the paper chromatographic fractionation of bitumens and Fazilov (*25G*, *26G*), the oxidation of asphalt using luminescence. McDonald and Cook (*62G*) used polyethylene as a dispersant when obtaining ultraviolet and visible spectra of high molecular weight asphaltenes.

Smith, Hodgson, and Scheutz (*81G*) used quantitative infrared data on specific structures in coating grade asphalts to predict weatherability; Yen and Erdman (*89G*) made infrared studies in the bending region of asphaltics; and Lisovskii, Portyanskii, and Gukhman (*57G*) studied the composition of phenol separated resins using infrared spectroscopy.

Molecular weights of asphalts and/or asphaltenes were determined by Sdobnov, Gutsalyuk, and Yatsenko (*80G*) using phenanthrene as a cryoscopic solvent; by Bekturov, Kemeleva, and Musabekov (*9G*) an ebullioscopic method; Bekturov, Kemeleva, *et al.* (*8G*) osmotic pressure and viscosity; and Lazare (*54G*) Staudinger's viscometric method. Corbett (*20G*) described a densimetric method for the characterization of asphalts which involved the measurement of the density, molecular weight, and H/C ratio of the petrolene fraction of the asphalt and Girdler (*28G*) attempted to determine the effects of asphaltene structure and content on asphalt behavior using elemental analysis, infrared and magnetic resonance spectroscopy, and molecular weight and color determinations. Poindexter (*70G*) used nuclear magnetic and electron paramagnetic resonance to show that a dynamic proton polarization in colloidal solutions of petroleum asphalt in mixed xylenes at 25° C arises from electron-proton dipolar coupling.

Antonishin and Grinenko (*6G*) studied the sulfonation of asphaltenes; Kolbanovskaya, Davydova, and Davydova (*49G*) the aging mechanism of asphalts using 5- to-50-micron thick layers exposed 10 hours in air at 40–160° C; Leibnitz, Hrapia, and Papp (*55G*) compared the composition of Nagylengyel and Romashkino asphalts and Vajta and Vajta (*85G*) used Traxler's type analysis with refractive index measurements on the fractions or groups to characterize the rheological and practical performance properties of asphalts. Neumann (*68G*) discussed the precipitation of asphaltenes in different solvents in relation to their concentration as a surface film at an oil–water interface.

Ariet and Schweyer (*7G*) separated products of pyrolyzed blown and unblown asphalts using a temperature-programmed gas chromatograph and Hrapia, Meyer, and Prause (*42G*) studied the effect of heat on ductility, penetration, viscosity, and adhesion of a Tatarian asphalt.

The glass transition temperatures of asphalts were measured by Schmidt and Barrall (*76G*) and Schmidt, Boynton, and Santucci (*77G*). Moavenzadeh and Brady (*65G*) developed an equation correlating plastic vsicosity with the asphaltene content and temperature of asphalt and Jones (*44G*) determined changes in the brittle point temperature during weathering to predict durability. Moavenzadeh and Stander (*66G*) described the effect of aging on the apparent activation energy and rheological properties of asphalts and Martin (*59G*) evaluated the degree of hardening of roofing asphalts and coal tar pitches exposed to both dark oxidation and solar exposure using a sliding plate microviscometer.

The effects of bacteria and/or fungi on asphalt were described by Traxler (*83G*), Harris (*33G*), and Jones (*45G*).

Using changes in infrared spectra, Campbell and Wright (*16G–19G*) reported the effects of oxidation of an asphalt flux with oxides of nitrogen, the ozonization of an asphalt flux, and described oxidation products in an oxygen-blown asphalt. Wright and Campbell (*88G*) reported the photo-oxidation of asphalts in presence of ozone. Rozental and Filippenko (*75G*) studied the effect of oxidation rate and catalysts in the preparation of roofing asphalts, and Tucker and Schweyer (*84G*) described the distribution and reactions of sulfur during air blowing and sulfurizing processes. Murayama (*67G*) studied differences in dielectric properties of asphalts.

Cox (*21G*) used a specific gravity method useful for micro quantities of bituminous substances; Brakey (*14G*) described a vacuum extractor for bituminous mixes; and Holland (*39G*) reported a rapid test suitable for field use for obtaining asphalt content of bituminous mixtures. Environmental testing of bituminous coatings was discussed by Wagner (*87G*) and Meany (*63G*); and Krenkler (*52G*) discussed the correlation of breaking point, softening point, etc., of a bitumen with a mastic prepared therefrom. King and Campbell (*47G*) devised a microscopic technique for evaluating the degree of oxidation of bituminous-based wire-rope lubricants and Rick and Temme (*74G*) related the phase separation observed in bituminous coatings to the colloid structure of the film, bleeding, and Oliensis Spot Test results.

Halstead (*31G*) described a relationship of asphalt ductility to performance; and Dalton (*23G*), Halstead (*32G*), and Buchanan (*15G*) discussed the sampling and acceptance of asphaltic products.

Kallas (*46G*) evaluated gyratory testing procedures for selecting the design asphalt content of paving mixtures; Krokosky and Chen (*53G*) used stress relaxation tests to conduct a viscoelastic analysis of the Marshall Test; and Herkenhoff (*34G*) correlated Marshall Test and Hveem Test properties at 110° and 140° F with tensile and compressive strength at 0.05 and 2.0 inch/minute loading rates. Lucas, Bazin, and Saunier (*58G*) described an apparatus for fatigue testing of bituminous mixes and Csanyi and Gumbert (*22G*) confirmed that the Calderon Test in which the results of a pure shear test and an unconfined compression test are combined to develop a Mohr envelope is a valid means for determining the physical properties of asphaltic concrete mixes.

Stephens and Breen (*82G*) used a split cylinder test to show that the tensile strength of a bituminous mixture increased with decreasing temperature from 40° to 20° F but from 20° to 0° F the increase was slight and Ganjula and Saliniuc (*27G*) discussed problems concerning the compactness, shape, and dimensions of specimens used for testing asphaltic concrete. Lettier and Metcalf (*56G*) discussed the application of design calculations to "black base" pavements; Heukelom and Klomp (*35G*) reviewed road design theories; and Kraemer (*51G*) reviewed several methods for testing asphaltic concrete. A vibratory pavement tester (*5G*) and an apparatus for testing soil samples were reported (*4G*). Bikerman (*10G*) reported a scraping test for determining the minimum temperature at which asphalt should be applied to joints between stone slabs.

Bohn (*11G*) investigated the effect of aggregate acidity on the breaking of a soap-based bituminous emulsion and Raudenbusch (*72G*) discussed stone coating tests for cationic emulsions.

Hydrocarbons and Hydrocarbon Types

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INSTRUMENTAL METHODS have continued to play a major role in the analysis of complex hydrocarbon mixtures. Methods that utilize gas chromatography, mass spectrometry, infrared spectroscopy, and combinations thereof, constitute the bulk of the contributions to the literature of hydrocarbon analysis during 1964 and 1965. However, there were few outstanding new developments during this period. Most of these reports are concerned with the application of instrumental techniques to the solution of particular analytical problems. Applications that deal with the lower-boiling (up to 650° F) petroleum fractions predominate, although there are a number of examples that attest to the usefulness of combinations of bulk separation, instrumental, and physical property methods for the analysis of the heavy gas oil and lubricant portion. A very timely review of past and present methods of hydrocarbon analysis, along with predictions of future trends, has been published by the American Society for Testing and Materials (*5H*). This volume should be of particular value to petroleum research chemists.

Accurate methods for the determination of carbon and hydrogen are basically useful in hydrocarbon analysis, and are a necessity for serious composition work on the viscous fractions. Consequently, the improvement of classical combustion procedures is a subject which continues to receive some attention. Van Leuven and Gouverneur (*83H*) describe a combustion-manometric method for the analysis of 3 to 4 mg of material that exhibits a repeatability standard deviation of about 0.07% C and 0.03% H. By modifying this procedure Gouverneur and others (*49H*) were able to analyze samples weighing as little as 5 to 50 μg with a repeatability standard deviation of 0.13% C and 0.10% H. Boes and Gouverneur (*19H*) have also given details of a special apparatus that allows the combustion of 0.8 gram of organic material. The equipment consists of a vertical catalyst-filled combustion tube and novel sample introduction system. When combined with a gravimetric finish, it is capable of producing data that exhibit a repeatability standard deviation of 0.008% C and 0.002% H. Instrumental techniques based upon neutron or x-ray scattering have also been explored by a fair number of investigators, possibly because of their potential in process-monitoring applications. Both Toussaint and Vos (*140H*) and Hasegawa *et al.* (*53H*) have discussed the use of the ratio of the coherent (Rayleigh) and incoherent (Compton) scattering of x-rays to determine the carbon and hydrogen content of hydrocarbons. Neutron scattering has been used for the same purpose by Finston and Yellin (*44H*), Braga and coworkers (*20H*) and Kovar, Hynst, and Koza (*76H*). The neutron source was either radium-beryllium or plutonium-beryllium.

Adsorption methods can still be numbered among the more useful and economical tools for the hydrocarbon-type analysis of petroleum fractions. Linear elution adsorption chromatography (LEAC) seems to be one of the more promising innovations in the field of liquid-solid chromatography, and the number of applications continues to grow. Snyder has described LEAC methods for the routine determination of aromatic hydrocarbon types in cracked gas oils (*131H*), for the determination of nonaromatic olefins in gasoline boiling range materials (*132H*), and for the routine compound class separation of heavy petroleum fractions (*133H*). When combined with mass spectrometry, this latter method is capable of generating a wealth of useful information at reasonable cost. A procedure similar to the aromatic type analysis applied to gas oils by Snyder has been reported by Siryuk and Zimina (*128H*). Snyder and Roth (*135H*) have also described an adsorption method for the rapid determination of the total percentage of saturated hydrocarbons in petroleum fractions having boiling points in excess of 400° F. Fundamental studies of the mechanism of the separation of hydrocarbons by adsorption have been reported by Snyder (*134H*) and Snyder and Warren (*136H*), and will in all likelihood lead to further applications of LEAC.

Conventional adsorption chromatography is still used extensively for the separation of the heavier petroleum oils. In many cases the final separated fractions are characterized by spectroscopic or physical property methods. Vámos and Kántor (*146H*) removed the *n*-paraffins from a 480–580° F Tuimazy gas oil by adduction with urea, isolated aromatic and nonaromatic fractions from the nonadduct forming portion by silica gel chromatography, and further separated these by elution chromatography on alumina and activated charcoal, respectively. The final fractions were characterized by physical properties and elemental analysis. Results of the application of a similar scheme to the adductable portion are reported in a second paper (*147H*). Vakhabova, Musaev, and Niyazov (*145H*) describe the isolation of a representative sample of *n*-paraffins from two gas oil fractions of Kotur-Tepe crude. The fractions were first dearomatized by adsorption chromatography over silica gel. The *n*-paraffins were then separated from the paraffin–cycloparaffin portion by urea complexing. The paraffins thus isolated were further investigated by gas chromatography. Eisen, Rang, and Kudryavtseva (*41H*) use both displacement and elution chromatography on silica gel to separate various classes of hydrocarbons from liquid fuels for further examination by gas chromatography. Bálint (*9H*) has used alumina containing picric acid to separate mononuclear, dinuclear, and trinuclear aromatic fractions from lubricating oil distillates.

The application of molecular sieves as selective adsorbents for *n*-paraffins has received some attention. A short review of petroleum applications has been prepared by Piatkiewicz (*103H*). Bacurova (*8H*) describes a volumetric method for the determination of *n*-paraffins in petroleum distillates of 300–650° F boiling range. The sample is refluxed with isooctane in the presence of 5A molecular sieves for four hours. The volume per cent of *n*-paraffin hydrocarbons is calculated from the decrease in the volume of the liquid in the system due to adsorption of *n*-paraffins. An accuracy of ±2% is claimed. Van der Will (*154H*) has reported the use of molecular sieves to isolate representative samples of C_{19}–C_{36} *n*-paraffins from the paraffin–cycloparaffin portion of lubricating distillates. A dilute isooctane solution of the sample was contacted with 5 A sieves at 99° C. Adsorption equilibrium was complete within one hour. About 97% of the lighter paraffins and 92–95% of the heavier paraffins could be recovered by desorption with *n*-heptane.

Liquid–liquid partition chromatography has not been widely practiced for the separation of petroleum hydrocarbons. Some interesting work with polynuclear aromatic systems that demonstrates the potential utility of partition methods has been reported by Tye and Bell (*143H*). They investigated the behavior of a number of complex polycyclic aromatics when chromatographed on a column of *s*-trinitrobenzene in polyethylene glycol supported on 100–200 mesh diatomaceous silica with isooctane as the mobile phase. Retention volumes and distribution data for thirteen aromatics are given.

Paper and thin layer chromatography have been applied principally for the separation and estimation of aromatic hydrocarbons. The use of thin layer chromatography in conjunction with infrared spectroscopy has been reviewed by Snavely and Grasselli (*130H*). Arro (*7H*) reports the use of thin layer and paper chromatography for the separation of 1,2-benzanthracene, 3,4-benzpyrene, and 1,2-benzpyrene. The application of these techniques to a shale oil tar allowed an estimate of the concentration of 3,4-benzpyrene and its alkyl derivatives. Köhler, Golder, and Schiesser (*74H*) use a mixed thin layer plate of alumina and acetylated paper to separate aromatic hydrocarbons. The plate is developed in one direction with hexane–toluene–pentane, dried, and developed at right angles with methanol–ethyl ether–water. Excellent separations of complex mixtures are obtainable, since not many aromatics exhibit the same R_f values on both cellulose acetate and alumina. Crump (*33H*) has applied a technique that he terms "loose-layer" chromatography to the analysis of gas oils. The separations are reportedly better than those obtained by conventional thin layer methods. Thin layer chromatography on silica gel impregnated with silver nitrate has been used by Gupta and Dev (*50H*) for the separation of olefins.

Judging from the number of contributions to the literature, gas chromatography is still the method of choice for the detailed analysis of light hydrocarbons, gasolines, kerosines, and light gas oils. It is often combined with other methods when wide boiling-range samples are examined. Tabulations of retention data for a number of hydrocarbons on several stationary phases have been reported by Churchwell and Zlatkis (*28H*), Eisen, Kudryavtseva, Rang, and Eisen (*40H*), by Eisen, Kirret, and Eisen (*39H*) and by Gerber and coworkers (*47H*). A correlation between elution characteristics and molecular structure for homologous series of hydrocarbons has been developed by Berezkin and Kruglikova (*13H*). Sakharov (*119H*, *120H*) has developed equations and graphs that can be used to identify unknown compounds from their retention characteristics. Partition coefficients have been used for identification purposes by Huber and Keulemans (*57H*). They have tabulated the partition coefficients for 80 hydrocarbons on squalane and dinonylphthalate.

The majority of reported applications of packed-column gas chromatography are concerned with the analysis of light hydrocarbons, trace analyses, or the separation of relatively specific mixtures of aromatics. Multiple packed-column arrangements for the analysis of mixtures of fixed gases and light hydrocarbons have been reported by Rokhlina (*115H*), Panson and Adams (*99H*), Kudryavtseva and Letunovskaya (*79H*), Dement'eva *et al.* (*36H*), Terry and Futrell (*138H*), and by Cerrone, Piatti, and Rio (*25H*). Apparatus for the chromatographic analysis of hydrocarbon and fixed-gas mixtures has been patented by Leggoe, Hoffman, Kahn, and Brewer (*82H*) and by Koegel *et al.* (*73H*). Packed columns have been used by a number of investigators for the separation of gasoline boiling-range olefins. Ryasnyanskaya and coworkers (*116H*) use a column of firebrick coated with a 1:10 mixture of valeric acid triester and pentaerythritol monochlorohydrin to analyze the olefins produced by wax cracking. Gas chromatography over β,β'-thiodipropionitrile supported on firebrick was used by Vigdergauz and Afanas'ev (*148H*) along with infrared spectroscopy to characterize the octenes produced by the dimerization of butenes. Lavrovskii, Brodskii, and coworkers (*81H*) describe the application of gas chromatography to the determination of cyclopentadiene, its dimer and codimers with acyclic dienes, and methylcyclopentadiene in pyrolysis distillates.

There are a number of reports that deal with the use of packed columns for the separation of aromatics. However, the majority of these are rather unimpressive. Hofmann, Leibnitz, and Koennecke (*56H*) have studied the separation of C_6 to C_8 aromatics on a number of stationary phases. They find that if a column containing a mixed phase of propyl 3,5-dinitrobenzoate and ethylene glycol bis(propionitrile ether) is used in parallel with a column containing 7,8-benzoquinoline or α,β-dinaphthylsulfide, a quantitative determination of C_6–C_8 aromatics in the presence of saturated hydrocarbons of similar boiling range can be achieved. Dal and Nabivach (*34H*) report using a single column of 1,3-dinitrobenzene for substantially the same purpose. Sidorov *et al.* (*126H*) find that the tetra-β-cyanoethyl ether of pentaerythritol exhibits a very strong selectivity for aromatics. By taking advantage of this, they were able to develop a gas chromatographic method for the determination of the total aromatic content of narrow petroleum fractions boiling below 480 °F.

Packed column methods for the separation of mixtures of naphthalenes have been reported by Pop, Barbul, and Beschea (*111H*), Garilli, Lombardo, and Zerbo (*46H*) and by Vaisberg and coworkers (*144H*). None of the methods described are capable of separating all of the C_{10}–C_{12} isomers. All three investigators conclude that highly polar phases provide the best separations, but that for a complete analysis it is necessary to combine gas chromatography and infrared spectroscopy.

The separation of mixtures of polyphenyls has been studied by Garcia (*45H*) and by Sanchez (*121H*). Both authors conclude that Apiezon greases give the best separation of isomers and that flame ionization is the most useful detection system. Abraham and Marks (*1H*) successfully separated a number of high-boiling condensed-ring aromatic hydrocarbons on lightly loaded packed columns at temperatures of 175–225° C. They report retention times relative to pyrene for 15 two- to four-ring aromatics.

The application of packed columns for the separation of high-boiling mixtures of saturated hydrocarbons has been largely confined to the separation of *n*-paraffins. Musaev *et al.* (*94H*) determine individual *n*-paraffins in concentrates isolated from petroleum by gas chromatography over silicone oil at temperatures of 225–275° C. Barrall and Baumann (*10H*) describe a subtractive method for C_7–C_{20} *n*-paraffins that uses a silicone column and molecular sieve column connected in series.

There are several reports that describe the use of packed columns for the analysis of rather wide-boiling petroleum fractions, although capillary columns are undoubtedly more useful for this type of application. The analysis of broad fractions of straight-run naphtha has been detailed by Lulova and coworkers (*86H*). Using an Apiezon column they were able to separate and identify 45 individual hydrocarbons. The identifications were confirmed by Raman spectroscopy and by repeating the gas chromatographic analysis after removing the *n*-paraffins, dehydrogenating, and dearomatizing. Blundell, French, and Griffiths (*18H*) describe the quantitative analysis of nonolefinic gasolines, particularly those prepared by blending straight-run stock with reformate, by the temperature programming of a nonpolar column. Jaworskii (*61H*) found it necessary to prefractionate gasoline samples by distillation before applying gas chromatography. The distillate fractions were then analyzed using a number of columns and the data combined to arrive at an estimate of the original composition.

Because they may be used with relatively high sample loadings, packed columns continue to be cited extensively for trace analysis. Methods for the determination of traces of acetylene in ethylene have been described by Vigdergauz and Andreev (*151H*) and by Rennhak (*112H*). The latter paper also reports methods for the determination of allene and methylacetylene in propylene, and methylacetylene, vinylacetylene, 1-butyne, and 1,2-butadiene in 1,3-butadiene. The determination of traces of vinylacetylene in butadiene has also been investigated by Pollard (*108H*) using a β,β'-oxydipropionitrile column with either thermal conductivity or flame ionization detection. As little as 5 ppm can be determined if the ionization detector is employed. Tago and Koudo (*137H*) use a stationary phase of formamide containing 15 wt. % β,β'-oxydipropionitrile to determine as little as 2–3 ppm of methyl-, ethyl-, vinyl-, and dimethylacetylene in mixtures of C_4 and C_5 hydrocarbons. Vigdergauz and Afanas'ev (*149H*) suggest that traces of propadiene and methylacetylene in propane–propylene fractions can be determined by using a column of heptadecane followed by one containing diisodecylphthalate as the stationary phase. Alekseeva, Gol'bert, and Fomina (*3H*) discuss the determination of C_1–C_4 hydrocarbons present as trace impurities in ethylene. They use a temperature-programmed $NaHCO_3$-modified alumina column followed by a partition column of diisodecylphthalate on firebrick. A sensitivity of 0.3–0.5 ppm is claimed. Kontorovich and Bobrova (*75H*) were able to determine traces of oxygen, nitrogen, carbon monoxide, carbon dioxide, methane, propadiene, butadiene, methylacetylene, ethylacetylene, vinylacetylene, dimethylacetylene, diacetylene, and benzene in acetylene by gas chromatography over silica gel at 20° and 80° C.

Traces of benzene, toluene, and cumene in ethylbenzene, and traces of ethyl- and butylbenzene in cumene have been successfully determined by Krichmar and Stepanenko (*78H*) using two packed columns in series. An accuracy of ±10% in the range of 0.01 to 0.10% is claimed. Dawson (*35H*) uses an electron capture detector in series with a hydrogen flame detector to determine as little as 1 ppm of polynuclear aromatics in petroleum products. The hydrogen flame chromatogram can be used to estimate the boiling point distribution, and, after suitable calibration, the electron capture chromatogram can be used to identify certain polynuclear aromatics.

Capillary gas chromatography has emerged as one of the most powerful tools at the petroleum chemists' disposal, and is especially well-suited for the analysis of wide-boiling mixtures or difficultly separable isomers. Bruderreck, Schneider, and Halasz (*21H*) have made an extensive study of the instrumental factors that affect the quantitative interpretation of capillary data. They conclude that standardization of detector geometry is highly desirable. McTaggart and Mortimer (*89H*) reported the results of a similar study from which they concluded that careful calibration is necessary in order to correct for differences in response due to variations in detector configurations. They also describe suitable capillary columns and operating conditions for analyzing straight-run petroleum fractions and mixtures of aromatics. Kunugi *et al.* (*80H*) describe the separation of light hydrocarbons using dibutyl maleate and propylene carbonate capillaries in series. They also report the separation of a complex mixture of C_5–C_{17} *n*-paraffins and *n*-olefins on a capillary coated with tricresylphosphate. A partial analysis of the C_4 and C_5 fractions from a cracked naphtha using a 300-foot × 0.01-inch i.d. polyethylene glycol capillary column has been described by Kagabe (*64H*). Miyake, Mitooka, and Matsumoto (*93H*) have studied the use of capillaries coated with mixed stationary liquids for the separation of C_6–C_{10} aromatics. Complete resolution of all of the isomers was achieved using a capillary coated with a 90:10 mixture of dinonylphthalate and 2,4-dinitrochlorobenzene. The capillary separation of xylene isomers on a number of stationary phases has been discussed by Vitt and coworkers (*152H*). They found that triethylene glycol butyrate gave exceptionally good results, especially when it was necessary to determine individual xylenes in the presence of saturated hydrocarbons of higher boiling point. Kabot and Ettre (*63H*) report the separation of a mixture of C_{10}–C_{12} naphthalenes on a 300-foot capillary column coated with DC-550 phenylsilicone. All of the methyl-, dimethyl- and ethylnaphthalenes except the 2,6- and 2,7-dimethyl isomers were resolved. Packed capillaries have been used by Schneider, Bruderreck, and Halasz (*124H*) to separate C_1–C_8 mixtures of hydrocarbons according to the number of carbon atoms in the molecule. The columns contained graphited carbon black whose adsorptive properties had been modified by coating with a small quantity of squalane. Vigdergauz and Andreev (*150H*) describe the use of packed capillaries for the high speed analysis of mixtures of petroleum hydrocarbons. Several examples are given, including the separation of a straight-run gasoline in 5 minutes on a 52- by 0.09-cm column containing pentadecane supported on diatomaceous silica. Karr and Comberiati (*65H*) have used capillary columns coated with a thin layer of γ-alumina to analyze mixtures of C_{15}–C_{33} *n*-paraffins isolated from coal tars. This type of column can be operated at temperatures as high as 400° C.

Temperature programming of both packed and capillary columns is now a fairly standard practice. It not only shortens analysis time, but permits the elution of components of wide-boiling mixtures under conditions that facilitate the quantitative measurement of low-boiling constituents and reduce the peak broadening of high-boilers. Shtof, Khomenko, and Ustenko (*125H*) describe a method for the determination of C_2–C_7 hydrocarbons in crude oil that utilizes a precolumn followed by an analyzer column that is programmed from 30° to 115° C at a rate of 2.3° C per minute. Both columns are packed with firebrick containing 10% petroleum jelly. Mixtures of C_8–C_{18} 1-olefins were analyzed by Poe and Kaelble (*106H*) on a 2-foot column of SE-30 silicone, programmed from 75° C at 9° C per minute. The authors claim that the method is more precise and accurate than mass spectrometry. The analysis of complex mixtures of alkylbenzenes of chain length C_{10}–C_{14} has been studied by Carnes (*23H*). A 150-foot × 0.01-inch i.d. capillary coated with DC-550 silicone oil and programmed from 120° to 170° C at 1.5° C per minute gave the most useful separation. Basili, Bordonali, and Patimo (*12H*) used a temperature-programmed capillary coated with SE-52 silicone rubber to resolve mixtures of *o*-, *m*-, and *p*-terphenyls. Various mixtures of quaterphenyls could also be analyzed using this technique. Cieplinski *et al.* (*30H*) report that the characterization of polymers can be accomplished in a shorter time if temperature-programmed capillary columns are used to analyze the products of pyrolysis. Flow programming is often a useful substitute for, or adjunct to, temperature programming. It sometimes can be used to advantage when the stationary phase exhibits high volatility or poor thermal stability at elevated temperatures. The results of some preliminary investigations of the use of programmed flow chromatography for the analysis of hydrocarbons have been reported by Neto, Koeffer, and de Alencar (*98H*).

The results of investigations dealing primarily with the evaluation of column packings have been described in a number of reports. The use of molecular sieves for the separation of mixtures of fixed gases and gaseous hydrocarbons has been studied by Kiselev, Chernen'kova, and Yashin (*69H*). Tsitsishvili *et al.* (*141H*) have investigated the changes in the adsorptive properties of type-X sieves when the sodium ion in these zeolites is exchanged for other alkali metal ions. They found that the separating power of a zeolite was strongly affected by both the nature and degree of substitution of the cation. The influence of silica gel structure on hydrocarbon separations by gas chromatography was examined by Kiselev and coworkers (*70H*), who concluded that the retention volumes of hydrocarbons are influenced chiefly by the pore size of the gel. The use of alumina for the gas chromatographic separation of hydrocarbons has been reported by Hoffmann, List, and Evans (*55H*), by Pollak and Uus (*107H*), and by Philippe and Honeycutt (*102H*). Coarse-pored glass has been suggested by Zhdanov, Kiselev, and Yashin (*156H*) as a packing for the separation of hydrocarbons having boiling points less than 150° C. It has the purported advantages of good thermal stability and uniform adsorbent surface. Halasz and Horvath (*51H*, *52H*) describe the performance of column packings prepared by coating glass microbeads with various powdered solids. These packings are useful in certain special applications, especially those where an extremely rapid separation of polar or high-boiling compounds is desired.

The preparation of columns containing modified Bentone 34 has been described in detail by Cassano and Burgos (*24H*), who use such columns for the separation of xylenes, and by Blake (*17H*), who reports their use for the separation of diethylbenzene mixtures. Some of the pitfalls that are likely to be encountered when using Bentone packings have been pointed out by Cieplinski (*29H*).

There are several reports that emphasize the usefulness of particular stationary phases in certain applications. Clemons, Leach, and Altshuller (*31H*) find that 1,2,3-tris-(2-cyanoethoxy) propane is an excellent stationary phase for the separation of C_6–C_{10} aromatics having saturated and unsaturated side chains. Its stability allows the direct analysis, with a flame ionization detector, of aromatic hydrocarbons at levels as low as 0.01 to 0.10 ppm. Jordan (*62H*) uses a mixture of 1,2,3-tris-(2-cyanoethoxy) propane and SE-30 gum rubber for a number of applications where hydrocarbons having olefinic or aromatic unsaturation are involved. Dibutyl tetrachlorophthalate is reported by Vsevolozhskaya, Morozova, and Petrov (*153H*) to be an extremely useful stationary phase for the separation of both saturated and aromatic hydrocarbons having from 7 to 10 carbon atoms.

There has been some interesting activity in the fields of instrument and detector development, although most of the devices described have yet to be field proved. Nerheim (*97H*) gives details of an indium encapsulation technique that facilitates the quantitative introduction of volatile liquid samples into a gas chromatograph. Examples of its application for the determination of naphtha in catalytic cycle oil and of gasoline diluent in motor oil are given. An electron drift–velocity detector which has high sensitivity to permanent gases was assembled and tested by Smith and Fidiam (*129H*). The device appears to be of limited utility. An extremely sensitive piezoelectric-sorption detector has been described by King (*68H*). The heart of the apparatus is a piezoelectric quartz crystal coated with a very thin film of high-boiling liquid. Weight changes brought about by sorption of eluting species in the coating cause a change in the amplitude and frequency of the vibrating crystal. By judicious choice of the coating the detector can be made selective for certain compound types. Winefordner, Williams, and Miller (*155H*) report details of a high sensitivity detector for gas analysis that depends upon changes in dielectric constant that result from the presence of other gases or organic vapors in a flowing helium stream. The device is extremely sensitive to such gases as oxygen, nitrogen, hydrogen, carbon dioxide, carbon monoxide, methane, acetylene, argon, nitrogen oxides, and sulfur dioxide. The use of a photoionization detector for gas chromatography was studied by Locke and Meloan (*85H*). They concluded that its main advantage is the inherent selectivity that results from differences in ionization potential among chemical compound types. Camin, King, and Shawhan (*22H*) describe the use of microvolume thermal conductivity detectors with 0.02-inch i.d. capillary columns. They report that simple, economical, and reliable capillary instruments can be assembled using commercially available components. Examples of the separation of a wide variety of hydrocarbon mixtures are given. A specialized apparatus for the determination of low-boiling aromatics in petroleum fractions has been assembled by Klayder (*71H*). He uses a column of Bentone 34 modified with Versilube F-50 to separate individual aromatics. A heated ultraviolet flow-through cell operated at 189.25 mμ is used as a detector to avoid interference from saturated hydrocarbons. A novel capillary method for the analysis of saturated hydrocarbons has been described by Piringer and Tatarn (*104H*). Detection is accomplished by combustion of the separated components to carbon dioxide which is then differentially determined by electrolytic conductivity in deionized water. The technique does not seem to offer any outstanding advantage over more conventional methods.

Chemical reactions such as sulfonation, hydrogenation, and dehydrogenation are now often used in combination with gas chromatography for the analysis of complex mixtures. The reactions may be conducted "in line" on a microscale, or performed in other apparatus on separate aliquots of the sample. Innes and Bambrick (*58H*) use chemical absorption in conjunction with packed columns and flame ionization detection to separate classes of hydrocarbons present in automotive exhaust. Sulfonation, dehydrogenation, and gas chromatography have been employed by Antheaume and Guiochon (*6H*) and Hines and Smith (*54H*) for the analysis of naphtha and light gas oil fractions. A study of microcatalytic hydrogenation and dehydrogenation and subsequent gas chromatography has been reported by Klesment, Rang, and Eisen (*72H*). Gordon (*48H*) uses in-line microhydrogenation to determine the various carbon structure isomers present in commercial heptenes. Two interesting papers by Revel'skii *et al.* (*113H*, *114H*), although only vaguely akin to reaction chromatography, seem worth mention. They describe an instrument that permits the continuous determination of the empirical formulas of hydrocarbons eluted from a gas chromatographic column. The molecular weights of the components are measured with a gas density detector, and the carbon and hydrogen contents determined using a special continuous elemental analysis apparatus.

Many of the problems encountered in the field of petroleum composition defy solution by the application of a single technique or instrumental method. Consequently, the use of a multiplicity of analytical tools in combination is now rather common practice. The combination of paper chromatography, and infrared and ultraviolet spectroscopy with gas chromatography for the identification of aromatic hydrocarbons has been the subject of a brief review by Derge (*37H*). Janak (*60H*) collects submicrogram amounts of high-boiling materials eluted from a capillary column by condensing them on glass or metal plates. The collected fractions are then characterized by microscope examination of their crystal structures, or by forming colored tetracyanoethylene–crystal complexes under the microscope. Capillary and preparative scale gas chromatography were used by King and Warren (*67H*) in conjunction with a number of other instrumental methods to identify the ultraviolet-sensitive impurities in petroleum-derived napthalene. By combining liquid-solid chromatography with capillary gas chromatography, Chapman and Kuemmel (*27H*) were able to characterize the high molecular weight internal olefins produced by the isomerization of dodecene and octadecene. The olefin mixtures were first separated into cis and trans fractions by liquid chromatography on silver nitrate–impregnated alumina. The bond-position isomers in these fractions were then determined from capillary column retention data along with data generated by oxidative cleavage followed by rechromatography.

Spectroscopic methods continue to play an important role in the characterization and analysis of petroleum hydrocarbons. Infrared spectroscopy was used by Nel'son and Stepanova (*96H*) to analyze the C_5 fraction resulting from the dehydrogenation of isopentane to isoprene. 1,3-Pentadiene had to be determined colorimetrically or chemically using a separate aliquot of sample. The infrared absorbances of the other olefins were then corrected for its presence. An accuracy of $\pm 5\%$ is claimed. Ishida, Uemura, and Kamijo (*59H*) report the use of an infrared method to determine the 1-olefin content of mixtures of octadecenes. The infrared spectra from 5 to 15 microns of 20 cyclopentane and 15 naphthalene derivatives have been recorded by Petrov and Osityanskaya (*101H*). The spectra were used for the partial characterization of various fractions isolated from a cracking oil. Pliev and coworkers (*105H*) provide details of an infrared method for the determination of total aromatics in products from the catalytic dehydrocracking of paraffins. The method is based upon measurement of the intensity of the characteristic absorption band at 1600 cm^{-1}. Infrared spectroscopy has been used successfully by Cornu, Bousquet, and Mantegazza (*32H*) to determine the concentration of 2,6-dimethylnaphthalene in admixture with other dimethylnaphthalenes. The method depends upon measurement of the intensity of the deformation band at 29.4 microns in the cesium bromide region. The presence of other dimethylnaphthalenes, hexamethylbenzene, or diphenylmethane does not interfere with the analysis. The infrared spectra of a number of C_{24} alkyl-aromatic hydrocarbons have been described in detail by Teterina and Petrov (*139H*). They regard these compounds, which are structural analogs of cyclano-aromatics, as models for the hydrocarbons present in the viscous fractions of petroleum. Berthold (*14H*) has described an infrared method for the determination of naphthenic carbon atoms in petroleum products. It is based upon an empirical logarithmic relationship between the extinction coefficient of the band at 970 cm^{-1} and the naphthenic carbon content. It can be applied to products of both high and low aromatic content and gives results in good agreement with values obtained by the n-d-M method. Berthold and Roesner (*15H*) also report the derivation of an equation and nomogram that permit the calculation of the number of naphthenic rings, the number of aromatic rings, and the number of chain carbons from infrared data. The results agree well with similar data derived from physical properties when the average number of aromatic rings exceeds one. When the average number of aromatic rings is less than one, the agreement is not satisfactory. In this situation, the authors recommend the application of an empirical equation designed to bring the data calculated from infrared spectroscopy into line with those calculated from physical properties. A microinfrared technique has been used by Estep and Karr (*42H*) to determine the total amounts of *n*-alkanes, 2-methylalkanes, 1-olefins, 2-methyl-1-olefins and/or trans-internal olefins in aliphatic neutral oil fractions separated from a low-temperature bituminous coal tar.

Ultraviolet spectroscopy has been used by Sadykhov, Zhirova, and Vavilova (*118H*) to determine the concentration of butadiene in gaseous products from the dehydrogenation of butene. The method involves a measurement of the intensity of the absorption bands at 225–235 mμ and is simple, rapid, and adaptable to the construction of a process analyzer. Neimark, Kogan, and Bragilevskaya (*95H*) describe an ultraviolet method for the analysis of mixtures of naphthalene, 1- and 2-methylnaphthalene, and benzothiophene.

The use of fluorescence spectroscopy for the analysis of condensed-ring aromatics seems to be gaining in popularity. Aladekomo and Birks (*2H*) report on a study of the fluorescence spectra of solutions of naphthalene and 14 of its alkyl derivatives. Birks and Christophorou (*16H*) also have recorded the fluorescence spectra of solutions of various alkyl derivatives of 1,2-benzanthracene and of dibenzanthracene, perylene, 1,12-benzoperylene, and anthanthrene. A sensitive fluorescence method for the determination of traces of naphthacene in anthracene has been described by Kreps, Druin, and Czorny (*77H*). Less than 1 ppm of naphthacene is easily detected. Fedonin *et al.* (*43H*) have used fluorescence spectroscopy to detect and estimate traces of polycyclic aromatics in reagent grade analytical solvents such as *n*-hexane, *n*-heptane, and *n*-octane. The suitability of fluorescence for the quantitative determination of small concentrations of perylene and 1,12-benzoperylene in complex mixtures has been discussed by Personov and Teplitskaya (*100H*). The fluorescence spectra of high molecular weight aromatic fractions isolated from a Noriisk crude oil have been studied by Chakrviani and Usharauli (*26H*). They conclude that the spectra of some of the fractions indicate the presence of anthracene, 1,2-benzanthracene, 1,2,5,6-dibenzanthracene, 1,2,3,4,5,6-tribenzanthracene and hydrocarbons of similar structure. Van Duuren and Bardi (*38H*) demonstrated that measurement of the fluorescence spectra of aromatics dispersed in KBr pellets is feasible. For some polycyclics the spectra are identical with those obtained using solutions; for others the maxima are shifted to longer wavelengths. The quenching action of pyridine and quinoline on the fluorescence of naphthalene derivatives has been studied by Miwa and Koizumi (*92H*). Schenk and Radke (*123H*) report the results of a similar investigation of the quenching behavior of tetracyanoethylene, trifluorenone and other pi-acceptors. Sawicki, Stanley, and Elbert (*122H*) take advantage of the quenching effect to detect the presence of hydrocarbons containing the fluoranthenic ring.

Nuclear magnetic resonance spectroscopy has been used by Bartz and Chamberlain (*11H*) to determine the structure of branched paraffins and/or the repeating units in predominantly saturated polymers. Charts and figures are given that correlate chemical shifts with chain structure and spectral pattern variations with chain length and structure. Martin (*90H*) has studied the NMR spectra of polycyclic aromatics containing three ortho-fused benzene rings. He introduces a nomenclature to describe the various types of protons that account for the resonances observed.

Mass spectrometer methods for the analysis of light hydrocarbons, mixtures of light hydrocarbons and inert gases, mixtures of inert gases and reactive gases, and of gasolines have been detailed by Zimina, Polyakova and Khmel'nitskii (*157H*). Saalfeld (*117H*) has devised a very simple mass spectrometer technique for determining the aromatic content of hydrocarbon mixtures of the type recovered from activated carbon absorbers. A method for the analysis of petroleum paraffins using standardized mass spectrometer coefficients has been proposed by Polyakova, Khmel'nitskii, and Medvedev (*109H*). It permits the determination of the carbon number distribution of *n*-paraffins, branched paraffins, and monocycloparaffins. These authors also report (*110H*) a mass spectrometer method for the determination of paraffins and mono- through hexacycloparaffins in saturated hydrocarbon fractions of distillate oils. The application of mass spectrometry for the determination of the composition of saturated fractions isolated from Russian low-sulfur, low-paraffin crudes and high-sulfur paraffinic crudes has been described by Mikhailov *et al.* (*91H*). King and Basila (*66H*) have developed a mass spectrometer method that allows determination of the isomer distribution of mixtures of C_{16}–C_{22} phenyl-*n*-alkanes. The data show good agreement with the results of analysis by gas chromatography. A new set of low voltage sensitivities for aromatic hydrocarbons has been compiled by Lumpkin and Aczel (*88H*). Compound types from alkylbenzenes to chrysenes are covered. Lumpkin (*87H*) has combined high resolution mass spectrometry and low voltage techniques to quantitatively analyze the trinuclear aromatic fraction of a high sulfur- and nitrogen-containing crude. Identifications were made by precise mass measurement of the molecule ion and then of the fragment ions as a test of the assumed structure. Tunnicliff and Wadsworth (*142H*) have described a stepwise regression program for the quantitative interpretation of mass spectra that does not require prior knowledge of sample composition. The program mathematically chooses, from a library of spectra, a suitable small group that, when multiplied by proper concentration factors and summed, gives the best least squares fit to the sample spectrum. The library may contain up to 150 reference spectra with data for 110 masses on each. The program has been used successfully for a two-year period on a wide variety of samples.

References to chemical or electrochemical methods for hydrocarbons are largely confined to the analysis of olefins or certain aromatic species. A colorimetric method for the determination of small amounts of acetylene using Ilosvay reagent has been described by Amamehyan and Moroz (*4H*). Siggia and Stahl (*127H*) have proposed a method for the detection of acetylenic compounds that involves formation of the mercuric acetate complex and measurement of the ultraviolet absorption of the addition product at 260 mμ. It was applicable to all compounds having terminal acetylenic groups that the authors tested. However, it failed in some cases when internal acetylenic linkages were involved. A polarographic method for the determination of napthalenes in petroleum products has been described by Lezhneva and Kruglov (*84H*). The total naphthalene content of gas oils can be determined with an accuracy approching 5%. Benzothiophene interferes with the analysis if it is present in concentrations greater than about 5%.

Nonmetal Elements and Compounds

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SULFUR

A review of the chemical and physical methods for concentrating and identifying sulfur compounds in petroleum was prepared by Thompson and coworkers (*153J*). Analytical methods for determining sulfur compounds in petroleum gases were reviewed by Donchenko (*37J*). Techniques for total sulfur by x-ray absorption, with a discussion of their application and accuracy, were surveyed by Servasier (*136J*).

Total sulfur in gases was determined by mass spectroscopy in a method described by Khvostenko and Sultanov (*73J*). Total sulfur in casing-head gases was determined by Radulescu (*128J*) using activated carbon absorption.

Microamounts of organic sulfur were determined by several new techniques. A combustion technique with a colorimetric measurement of sulfur dioxide formed was reported by Dokladalova (*35J*), and by Chen, Tan, and Chou (*26J*). Durbetaki and Alvarez (*39J*) reported a novel method for the determination of 0.2–500 ppm total reducible sulfur based on desulfurization via activated Raney nickel in a specially designed apparatus; the nickel sulfide formed reacts with mineral acids to generate hydrogen sulfide which is absorbed in an alkaline potassium iodate solution; iodine is liberated by acidification, extracted with toluene, and measured spectrophotometrically at 307 mμ. Traces of sulfur in organic and inorganic substances were determined by Takeuchi and Tanaka (*151J*), using catalytic hydrogenation followed by measurement of hydrogen sulfide by the methylene blue method. Microgram quantities of sulfur in low-boiling petroleum distillates were measured by Pitt and Rupprecht (*123J*) in a colorimetric method based on reduction of sulfur by Raney nickel in an oxygen-free atmosphere. Krafft and Wagner (*79J*) discussed the application and accuracy of a method for determination of small quantities of sulfur using Raney nickel reduction and titration of the hydrogen sulfide with mercuric acetate. Traces of sulfur and halogens were determined in a new combustion apparatus described by Pichl (*121J*), with a photometric measurement for halogens and a gravimetric finish for sulfur. Combustion of the sample followed by reduction of the resulting sulfur oxides was recommended in a procedure developed by Takeuchi, Fujishima, and Wakayama (*150J*) for the determination of microamounts of sulfur in organic compounds. Dokladalova, Korbel, and Vecera (*36J*) calcined samples in an open quartz tube at 750° C in a current of air to determine small amounts of sulfur in organic substances.

In determining macroamounts of sulfur in petroleum and its products, Malissa, Machherndl, and Pell (*95J*) found combustion rates to be an important parameter and discussed the relation of sulfur percentage and sample size to combustion rates. To determine sulfur in volatile organic substances, Stoerzbach (*147J*) used a calorimetric bomb with complexometric back-titration of excess barium added to the ignition products. A rapid method for determination of sulfur, adaptable for the measurement of halogens and metals, which was developed by Barat and Laine (*8J*), uses a special stainless steel injection burner, and titration of the sulfuric acid with barium chlorate. The Schoniger method was modified by Farley and Winkler (*46J*) to provide a flow of oxygen to the combustion bottle, the products from which were passed into a Wickbold absorber for analysis. The Société Nationale des Pétroles d'Aquitaine (*142J*) published a method for the continuous determination of sulfur in gas streams; the method employs catalytic oxidation followed by conductometric determination of the sulfur dioxide formed. In'kova, Makarov, and Klement'eva (*63J*) propose a rapid method for sulfate determination which uses barium chloride and potassium chromate. Total sulfur determination by reduction to hydrogen sulfide was proposed by Krumbholz and Jaeckel (*81J*) who reduced the sulfur catalytically; small amounts of hydrogen sulfide were determined by the methylene blue method, and larger amounts measured by iodometric titration.

Methods employing x-ray and gamma-radiation absorption were discussed by Gorski and Grabczak (*52J*) who compared several techniques for determination of total sulfur in organic products. Servasier (*136J*), who used iron-55, tritiated zirconium, or tritiated titanium as radiation sources, measured sulfur in petroleum gases. The sulfur impurities in naphthalene were measured by Maklakov and coworkers (*94J*) using iron-55 as the radiation source; Matsushima (*101J*) used a similar method to determine sulfur in benzene and heavy oil. Pyrah (*127J*) recommended x-ray absorption using a titanium target and a Geiger tube for quick determination of sulfur in heavy distillates and residual fuels. Improved accuracy was obtained in sulfur analyses by Lyast and Vshivtsev (*92J*) when two radiation sources were used, one being maintained as a reference

A scheme of analysis for determining types of sulfur compounds in petroleum products was the subject of several papers. Koch and Paul (*78J*) presented a scheme for the analysis of sulfur compounds in petroleum gases. A method for the group analysis of sulfur compounds in low-boiling petroleum distillates was developed by Maskova and Loipersbergerova (*100J*). The characterization of the sulfidic structures in petroleum products was described by Prinzler (*126J*). The sulfur compounds in transformer oils were characterized in a procedure reported by Krol, Rozhdestvenskaya, and Kucheryavaya (*80J*). A discussion of methods for the group analysis of sulfur compounds in petroleum was presented by Luk'yanitsa (*90J*). A colorimetric method for determination of disulfides, developed by Cuzzoni and Lissi (*33J*) distinguished between aliphatic, heterocyclic, and aromatic disulfides. Free sulfur, hydrogen sulfide, thiols, sulfides, and disulfides were determined qualitatively and quantitatively in a method recommended by Rubinshtein and Krivova (*132J*). Microwave spectra were used by Pozdeev and coworkers (*125J*) for qualitative analysis of complex mixtures of organosulfur compounds.

Corrosive sulfur was determined by oscillographic polarography in a method described by Kiselev (*74J*). Total and corrosive sulfur in insulating oils were determined by Rey and Sutter (*130J*) using a combination of metallic mercury treatment and combustion.

Hydrogen sulfide was determined by Baranenko and Krivosheeva (*7J*) using cadmium acetate absorber solution and back-titration of excess cadmium, thus avoiding interference from olefins. Hydrogen sulfide in gases was measured by Shul'gina, Arutyunova, and Blyumshtein (*138J*) using cadmium acetate absorption and iodometric titration. Low concentrations of hydrogen sulfide were determined by Malkova and Radovskaya (*96J*) by colorimetric measurement of the blue complex formed with ammonium molybdate. LeRosen (*86J*) determined hydrogen sulfide and thiols in alkaline solution by gas chromatography using acid-coated Chromosorb ahead of the analytical column. A mixture of gases containing hydrogen sulfide was analyzed by Staszewski, Pompowski, and Janák (*145J*) using gas chromatography; results from the use of several stationary phases were reported. Hodges and Matson (*59J*) reported the results of using combinations of two supports and four stationary phases in the gas chromatographic analysis of a mixture containing carbon dioxide, carbon oxysulfide, hydrogen sulfide, carbon disulfide, and sulfur dioxide. A qualitative potentiometric determination of hydrogen sulfide in jet fuels, reported by Englin and coworkers (*42J*), established detection limits as low as 10 ppb. Jacobs, Braverman, and Hochheiser (*66J*) determined hydrogen sulfide in the air in the parts-per-billion range using an alkaline cadmium hydroxide absorber. Huygen (*62J*) collected samples of hydrogen sulfide from the air using filter paper impregnated with potassium zincate; efficiencies at various temperatures and humidities were reported. Measurement of the fluorescence of a fluorescin dye before and after its reaction with hydrogen sulfide in air samples was used by Andrew and Nichols (*3J*) to determine concentrations as low as 0.5 ppb. Microamounts of hydrogen sulfide in petroleum waters were determined by Nedorost and Paralova (*109J*) by photometric measurement after reaction with *p*-aminodimethylaniline. A patent was issued to the Union Carbide Corp. (*156J*) for equipment designed for continuous polarographic determination of low concentrations of hydrogen sulfide in nitrogen streams.

Thiols in gasoline were determined by Bloembergen and Vermaak (*16J*) using alkaline extraction and gas chromatography. Brand and Keyworth (*22J*) identified and estimated amounts of thiols in gasoline by precipitation with silver nitrate followed by regeneration with hydrogen sulfide and resolution by gas chromatography. A procedure for amperometric titration of thiol groups with the rotating platinum electrode was described by Nedić and Berkeš (*108J*), and by Romovacek and Holub (*131J*). A potentiometric alkali-acidimetric determination of thiols was developed by Singh and Varma (*139J*); the hydrogen ion was liberated by iodoacetate solution and reacted with excess sodium hydroxide, which was back-titrated with hydrochloric acid. Suchomelová and Zýka (*148J*) used lead tetra-acetate in glacial acetic acid for potentiometric titration of thiols. Thiols in gasoline were determined by Oelsner and Heubner (*114J*) using plumbite extraction followed by complexing with Komplexon(III) and back-titration of the excess Komplexon with a zinc salt. Ellman (*41J*) found that when a thiol was treated with a reagent with a bis (substituted-4-nitrophenyl) disulfide structure a brilliant yellow color developed which was proportional to the concentration of the thiol.

Seventeen aliphatic and aromatic sulfides, plus other sulfur compounds, were studied by Ratovskaya and Gavrilova (*129J*) using anodic derivative polarography. Thompson and coworkers (*152J*) evolved a systematic procedure of concentration and identification of sulfur compounds in petroleum fractions, and identified a group of chain sulfides in the distillate boiling from 111° to 150° C. Disulfides were determined polarographically by Obolentsev, Gavrilova, and Bulatova (*112J*), and studies of the interference caused by the presence of elemental sulfur and thiols were reported. The ultraviolet absorption spectra of 10 sulfides, 2 disulfides, and 3 thiophenes were reported and discussed by Obolentsev and Lyubopytova (*113J*). A novel qualitative test for organic compounds containing bivalent sulfur, based on the color development with bromine vapor in the presence of aniline, was described by Bayfield, Clarke, and Cole (*9J*).

Organic sulfur compounds were separated chromatographically, converted to sulfur dioxide in a flame ionization detector and determined conductimetrically by Cowper (*31J*). A sulfur-specific microcoulometric cell was described by Adams and Jensen (*1J*) for measurement of sulfur compounds from gas chromatographic separations. The sulfur compounds in petroleum samples were determined by Martin and Grant (*98J*) using gas chromatography with microcoulometric sulfur detection.

Thiophene was determined polarographically by Jaworski and Bogaczek (*67J*) in a procedure designed to minimize interferences. Martin and Grant (*99J*) developed a method for thiophene determination based on the decomposition of nonthiophenic sulfur over alumina at 500° C, followed by gas chromatography with microcoulometric detection of sulfur. The mass spectra of 23 alkylthiophenes were obtained by Foster and coworkers (*47J*), who discussed the correlations of the spectra with compound structure.

Carbon disulfide in benzene was determined by Grant and Vaughn (*53J*) using a gas chromatograph with an electron capture detection system. Low concentrations of carbon disulfide in benzene were measured by White (*161J*) and by Maklakov and Gorelov (*93J*), using ultraviolet spectrophotometry.

The measurement of the amounts of sulfur oxides in the atmosphere received increasing attention. Stephens and Lindstrom (*146J*) proposed a spectrophotometric determination based on the reaction of the sulfur dioxide with ferric iron and 1,10-phenanthroline. Sulfur dioxide and sulfur trioxide were determined by Bond, Mullin, and Pinchin (*18J*); the gas sample was passed over crystalline oxalic acid where sulfur trioxide reacted to form carbon dioxide and carbon monoxide which were resolved gas chromatographically. A tracer technique was used by Urone, Evans, and Noyes (*157J*) to evaluate colorimetric and conductometric methods for sulfur dioxide analysis. Lisle and Sensenbaugh (*89J*) proposed modifications in the method for determination of sulfur trioxide and acid dew points in flue gases. Studies of color enhancement and stability in the spectrophotometric determination of sulfur dioxide in air were carried out by Scaringelli, Saltzman, and Nieman (*133J*), and by Huitt and Lodge (*61J*). Omichi (*116J*) improved the procedure for the colorimetric determination of atmospheric sulfur oxides by lead dioxide cylinder method. Sulfur dioxide in air was measured by the change in the impedance of the sample when the sulfur dioxide was absorbed in aqueous acidified hydrogen peroxide solution, as reported by Leong, Dowd, and MacFarland (*85J*). Strips of filter paper impregnated with potassium carbonate were used by Fukui (*49J*) to collect samples of sulfur dioxide, nitrogen dioxide, and chloride ion from the atmosphere; conventional methods of analysis were then employed. Ammonia interference in the acidimetric determination of sulfur dioxide in the atmosphere was studied by Paccagnella (*118J*); the interferences caused by the presence of nitrogen dioxide were investigated by West and Ordoveza (*160J*), and by Pate and coworkers (*120J*). Hesse (*57J*) developed a special packing for a sulfur dioxide detection tube.

NITROGEN

A method for classifying nitrogen compounds in petroleum into five different types—strongly basic nitrogen, three types of weakly basic nitrogen, and nontitratable nitrogen—was developed by Okuno, Latham, and Haines (*115J*). The method involves titration of the crude oil with perchloric acid in acetic anhydride solution before and after reduction with lithium aluminum hydride. Prior separation or concentration of the nitrogen compounds is not required.

Bender, Sawicki, and Wilson (*11J*) developed a method for the fluorescent spot test detection and spectrophotometric determination of carbazoles and polynuclear carbazoles. Tetraethylammonium hydroxide gave a more stable fluorescent color and fluorescence spectra than reagents previously used. A method was developed by Snyder and Buell (*141J*) to characterize and routinely determine certain types of nonbasic nitrogen compounds in high-boiling petroleum distillates. Ion exchange, linear elution adsorption chromatography, and ultraviolet spectrophotometry were used for determining indoles, carbazoles, and benzocarbazoles in straight run, heavy gas-oil distillates.

The composition of the neutral nitrogen compounds in Russian crude oils was investigated by Bezinger, Gal'pern and Abdurakhmanov (*15J*). Using potentiometric titration and infrared spectral analysis, the authors found that 85–100% of these neutral nitrogen compounds were disubstituted aromatic amides. Copelin (*29J*) identified 2-quinolones in the heavy gas-oil fraction of a California crude oil. These compounds comprise 0.4 weight % of the gas-oil and 0.05 weight % of the crude oil. Latham, Okuno, and Haines (*84J*) reviewed the literature on the nonbasic nitrogen compounds in petroleum; pyrroles, indoles, carbazoles, benzocarbazoles, phenazines, benzonitriles, and amides have been identified in petroleum stocks.

Several investigators modified the Dumas method for determining nitrogen. Farley, Guffy, and Winkler (*45J*) used a Coleman analyzer for the automatic analyses of lubricating oils and additives at the 20-ppm level; the nitrogen compounds were adsorbed on alumina, which was then analyzed. Mitsui (*105J*) developed an improved nitrometer that is operated without a leveling bulb and has a large capacity for potassium hydroxide solution. A simplified apparatus which permits rapid and accurate nitrogen analysis by the Dumas method was described by Tuchscheerer (*155J*); the advantages of the method are that the potassium hydroxide solution cannot be drawn back into the combustion section, the liquid surface has constant absorption characteristics, and the time required for analysis is reduced without loss of accuracy. Pippel and Römer (*122J*) described a modified Dumas micromethod for determining nitrogen in compounds that normally do not react readily with carbon dioxide during combustion; alkali-free nickel oxide was used as the catalyst. Otea (*117J*) used sodium hydrogen carbonate as the source of carbon dioxide in the method. Inoue, Sayama, and Takata (*65J*), in determining high percentages of nitrogen, used 60- to 100-mesh copper oxide and maintained the stationary furnace at 900° to 975° C.

Weitkamp and Korte (*159J*) developed a fully automatic apparatus for the combustion analysis of carbon, hydrogen, and nitrogen in 0.1- to 0.9-milligram samples. A method to determine carbon, hydrogen, and nitrogen in samples in the 0.01- to 1.0-milligram range was developed by Koch and Jones (*77J*).

The applicability of the Kjeldahl procedure for estimation of nitrogen in heterocyclic ring compounds was studied by Mookherjea (*106J*) who found that the digestion period could be reduced two to four hours by using a combination of vanadium pentoxide catalysts with selenium. Kuehnert and Bauer (*82J*) discussed the advantages of using boric acid as the absorbing solution during the distillation step of the Kjeldahl procedure. Lunt (*91J*) used an alkaline stannite to reduce nitro groups and make the nitrogen available for Kjeldahl determination.

Chumachenko and Mukhamedshina (*27J*) determined nitrogen by heating the organic materials with metallic potassium in a steel bomb at 1000°, then extracting with ethanol. Nitrogen in the extract was converted to tetracyanonickel anion with nickel sulfate, and excess nickel was titrated complexometrically with a nickel-complexing reagent.

A microcombustion technique developed by Norris and Flynn (*111J*) to determine nitrogen in petroleum down to the 1-ppm level involves burning the sample in an oxygen atmosphere over platinized asbestos; the nitric oxide formed is treated with sulfanilic acid and 1-naphthylamine to produce a red dye, whose absorbance at 535 mμ is compared to a calibration standard. Trutnovsky (*154J*) automated a previously described microcombustion method for determining nitrogen in organic compounds.

Klemm and Airee (*75J*) investigated the geometry of adsorption of heterocyclic nitrogen compounds by gas–solid adsorption chromatography. Columns of alumina and quinolidine-treated alumina were used to obtain retention times and heats of adsorption for a number of nitrogen compounds.

Baker, Lachman, and Corwin (*5J*) separated a number of porphyrin compounds by column-partition chromatography. A column consisting of dimethylsulfoxide on a silica support with cyclohexane as the mobile phase was used to make the separation.

OXYGEN

The Unterzaucher procedure for the determination of total oxygen was the subject of several studies. Schoeniger (*135J*) summarized 10 years' experience with the titrimetric microdetermination by the Unterzaucher method. Mandrykina (*97J*) suggested removal of sulfur from the pyrolysis products before conversion to carbon monoxide; sulfur is removed by passage over silver at 700–720° C. Mietasch and Horacek (*103J*) preferred hydrogen to nitrogen as carried gas, particularly when sulfur was present, because of ease of removal of hydrogen sulfide. Campiglio (*24J*) suggested several improvements in the Unterzaucher procedure. Kuznetsova, Stolyarova, and Dobychin (*83J*) pyrolyzed the sample at 900° C in an atmosphere of helium in the presence of nickel-containing carbon black and determined carbon monoxide by gas chromatography. Boys and Dworak (*19J*) pyrolyzed in the presence of carbon, eluted the pyrolysis products through a molecular sieve column with helium for separation of the carbon monoxide, which was measured by a katharometer. Goetz (*51J*) decomposed a 2- to 30-mg sample in a quartz vessel at 1120° C and, using hydrogen as a carrier gas, swept the products over carbon to a chromatographic column for measurement of carbon monoxide.

After examination of several modifications of the Scheutze–Unterzaucher method, Belcher, Davies, and West (*10J*) suggested pyrolysis over platinized carbon at 900° C, use of copper and soda asbestos to remove interfering gases, and gravimetric determinations after conversion of carbon monoxide to carbon dioxide with Scheutze reagent at room temperature.

An automated instrument for compounds dissociable below 900° C was reported by Ebeling and Marcinkus (*40J*); the sample was pyrolyzed at 900° C in an alumina reaction tube in the presence of platinized carbon; after removal of the halogens and the sulfur, the carbon monoxide was oxidized to carbon dioxide and determined gravimetrically. The oxidation of carbon monoxide and other pyrolysis products by iodine pentoxide and anhydroiodic acid was studied by Kainz and Scheidl (*70J*, *71J*).

Other approaches to the determination of total oxygen included a method by Frazer (*48J*) which involved mixing the sample with an organic reducing agent such as phenyl thiourea and reacting in a bomb at 1050° C; the volumes of hydrogen and carbon monoxide are then measured. Berezkin, Mysak, and Polak (*13J*) suggested converting the oxygen to carbon monoxide by reaction with carbon black, hydrogenating the carbon monoxide on nickel to methane for detection, using a gas chromatograph with a flame ionization detector. Yoshida and Goshi (*162J*) studied the sources of error in the activation analysis by fast neutrons; nitrogen-16 produced by exposing oxygen to 14-Mev neutrons was measured. Mott and Orange (*107J*) suggested reducing the beam and target effects on the precision of activation analyses with fast-neutron generators by using a defocused D beam with beam aperture and by rotating the sample and comparator around an axis parallel to the beam axis.

Oxygen dissolved in liquids or mixed in gases continued to receive attention. Dowson and Buckland (*38J*) described a process in which the oxygen concentration is continuously determined in a flowing fluid medium; the apparatus consists of a measuring sensor responding to oxygen, an oxygen-absorbing galvanic cell with a silver wire cathode and an anode consisting of finely divided cadmium, which is arranged in a tube filled with an inert carrier gas. A patent by Courtaulds Ltd. (*30J*) described a method for continuous detection of high oxygen concentration in combustible gases; the mixture is brought in contact with a high temperature element, thus inducing a reaction between oxygen and the combustible gases in the mixture; the temperature of the gases is taken after their contact with the element. Hersch (*56J*) patented a trace analyzer which consists of two conductive electrodes contacting a nonconducting porous diaphragm between them; the cell is filled with an electrolyte such as 5*N* potassium hydroxide; an external electric current, sufficient to cathodically reduce the oxygen, but insufficient to decompose the water is applied during use and the current is measured for the determination. A device for continuous measurement of dissolved oxygen in organic liquids and in water was developed by Capuano (*25J*); it involves a thallium and calomel electrode pair; the oxygen, in the presence of water, reacts to form thallium ions which are reduced at the thallium electrode. Collins and Hoenig (*28J*) reported an oxygen detector, which is sensitive to oxygen at 10^{-11} torr, that depends upon the increase in work function of a tungsten filament when chemisorption of oxygen occurs. Paris, Gorsuch, and Hercules (*119J*) developed a procedure for the titration of uncombined oxygen and antioxidants which involves potentiometric or photometric titration with 2,4,6-tri-*tert*-butylphenoxy free radicals.

Several workers determined water in hydrocarbons by its reaction to produce hydrogen. Starshov and Galeeva (*143J*) passed light hydrocarbon vapors through a tube of calcium hydride; the hydrogen produced was separated from the light hydrocarbons using molecular sieves and the hydrogen volume measured. An automatic microanalyzer described by Liebetrau, Daehne, and Mohnke (*87J*) is based on the reaction of water with calcium hydride; the resulting hydrogen reacts with palladium dichloride to give hydrogen chloride, which is absorbed in water and determined by measuring the electrical conductivity, the pH, or by titration. Berezkin and coworkers (*12J*) determined traces of water in olefins by the quantitative chromatographic determination of hydrogen evolved in the reaction of the water with a solution of sodium aluminum hydride in diethylene glycol dimethyl ether. Starshov and Voevodkin (*144J*) determined traces of water in gases and liquids, passing the sample through a column filled with sodium and determining the resulting hydrogen by gas chromatography.

Modifications of the Karl Fischer technique for the determination of water continued to appear. Archer and Jeater (*4J*) suggested using *N*-ethylpiperidine as the catalyst, detecting the end points with a polarized electrode system applied to a pH meter, and making 5-minute drift measurements after completion of a titration. Schober and Strittmatter (*134J*) found direct Fischer titration by the deadstop method unsuitable for insulating oils with less than 20 ppm water and suggested an indirect nitrogen-purge Fischer method. Gruess (*54J*) described an automatic micropipet for the simple and exact determination of the standard strength of Karl Fischer reagent solutions.

Small amounts of water were determined by Polyakov (*124J*) using the reaction with ethyllithium to form lithium hydroxide and ethane followed by either fixing the excess ethyllithium with vanadium pentoxide and determining the reacted ethyllithium, or by measuring the amount of liberated alkali after taking up the excess ethyllithium in a benzyl chloride–tetrahydrofuran mixture. Huebl, Schatzberg, and Glassman (*60J*) determined small amounts of suspended water by mixing water-soluble bicarbonates and organic acids with the water-insoluble organic liquid in a gas-tight bomb and measuring the pressure increase due to release carbon dioxide. Fallah (*43J*) described a manometric method for determining water and dissolved gases in mineral oil in which water is condensed at −78° C and the amount (down to 0.5 ppm) is determined by vapor pressure; noncondensible gases are measured (down to 0.25 ml/liter) volumetrically; oxygen is then eliminated with white phosphorus; and the oxygen content calculated from the pressure difference. Medveczky and Sebestyen (*102J*) prepared an indicator reagent capable of detecting about 0.002% water by adsorbing bromochlorophenol blue on barium sulfate and adding sodium diethylbarbiturate. Voelker and White (*158J*) developed equipment to monitor the moisture content of refinery streams continuously by detecting changes in the electrical capacitance of a remote cylindrical condenser immersed in the stream; the condenser is packed with a granular desiccant, which permits measurement of less than 1 ppm of dissolved moisture.

Szalay, Varallyai, and Porzsolt (*149J*) suggested the Szabo-Nagy apparatus for the determination of the water content in mineral oils by the measurement of the dielectric constant. Klugman (*76J*) studied the accuracy of electronic apparatus for moisture determination and suggested several improvements. In connection with a study of the bubble column slurry reactor, Farkas (*44J*) developed a new method for determination of hydrocarbon-in-water solubilities which requires commonly available equipment.

Carbon monoxide may be determined in gas mixtures by means of its color reaction with bis-(1,10-phenanthroline)-palladium chloride and subsequent measurement of the extinction of 550 mμ, according to a paper by Burianec and Burianova (*23J*). Mirzayanov and Berezkin (*104J*) determined traces of oxygen and carbon monoxide in propylene chromatographically on a molecular sieves column. Jobahazi (*69J*) compared absorbents used for carbon monoxide in the Orsat apparatus and suggested a 2-pipet system, consisting of a sulfuric acid–cuprous oxide suspension and ammoniacal copper solution. Doering and Mueller (*34J*) determined carbon oxysulfide in synthesis gas by the iodine–azide reaction after removal of carbon dioxide and hydrogen sulfide.

Mixtures of monohydric phenols (C_6–C_8) were analyzed by Adlard and Roberts (*2J*) by silylation of the phenols before gas–liquid chromatography in a capillary column. Lindeman and Nicksic (*88J*) suggested the characterization of individual alkylphenols by proton magnetic resonance after acetylation. Crump (*32J*) resolved mixtures of simple alkylphenols from kerosine; the phenols, coupled as *p*-nitrophenylazo dyes, were separated by two-dimensional chromatography on silica gel impregnated with sodium hydroxide. Skrynnikova and Matveeva (*140J*) separated phenols by steam distillation and bromination; the end of bromination was determined amperometrically. Zubkova and coworkers (*163J*) used thin layer chromatography on alumina for the separation and identification of alkylphenols and aminophenols in mineral or synthetic oils.

The determination of the phenols used as additives or solvents was the subject of several papers. Berthold and Drescher (*14J*) determined 2,6-di-*tert*-butyl-*p*-cresol in turbine oils by infrared spectroscopy, measuring the intensity of the 1168 cm^{-1} band of the hydroxyl group on the tertiary carbon atom. The same additive was determined by Braithwaite and Penketh (*20J*) using a procedure involving preliminary separation by distillation with methanol followed by analysis based on colorimetry, infrared spectroscopy, or gas-liquid chromatography. Braithwaite, Penketh, and Underwood (*21J*) separated 2-methyl-6-*tert*-butyl-*p*-cresol from gasoline by column chromatography, oxidized with potassium ferricyanide, coupled with diazotized *p*-nitroaniline, and determined spectrophotometrically. In'kova and Piyunkina (*64J*) described a method for the determination of phenol and alkylphenols in additives. Norikov and Vetchinkina (*110J*) separated various *tert*-butylphenol additives gas chromatographically using Apiezon N as the stationary liquid phase. Geller, Shevchenko, and Rastorguev (*50J*) suggested two methods for the determination of phenol and cresol used in solvent extractions; one method is based on removing the solvents from the oil with aqueous sodium carbonate and the other removes them with steam distillation; in both methods phenol and cresol concentration is determined colorimetrically.

Mixtures of petroleum acids were separated by Bock and Behrends (*17J*) by esterifying and then separating the methyl esters by distillation and gas chromatography with polyethylene glycol and 2-ethylhexyl sebacate as the stationary phase. Jenkins (*68J*) described the determination of carboxylic acids and esters in petroleum distillates and residues by infrared spectroscopy; this paper includes a survey of recent reports in which infrared spectroscopy was applied to petroleum. Hill and Hill (*58J*) separated mixtures of free aromatic acids by gas chromatography with hexamethyldisilazane-treated Chromosorb W coated with 15% Carbowax 20M. Harlow and Morman (*55J*) described automatic ion exclusion–partition chromatography for acids involving separation on a column of sulfonic acid exchange resin and continuous automatic titration to pH 8.5.

The oxygen-containing compounds resulting from the pyrolysis of light oil were isolated and characterized by Shor, Chertkov, and Gol'din (*137J*) using chromatography on KSM silica gel. Keil and Rentrop (*72J*) found that the chief products of mineral oil aging (acids, esters, lactones, and alcohols) could be determined by infrared spectroscopy; the undiluted oils, after removal of sludge, were examined in the regions 1700 to 1800 and 3100 to 3600 cm^{-1}. Banerjee and Budke (*6J*) extended a spectrophotometric method for the determination of peroxide for use with unsaturated compounds.

Metals in Oil

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Among the instrumental methods which recently have evolved, activation analysis and atomic absorption spectrometry are gaining popularity among analysts for the determination of a variety of chemical elements in petroleum and related materials. In the 3.25-year period covered by the last two reviews, only two papers in each one of the above-mentioned techniques were abstracted, while in the 2-year period covered by the present review, six papers dealing with atomic absorption and eight involving activation analysis were published. The following papers are arranged by analytical methods.

Activation Analysis. Fast neutron activation associated with γ-ray spectrometry and computer programming has been applied by Hull (*23K*) to the determination of nitrogen, phosphorus, chlorine, zinc, and barium in lubricating oils. The use of computer data reduction in neutron activation analysis has been described by Steele (*44K*). Oxygen in hydrocarbons and barium in lubricating oils were determined by Gibbons (*13K*) as examples of the application of 14-m.e.v. neutron generators in activation analysis. Additives in lubricating oils have been indirectly measured by determining barium, phosphorus, iron, sulfur, and chlorine by fast neutron activation (*12K*). Phosphorus, sulfur, and zinc were also determined in motor oil additives by radioactivation analysis by Nagy and Szokolyi (*35K*). Nuclear reactor irradiation followed by chemical separation and γ-ray spectrometry has been used by Malvano (*32K*) to analyze crude oils, crude asphalts, and polyphenyls for traces of nickel and cobalt. A similar approach was successfully applied by Colombo *et al.* (*7K*) to the systematic analysis of 11 metals in petroleum products. Ten elements in the parts-per-million and parts-per-billion range were identified and measured in some Italian oils and asphalts by Ciuffolotti *et al.* (*6K*) using an elaborated reactor irradiation technique.

Atomic Absorption Spectrometry. This analytical technique has been used to rapidly determine traces of iron, copper, and nickel in gas oil feedstocks (*1K*); and nickel, down to 0.05 parts per million in catalytic cracking feedstocks by Trent and Slavin (*46K*). Several problems affecting the evaluation of lead in gasolines, especially the chemical nature of the lead standard, have been studied by Dragnall and West (*9K*). A procedure for atomic absorption determination of lead in gasoline has been described by which lead can be evaluated with an accuracy of about 1% of the amount present (*47K*). Atomic absorption spectrometry was applied to the determination of wear metals in lubricating oils: copper, chromium, iron, lead, and silver in concentrations ranging from 0–100 ppm have been determined by Burrows *et al.* (*5K*) in used lubricating oils with good reproducibility and without interference problems. The rapid determination of iron, copper, silver, magnesium, chromium, tin, lead, and nickel in lubricating oils has been reported by Means and Ratcliff (*33K*). Finally a procedure has been described by Sprague and Slavin (*43K*) for the rapid analysis of used aircraft lubricating oils for nickel, chromium, lead, copper, silver, and magnesium, at the rate of 14 seconds per sample per element, with a precision of about 5%.

Emission Spectrometry. A technique was described by Kahonitz (*26K*) for emission spectrographic determination of a variety of metals in liquid mineral oil products, using a rotating electrode. An indirect determination of vanadium and nickel has been reported by Petho (*36K*) in which a pair of graphite electrodes are immersed in the oil, and heated to 1000° C before being excited by a high tension spark. Nine elements, including nickel and vanadium, have been estimated in petroleum and bitumens by Berman (*2K*) first combusting the sample with a buffer mix and then exciting the ashes in a 5-ampere d.c. arc. Determination of trace elements in crude oils has been achieved by

Korosteleva *et al.* (*27K*) first transforming the oil into coke and then exciting it with a 20-ampere d.c. arc. Zinc, barium, and vanadium were estimated in petroleum products without preliminary ashing by Hauptman (*19K*), using copper electrodes. An apparatus that can be attached to any spectrograph and that permits the safe spark excitation of petroleum products and flammable liquids has been described by Buncak (*3K*). Crankcase oil contamination was studied by Sokolov (*42K*) who determined iron, lead copper, chromium, silicon, aluminum, and tin after ashing the samples and using nickel as internal standard. The analysis of diesel oil to determine metals worn away by friction from engine parts has been reported (*40K*) using conventional sample handling and spectrographic techniques. To avoid sample ashing, a method has been described by Pforr *et al.* (*37K*) for zinc and calcium in compounded motor oils in which the sample is sprayed into the spark gap by means of a rotating carbon disk. Wear metals—iron, aluminum, lead, copper, chromium, and silicon—were determined in locomotive diesel oils by Sokolov (*41K*). Boron in heavy hydrocarbon mixtures has been measured by Giorgini (*15K*) with no previous ashing by diluting the sample in a graphite matrix. Nickel, copper, vanadium, and iron in the parts-per-billion range were determined in ashes from catalytic feedstocks by Hoggan *et al.* (*21K*), using cobalt as internal standard and a direct-reading spectrometer. A general computer program was developed by Tunnicliff and Weaver (*48K*), which calculates and reports results from the common matrix method of emission spectrographic analysis.

X-Ray. A review covering problems encountered in x-ray analysis, with emphasis on applications to mineral oil products, has been reported by Louis (*29K*). In this paper, x-ray tubes, crystal analyzers, collimators, and characteristics of crystals are critically discussed. Detection limits in mineral oils for elements of atomic numbers between 15 and 82, using x-ray emission analysis, have been described by Louis (*30K*). For elements heavier than chlorine a detection limit below 1 ppm is possible, and between 1 and 3 ppm for sulfur and phosphorus. X-ray emission spectroscopy was applied by the same author (*28K*) to the determination of zinc, barium, calcium, chlorine, sulfur, and phosphorus in lubricating oils. A direct simple channel method for analyzing 0.2–10 ppm of nickel in catalytic cracking feedstocks, in about 23 minutes, with a standard deviation of 0.1 ppm and within 0.1 ppm agreement with chemical results has been reported by Gunn (*17K*). Calcium, barium, zinc, and lead were determined by Burke *et al.* (*4K*) using x-ray spectrography in lubricating oils, white oils, and gasolines. This technique uses a pellet of a suitable reference element in a conventional Phillips cup containing the sample. Catalytic cracking feedstocks have been analyzed by Rowe and Yates (*38K*) using x-ray fluorescence to determine copper, iron, nickel, and vanadium; the samples are prepared by ashing with xylene sulfonic acid. A discussion on the application of x-ray fluorescence spectrometry to the determination of barium, zinc, and calcium in lubricating oils and additives has been reported by Haycock (*20K*). An internal standard technique to eliminate inter-element effects is used.

Miscellaneous. A procedure for ash determination in hydrocarbons that is faster than the ASTM ash analysis D482-63 has been described by Dubeau *et al.* (*8K*). In this method the oil evaporation time is decreased from 90 to 9 minutes by using a slight vacuum. Zinc calcium, and bromine in unused lubricating oils have been selectively estimated by Fisher (*11K*) using complexometric titration with EDTA in an alcoholic–hydrochloric acid extract of the sample. Several procedures for the complexometric determination of zinc and calcium in oils containing additives have been studied, although no satisfactory results were reported by Studeny (*45K*). A gravimetric method by Muzychenko *et al.* (*34K*) for the determination of carbonates in oil additives containing calcium or barium involves treating the samples with sulfuric acid and determining the liberated carbon dioxide. A chemical method for determining barium in lube oils additives concentrates has been extensively tested by several laboratories (*10K*). In this procedure (IP Inorganic Analysis Panel ST-G-3), which is simpler and more rapid than the IP-110, the samples are treated with sulfuric and nitric acids and the barium is weighed as sulfate. Routine photometric analysis for the determination of zinc additives in unused lubricating oils has been carried out by Howard (*22K*) forming the zinc dithizonate complex in a carbon tetrachloride solution of the oil. A pulse polarographic determination of microgram amounts of nickel and vanadium in petroleum stocks has been reported by Gilbert (*14K*) which can be effected on a single polarogram in a short time, after chemically treating the samples. Vanadium contained in petroleum as a porphyrin complex was determined by Ishii (*25K*) using an a.c. polarographic technique after extracting the vanadium with a $KSCN\text{-}H_2\text{-}SO_4$ solution. Simultaneous polarographic behavior of nickel, cobalt, iron, and manganese has been studied in mineral residues of crude oils and a simultaneous determination of nickel and cobalt in several Romanian oils has been described by Serbanescu (*39K*). A review covering the subject of polarographic analysis has been written by Ishii (*24K*) containing 57 references. Potentiometric acid–base titration has been applied by Lyashenko *et al.* (*31K*) to the determination of calcium salts in lubricant oil additives. Two analytical procedures have been described by Hammerich and Gonderman (*18K*) to analyze combustion residues from engines. In one of them, lead, barium, and silicon are gravimetrically determined after insolubilization with acids; iron, zinc manganese, molybdenum, copper, and phosphorus are spectrophotometrically determined and a potentiometric method is used for chlorine, bromine, and boron. In the other procedure by Grant (*16K*), silicon and aluminum are colorimetrically determined, calcium and magnesium by complexometric methods and chloride by potentiometric titration.

Catalysts

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Acidity. The measurement of acid sites by the titration technique using a series of color indicators continues to be used as reported by Bertolacini (*2L*)and Hirschler (*13L*). Spectral absorption and electron paramagnetic resonance measurements of anthracene adsorbed on silica–alumina catalysts have been employed by Karakchiev *et al.* (*14L*) to elucidate protonic and aprotonic acidities. The infrared spectrum of coordinately bonded pyridine has also been utilized by Basila *et al.* (*1L*) and by Parry (*18L*) to differentiate between the types of acid sites. The distribution of acid centers was reported by Strich and Becker (*20L*) by potentiometric titration with potassium methoxide in a non-aqueous system.

Metals. Emission spectroscopy was used by Biktimirova and Baĭbazarov (*3L*) to determine sodium in catalysts, and copper and palladium in sediments and deposits. Townsend (*23L*) reported the application of a fusion-cast disk technique for analyzing catalysts by emission x-ray spectrography.

A modification of the Sen titrimetric procedure for determining nickel has been reported by Danowski and Lewandowska (*7L*). Svajgl (*21L*) devised a colorimetric and a titrimetric method for tracing vanadium in hydrogenation processes. Potentiometric titration with ceric sulfate has been employed by Giuffre and Cassani (*10L*) to determine the V^{+2} and V^{+3} contents of catalysts prepared from vanadium trichloride and aluminum alkyls. And, a complexometric method for molybdenum was devised by Uvarova and Rik (*24L*).

Physical Properties. The measurement of surface area of catalysts utilizing the gas chromatographic technique continues to be reported as by Chu (*6L*), Kuge and Yoshikawa (*16L*), and Liu *et al.* (*17L*) as does the x-ray diffraction technique by Keely (*15L*). Rapid methods using a simple calorimeter has been studied by Taylor (*22L*) and the Haul and Deumbgen apparatus for surface area has been studied by Grundke and Frenzel (*11L*).

Determination of the total metallic area of supported catalysts using chemisorption of hydrogen and carbon monoxide also received further attention by Germain *et al.* (*9L*). In similar type studies, Weigel *et al.* (*25L*) compared the BET and small-angle x-ray scattering techniques for absolute values and other information that could be derived from the data.

A rapid, continuous flow method for the pore size distribution of catalysts has been described by Cahen and Fripiat (*4L*). And Harris (*12L*) discussed the source of error in the calculation of pore size data from nitrogen isotherms.

The density of porous solids by immersion in mercury in a pycnometer has been published by Cartan (*5L*) and a mathematical treatment was developed by Princon (*19L*) for coincidence corrections necessary in determining particle size distribution of catalysts using the Coulter counter.

Miscellaneous. A study of the distribution of coke on catalyst beads as a function of the radius of the beads has been reported by Galimov *et al.* (*8L*); two different techniques for measuring the coke were evaluated.

Physical Properties

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LANGTON AND VAUGHAN (*29M*) studied flow time measurements in Ostwald viscometers tilted out of the vertical; they found variations can be explained as a function of the geometry of the viscometer. An empirical formula developed by Bondarenko (*10M*) correlated rate of slipping of a drop of liquid along a vertical filament with the viscosity of the liquid. Results compared favorably with measured data in an Ostwald viscometer.

Kinoshita and Onoyama (*27M*) postulated that surface active material from solvent refined oil adsorbed onto capillary walls, resulting in reduced effective bore size of the tube for subsequent tests. Flow rate changed gradually with repeated measurements on the same oil. A two-way capillary viscometer measuring bulk viscosity at shear rates from less than 0.1 to over 1,000,000 per second and pressure drop up to 1000 psi was described byTzentis (*55M*). After calibration with Newtonian oils it was also applied to non-Newtonian salt solutions of polyacronitrile.

Lim and Johnson (*30M*) made precise measurements electronically of the time required for commercially available solid steel balls to flow through a known distance in liquid samples. Seibert, Johnson, and Stross (*51M*) adapted falling ball viscometry for more rapid routine use. They found good agreement with capillary viscometry with precision largely determined by the weight range of the balls.

A new constant rate of flow viscometer described by Bianchi, Patrone, and Babini (*7M*) permitted static measurements of dilute polymer solutions over a wide velocity gradient range even at high temperatures and under pressure.

Use of the falling cylinder for non-Newtonian fluids was evaluated by Ashare, Bird, and Lescarboura (*4M*). The non-Newtonian viscosity is estimated by treating the velocity-of-fall measurements by a differentiation procedure.

Billington (*8M*) developed a coaxial cylinder viscometer for use under oscillatory and transient conditions as well as steady-state conditions. The time-dependent solutions to the shear stress *vs.* shear rate relationships were confirmed on a series of Newtonian lubricating oils.

Ramarkrishna (*46M*) made measurements of absolute viscosity by three computational methods based on the rotation of a cylinder suspended in the liquid by a wire. Equations for the velocity distribution and couple when a sphere is rotated in an inelastic non-Newtonian fluid were presented by Bourne (*11M*). He covered their application to the measurement of fluid parameters at low shear rates. Jaffe and Ruysschaert (*21M*) described a recording rotational viscometer with glass-laden Teflon cylinders; accuracy and precision are discussed both for water and benzene.

An extension to power-law fluid of Lewis' theory for rolling ball viscometers was presented by Bryd and Turian (*12M*). They found a correlation of the speed of the rolling ball with fluid density and with physical and geometrical parameters when applied to non-Newtonian flow. A rolling ball viscometer for use at temperatures to 400° C under pressures to 5 kilobar was developed by Harrison and Grosser (*18M*). Pressure medium is compressed argon and data covers glycerol at 75° C.

Marcelin (*36M*) designed a microviscometer consisting of a collar centered around a conical wedge of a very small conical angle where the thickness of the oil film depends on the depth of the wedge in the collar. His data showed viscosity coefficient to vary with film thickness. He suggested (*37M*) the lubricant films consist of monomolecular layers on the solid surfaces, dense stratified layers, then diffuse stratified layers separating the dense layers and the bulk liquid. The effect of the structural changes under varying lubrication conditions is discussed.

A series of equations covering the relationship of pressure and viscosity were developed by Amirbekov (*3M*).

The viscosity of oil emulsions was reported by Muratova and Korobkova (*41M*); they used a rheoviscometer in the form of a balance. The shear stress of oil emulsions was compared with their viscosity by adding weights to a tray attached to the balance arm of the viscometer. At above 40° C, the viscosity did not depend on shear stress and the liquids were Newtonian.

Porter, Klaver, and Johnson (*44M*) developed a recording high-shear viscometer for measurements near a million reciprocal seconds. Accurate measurements were made at shear rates of 500 to 2 million per second within an extended temperature range of −20° to 150° C. Viscosity range varies inversely with the shear rate selected for measurement. The shear-rate range is greater and its upper limit higher than previously reported. A simple flexible viscometer for testing pseudoplastic liquids at pressures as high as 1500 psi was described by Ram and Tamir (*45M*). Equations and nomographs are given for designing viscometers or for selecting operating conditions with an existing one. Freund, Csikos, and Mozes (*16M*) studied anomalous flow behavior of lubricating oils and slack wax at low temperatures. No clear-cut relationship could be obtained between instantaneous shear stress values or shear rates and the corresponding rheological conditions.

A comparison of the (U.K.) National Physical Laboratory and the (U.S.) National Bureau of Standards viscosity scales reported by Daborn (*14M*), showed results agreed with NBS values by 0% to +0.5%. A 10-year test for stability of viscosity of petroleum reference oils was carried out by Weber (*56M*). Samples exposed to daylight showed more viscosity increases than those kept in the dark; little effect resulted from subjecting another series of samples to temperature variations.

An improved equation for converting Engler to Kinematic viscosity was reported by Martens (*38M*). It is recommended particularly for Engler values below 2.5. A circular slide rule which can be used to convert viscosity units between Kinematic, Engler, Redwood, and Saybolt was reported in *Science Lubrication* (*52M*). This nomogram developed by Angleroth may also be used for determining viscosity index and SAE classifications. Nagypal (*42M*) reported a linear relation between the cube root of the viscosity logarithm as expressed in Engler degrees and the ratio of components in mineral oil blends.

Wright (*57M*) reviewed existing scales for expressing the viscosity temperature relationship of commercial lubricants, particularly VI, and proposed a new Viscosity Index extension (VI_E) for use above 100 VI. The new scale eliminates anomalous reverse curvatures and gives approximately constant VI for all numbers of a series. It has subsequently been incorporated into ASTM Method D2270, Calculating Viscosity Index from Kinematic Viscosity.

Zuidema (*58M*) discussed the effect of precise temperature control on viscosity with particular attention to use of viscosity temperature charts described in ASTM Method D341, Standard Viscosity-Temperature Charts for Liquid Petroleum Products. He also covered some aspects of viscosity index and deviations from linearity. "Slope Index," that is the slope common to a family of straight parallel lines obtained by plotting viscosity-temperature relationships for members of a naturally homologous group of mineral oils, was prepared by Roelands, Blok, and Vlugter (*47M*) as a new viscosity temperature criterion. A low slope index corresponds to a high dynamic viscosity index. Saal (*50M*) prepared a theoretical review of the principles underlying the various systems of classification of lubricating oils according to their viscosity-temperature relation.

A method for measuring vapor pressure in the range 10^{-5} to 10^{-1} millimeter Hg using an effusion manometer system has been suggested by Morecroft (*40M*). Vapor pressure is obtained by measuring the recoil force produced by vapor effusing through a small hole into a high vacuum. Bell (*6M*) working in the same area, measured volatility of high boiling petroleum products in lubricating oil range using a saturator containing the sample over which a slow stream of nitrogen is passed. Flame ionization detector technique was used as a measurement of weight of vapor removed from the sample. Brzoska (*13M*) worked on low vapor pressures of diffusion pump oils. The method is applicable where a low flow rate of vapor from sample to vacuum pump can be maintained and regulated. Saturated vapor pressure of lubricants in which the oil or grease sample is vaporized in a bulb in an inert gas atmosphere was carried out by Martynov and Morozova (*39M*). They reported saturated vapor pressure of a grease is practically equal to the saturated vapor pressure of the base oil.

Popov, Tsederberg, and Morozova (*43M*) worked on thermal conductivity of diesel fuel and lubricants using a heated tube with a platinum fiber passing along its axis. Jobst (*23M*) described a nonstationary absolute technique for measuring the thermal conductivity of aliphatic compounds by measuring temperature of a uniformly heated electric wire immersed in the sample. Correlations are presented for water, glycerol, ethanol, gasolines, and paraffins. A simple device for measuring thermal conductivity of liquids with moderate accuracy was employed by Jamieson and Tudhope (*22M*). A steady-state hot-wire method is used with a straight-platinum-wire conductivity cell and temperatures from 0 to 75° C.

Dukhovnyi (*15M*) determined molecular weight cryoscopically with improvements including automatic measurement and recording of crystallization temperature. Kemeleva and Bekturov (*24M*) determined molecular weight of tar separated after oxidizing heavy petroleum residues Osmometric, cryoscopic, and viscosimetric measurements were carried out in benzene solution. A vapor pressure osmometer was used by Kume and Kobayashi (*28M*) to measure molecular weight of polybutadienes.

Lindhe and Thorsson (*31M*) lectured on the aniline point as a measure of the content of aromatics in mineral oils and the aromatic content and hydrocarbon-type analysis of low-boiling mineral oil fractions. Reversing the usual process of determining other characteristics from aniline point, Hollinghurst (*20M*) has prepared nomograms in which the aniline point is given either as a function of viscosity and VI or gravity and viscosity.

Mackle and McClean (*34M*) extended previous work with refinements and improvements of apparatus for measurement of vaporization heats by gas liquid chromatography. Results on thiaalkanes were in good agreement with values obtained by twin ebulliometry as well as other empirical methods.

A single stage centrifugal molecular still was used by Thomas (*54M*) for distillation of lube oils. Results were compared with those of a high vacuum pot still. Kerenyi and Borzsonyi (*25M*) described glass distillation equipment suitable for recording the equilibrium boiling point curve both at atmospheric and reduced pressures. Aleksandrova and Zykov (*1M*) used a 30-plate column for determining true boiling point curves of heavy fractions boiling in the range of 250–550° C, incorporating several automatic features. The importance of intervals of time and temperature increments in interpreting distillation data when using a single stage centrifugal still were discussed by Hickman and Thomas (*19M*). Barrall, Porter, and Johnson (*5M*) developed a micro boiling point technique by differential thermal analysis. Boiling points were obtained either by direct extrapolation of individual thermograms or by bracketing samples between API standard hydrocarbons. The method has been used for high-resolution gas chromatographs. Green, Schmauch, and Worman (*17M*) used a gas chromatographic technique to simulate distillation equivalent to a 100-theoretical plate distillation. This was applied to distillates with boiling points up to 1000° F and to samples containing as much as 85% nonvolatiles.

Bodan and Boichuk (*9M*) developed a rapid method for determination of density of liquid and solid products where refractive indices are measured on binary mixtures. Shraiber and Kukolenko (*53M*) described a method for the precise measurement by pycnometer of density of liquids such as xylene isomers within a broad temperature range.

Russian workers reported on cryoscopic methods for determining purity of aromatics. Mamedova, Akhmedov, and Aliev (*35M*) published work on xylene isomers featuring sealed ampules for avoiding moisture problems. The same group (*2M*) extended their work to doubly sublimed and recrystallized naphthalene. Lyashkevich (*32M, 33M*) studied the influence of several variables on precision and developed a polymicro method applicable to samples of several hundredths of a gram and at a temperature range of between −170° and +350° C.

Ross and Dixon (*48M*) modified previously used techniques in the purity analysis of highly purified material by time-temperature cryometry. Dielectric cryometry was used by Ross and Frolen (*49M*); they suggest this might be adaptable for automatic control of temperature within extremely narrow limits. Kieffer *et al.* (*26M*) used thermal analysis for the determination of the purity of very low melting hydrocarbons with a sensitivity greater than gas chromatographic analysis for trace contaminants.

Analytical Instrumentation

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THE DEVELOPMENT of automatic types of instruments for petroleum analysis and testing continues strongly. Laboratory instrumentation has received the major share of attention; new in-stream analyzers have appeared, and previously developed instruments are finding more numerous and wider application.

Among elemental analyses, automatic determination of carbon, hydrogen, and nitrogen has received greatest attention. These elements are measured with excellent precision by an instrument developed originally by Korte and Weitkamp (*40P*). Takeuchi and Mori (*70P*) have statistically examined many of the factors influencing precision and accuracy of C.H.N. analyses. Francis (*25P*) has reviewed the several commercial automatic microanalyzers for carbon, hydrogen, and nitrogen. Farley, Guffy, and Winkler (*22P*) have adapted one of the commercial analyzers to permit determining nitrogen in lubricating oils and additives at concentrations near 20 ppm. The continuous, simultaneous determination of sulfur and hydrogen in oils is described by Dahn (*17P*), utilizing the attenuation of β-rays from a weak iron-55 source as they travel through the sample. Coggeshall and coworkers (*15P*) developed a process analyzer for vanadium in gas oils, using electron spin resonance; an ingenious flow-through cell makes the in-stream analysis possible. Hoggan (*33P*) has described improvements to the direct-reading spectrochemical analysis for trace metals after ashing samples of charge stocks to catalytic processes. Dwiggins (*19P*) has developed correction methods suitable for automated x-ray emission spectroscopic analyses for elements in organic materials. Munch (*50P*) reviewed in-stream applications of automatic x-ray spectroscopic analyses to process streams.

The continuous measurement of water in petroleum liquids has been described by Bublikov, Klugman, and Stepanenko (*10P*) who utilize dielectric constant in a process instrument; the sample is thermostated and turbulent flow through the measuring cell ensures that the water (0–3%) is dispersed in spherical droplets. Instruments are now commercially available for determining water in gases and vapors semiautomatically in the laboratory or continually in-stream, utilizing King's piezoelectric sorption detector (*38P*); rapid and precise analyses are obtained in the range from zero to a few thousand parts per million. Choice of suitable substrates for coating the piezoelectric crystal allows determining CO_2, SO_2, or SO_3 in gases (*21P*). The analysis of gas mixtures by a combination of automatic instruments employing thermal conductivity, infrared absorption, and paramagnetic-diamagnetic principles has been described and diagrammed by Kohli (*39P*).

Hydrocarbon gases and vapors in air were continuously measured by Lovelock (*45P*), using a device based on the energy in electron volts required to produce an ion-pair in the gas stream; it was applied to relatively high hydrocarbon contents, for example, fuel:air mixtures in the combustible range. Decristoforo (*18P*) described a more sensitive continuous hydrocarbon detector which employs flame ionization to measure concentrations as low as one part per billion. A somewhat similar instrument (*13P*) is suitable for continuous analysis for organic vapors and gases in air or in process streams; its range is from trace amounts to several per cent. A Russian patent (*41P*) covers an automatic instrument for determining the total concentration of aromatic hydrocarbons in a liquid sample by differential absorption of infrared energy: The temperature of the stirred sample in a measuring cell is compared against that of a stirred reference material in an identical cell while both cells are exposed to an infrared source; no precision data are given.

T. R. Mueller and colleagues (*49P*) have developed a recording conductimetric titrator having automatic temperature compensation. Several continuous titrators have resulted from the work of Blaedel and Laessig (*4P*). Metal ions that can be chelated with EDTA have been continuously determined by electrolysis of a novel tubular electrode made of HgEDTA. In another continuous titrator, no burets are needed for determining Fe(II) by titration with Ce(IV) reagent (*5P*); direct readout is obtained by varying the rate of titrant addition to maintain endpoint conditions while the sample is introduced at constant rate. Blaedel and Strohl have described a polarographic cell for the continuous analysis of flowing solutions (*6P*), as well as an instrument and system for continuous electrolysis (*7P*). Automatic titration of acids following separation by ion exchange from a column of sulfonic acid resin has been achieved by Harlow and Morman (*30P*); the eluent is water and the time-integral of titrant volume permits continuous measurement of acid concentration. The use of an automatic in-stream titrator to monitor the addition of TEL/TML while blending gasolines in-line has been described by Skogan (*69P*).

Applications of gas–liquid chromatography (GLC) to analytical problems related to petroleum processes and products were surveyed by Puzniak and Lichtenfels (*64P*). How best to use chromatographs for automatic control of distillation processes was discussed by Oglesby and Hobbs (*52P*), embracing principles of feedback control and giving attention to the design of the sampling systems, selection of sample points, and choice of sampling rate. Examples of installations are given, together with data on economic justifications. A survey by Noebels (*51P*) covered applications of chromatographs to chemical processes and much of his material is applicable to refinery processes, notably distillation. A comprehensive picture is given by Pine (*62P*) of the development and use of chromatographs for in-stream analyses at the parts-per-million level in chemical processing.

The design of sampling systems and the selection of sample-conditioning components has been systematically treated by Barnes (*3P*). A special sampling valve for transferring microliter samples with excellent precision from process streams to an automatic capillary gas chromatograph was devised by Grant (*27P*).

Simulated distillation by temperature-programmed GLC has received widespread attention. An automatic in-stream analyzer was developed by Petrocelli, Puzniak, and Clark (*60P*) to present data corresponding to the ASTM D-86 distillation method. Maier, Bossart, and Heller (*48P*) have described a somewhat similar instrument. The boiling-point distributions of petroleum distillates having end points above 1000°F have been obtained by Worman and Green (*76P*), employing simulated distillation; results are expressed as true-boiling-point curves. The Special Design Office for Automation in Petroleum Refining (USSR) have developed two new gas chromatographs (*2P*) for the analysis of gasoline, kerosene and other petroleum products. Kusy and Prochazka (*43P*) have described in detail the use of gas chromatographs equipped with electronic integrators for automatic control of butane and certain aromatic hydrocarbons.

Other instrumental developments based on GLC include a portable instrument for field analysis of natural gas (*34P*); the measurement of hydrogen content of process streams (*72P*); and an inexpensive but highly sensitive electric discharge detector (*23P*). A relatively large-scale preparative chromatograph was described and future developments have been discussed by Perkins and Carel (*58P*); their instrument combines good resolution and large throughput with efficient recovery of the fractions and low operating cost.

Automation of laboratory distillations has claimed the attention of numerous investigators. Ostrovskaya, Smol'nik, and Fainerman (*55P*) base their automatic distillation tester for petroleum products on a weight rather than a volume basis. Their apparatus exhibits a precision of about 2°F in the gasoline range, and analysis time is reduced to 20–25 minutes. Rhodes and Jacobs (*66P*) have invented a cyclic distillation apparatus for automatic in-stream monitoring; it measures and records four or more vapor temperature corresponding to a succession of distillation points per ASTM D-86. Wochlisch (*75P*) has devised a simple automatic instrument to measure the ASTM 90% distillation point for quality control of petroleum solvents. Reflux rate is automatically programmed and controlled in laboratory and pilot plant fractionating columns by a device described by Pascalau and Peculea (*57P*). For batch distillations in the laboratory, Perry (*59P*) has developed a modular system to start up and equilibrate columns, stabilize the top reflux rate, and signal the operator when cut-points are reached. Automatic control of heat input and reflux rate for simple laboratory distillation columns has been demonstrated by Tupchii (*71P*).

Maelzer (*47P*) has published the design of a continuous laboratory vacuum still for topping residua at 3 torr absolute pressure. The apparatus is used to separate the toppped residuum into eight fractions for further examination, and it was found that true-boiling-point curves can be derived from temperatures at the side-stream draw-offs. Reckhard and Kurtenbach (*65P*) have developed an automatic vacuum-distillation tester for petroleum products and coal tar. A continuous vacuum-distillation analyzer (*54P*) has reached commerical status in the USA; it monitors a single (preselected) distillation point and is applicable to hydrocracker feed stocks, catalytic cracking feeds, and lube oil fractions.

Seibert, Johnson, and Stross (*68P*) have provided additional data on the theoretical as well as practical aspects of their automatic falling-ball viscometer. Automation of the conventional glass-capillary viscometers has been partially achieved by the French researchers Leray and Gramain (*44P*) and in America (*12P*).

The specific gravity of diesel fuel and gasoline flowing in a products pipeline has been monitored by Frolov and Osipov (*26P*), using a float-type instrument with remote readout.

A fully automatic system that employs refractive index to monitor countercurrent distribution has been described in detail by Butterfield and Dutton (*11P*). The use of continuous gas-density meters for controlling an oxidation process has been reported by Hawkes (*31P*).

Rolfson and Coll (*67P*) described an automatic osmometer for determining the average molecular weight of polymers rapidly and with a high degree of precision.

Automatic in-stream instruments for determining the ASTM pour point of distillate fuels have been invented by Kapff (*36P*) and modified by Cropper and Hammond (*16P*); precision of 1° F has been achieved in these cyclic instruments which reduce analysis time to about 15 minutes.

A Russian patent (*56P*) describes an automatic instrument for measuring the solidification temperature of petroleum products by cooling the sample and recording the temperature at which it is unable to transmit pressure pulses through a manometer-like test cell.

In-stream monitoring of octane number of gasolines has proved highly successful. Green and Jones (*28P*) have presented detailed descriptions of two methods of monitoring and controlling the anti-knock quality of gasoline blends. Hoffman and Morris (*32P*) have reported at length on three field trials of another monitoring and control system that utilizes repetitive comparative knock intensity of the sample *vs.* a reference material.

An automatic laboratory instrument (Absorptomat) for determining the pore volume, pore size distribution, and surface area of cracking catalyst has been made commercially available (*35P*). An automatic process monitor has been developed by Kapff (*37P*) to measure the strength of sulfuric acid catalyst in alkylation processes; H_2SO_4 strength is a function of the maximum conductivity of the solution while the sample is being diluted to less than 20%. The H_2SO_4 concentration is directly recorded with an accuracy of 0.5%. An automatic analyzer for coke content of regenerated cracking catalyst has been described by Kurochkin and Kirillov (*42P*) and is based on light reflection from the surface of spherical particles of the catalyst.

Examples of applications of in-stream monitors to petroleum processing operations have been described in several survey articles. Alston, Hallikainen, and Waner (*1P*) summarized instruments available for monitoring composition or quality and presented some data on pay-out periods and profitability as well as several flow diagrams and charts. Bozeman discussed the advantages of process monitors for light-ends processing (*8P*) and for in-line blending of gasolines and distillate products (*9P*). Problems encountered in the development, evaluation, and maintenance of process analyzers were illustrated by Clark (*14P*), using case histories. Escher (*20P*) emphasized the trends in use of stream analyzers for process control and computer surveillance and Fraade (*24P*) presented brief descriptions of the operating principles of several commercial stream analyzers. Gurrola, Fritsch, and Dubner (*29P*) gave a complete description of their use of continuous volatility monitors for in-line blending; precision data for these monitors are also reported. The use of continuous analyzers for pilot-plant operations was discussed by Lyon and Shields (*46P*). The present status and probable future trends in the use of continuous analyzers were summarized by Pomper (*63P*), who mentioned as an example the highly instrumented Shell Refinery at Oakville, Canada (*53P*). Automatic control in refinery processes in the U.K. and in Europe was surveyed (*61P*), including in-line product blending and loading facilities and possibilities for computer control. The use of continuous recording viscometers to improve the in-line blending of asphaltic products was described by Waner, Hallikainen, and Alston (*74P*). Votlokhin and Shots (*73P*) summarized several instruments that are used in Russia for process control based on such properties as density, viscosity, catalyst flow-rate, and apparent pressure, and solidfication or freezing point.

Miscellaneous

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Refinery Waste Water. The determination of oil content is still an unsatisfactory analytical process and two more papers have been offered. Fastabend (*5R*) extends the infrared spectrophotometric method to 30 ppm with an average error of 9%; a single extraction of a 1000-cc sample was made with 50 cc of carbon tetrachloride, followed by spectrometry of the extract. Edeline and Heuze (*4R*) introduced a simple novelty for measuring surface oil. A copper cylinder, lined with chromatography paper to absorb the oil, defines the area of water surface; the oil film was driven toward the periphery of the circle by adding a trace of detergent. The paper was dried and extracted; a mean oil recovery of 93% with an 8% coefficient of variation was claimed.

A larger number of papers concerned themselves with oxygen demand. Busch and associates (*1R, 2R*) suggested means for rapidly measuring BOD; assuming that only dissolved carbon compounds require biological oxidation for their removal, he used a new aerated mass culture technique and measured the change in dissolved carbon. The test can be completed in 4 hours, as compared to 5 days by the usual BOD test, with a precision of ±10%. Gaudy and Ramanathan (*8R*) compared the titrimetric procedure and by a new colorimetric dichromate oxidation procedure; correlation was excellent when the turbidity of the sample was low. Van Hall and Stenger (*25R*) concerned themselves with measurement of total carbon by introducing the waste water into a heated tube together with oxygen; the carbon dioxide formed was measured in a nondispersive infrared analyzer. No definite relationship with BOD or COD was found. Weber and Morris (*27R*) also tried an oxidation technique for total carbon; the sample was passed through a chromic sulfuric acid medium at 175° C and the combustion product passed into a standard barium hydroxide solution.

Sharonova and Shul'man (*23R*) described a procedure for determining the phenol content of waste water by treating the sample with diazotized *p*-nitroaniline, and measuring the dye formed colorimetrically. Hydrogen sulfide, calcium and magnesium, and oil emulsions were removed prior to color development. A unique method for the measurement of small amount of sulfates and hydrogen sulfide was described by Nedorost and Paralova (*19R*).

Furfural. Furfural is sometimes used as an odorant for domestic liquefied petroleum gas: To determine its concentration, Marchetti and Saracco (*17R*) described a procedure based on the furfural–aniline reaction in acetic acid with the formation of a red Schiff base. The determination was a colorimetric one; the most critical part of the analysis was that of sampling.

In the furfural extraction of lubricating oils, the important analysis is the furfural content of the raffinate. For high concentrations encountered in column operation, Kyazimov *et al.* (*16R*) found the measurement of the refractive index to be adequate; the variation of this index was linear with temperature between 15° and 30° C. For the finished product, Palframan and Rose (*20R*) extended a previously published chromatographic method (*11R*) down to 2 ppm. The sample was extracted with sodium bisulfite, the bisulfite washed with legroine, and then ethylene oxide; the latter is injected into a column containing glass beads on which a thin film of 2,2′-iminodipropionitrile was deposited. Ratovskaya and Kuz'menko (*22R*) describe a photometric method applicable over the same range as the previous authors; it is based on the reaction of phloroglucinol in the presence of ammonia to give 5-aminoresorcinol which forms an orange-yellow compound with furfural. The sample is dissolved in a large amount of ethyl alcohol in which the reaction takes place; the absorption maximum was at 410 mμ. To control loss of furfural in plant wash water, Jacquemain and Remy (*12R*) described a photometric process applicable in the range of 5 to 3 mg/1000 cc; the reaction in a neutral saline solution was with a mixture of aniline and acetic acid, the maximum absorption being at 490 mμ.

Sulfonates. In refining petroleum fractions with sulfuric acid to obtain neutral oils, sulfonates are formed. In a procedure developed by Vamos and Simon (*26R*) the samples were diluted with benzene and chromatographed through silica gel with large pore and gain size. Elution was made with benzene then acetone, in which the neutral oil was found, and then with ethyl alcohol, which contained the sulfonates.

Halogens. For a simple rapid estimation of chlorides in marine lubricating oils, McKeon and O'Hara (*18R*), precipitated silver chromate on silica gel which was then packed in a narrow transparent tube. The oil was shaken with buffered water and the tip of the packed tube touched to the surface of the water solution; the length of discoloration correlated with the chloride content. High frequency conductometric titration of chloride in petroleum oils dissolved a benzene–methyl alcohol media with an alcoholic solution of silver nitrate was described by Stubec (*24R*). Kainz (*13R*) described a combination-element analysis procedure in which natural gas, petroleum distillates, and solid organic materials are burned in a jet. The combusted gases were passed over silver wool at 650° C and then through absorber solutions; it is claimed that trace amounts of chlorine, bromine, iodine, phosphorus, and sulfur may be determined together in this manner.

Rubber Extender Oils. Procedures were described by Ford (*7R*) for extending the clay-gel chromatographic to oils containing more than 20% polar compounds and to permit its use to classify the aromatic fraction.

Trace Oils. Krummel (*15R*), and Radler and Grnearevic (*21R*) describe extraction methods for determination of small amounts of paraffin on raisins, previously dressed with the oil to prevent stickiness. Krieger (*14R*) uses thin layer chromatography to determine small amounts of mineral oil products in soil. Giebler, Koppe, and Kempf (*9R*) describe a gravimetric procedure for separating oily material from soils or water and then by thin layer chromatography (*10R*) of separating mineral oils from vegetable and animal oils and determining this amount. Fletcher (*6R*) described the determination of mould release oils on concrete surfaces.

Coke. Procedures for the determination of 13 elements (Al, B, Ca, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Ti, V) in petroleum coke in ppp amounts were described by Chu and Chen (*3R*); special ashing procedures were used to produce solutions for colorimetric analysis.

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Pharmaceuticals and Related Drugs

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THIS REVIEW includes analytical methods and subjects related to pharmaceutical analysis which have appeared since the last published survey (*2442*), or were not included in previous reviews. The literature through June 1966 is covered.

The same tabular format is retained from the last review. An addition suggested by Florey (*621*) is the use of a superscript^B denoting references to the analysis of a drug in biological media. Such references are restricted to the determination of administered pharmaceutical agents. This will provide information to the pharmaceutical analytical chemist working with biological media, as well as offer alternate methods of analysis and separation to other workers.

Numerous reviews of interest have appeared. Thin layer chromatography (TLC) of pharmaceuticals has received much attention (*421, 648, 665, 825, 1104, 1357, 1383, 1476, 1547, 1687, 1938, 2011, 2503*). Applications of TLC to alkaloids have been described (*844, 2005, 2256, 2502*), as well as TLC of steroids (*831, 1298, 2134*), barbiturates (*2013*), dyes (*84*), sulfonamides (*747*), sulfonamide-type diuretics (*691*), and vitamins (*950, 1064, 1065, 1066*). An English translation of Stahl's book (*2107*), and a book by Bobbitt (*208*), are useful volumes on the subject of TLC and contain many references to drugs.

The uses of chromatography in pharmacy have been discussed (*1380, 2014*), and reviews on gas chromatography (*276, 1620*), ion exchange resins (*698*), and paper electrophoresis (*983, 984*), all as applied to analysis of medicinal substances, were noted.

General articles discussing the present status and progress of pharmaceutical analysis are included in the following papers (*270, 834, 980, 1135, 1424, 1597, 1892, 1916, 2405*), as well as instrumental methods (*1386*) and physicochemical methods applicable to drugs (*1671*).

Analytical automation in drug manufacture and quality assurance were discussed (*60, 123, 2457*).

Zone melting was reviewed as a means of achieving ultrapurity of pharmaceuticals (*650, 651, 966*).

In the field of spectrophotometric analysis, the following area reviews have appeared: ultraviolet (*1182, 1527, 1930*), infrared (*72, 1527*), and fluorometric approaches (*325, 558, 1244*). The characterization of drugs by their self emitted infrared radiation was reviewed (*1302*). Winefordner and Tin (*2444*) reported on the use of phosphorimetry in drug analysis, and spectropolarimetric determinations have been described (*1761*).

Nonaqueous titrimetry was reviewed (*98, 389, 834*). Kucharsky's and Safarik's book "Titrations in Nonaqueous Solvents," appeared (*1204*), and is devoted primarily to applications of the technique. A reversible internal indicator for sodium nitrite titrations was described by Schilt and Sutherland (*1951*).

Ashworth's "Titrimetric Organic Analysis," Part II (*61*), is an extensive bibliographic compilation of indirect

(*Text continued on page 212*)

Table I. Alkaloids

Compound or class	Method or subject	Reference
Alkaloidal Amines		
Ephedrine	Chromatographic, gas	(*1238, 2357*)
	Chromatographic, ion exchange	(*203*)
	Chromatographic, paper	(*11, 466, 649*)
	Chromatographic, partition column	(*1464*)
	Chromatographic, thin layer	(*190, 1638, 2284*)
	Colorimetric	(*294, 378, 649, 1143, 1901*)
	Distribution coefficients	(*511*)
	Extractive, from mixtures	(*254*)
	Identity	(*1474, 2486*)
	Optical rotatory dispersion	(*1052*)
	Phosphorimetric	(*2444*)
	Polarographic	(*1349*)
	Reaction, with chloroform	(*2439*)
	Spectropolarimetric	(*1761*)
	Volumetric, acid–base	(*488*)
	Volumetric, mercurometric	(*2237*)
	Volumetric, ox.–red.	(*326*)
Ephedrine derivatives	Chromatographic, paper	(*649*)
	Chromatographic, thin layer	(*1638*)
	Fluorometric	(*1983*^{B})
	Optical rotatory dispersion	(*1052*)
	Polarographic	(*1349*)
Mescaline	Fluorometric	(*1983*^{B})
Methylephedrine	Identity	(*2423*)
	Optical rotatory dispersion	(*1052*)
Pseudoephedrines	Chromatographic, paper	(*649*)
	Identity	(*649, 1474*)
	Polarographic	(*1349*)
	Optical rotatory dispersion	(*1052*)
	Spectropolarimetric	(*1761*)
Alkaloids, general methods		
	Chromatographic, adsorption	(*46*)
	Chromatographic, gas	(*277, 278, 1103, 1381*)
	Chromatographic, paper	(*399, 428, 836, 1032, 1209, 2091, 2472*)
	Chromatographic, partition	(*1257*)
	Chromatographic, thin layer	(*561*^{B}, *790*^{B}, *836, 844, 1032, 1383, 1487*^{B}, *1547, 1621, 1978, 2005, 2010, 2012, 2091, 2120, 2164, 2256, 2296, 2318, 2419, 2503*)
	Colorimetric	(*243, 873, 1143, 1959, 2392*)
	Countercurrent, distributive	(*406*)
	Electrophoretic, paper; review	(*984*)
	Extractive	(*331, 1143, 1187*)
	Fluorometric	(*325, 1215, 1244*)
	Identity	(*873, 1389, 1393, 1839, 1939, 2192, 2264*^{B})

(*Continued*)

Table I. Alkaloids (*Continued*)

Compound or class	Method or subject	Reference
	Incompatibility, pH	(*1900*)
	Mixtures, complex; review of analysis	(*2111*)
	Physicochemical methods, review	(*1373*)
	Polarographic, review	(*1915*)
	Review	(*305*)
	Spectrophotometric	(*1143, 1373, 1700, 1913*)
	Volumetric, acid–base	(*1566, 1760, 1884, 1885, 1972, 1973, 2087, 2110, 2409*)
	Volumetric, amperometric	(*1734*)
	Volumetric, complexometric	(*835*)
	Volumetric, with tungstosilicic acid	(*1337*)
	Volumetric, two-phase	(*1657*)
	Water methods, comparative study	(*1756*)
	X-ray	(*1373*)
Alkaloids, miscellaneous		
Aconitum	Chromatographic, thin layer	(*387*)
Aconitine	Chromatographic, adsorption	(*15, 1858*)
	Chromatographic, thin layer	(*15, 387, 489*)
Anabasine	Identity	(*1536*)
Aristolochic acid	Polarographic	(*882*)
Catharanthus roseus	Chromatographic; colorimetric	(*771*)
	Chromatographic, thin layer	(*418, 961*)
Chenopodium botrys	Chromatographic, adsorption	(*534*)
Cissampelos	Spectrophotometric, ultraviolet	(*118*^{B})
Coptis	Amperometric	(*884*)
Dionine	Chromatographic, thin layer	(*2284*)
	Extractive; spectrophotometric	(*1143*)
	Identity	(*2018*)
Galanthamine	Chromatographic, paper	(*2375*)
	Colorimetric	(*2383*)
	Identity	(*1433*)
Harmine; derivatives	Chromatographic, adsorption	(*1636*)
	Identity	(*2481*)
Heroin	Spectrophotometric, ultraviolet	(*1699*)
Leguminosae	Chromatographic, thin layer	(*1793*)
N-Methylhayatinium iodide	Spectrophotometric, ultraviolet	(*118*^{B})
Pachycarpine	Chromatographic, adsorption	(*534*)
	Identity	(*2481*)
Pethidine	Activation analysis	(*120*)
	Volumetric	(*1383*)
Promedol	Extractive; spectrophotometric	(*1143*)
	Identity	(*2481*)
Sarracine	Extractive; pH effect	(*551*)
Securinine	Colorimetric; extractive	(*1432*)
	Polarimetric	(*1849*)
Spherophysine	Identity	(*2481*)
Tilia	Chromatographic, thin layer	(*2124*)
Glyoxaline Alkaloids		
Pilocarpine	Chromatographic, thin layer	(*1376*)
	Colorimetric	(*280, 579, 1857, 2057, 2506*)
	Identity	(*2481*)
	Spectrophotometric	(*326*)
	Volumetric, mercurometric	(*2237*)
Indole Alkaloids		
Ajmaline	Colorimetric	(*2006*)
	Volumetric, acid–base	(*699*)
Brucine	Chromatographic, adsorption	(*1858*)
	Chromatographic, paper	(*1921*)
	Chromatographic, thin layer	(*1723, 1866*^{B}*, 1925*)
	Colorimetric	(*127*)
	Electrophoretic, paper	(*1921*)
	Extractive; spectrophotometric	(*1674*)
	Extractive; volumetric	(*1567*)
	Identity	(*1611*)
	Nuclear magnetic resonance	(*1712*)
Ergotamine	Chromatographic, adsorption	(*1684, 1792*)
	Chromatographic, ion exchange	(*1778*)
	Chromatographic, paper	(*94, 690*)
	Chromatographic, thin layer	(*861, 1263, 1264, 1327, 1887, 2512*)
	Colorimetric	(*94, 1778, 2512*)
	Fluorometric	(*1887*)
	Identity, flame test	(*1472*)
	Spectrophotometric, infrared	(*2460*)
	Stability	(*1217*)
Ergot alkaloids	Chromatographic, adsorption	(*1684, 1792*)
	Chromatographic, ion exchange	(*583, 1792*)
	Chromatographic, paper	(*690, 1157*)
	Chromatographic, thin layer	(*644, 861, 1157, 1263, 1264, 1327, 1887*)
	Colorimetric	(*640, 1778, 2512*)
	Fluorometric	(*1887*)
	Spectrophotometric, infrared	(*2460*)
	Spectrophotometric, ultraviolet	(*690*)
Hydrogenated ergot alkaloids		
	Chromatographic, paper	(*690*)
	Chromatographic, thin layer	(*861*)
	Spectrophotometric, ultraviolet	(*690*)
Indoles, general	Phosphorimetric	(*865*)
Physostigmine	Chromatographic, paper; stability	(*1325*)
	Colorimetric	(*1858*)
	Phosphorimetric	(*2444*)
	Volumetric	(*2088*)
Rauwolfia alkaloids	Chromatographic, adsorption	(*2289, 2322*)
	Chromatographic, paper	(*1374, 2271, 2272, 2322*)
	Chromatographic, thin layer	(*932, 1876*)
	Electrophoretic	(*1374*)
	Fluorometric	(*2272*)
	Spectrophotometric, ultraviolet	(*2007*)
Reserpine	Chromatographic, adsorption	(*2322*)
	Chromatographic, paper	(*1374, 2271, 2273*)
	Chromatographic, thin layer	(*125, 1789, 1876*)
	Colorimetric	(*284, 1557, 1876, 2271, 2273*)
	Extractive	(*236, 613, 1789*)
	Identity	(*1675, 2322*)
	Polarographic	(*528*)
	Spectrophotometric, ultraviolet	(*33, 284*)
Strychnine	Chromatographic, paper	(*11, 1921, 2068*)
	Chromatographic, ion exchange	(*1, 240, 1028*)
	Chromatographic, thin layer	(*742, 751, 1274, 1723, 1859*)
	Colorimetric	(*1866*^{B}*, 1929, 2284*)
	Complexometric	(*2170*)
	Electrophoretic	(*1921*)
	Extractive	(*1273*^{B}*, 1567, 1674*)

(*Continued*)

Table I. Alkaloids (*Continued*)

Compound or class	Method or subject	Reference
	Gamma ray irradiation, effect on	(*230*)
	Nuclear magnetic resonance	(*1712*)
	Radiometric	(*1925*)
	Spectrophotometric	(*1674*)
	Spectrophotometric, infrared	(*230*)
	Spectrophotometric, ultraviolet	(*1*)
	Volumetric	(*2088, 2206*)
	Volumetric, acid–base	(*1567, 1759, 1760*)
Strychnos alkaloids	Chromatographic, paper	(*1921*)
	Chromatographic, thin layer	(*751*)
	Colorimetric	(*127*)
	Electrophoretic	(*1921*)
	Extractive	(*1674*)
	Radiometric	(*1925*)
	Volumetric, acid–base	(*1567*)
Vincamine	Chromatographic, thin layer	(*1041*)
Vinca rosea	Chromatographic, thin layer	(*418, 961*)
Yohimbine	Chromatographic, gas	(*199*)
	Chromatographic, paper	(*2068*)
	Colorimetric	(*1835*)
	Polarographic	(*528*)
Isoquinoline Alkaloids		
Apomorphine	Chromatographic, paper; identity	(*1134*)
	Polarographic	(*504*)
	Spectrophotometric, ultraviolet	(*1699*)
	Stability kinetics	(*320*)
	Volumetric	(*1758*)
	Volumetric, acid–base	(*504*)
Berberine	Chromatographic, ion exchange	(*2421*)
	Chromatographic, paper	(*723, 2068*)
	Chromatographic, thin layer	(*1218*)
	Spectrophotometric	(*380*)
	Volumetric	(*2421*)
Codeine	Chromatographic, adsorption	(*301*)
	Chromatographic, gas	(*2357*)
	Chromatographic, ion exchange	(*486*)
	Chromatographic, miscellaneous	(*279*)
	Chromatographic, paper	(*455, 473, 1134, 2068*)
	Chromatographic, partition column	(*375, 908, 909*)
	Chromatographic, thin layer	(*126, 190, 371, 663, 1242, 1432, 1456, 1719, 1722, 1922, 2203, 2239, 2284*)
	Colorimetric	(*190, 378, 1496, 1551, 2040*)
	Complexometric	(*889, 2170*)
	Extractive	(*113, 116, 293*)
	Fluorometric	(*266*)
	Identity	(*1377, 2018, 2347, 2350*)
	Mass spectrometric	(*2225, 2226*)
	Polarographic	(*1875*)
	Reaction with iodine monochloride	(*1095*)
	Sodium in, by flame photometric	(*298*)
	Spectrophotometric, infrared	(*695*)
	Spectrophotometric, ultraviolet	(*909, 1003, 1242, 1437, 1699, 1703*)
	Volumetric, acid–base	(*189, 973, 1565, 1654, 1758, 1929*)
	Volumetric, ox.–red.	(*2270*)
Cotarnine	Volumetric, mercurometric	(*2237*)
Diacetylmorphine	Identity	(*2350*)
	Reaction with iodine monochloride	(*1095*)
Dihydrocodeine	Colorimetric	(*1893*)
	Mass spectrometric	(*2225*)
	Volumetric, acid–base	(*1758*)
Dihydrocodeinone	Chromatographic, paper	(*181*^{B}, *1134*^{B})
	Chromatographic, partition column	(*1258*)
	Chromatographic, thin layer	(*181*^{B})
	Identity	(*1134*^{B}, *1377*)
Dihydrohydroxycodeinone	Identity, color reactions	(*2163*)
Dihydroxycodeinone	Chromatographic, paper; identity	(*1134*^{B})
Dihydromorphinone	Chromatographic, paper	(*1134*)
	Identity	(*1377*)
	Polarographic	(*504*)
	Volumetric, acid–base	(*291*)
Ethyl morphine	Chromatographic, gas	(*2357*)
	Colorimetric; extractive	(*1551*)
	Extractive	(*116*)
	Identity	(*1881, 2347*)
	Spectrophotometric, ultraviolet	(*1003*)
	Volumetric	(*1758*)
	Volumetric, mercurometric	(*2237*)
Hydrastine	Chromatographic, thin layer; fluorometric	(*1789*)
Hydrastinine	Spectrophotometric, ultraviolet	(*1699*)
Meconic acid	Colorimetric	(*695*)
Morphine	Chromatographic, adsorption	(*249, 1071, 1730, 1956, 1966, 2181, 2186*)
	Chromatographic, ion exchange	(*240; 486*)
	Chromatographic, paper	(*976, 1134, 1336, 1663*)
	Chromatographic, partition column	(*1947*)
	Chromatographic, thin layer	(*126, 371, 663, 843, 844, 845, 1072, 1242, 1456, 1722, 1935, 2203, 2239, 2284*)
	Colorimetric	(*1895*)
	Extractive	(*293, 695, 1693*)
	Fluorometric	(*266, 1215*)
	Gravimetric	(*249, 1956, 2181*)
	Identity	(*1336, 1377, 2018, 2347, 2350*)
	Mass spectrometric	(*65*)
	Oxidation, enzyme	(*1936*)
	Phosphorimetric	(*865*)
	Polarographic	(*504, 716, 845, 976*^{B}, *1601*)
	Review, comparison of methods	(*209*)
	Reaction with iodine monochloride	(*1095*)
	Spectrophotometric, infrared	(*230, 695, 1242*)
	Spectrophotometric, ultraviolet	(*1003, 1699, 1947*)
	Stability	(*230, 1780, 2186*)
	Volumetric, acid–base	(*549, 1758*)
	Volumetric, amperometric	(*2353*)
	Volumetric, ox.–red.	(*1693*)
	Volumetric, mercurometric	(*2237*)
Morphine derivatives	Chromatographic, paper	(*1663*)
	Mass spectrometric	(*65*)
	Polarographic	(*504*)
	Reaction with iodine monochloride	(*1095*)
	Volumetric, acid–base	(*1758*)
	Volumetric, amperometric	(*2353*)

(*Continued*)

Table I. Alkaloids (*Continued*)

Compound or class	Method or subject	Reference
Nalorphine	Chromatographic, ion exchange	(*1663*)
	Chromatographic, paper	(*2377*)
	Polarographic	(*504*)
	Volumetric, acid–base	(*504, 1758*)
Narceine	Chromatographic, thin layer	(*1242*)
	Spectrophotometric, ultraviolet	(*1242, 1699*)
Narcotine	Chromatographic, adsorption	(*301*)
	Chromatographic, ion exchange	(*486*)
	Chromatographic, paper	(*455, 956, 1134*)
	Chromatographic, thin layer	(*126, 371, 663, 1719, 1720, 1722*)
	Extractive	(*293, 695, 1837*)
	Identity	(*2018, 2019*)
	Spectrophotometric, infrared	(*695*)
	Spectrophotometric, ultraviolet	(*1699*)
Normorphine	Chromatographic, paper	(*2377*)
Opium alkaloids, general		
	Chromatographic, adsorption	(*301*)
	Chromatographic, gas	(*279*)
	Chromatographic, paper	(*279, 455, 1134*)
	Chromatographic, partition column	(*301, 956*)
	Chromatographic, thin layer	(*126, 187, 279, 371, 663, 1242, 1456, 1719, 1720, 1722, 1790, 2203, 2239*)
	Extractive	(*293*)
	Identity	(*1134, 1681, 2018*)
	Mass spectrometric	(*478, 2224*)
	Optical rotatory dispersion	(*121*)
	Spectrophotometric, infrared	(*695*)
	Spectrophotometric, ultraviolet	(*455, 1003, 1242, 1699*)
	Volumetric, acid–base	(*293*)
Papaverine	Chromatographic, adsorption	(*301*)
	Chromatographic, gas	(*56*)
	Chromatographic, paper	(*455, 956, 1134, 2325*)
	Chromatographic, thin layer	(*126, 371, 461, 663, 1242, 1456, 1719, 1720, 1722, 1922, 1935, 2203, 2284*)
	Colorimetric	(*483, 1144, 1211*)
	Complexometric	(*2498*)
	Extractive	(*116, 189, 293, 695, 1144, 1211*)
	Identity, flame tests	(*1134, 1472*)
	Spectrophotometric, infrared	(*695*)
	Spectrophotometric, ultraviolet	(*956, 1003, 1242, 1699, 1703*)
	Volumetric, acid–base	(*189, 295, 376, 377, 1495, 1670, 2342*)
	Volumetric, mercurometric	(*2237*)
Papaverine alkaloids	Chromatographic, thin layer	(*461*)
Porphyroxine	Chromatographic, adsorption; colorimetric	(*695*)
	Chromatographic, in opium	(*694*)
Pseudomorphine	Chromatographic, paper	(*1336*)
	Polarographic	(*504*)
	Spectrophotometric, infrared	(*1936*)
Salsolidine	Chromatographic, ion exchange	(*1096*)
	Chromatographic, thin layer	(*2284*)
	Distribution, liquid-liquid	(*1145*)
	Identity	(*2481, 2486*)
	Spectrophotometric, ultraviolet	(*1699*)
	Volumetric, acid–base	(*1495*)
	Volumetric, mercurometric	(*2237*)
Salsoline	Chromatographic, ion exchange	(*1096*)
	Identity	(*1657, 2481*)
	Spectrophotometric, ultraviolet	(*1699*)
	Volumetric, acid–base	(*1495*)
	Volumetric, mercurometric	(*2237*)
Tecodine	Spectrophotometric, ultraviolet	(*1003*)
Thebaine	Chromatographic, absorption and partition	(*301*)
	Chromatographic, ion exchange	(*486*)
	Chromatographic, paper	(*455, 956*)
	Chromatographic, thin layer	(*126, 371, 663, 1456, 1719, 1722, 2239*)
	Colorimetric	(*1838, 1894*)
	Extractive	(*293, 695, 1838*)
	Spectrophotometric, infrared	(*695*)
	Spectrophotometric, ultraviolet	(*455, 1699*)
Isoquinoline, bis-benzyl, alkaloids		
Cephaeline	Chromatographic, paper	(*1324, 2117*)
Curare	Review	(*1561*)
Emetine	Chromatographic, paper	(*1324, 2117*)
	Chromatographic, thin layer	(*2203*)
	Colorimetric	(*2386*)
	Extractive	(*1145*)
	Identity	(*1881*)
	Spectrophotometric, infrared	(*230*)
	Spectrophotometric, ultraviolet	(*1699*)
	Stability	(*230, 1324*)
General	Chromatographic, thin layer	(*187*)
	Optical rotatory dispersion	(*121*)
Ipecacuanha alkaloids	Chromatographic, adsorption	(*1782*)
	Chromatographic, paper	(*2117*)
	Extractive	(*2087*)
	Stability	(*2117*)
	Volumetric, acid-base	(*1782, 2087*)
Tubocurarine	Fluorometric	(*408*^{B})
	Spectrophotometric, ultraviolet	(*2429*)
Lupinane Alkaloids		
General	Chromatographic, paper	(*2078*)
	Polarographic	(*1156*)
Sparteine	Chromatographic, paper	(*2078*)
	Colorimetric	(*483, 2130*)
	Polarographic	(*1156*)
	Spectrophotometric, infrared; stability	(*230*)
Sparteine beta camphorsulfonate	Volumetric	(*1731*)
Pyridine Alkaloids		
Lobelia alkaloids	Chromatographic, thin layer	(*237, 1623*)
	Polarographic	(*1623*)
Lobeline	Chromatographic; colorimetric	(*473*)
	Chromatographic, thin layer	(*237, 1623*)
	Colorimetric; extractive	(*880*^{B})
	Extractive	(*1145, 1219*^{B})
	Polarographic; stability	(*1623*)
Thermopsis alkaloids	Extractive; volumetric	(*474*)

(*Continued*)

Table I. Alkaloids (*Continued*)

Compound or class	Method or subject	Reference
Quinoline Alkaloids		
Cinchona alkaloids	Chromatographic, ion	(*1030*)
	Chromatographic, thin layer	(*259, 1999, 2171, 2172, 2173*)
	Colorimetric	(*1550*)
	Phosphorimetric	(*865*)
	Spectrophotometric, ultraviolet	(*1034, 1698*)
Cinchonidine	Chromatographic, paper	(*2068*)
	Chromatographic, thin layer	(*1999, 2171*)
	Determination, in quinine	(*838*)
	Fluorometric	(*1030*)
	Spectrophotometric, ultraviolet	(*1034, 1698*)
Cinchonine	Chromatographic, thin layer	(*1999, 2171*)
	Colorimetric	(*1550*)
	Spectrophotometric, ultraviolet	(*1034, 1698*)
Cinchoninic acid	Identity	(*1478*)
Equinine	Chromatographic, ion exchange; fluorometric	(*2071*)
	Volumetric, ox.-red.	(*1489*)
Hydroquinidine	Chromatographic, thin layer	(*2171*)
	Fluorometric	(*259*)
	Identity	(*1030*)
	Spectrophotometric, infrared	(*641*)
Quinic acid; natural esters	Colorimetric	(*1413*)
Quinicine	Polarographic	(*716*)
Quinidine	Chromatographic, thin layer	(*1999, 2171*)
	Colorimetric	(*1550*)
	Fluorometric	(*330*)
	Identity	(*1030, 1377*)
	Monograph, proposed	(*1727*)
	Nuclear magnetic resonance	(*475*)
	Phosphorimetric	(*2444*)
	Spectrophotometric, infrared	(*641*)
	Spectrophotometric, ultraviolet	(*1034, 1698*)
Quinine	Assay, in mixture	(*100*)
	Chromatographic, ion exchange	(*1028, 1030*)
	Chromatographic, thin layer	(*259, 1789, 1922, 1935, 1999, 2171*)
	Cinchonidine, determination in	(*838*)
	Colorimetric	(*1211, 1550, 2171*)
	Fluorometric	(*259, 1789*)
	Identity	(*330, 1030, 1377, 1472, 1999*)
	Monograph, proposed	(*1726*)
	Phosphorimetric	(*2444*)
	Polarographic	(*716*)
	Spectrophotometric, infrared	(*641*)
	Spectrophotometric, ultraviolet	(*780, 1034, 1698, 2376*)
	Volumetric, acid-base	(*330*)
	Volumetric, ox.-red.	(*489*)
Quinine derivatives	Colorimetric	(*2022*)
Senecio Alkaloids		
Platyphylline	Chromatographic, thin layer	(*2284*)
	Colorimetric	(*2268*)
	Extractive	(*113, 116, 550, 1145*)
	Identity	(*1211, 2486, 2517*
	Stability	(*2268*)
Pyrrolizidine alkaloids	Chromatographic, thin layer	(*2009*)
Steroidal Alkaloids		
Holarrhena alkaloids	Chromatographic, thin layer	(*1226*)
Sanguinarine	Chromatographic, for impurities	(*287*)
Solanidine; solanine	Chromatographic, thin layer	(*1619*)
	Polarographic	(*1688*)
Solanum alkaloids	Chromatographic, thin layer	(*784, 1619, 1960, 2399*)
	Colorimetric	(*784, 2399*)
	Polarographic	(*1688*)
	Review	(*2291*)
Solasodine	Chromatographic, thin layer; colorimetric	(*2399*)
beta-Tomatine	Chromatographic, paper	(*1958*)
Tropane Alkaloids		
Atropine	Adsorption, by antacids	(*204*)
	Chromatographic	(*2509*)
	Chromatographic, gas	(*1238*)
	Chromatographic, ion exchange	(*240*)
	Chromatographic, ion exchange, paper	(*1261, 1354*)
	Chromatographic, paper	(*85, 86, 128, 304, 305, 306, 445, 473, 875, 1261, 1354, 1532, 1799, 2143, 2325*)
	Chromatographic, review	(*305*)
	Chromatographic, thin layer	(*15, 305, 306, 307, 1002, 1006, 1603, 2203, 2284, 2348*)
	Colorimetric	(*85, 86, 445, 473, 875, 1006, 1858, 2143, 2473*)
	Complexometric	(*2170*)
	Extractive	(*115, 116, 2325*)
	Fluorometric	(*1577*)
	Hydrolysis of	(*2063*)
	Identity	(*128, 1002, 2017, 2348, 2350, 2483*)
	Impurities in	(*2385*)
	Phosphorimetric	(*2444*, 2445^{B})
	Spectrophotometric	(*2491*)
	Spectrophotometric, ultraviolet	(*585, 2385*)
	Stability	(*304, 585, 1006, 2063, 2142, 2143*)
	Volumetric, acid-base	(*1757, 1760, 2061, 2088, 2342*)
Belladonna alkaloids	Chromatographic, adsorption	(*748*)
	Chromatographic, ion exchange	(*20*)
	Chromatographic, ion exchange paper	(*1261*)
	Chromatographic, paper	(*305, 306, 1261, 1353, 1799*)
	Chromatographic, partition column	(*413*)
	Chromatographic, thin layer	(*305, 306, 307, 1353, 1603*)
	Colorimetric	(*2473*)
	Electrophoretic, paper; fluorometric	(*1353*)
	Identity	(*1353, 2017*)
	Monograph, proposed	(*1717*)
	Volumetric, acid-base	(*20, 748, 1506, 1567, 1957*)
Cocaine	Chromatographic, gas	(*2357*)
	Chromatographic, ion exchange	(*1536*)
	Chromatographic, paper	(*875, 1338, 1339, 2238*)
	Chromatographic, thin layer	(*665, 788*)
	Colorimetric	(*244, 246, 482, 483, 1049*)

(*Continued*)

Table I. Alkaloids (*Continued*)

Compound or class	Method or subject	Reference
	Gravimetric	(*2255*)
	Phosphorimetric	(*2444*)
	Review of methods	(*2255*)
	Spectrophotometric, ultraviolet	(*1004*)
	Stability	(*482, 788, 1338*)
	Volumetric, acid-base	(*482, 1757, 2445^{B}*)
	Volumetric, mercurometric	(*2237*)
Ecgonine	Chromatographic, paper	(*13 8, 1339, 2255*)
	Chromatographic, thin layer	(*788*)
	Extractive; Kjeldahl nitrogen	(*2255*)
Homatropine	Adsorption, on antacids	(*204*)
	Chromatographic, paper	(*128, 875*)
	Chromatographic, thin layer	(*1002*)
	Colorimetric	(*875*)
	Identity	(*2350*)
	Volumetric, acid-base	(*1565, 1757*)
Homatropine methylbromide	Chromatographic, ion exchange paper	(*1626*)
	Chromatographic, paper	(*128, 1626*)
	Spectrophotometric, ultraviolet	(*349*)
	Volumetric, acid-base	(*1565, 1757*)
Hyoscyamine	Chromatographic, absorption	(*748*)
	Chromatographic, paper	(*114, 306, 473, 875, 1043*)
	Chromatographic, partition column	(*133*)
	Chromatographic, thin layer	(*305, 306, 307*)
	Colorimetric	(*133, 875*)
	Extractive	(*116*)
	Volumetric, acid-base	(*114, 748*)
Methyl homatropine	Identity	(*2350*)
Pseudotropine	Chromatographic, gas	(*2373*)
	Chromatographic, thin layer	(*1528*)
Scopolamine	Chromatographic	(*2509*)
	Chromatographic, gas	(*2357*)
	Chromatographic; ion exchange, paper	(*1355*)
	Chromatographic, paper	(*85, 86, 114, 128, 305, 306, 875, 1043*)
	Chromatographic, partition column	(*115*)
	Chromatographic, thin layer	(*305, 306, 307, 1002, 1603, 2203, 2510*)
	Colorimetric	(*85, 86, 875, 1858, 2129, 2510*)
	Complexometric	(*2170*)
	Identity	(*1002, 2350*)
	Spectrophotometric	(*2491*)
	Volumetric, acid–base	(*114, 1565, 1757, 2088, 2509*)
	Volumetric, mercurometric	(*2237*)
Tropane, general	Chromatographic, gas	(*637*)
	Chromatographic, ion exchange paper	(*1261*)
	Chromatographic, paper	(*874, 875, 1043, 1261, 1685*)
	Chromatographic, thin layer	(*305, 1002, 1602, 1603*)
	Colorimetric	(*1685*)
	Volumetric, acid-base	(*1757*)
Tropic acid	Spectrophotometric, ultraviolet	(*585*)
Tropine	Chromatographic, paper	(*1043*)
	Chromatographic, thin layer	(*305, 1002, 1528, 1603*)
	Luminescent	(*2020*)
	Review	(*624*)
Tropinone	Chromatographic, thin layer	(*1528*)
	Reineckate method, evaluation	(*184*)

Table II. Antibiotics

Compound or class	Method or subject	Reference
Actinomycin	Chromatographic, paper	(*533*)
	Chromatographic, thin layer	(*334*)
	Stability	(*435*)
Aureomycin	Spectrophotometric, ultraviolet	(*763*)
Bacitracin	Chromatographic, thin layer	(*1560*)
Cephaloridine	Review, methods	(*1372*)
Chloramphenicol	Chromatographic, paper	(*1556, 1852, 2002*)
	Chromatographic, thin layer	(*1229, 1278, 1888*)
	Colorimetric	(*231, 810, 936, 1428, 2056*)
	Microbiological	(*2501*)
	Polarographic	(*1867, 1869, 1870, 1871, 2161*)
	Spectrophotometric, ultraviolet	(*620, 1555, 2371*)
	Stability	(*404*)
	Volumetric, ox.–red.	(*1001, 1993*)
	Volumetric, precipitation	(*608, 623, 1485, 2100*)
Chlortetracycline	Chromatographic, column	(*881*)
	Chromatographic, paper	(*1342*)
	Chromatographic, thin layer	(*1037, 2096*)
	Colorimetric	(*921, 1086, 1126, 1344, 1709*)
	Identification	(*1709*)
	Microscopic	(*1045*)
	Polarographic	(*328, 582*)
	Refractometric	(*2415*)
	Review, feed additives	(*715*)
	Stability	(*2416*)
	Volumetric, acid-base	(*149*)
	Volumetric, ox.–red.	(*1707, 2169*)
Colimycin	Chromatographic, ion exchange	(*1223, 1783*)
	Stability	(*1223*)
Colistin	Identification	(*2437*)
Cranomycin	Stability	(*1137*)
Cycloheximide	Stability	(*681, 682*)
Cycloserine	Colorimetric	(*1050, 1159*)
	Polarographic	(*2180*)
	Stability	(*1159, 1347, 2312*)
Dactinomycin	Review, official methods	(*594*)
Desertomycin	Chromatographic, paper	(*2198*)
	Colorimetric	(*2198*)
	Identification	(*2198*)
Erythromycin	Chromatographic, thin layer	(*45, 1863*)
	Spectrophotometric, ultraviolet	(*1147*)
	Stability	(*367, 2034, 2302*)
	Volumetric, acid-base	(*619*)
Furacillin	Colorimetric	(*2297*)
	Identification	(*2274*)
	Volumetric, ox.–red.	(*2166*)
Furadonin	Identification	(*2274*)
Furazolidone	Identification	(*2274*)

(*Continued*)

Table II. Antibiotics (*Continued*)

Compound or class	Method or subject	Reference
General	Calorimetric, review	(*2305*)
	Chromatographic, absorption column	(*438*)
	Chromatographic, ion exchange	(*941*)
	Chromatographic, paper	(*179, 206, 207, 283, 922, 1824, 2068*)
	Chromatographic, thin layer	(*178, 283, 903, 953, 1499*)
	Countercurrent distribution	(*1350*)
	Electrophoretic	(*443, 1271*)
	Identification	(*207, 2068*)
	Polarographic	(*1186*)
	Review	(*591, 592, 850*)
	Spectrophotometric, infrared	(*2304, 2422*)
	Spectrophotometric, ultraviolet	(*1443, 2304*)
	Stability	(*405, 464*)
	Turbidimetric	(*1713*)
Gramicidin	Chromatographic, paper	(*2049*)
	Chromatographic, thin layer	(*1559*)
Griseofulvin	Colorimetric	(*1330, 2300*)
	Fluorometric	(*559, 1188*)
	Identification	(*2199*)
	Spectrophotometric, luminescence	(*1530*)
	Spectrophotometric, ultraviolet	(*542, 862, 2307*)
Hamycin	Stability	(*186*)
Kanamycin	Chromatographic, column	(*1146*)
	Colorimetric	(*1146*)
Linomycin	Stability	(*663*)
Mycerin	Chromatographic, ion exchange	(*1223*)
	Stability	(*1223*)
Neomycin	Chromatographic, ion exchange	(*1223, 1329, 1914*)
	Chromatographic, paper	(*1005, 1914*)
	Chromatographic, thin layer	(*631*)
	Colorimetric	(*954*)
	General	(*2093*)
	Microbiological	(*758*)
	Radiographic	(*1005*)
	Review, official methods	(*593*)
	Stability	(*1223*)
Novobiocin	Chromatographic, paper	(*1149*)
	Colorimetric	(*1412*)
	International Standard	(*1272*)
Nystatin	Colorimetric	(*356, 1158*)
	Spectrophotometric, ultraviolet	(*1148*)
	Stability	(*142, 1112*)
Oleandomycin	Chromatographic, thin layer	(*677*)
	Colorimetric	(*1150*)
	Microbiological	(*2232*)
	Spectrophotometric, ultraviolet	(*661*)
	Stability	(*661*)
Olivomycin	Chromatographic, paper	(*1200*)
Oxacillin	Stability	(*368*)
Oxtetracycline	Biological	(*2127*)
	Chromatographic, column	(*881*)
	Chromatographic, thin layer	(*1037, 2096*)
	Colorimetric	(*607, 1639, 1709*)
	Identification	(*1708, 1709*)
	Polarographic	(*328*)
	Review, feed additives	(*715*)
	Review, methods	(*2295*)
	Spectrophotometric	(*2127*)
	Stability	(*793, 2127*)
	Volumetric, ox.–red.	(*2169*)
Paromomycin	Chromatographic, paper	(*904*)
	Stability	(*904*)
Penicillins	Chromatographic, gas	(*577, 1073, 2328*)
	Chromatographic, ion exchange	(*239*)
	Chromatographic, paper	(*618, 1098, 1834, 2334*)
	Chromatographic, pH	(*177*)
	Chromatographic, thin layer	(*982, 1558*)
	Colorimetric	(*2178*)
	Sedimentation	(*1686*)
	Solubility	(*251*)
	Spectrophotometric, flame	(*1106*)
	Spectrophotometric infrared	(*2518*)
	Spectrophotometric, ultraviolet	(*906, 2306*)
	Stability	(*385, 611, 982, 1514, 1515, 1588, 1612, 1697, 1775, 1974, 1975, 1976, 2229, 2244, 2417*)
	Volumetric, ox.–red.	(*229, 1410*)
Phalamycin	Stability	(*1085*)
Phenethicillin	Chromatographic, paper	(*430*)
	Stability	(*1977*)
Polymyxin	Agar diffusion	(*508*)
	Identification	(*2437*)
	Stability	(*2050*)
Porfiromycin	Stability	(*684*)
Primycin	Chromatographic, paper	(*2197*)
Ramycin	Identification	(*2327*)
Rifamycin	Chromatographic, paper	(*2003*)
	Colorimetric	(*674*)
	Spectrophotometric, ultraviolet	(*2004*)
Ristomycin	Chromatographic, ion exchange	(*1237*)
	Electrophoretic	(*1905*)
Sekazin	Colorimetric	(*1785*)
Spiramycin	Chromatographic, paper	(*617*)
Streptomycin	Chromatographic, ion exchange	(*417, 1783*)
	Chromatographic, thin layer	(*1562, 1563*)
	Colorimetric	(*417, 2155*)
	Fluorometric	(*588*)
	Identification	(*1562*)
	Microbiological	(*442, 1563*)
	Volumetric, ox.–red.	(*900*)
Streptothricins	Chromatographic, ion exchange	(*214, 1099*)
	Chromatographic, paper	(*214*)
Tetracycline	Chromatographic, column	(*881*)
	Chromatographic, ion exchange	(*1904*)
	Chromatographic, paper	(*885, 1239, 1342, 1343, 1931*)
	Chromatographic, thin layer	(*1037, 1874, 2058, 2096*)
	Colorimetric	(*228, 1087*)
	Fluorometric	(*1692*)
	Polarographic	(*328*)
	Review, feed additives	(*715*)
	Spectrophotometric, ultraviolet	(*1692*)
	Spectroscopic, mass	(*855*)
	Stability	(*151, 1040, 1813*)
	Thermal analysis	(*940*)
	Volumetric, ox.–red.	(*2169*)
Trichothecin	Colorimetric	(*1735*)
Tylosin	Extractive	(*1850*)
Tyrothricin	Chromatographic, thin layer	(*1559*)
Viomycin	Colorimetric	(*2037, 2038*)
	Stability	(*1269*)
Zygomycin	Chromatographic, column	(*876*)

Table III. Inorganic and Metal–Organic Compounds

Compound or class	Method or subject	Reference
Aluminum	Colorimetric	*(1397)*
	Identification	*(1449)*
	Neutron activation analysis	*(2281)*
	Spectrographic	*(948)*
	Volumetric, complexometric	*(1718)*
Ammonia	Review, official methods	*(1447)*
Antimony	Radiometric	*(1949)*
	Volumetric, ox.–red.	*(96)*
Arsenic	Colorimetric	*(530)*
	Identification	*(201)*
	Review, official methods	*(1421, 2351)*
	Volumetric, complexometric	*(635)*
	Volumetric, ox.–red.	*(2027, 2167)*
Barium	Volumetric, complexometric	*(23)*
Bismuth	Colorimetric	*(1253)*
	Identification	*(800)*
	Neutron activation analysis	*(773)*
	Radiometric	*(1950)*
	Volumetric, complexometric	*(947, 1370)*
	Volumetric, ox.–red.	*(2025)*
Boron	Spectrophotometric, flame	*(1123)*
	Volumetric, ox.–red.	*(2282)*
Bromine	Amperometric	*(4)*
	Identification	*(219, 220, 599)*
	Neutron activation analysis	*(586)*
Cadmium	Gravimetric	*(756)*
	Volumetric, complexometric	*(1113)*
Calcium	Amperometric	*(6, 2524)*
	Chromatographic, paper	*(1649)*
	Identification	*(1448)*
	Radiometric	*(1924, 1949)*
	Stability	*(49)*
	Volumetric complexometric	*(141, 217, 323, 721, 1384, 1650, 1662, 2118, 2365)*
Chlorine	Colorimetric	*(812)*
	Identification	*(220)*
	Volumetric, ox.–red.	*(812, 1856)*
Chromium	Identification	*(2459*^{B}*)*
Cobalt	Colorimetric	*(816, 1418)*
	Fluorometric	*(458)*
Copper	Colorimetric	*(866)*
	Gravimetric	*(757)*
	Identification	*(867)*
	Radiometric	*(1919, 1924)*
	Spectrographic	*(948)*
Fluorine	Colorimetric	*(829, 987)*
	Volumetric, complexometric	*(892)*
General	Neutron activation analysis	*(652, 978)*
	Polarographic	*(369)*
	Radiometric	*(2343)*
	Volumetric, complexometric	*(202)*
Gold	Radiometric	*(1689, 1949)*
	Spectrophotometric, X-ray	*(1830)*
Halogens, general	Radiometric	*(2258)*
	Review, methods	*(1388)*
	Volumetric, ox.–red.	*(1554)*
Heavy metals, general	Identification	*(1450, 1961)*
Hydrogen peroxide	Biological	*(1395)*
	Stability	*(2500)*
Iodine	Chromatographic, ion exchange	*(636, 1445, 2230)*
	Identification	*(219)*
	Review	*(138*^{B}*)*
	Spectrophotometric, ultraviolet	*(718)*
	Volumetric, ox.–red.	*(1252, 1832)*
Iodine, organic	Chromatographic, paper	*(730, 1971)*
	Electrophoretic	*(730)*
	Volumetric, ox.–red.	*(1266)*
Iron	Chromatographic, paper	*(1649)*
	Chromatographic, thin layer	*(2496)*
	Colorimetric	*(866)*
	Identification	*(867)*
	Neutron activation analysis	*(586)*
	Spectrographic	*(948)*
	Volumetric, complexometric	*(227, 2101)*
	Volumetric, ox.–red.	*(97, 2282)*
Lead	Colorimetric	*(866)*
	Identification	*(867)*
	Volumetric, complexometric	*(1307, 1403)*
	Volumetric, ox.–red.	*(1451)*
Magnesium	Volumetric, complexometric	*(2118)*
Manganese	Neutron activation analysis	*(586)*
	Spectrographic	*(948)*
Mercury	Chromatographic, thin layer	*(2458)*
	Colorimetric	*(322, 656, 951)*
	Polarographic	*(200, 870)*
	Volumetric, acid–base	*(419)*
	Volumetric, ox.–red.	*(523, 1267, 2025)*
Molybdenum	Neutron activation analysis	*(586)*
Nitrogen	Chromatographic, gas	*(1251)*
	Identification	*(1488)*
Phosphorus	Colorimetric	*(1416, 2090)*
	Radiometric	*(2258)*
	Review, methods	*(1383)*
	Spectrophotometric, X-ray	*(2258)*
Potassium	Amperometric	*(2524)*
	Spectrophotometric, flame	*(297)*
Selenium	Colorimetric	*(99)*
	Neutron activation analysis	*(180)*
	Spectrophotometric, ultraviolet	*(737)*
Silver	Chromatographic, thin layer	*(1406)*
	Radiometric	*(1949)*
Sodium	Spectrophotometric, flame	*(296)*
Sulfur	Amperometric	*(7)*
	Identification	*(1448, 2352)*
	Radiometric	*(2258)*
	Review, methods	*(1388)*
	Stability	*(2195)*
Tellurium	Radiometric	*(1197)*
Tin	Colorimetric	*(829)*
Water	Neutron activation analysis	*(1652)*
	Radiometric	*(2204)*
	Refractometric	*(2451)*
	Volumetric, ox.–red.	*(517, 518)*
Zinc	Colorimetric	*(866, 1346)*
	Identification	*(867)*
	Polarographic	*(1346)*
	Radiometric	*(1919, 1924, 1949)*
	Volumetric, complexometric	*(1023, 1113)*

Table IV. Nitrogen-Containing Compounds

Compound or class	Method or subject	Reference
Acridines	Colorimetric	(*516, 2098*)
	Fluorometric	(*557, 1124*)
	Thermogravimetric	(*516*)
	Volumetric, conductometric	(*515, 516*)
	Volumetric, ox.–red.	(*1751*)
Amides	Chromatographic, gas	(*1167, 1624*)
	Chromatographic, ion exchange	(*139, 1991*)
	Chromatographic, paper	(*1829, 1842*)
	Chromatographic, thin layer	(*646, 1573, 1932, 2194*)
	Crystallographic	(*1846*)
	Identification	(*340, 1573, 1710, 2051*)
	Polarographic	(*282*)
	Review, analysis of Phenacetin Intermediates	(*1131*)
	Spectrophotometric, ultraviolet	(*289, 439*)
	Stability	(*2467*)
	Volumetric, acid–base	(*139, 163, 1276*)
	Volumetric, ox.–red.	(*1020*)
Amine oxides	Chromatographic, gas	(*431*)
Amines		
Alicyclic Amines	Gasometric	(*171*)
	Identification	(*166*)
	Stability	(*2456*)
Aliphatic Amines	Colorimetric	(*434, 1678*)
	Volumetric, acid–base	(*434*)
Antihistamines	Chromatographic, gas	(*1320, 1321*)
	Chromatographic, paper	(*713*^{B})
	Fluorometric	(*972*)
	Identification	(*994*)
Miscellaneous Amines	Chromatographic, gas	(*1238*)
	Colorimetric	(*1980*)
	Colorimetric, dye transfer	(*1941, 1942, 1944, 1945*)
	Identification	(*2246*)
	Spectrophotometric, infrared	(*2246*)
	Spectrophotometric, ultraviolet	(*469*)
	Volumetric, dye transfer	(*1836*)
	Volumetric, precipitation	(*426*)
Morpholines	Identification	(*642*)
	Volumetric, acid-base	(*642*)
Nitrogenous Organic Bases	Chromatographic, paper	(*676, 707, 1480, 1953*)
	Chromatographic, thin layer	(*134, 1572*)
	Colorimetric	(*535, 1270, 2039*)
	Crystallographic	(*1153, 1765*)
	Differential thermal analysis	(*1985*)
	Fluorometric	(*1224*^{B}*, 1480, 2370*)
	Identification	(*359, 598*)
	Polarographic	(*1572*)
	Review, methods	(*2497*)
	Spectrophotometric, ultraviolet	(*349, 2303, 2361*)
	Stability	(*411, 1138, 1245*)
	Volumetric, acid–base	(*563, 2476*)
	Volumetric, amperometric	(*1303, 2069*)
	Volumetric, conductometric	(*2321*)
	Volumetric, ox.–red.	(*1462, 1951*)
	Volumetric, precipitation ($^{110}AgNO_3$)	(*1923*)
	Volumetric, turbidimetric	(*2499*)
Phenylalkylamines	Chromatographic, adsorption	(*275*)
	Chromatographic, gas	(*136*^{B}*, 137*^{B})
	Chromatographic, thin layer	(*43*)
	Colorimetric	(*2079, 2390, 2398*)
	Identification	(*398*)
	Polarographic	(*2079*)
	Stability	(*2254*)
	Volumetric, acid–base	(*2338*)
Piperazines	Volumetric, acid–base	(*2337*)
Piperidines	Identification	(*726, 1855*)
	Polarographic	(*452*)
	Spectrophotometric, infrared	(*1455*)
	Stability	(*2154, 2202*)
	Volumetric, acid–base	(*946, 2339*)
	Volumetric, conductometric	(*55*)
Procaine and analogs	Chromatographic, gas	(*2183*^{B})
	Chromatographic, paper	(*2062, 2448*)
	Colorimetric	(*92*^{B}*, 119, 1221, 1774, 2301, 2387*)
	Identification	(*2035*)
	Spectrophotometric, infrared	(*1128*)
	Spectrophotometric, ultraviolet	(*487, 2062*)
	Stability	(*17, 271, 612, 761, 1203, 1227, 1781, 1873, 1883, 2044, 2064, 2279, 2448, 2514, 2515*)
	Volumetric, acid–base	(*791; 1128, 2335*)
	Volumetric, ox.–red.	(*785*)
Pyridines	Chromatographic, paper	(*894, 1648*)
	Colorimetric	(*894, 2212*)
	Identification	(*596, 2314*)
	Polarographic	(*806, 1142*)
	Spectrophotometric, ultraviolet	(*437*)
	Stability	(*1648, 2212*)
	Volumetric, acid-base	(*2341*)
Sympathomimetic Amines	Chromatographic, thin layer	(*21*)
	Gravimetric	(*1107*)
	Identification	(*21*)
	Volumetric, amperometric	(*2083*)
Amino Acids	Chromatographic, gas	(*714, 955*)
	Chromatographic, ion exchange	(*273, 420, 1193, 1315, 1828*)
	Chromatographic, paper	(*32, 74, 233, 968, 970, 1054, 1391, 1647*)
	Chromatographic, thin layer	(*233, 495, 1631, 1632, 1797, 1955, 2231*)
	Colorimetric	(*95, 109, 539, 804*^{B}*, 957, 1666, 2033*)
	Fluorometric	(*1578, 2326*)
	Identification	(*600, 1417*)
	Polarographic	(*2139, 2507*)
	Review, chromatography	(*998*)
	Separation, gel filtration	(*1477*)
	Spectrophotometric, reflectance	(*638*)
	Spectrophotometric, ultraviolet	(*1170, 1391*)
	Stability	(*477, 1482, 1647, 2121, 2434*)
	Volumetric, acid–base	(*1669, 1962, 2036*)
	Volumetric, amperometric	(*2524*)
	Volumetric, complexometric	(*538*)
	Volumetric, ox.–red.	(*1616, 1962*)
Amino Alcohols	Chromatographic, gas	(*2428*)
	Chromatographic, ion exchange	(*360*)
	Chromatographic, thin layer	(*770*)
	Colorimetric	(*770, 1415, 1984*)
	Identification	(*1365*)
	Ionization constants	(*803*)
	Polarographic	(*1348, 1349*)
	Spectrophotometric, infrared	(*1365*)
	Spectrophotometric, ultraviolet	(*1365*)
	Spectropolarimetric	(*1762*)
	Volumetric, acid–base	(*1365*)
	Volumetric, ox.–red.	(*1365*)

(*Continued*)

Table IV. Nitrogen-Containing Compounds (*Continued*)

Compound or class	Method or subject	Reference
Amino Esters	Volumetric, acid–base	(1431)
Amino Ethers	Chromatographic, paper	(513)
	Colorimetric	(1987)
	Spectrophotometric, ultraviolet	(2435)
Amino Phenols	Biological determination	(1051^B)
	Chromatographic, paper	(16, 1390, 1473, 1827)
	Chromatographic, thin layer	(1543, 1763)
	Colorimetric	(232, 670, 779, 802, 1130, 1473)
	Electrophoretic	(552^B)
	Fluorometric	(1409^B)
	Gravimetric	(1038)
	Identification	(996)
	Identification, review	(1461)
	Ionization constants	(802)
	Polarimetric	(1038)
	Polarographic	(869)
	Review, methods	(232)
	Spectrophotometric, ultraviolet	(401, 1171)
	Stability	(73, 578, 878)
Azauridines	Chromatographic, paper	(1441)
	Spectrophotometric, ultraviolet	(1441)
Azepins	Chromatographic, thin layer	(13, 456, 1234^B, 1773, 1831)
	Colorimetric	(456, 878)
	Gravimetric	(456, 469)
	Identification	(1773, 1831, 2257)
	Polarographic	(392^B, 1569, 1570, 1571, 1996)
	Spectrophotometric, ultraviolet	(456, 469, 963)
	Stability	(1608)
	Volumetric, acid–base	(153, 456)
	Volumetric, ox.–red.	(963)
Azomethines	Polarographic	(57)
Barbituric acids and Barbiturates	Chromatographic, gas	(256, 959, 1371, 1524, 1525, 1809^B)
	Chromatographic, paper	(1114, 1164, 2176^B, 2248, 2400)
	Chromatographic, partition	(1840)
	Chromatographic, thin layer	(78, 213^B, 1084^B, 1683^B, 1752, 1886, 2410, 2426)
	Colorimetric	(79, 545, 1288, 1292, 1304^B, 1402, 1903)
	Colorimetric, phase titration	(444^B)
	Crystallographic	(388, 943)
	Differentiation	(1101)
	Extraction	(1291)
	Fluorometric	(2016, 2185^B)
	Identification	(18, 78, 388, 943, 1290, 1293, 1294, 1295, 1683, 1771)
	Microsublimation	(78)
	Oxidative resolution (mixtures)	(2131, 2132)
	Polarographic	(407, 657)
	Refractometric	(1438, 1439)
	Review, methods	(155, 2252)
	Solubility (^{14}C labeling)	(1982)
	Spectrophotometric, reflectance	(257)
	Spectrophotometric, ultraviolet	(288, 1840, 2426)
	Stability	(494, 572, 573)
	Sublimation	(782)
	Volumetric, acid–base	(1332, 2260)
	Volumetric, amperometric	(162, 1491)
	Volumetric, complexometric	(942)
	Volumetric, coulometric	(1021, 1537)
	Volumetric, ox.–red.	(964)
	Volumetric, photometric	(1660)
	X-Ray diffraction	(77)
Carbamates	Chromatographic, thin layer	(846, 1279)
	Colorimetric	(274, 846)
	Identification	(554)
	Review, methods	(554)
	Spectrophotometric, infrared	(554)
	Spectrophotometric, NMR	(2080)
	Stability	(507)
	Volumetric, acid–base	(173)
Hydantoins	Colorimetric	(1070)
	Identification	(1053, 1665)
	Microsublimation	(1053)
	Polarographic	(1585)
	Spectrophotometric, ultraviolet	(1665)
Guanidines	Chromatographic, paper	(1617^B)
	Colorimetric	(241, 1617^B)
	Volumetric, acid–base	(1732)
	Volumetric, ox.–red.	(944)
Hydrazides	Chromatographic, adsorption	(102^B)
	Chromatographic, ion exchange	(1010)
	Colorimetric	(19, 484, 555, 1010, 2235, 2277, 2516)
	Complex with *p*-aminosalicylic acid	(1811)
	Dissociation constants	(1902)
	Gasometric	(352, 2359)
	Gravimetric	(756, 1695)
	Identification	(340)
	Mass spectroscopic	(352)
	Polarographic	(267)
	Spectrophotometric, ultraviolet	(531)
	Stability	(1010, 1011, 2384)
	Thiocyanato–metal complexes	(755)
	Volumetric, acid–base	(2236)
	Volumetric, complexometric	(393)
	Volumetric, conductometric	(54, 891)
	Volumetric, ox.–red.	(741, 1694, 1695, 2277, 2384, 2493)
Hydrazines	Gasometric	(2360)
	Volumetric, amperometric	(1024)
	Volumetric, coulometric	(1586)
	Volumetric, ox.–red.	(914, 1024)
Hydrazones	Volumetric, acid–base	(224)
	Volumetric, ox.–red.	(157, 913)
Hypnotics	Chromatographic, gas	(1928)
	Chromatographic, thin layer	(2278^B)
	Identification	(639)
	Spectrophotometric, ultraviolet	(639)
Imidazoles	Chromatographic, paper	(868)
	Chromatographic, thin layer	(762)
	Colorimetric	(176, 772)
	Stability	(1880)
	Volumetric, acid–base	(1880)
	Volumetric, ox.–red.	(1518)
Imidazolines	Chromatographic, paper	(1645)
	Colorimetric	(1645, 2316)
	Stability	(1645)
	Volumetric, acid-base	(764)
Imides	Chromatographic, gas	(1911)
	Chromatographic, paper	(470)
	Chromatographic, partition	(2082)
	Identification	(2298)
	Polarographic	(840, 841, 842)
	Review, methods	(1232)
	Spectrophotometric, infrared	(1738)
	Spectrophotometric, ultraviolet	(355, 2082)
	Stability	(355, 683)
	Volumetric, acid–base	(945)

(*Continued*)

Table IV. Nitrogen-Containing Compounds (*Continued*)

Compound or class	Method or subject	Reference
Indoles	Chromatographic, ion exchange	(*2470*)
	Colorimetric	(*117, 826*^{B})
	Dissolution rate–solubility	(*805*)
Indolines	Colorimetric	(*1340*)
	Review, methods	(*471*)
	Volumetric, ox.–red.	(*471*)
Naphthyridines	Colorimetric	(*1493*)
	Spectrophotometric, infrared	(*2053*)
	Spectrophotometric, ultraviolet	(*2053*)
	Volumetric, acid–base	(*923*)
Narcotics	Chromatographic, thin layer	(*2296*)
Nitro	Chromatographic, thin layer (^{14}C labeling)	(*506*)
	Colorimetric	(*1129*)
	Distribution coefficients	(*147*)
	Gravimetric	(*1129*)
	Polarographic	(*1627, 2362*)
	Review, methods	(*268*)
	Spectrophotometric, infrared	(*823*)
	Spectrophotometric, ultraviolet	(*544, 877*)
	Volumetric, acid–base	(*1081, 1129*)
	Volumetric, ox.–red.	(*543*)
Nucleic Acids	Chromatographic, thin layer	(*1595*)
	Stability	(*680*)
Nucleosides	Chromatographic, paper	(*329*)
	Spectrophotometric, ultraviolet	(*329*)
Oxazines	Review, methods	(*1183*)
Oxazolidones	Colorimetric	(*454*)
	Polarographic	(*454*)
	Review, methods	(*993*)
	Volumetric, precipitation	(*454*)
Oxazolidinediones	Spectrophotometric, infrared	(*673*)
	Stability	(*673*)
Oxazolinones	Volumetric, ox.–red.	(*156*)
Peptides	Chromatographic, ion exchange, review	(*985*^{B})
Phenanthrenes	Chromatographic, thin layer	(*1637*)
	Spectrophotometric, ultraviolet	(*1637*)
Phthalazines	Colorimetric	(*1111*)
	Identification	(*143*)
	Volumetric, conductometric	(*53*)
	Volumetric, coulometric	(*1019*)
	Volumetric, precipitation	(*754*)
Purines	Chromatographic, thin layer	(*1823*)
	Colorimetric	(*1787, 2395*)
	Deamination	(*2060*^{B})
	Review, methods	(*1280*)
Pyrazolidines	Identification	(*112, 2478*)
	Volumetric, coulometric	(*1025*)
	Volumetric, ox.–red.	(*590*)
Pyrazolones	Chromatographic, paper	(*1378, 1651*)
	Chromatographic, partition	(*1260*)
	Chromatographic, thin layer	(*1651*)
	Colorimetric	(*235, 397, 686, 776, 879, 1058, 1444, 1690, 2112, 2275*)
	Flame photometric	(*299*)
	Identification	(*2331, 2382*)
	Spectrophotometric, infrared	(*1581*)
	Spectrophotometric, ultraviolet	(*1260*)
	Stability	(*1378, 1651*)
	Volumetric, acid–base	(*34*)
	Volumetric, complexometric	(*71*)
	Volumetric, ox.–red.	(*1435, 1436*)
	Volumetric, precipitation	(*2100*)
Pyridoindoles	Chromatographic, paper	(*1541*)
	Spectrophotometric, ultraviolet	(*1541*)
	Volumetric, acid–base	(*1541*)
Pyrimidines	Identification	(*1889*^{B})
	Polarographic	(*253*)
	Review, methods	(*131*)
	Stability	(*87*)
	Volumetric, ox.–red.	(*87*)
Pyrrolidines	Colorimetric	(*165*)
Quaternary Ammonium Compounds	Chromatographic, paper	(*1816*)
	Chromatographic, thin layer	(*685*)
	Colorimetric	(*1141*)
	Crystallographic	(*727, 1766*)
	Identification	(*1764, 1817, 2396, 2424*)
	Microbiological	(*1500*)
	Review, indicator–complex methods	(*174, 1943, 1946*)
	Review, methods	(*1181*)
	Stability	(*1181*)
	Volumetric, acid–base	(*312*)
	Volumetric, thermometric	(*2427*)
Quinazolones	Colorimetric	(*1265, 1510*)
	Identification	(*1331*)
	Volumetric, ox.–red.	(*1331*)
Quinolines	Chromatographic, paper	(*460*)
	Chromatographic, thin layer	(*1162*)
	Colorimetric	(*2484*)
	Fluorometric	(*822*)
	Polarographic	(*324*)
	Review, methods	(*1814*)
	Spectrophotometric, ultraviolet	(*1240*)
	Volumetric, indicator–complex	(*1240*)
Tetrazoles	Colorimetric	(*462*)
	Precipitation with tungstophosphoric acid	(*2247*)
	Volumetric, acid–base	(*2247*)
	Volumetric, complexometric	(*1133*)
Triazines	Chromatographic, paper	(*2200*)
	Stability	(*2200*)
Uracils	Chromatographic, paper	(*40*)
	Chromatographic, partition	(*2067*)
	Colorimetric	(*2179*)
	Review, methods	(*991*)
	Spectrophotometric, ultraviolet	(*205, 1009*)
	Stability	(*1801, 2067, 2179*)
	Stability, gamma radiation	(*2366*)
Urea and Derivatives	Colorimetric	(*917*)
	Identification	(*2446*)
	Review, color reactions	(*2046*^{B})
	Review, methods	(*2369*)
	Stability	(*2280*)
	Tensiometric	(*101*)
	Volumetric, precipitation	(*2446*)
Ureides	Chromatographic, thin layer	(*78*)
	Colorimetric	(*2189*)
	Identification	(*78, 857, 2356*)
	X-ray diffraction	(*77, 1854*)
Xanthenes	Chromatographic, thin layer	(*1653*)
	Spectrophotometric, ultraviolet	(*1184*)
	Stability	(*1653*)
Xanthines	Chromatographic, paper	(*223, 819, 820*)
	Chromatographic, thin layer	(*1262*)
	Colorimetric	(*338, 1981*)
	Identification	(*820, 1682, 1737*)
	Stability	(*2126*)
	Volumetric, amperometric	(*252, 255*)
	Volumetric, complexometric	(*717, 1465, 2024*)
	Volumetric, coulometric	(*1017, 1018*)
	Volumetric, ox.–red.	(*731*)

Table V. Oxygen-Containing Compounds

Compound or class	Method or subject	Reference
Acids and derivatives		
Carboxylic acids, aliphatic	Chromatographic, paper	(*218, 281*)
	Colorimetric	(*195, 281, 321, 382, 1786, 2109, 2323*)
	Fluorometric	(*1453*)
	Gravimetric	(*2495*)
	Spectrophotometric, infrared	(*345*)
	Stability	(*50, 847*)
	Volumetric, acid–base	(*724, 2174, 2495*)
	Volumetric, complexometric	(*538, 1926*)
Carboxylic acids, aromatic	Chromatographic, ion exchange	(*2076*)
	Chromatographic, thin layer	(*1468, 1898*)
	Colorimetric	(*630*)
	Fluorometric	(*2028*)
	General	(*798*)
	Polarographic	(*1007*)
	Spectrophotometric, ultraviolet	(*1007, 2435*)
	Volumetric, complexometric	(*394*)
Esters	Chromatographic, gas	(*2135*)
	Chromatographic, paper	(*2147*)
	Chromatographic, thin layer	(*1898*)
	Colorimetric	(*1988, 2479*)
	Fluorometric	(*2028*)
	Stability	(*507, 1481, 1483, 1502*)
	Spectrophotometric, ultraviolet	(*1241*)
	Volumetric, acid–base	(*2475*)
Salicylic acid and derivatives	Chromatographic, gas	(*1534*)
	Chromatographic, ion exchange	(*2076*)
	Chromatographic, thin layer	(*82, 647, 653, 1705*)
	Colorimetric	(*1345*)
	Coulometric	(*1538*)
	Fluorometric	(*2028*)
	Identification	(*2333*)
	Spectrophotometric, ultraviolet	(*358, 653, 1802*)
	Stability	(*743, 744, 745, 930, 1368*)
Aldehydes	Chromatographic, thin layer	(*2000*)
	Colorimetric	(*1120, 1988, 2487*)
	Identification	(*597*)
	Spectrophotometric, infrared	(*381*)
Carbohydrates	Chromatographic, gas	(*1820*)
	Chromatographic, paper	(*196, 335, 532*)
	Chromatographic, thin layer	(*8, 448, 2378, 2420*)
	Colorimetric	(*152, 1667*)
	Identification	(*153*)
	Physical methods	(*496*)
	Stability	(*1166, 1228, 1460, 1513, 2466*)
	Volumetric, ox.–red.	(*169, 563*)
Chromones	Chromatographic, paper	(*1747, 1933*)
	Chromatographic, thin layer	(*546, 666, 853*)
	Colorimetric	(*91, 1747, 1933, 2367*)
	Fluorometric	(*725*)
	Polarographic	(*1598*)
	Nuclear magnetic resonance	(*1319*)
	Review	(*851*)
	Spectrophotometric, ultraviolet	(*1285, 2052*)
	Volumetric, ox.–red.	(*1080*)
Coumarins	Chromatographic, gas	(*977*)
	Chromatographic, paper	(*383, 384^{B}, 654, 1599, 1933*)
	Colorimetric	(*1779, 1933, 2114*)
	Fluorometric	(*2488*)
	Fractional separation	(*185*)
	Identification	(*47, 643*)
	Polarographic	(*1599, 1600*)
	Spectrophotometric, ultraviolet	(*2488*)
Ethers	Chromatographic, gas	(*14*)
	Chromatographic, paper	(*712, 2147*)
	Chromatographic, thin layer	(*2241, 2242, 2418*)
	Colorimetric	(*14, 634*)
	Polarographic	(*678*)
	Review, methods	(*1794*)
	Review, spectrophotometric	(*1812*)
	Stability	(*1844*)
	Volumetric, ox.–red.	(*1890, 2299, 2404*)
Fats and oils	Chromatographic, adsorption column	(*2146*)
	Chromatographic, gas	(*1379*)
	Chromatographic, paper	(*997, 1566, 2147*)
	Stability	(*290*)
Glycosides		
Cardiac glycosides	Chromatographic, paper	(*198, 560, 2102, 2251^{B}*)
	Chromatographic, thin layer	(*198, 706, 1805, 2097*)
	Colorimetric	(*2072, 2102, 2215, 2217, 2218, 2219, 2220, 2251^{B}, 2508*)
	Identification	(*1458, 2216*)
	Extractive	(*1198*)
	Polarographic	(*2379*)
	Review, identification	(*346*)
	Stability	(*789, 1341, 1587, 2215, 2218, 2219, 2220, 2504*)
Digitoxin	Chromatographic, column	(*1427*)
	Chromatographic, gas	(*969*)
	Chromatographic, paper	(*37, 39, 300, 872, 1088, 1426, 1427, 2191, 2411, 2455*)
	Chromatographic, paper review	(*38*)
	Chromatographic, thin layer	(*258, 587, 967, 1236, 1804, 2095, 2519, 2520*)
	Colorimetric	(*107, 108, 821, 872, 1426, 1728, 2191*)
	Fluorometric	(*927*)
	Identification	(*645*)
	Review, methods	(*347, 1220*)
	Stability	(*258, 1907, 2191*)
Flavones	Chromatographic, column	(*1877, 2125*)
	Chromatographic, paper	(*1284, 1680, 1748, 2125*)
	Chromatographic, thin layer	(*547, 1284, 1711, 2125*)
	Colorimetric	(*1680, 1748*)
	Identification	(*2367*)
	Spectrophotometric, ultraviolet	(*1877*)
General	Chromatographic, gas	(*2210*)
	Colorimetric	(*1668*)
Lanatosides	Chromatographic, thin layer	(*2512, 2522*)
	Stability	(*1199*)
Miscellaneous	Chromatographic, paper	(*2525*)
	Chromatographic, thin layer	(*740, 1136, 1208*)
	Colorimetric	(*509, 581, 2391*)
	Extractive	(*581*)
	Fluorometric	(*2391*)
	Spectrophotometric, ultraviolet	(*671, 1810*)
Phenol	Chromatographic, column	(*2240*)
	Identification	(*2240*)

(*Continued*)

Table V. Oxygen-Containing Compounds *(Continued)*

Compound or class	Method or subject	Reference
Quinones	Chromatographic, paper	*(210, 212, 1177, 1202, 1249, 1680)*
	Chromatographic, thin layer	*(210, 212, 852, 1190, 1297, 2045)*
	Colorimetric	*(1247, 1680, 2045)*
	Polarographic	*(1716)*
	Review	*(580)*
	Review, official methods	*(211)*
	Spectrophotometric, ultraviolet	*(1177, 1297)*
	Volumetric, acid–base	*(1177)*
Santonin	Chromatographic, gas	*(1927)*
	Fluorometric	*(2394)*
	Spectrophotometric, infrared	*(194)*
Saponins	Chromatographic, paper	*(446)*
	Chromatographic, thin layer	*(366, 1078, 1100, 2207)*
	Colorimetric	*(1614)*
	Review	*(893)*
Strophanthin	Chromatographic, column	*(1401)*
	Chromatographic, thin layer	*(423, 424, 697, 1401)*
	Colorimetric	*(423, 697, 1401, 2073)*
	Identification	*(424)*
	Stability	*(2145)*
Hydroxyl, alcoholic	Chromatographic, gas	*(1281, 1808, 1864)*
	Chromatographic, paper	*(281, 467)*
	Chromatographic, thin layer	*(770)*
	Colorimetric	*(242, 281, 309, 350, 634, 770)*
	Identification	*(467)*
	Interactions	*(80)*
	Refractometric	*(2454)*
	Review, methods	*(992)*
	Volumetric, phase measurement	*(709)*
	Volumetric, ox.–red.	*(783, 1048)*
Hydroxyl, phenolic	Chromatographic, gas	*(2156)*
	Chromatographic, ion exchange	*(1075, 1423, 2076)*
	Chromatographic, paper	*(1246, 1535)*
	Chromatographic, thin layer	*(647, 1423, 2108, 2175)*
	Colorimetric	*(140, 238, 935, 1036, 1091, 1743, 2081, 2162, 2363, 2484)*
	Identification	*(1036, 2081, 2368)*
	Polarographic	*(58, 2162)*
	Spectrophotometric, infrared	*(1075)*
	Spectrophotometric, ultraviolet	*(1246, 1382, 1517)*
	Stability	*(58, 837)*
	Volumetric, acid–base	*(688)*
	Volumetric, complexometric	*(890)*
	Volumetric, ox.–red.	*(154, 1454, 2165, 2438)*
	X-ray analysis	*(1845)*
Hydroxyl, polyhydric	Chromatographic, column	*(1452)*
	Chromatographic, paper	*(467)*
	Colorimetric	*(634, 1505, 2104)*
	Identification	*(467)*
	Review, methods	*(541, 916)*
	Spectrophotometric, infrared	*(823)*
	Volumetric, ox.–red.	*(168, 1047)*
Ketones	Chromatographic, gas	*(1864)*
	Chromatographic, paper	*(2031)*
	Chromatographic, thin layer	*(1201)*
	Colorimetric	*(1772)*
	Fluorometric	*(2028)*
	Identification	*(863)*
	Polarographic	*(449, 450, 453)*
	Volumetric, ox.–red.	*(1044, 1189)*
Lactones	Spectrophotometric, infrared	*(2313)*
	Volumetric, acid–base	*(2313)*
	Volumetric, ox.–red.	*(2313)*
Quinones	Chromatographic, paper	*(1102, 1162, 1250)*
	Chromatographic, thin layer	*(459, 2432)*
	Colorimetric	*(2317, 2412)*
	Identification	*(1250)*
	Polarographic	*(1716)*
	Stability	*(1248)*
	Volumetric, acid–base	*(1815)*

(Continued)

Table VI. Stability (General)

Compound or class	Method or subject	Reference
Aerosols	Stability testing, reaction kinetics	*(1979)*
Drugs	Autooxidation and antioxidants	*(1093)*
	Review, use of preservatives in	*(2505)*
	Stability in polyethylene	*(1520)*
Drug Stability	Kinetics of	*(285, 883, 1299, 1729)*
	Prediction of	*(571, 679, 895, 1090, 1108, 1300, 2492)*
	Review of	*(70, 1318, 2315, 2346, 2433)*
	Solubility analysis for measurement of	*(416)*
Fat Bases	Stability testing of	*(2449)*
Glass Containers	Physical and chemical control of	*(1469)*
Liquid Pharmaceuticals	Effect on plastics and rubbers	*(1833)*
Parenterals	Stability of preservatives in	*(1795)*
Pharmaceuticals	Radioactive irradiation of	*(302, 303, 2440)*
Tablet Formulations	Physical and chemical stability testing of	*(1230)*

Table VII. Steroids and Hormones

Compound or class	Method or subject	Reference
Adrenocorticals		
Aldosterone	Chromatographic, thin layer	(722)
Betamethasone	Chromatographic, column	(2355)
	Chromatographic, thin layer	(801, 1934)
Corticosterone	Chromatographic, paper	(2115^B)
	Chromatographic, thin layer	(722)
Cortisone	Chromatographic, paper	(362, 503, 1213, 2115^B)
	Chromatographic, thin layer	(722)
	Colorimetric	(1174, 1213, 1548)
	Nuclear magnetic resonance	(436)
	Optical rotatory dispersion	(1504)
	Spectrophotometric, ultraviolet	(269, 1174, 1504)
Dexamethasone	Chromatographic, thin layer	(801)
	Colorimetric	(759)
	Polarographic	(370)
	Spectrophotometric, ultraviolet	(269)
Fluorocortisone	Chromatographic, thin layer	(415)
	Stability	(415)
General	Chromatographic, gas	(286)
	Chromatographic, paper	(41^B, 342^B, 981, 1213)
	Chromatographic, thin layer	(343, 403, 801, 1312, 2113, 2345)
	Colorimetric	(41^B, 1213)
	Electrophoretic	(342^B)
	Review	(76)
	Spectrophotometric	(41^B)
Hydrocortisone	Chromatographic, gas	(1848)
	Chromatographic, paper	(503, 1213, 2115)
	Chromatographic, thin layer	(722)
	Colorimetric	(182, 1548)
	Crystallographic	(197)
	Spectrophotometric, infrared	(197)
	Spectrophotometric, ultraviolet	(197, 269)
	Spectrophotometric, X-ray	(197)
Prednisolone	Chromatographic, ion exchange	(899)
	Chromatographic, paper	(1213)
	Chromatographic, thin layer	(1529)
	Colorimetric	(182, 1175, 1213, 1214, 1314)
	Crystallographic	(197)
	Spectrophotometric, infrared	(197)
	Spectrophotometric, ultraviolet	(269, 1498)
	Spectrophotometric, X-ray	(197)
	Stability	(971, 1574)
Prednisone	Chromatographic, paper	(1213)
	Chromatographic, thin layer	(1529)
	Colorimetric	(1175, 1213, 1498)
	Spectrophotometric, ultraviolet	(269, 1498)
Triamcinolone	Colorimetric	(59)
	Volumetric, precipitation	(987)
Androgens		
Androsterone	Chromatographic, paper	(595)
	Chromatographic, thin layer	(357, 728)
Adrosteneol	Chromatographic, paper	(739)
	Chromatographic, thin layer	(337, 739)
	Colorimetric	(1986)
Fluandrenolone	Chromatographic, column	(960)
General	Chromatographic, thin layer	(701)
Methandrostenolone	Polarographic	(370)
	Spectrophotometric	(1173)
Testosterone	Colorimetric	(492, 1059, 1139, 1140, 1882)
	Fluorometric	(702, 1868)
	Nuclear magnetic resonance	(436)
	Spectrophotometric, ultraviolet	(1035)
	Volumetric, ox.–red.	(1879)
Estrogens		
Estradiol	Chromatographic, column	(260, 1568)
	Chromatographic, gas	(1196, 2267)
	Chromatographic, paper	(1776)
	Chromatographic, thin layer	(615, 958)
	Colorimetric	(492, 1311)
	Fluorometric	(958, 1317)
	Optical rotatory dispersion	(1504)
	Spectrophotometric	(1504)
Estrenol	Chromatographic, thin layer	(337)
	Colorimetric	(669, 1055, 1057)
Estriol	Chromatographic, column	(260, 1568)
	Chromatographic, gas	(1196, 2267)
	Chromatographic, thin layer	(615, 958)
	Colorimetric	(1311)
	Fluorometric	(958)
Estrone	Chromatographic, column	(260, 1110, 1568)
	Chromatographic, gas	(1196, 2267)
	Chromatographic, thin layer	(958, 1109, 1110, 1311, 1430, 1503)
	Fluorometric	(958, 2085)
	Optical rotatory dispersion	(1504)
	Spectrophotometric, ultraviolet	(1504)
	Volumetric, acid–base	(735)
Ethinylestradiol	Chromatographic, gas	(1967, 2209)
	Chromatographic, paper	(1000)
	Chromatographic, thin layer	(568)
	Colorimetric	(568)
	Fluorometric	(1396)
General	Chromatographic, paper	(1000)
	Chromatographic, thin layer	(1313)
Mestranol	Colorimetric	(373)
General	Chromatographic, absorption	(1531)
	Chromatographic, gas	(463, 871, 1046, 1121)
	Chromatographic, paper	(512, 1155)
	Chromatographic, thin layer	(525, 708, 830, 1225, 1664, 2133, 2208, 2372)
	Dichroimetry	(1677)
	Identification	(605)
	Nuclear magnetic resonance	(436)
	Radiometric	(512, 1046)
	Review	(1821)
	Spectrophotometric, infrared	(519)
	Volumetric, ox.–red.	(732, 733, 734, 2086)
Insulin	Polarographic	(2374)
	Radiometric	(124)
Keto steroids	Chromatographic, paper	(2075)
	Chromatographic, thin layer	(1906)
	Colorimetric	(106, 1286, 1544, 1676 1906, 1969)
	Volumetric, ox.–red.	(1082)
Miscellaneous	Amperometric	(606)
	Chromatographic, paper	(341, 432)
	Chromatographic, thin layer	(658, 797, 1195)
	Colorimetric	(341)
	Review	(2227)
	Spectrophotometric, ultraviolet	(658)
Oxosteroids	Chromatographic, gas	(354)
	Chromatographic, thin layer	(2116^B)
	Colorimetric	(898, 1233, 1512, 1679, 1968, 1969, 1970)
	Identification	(898, 1282)
	Review	(1419^B)
Oxytocin	Chromatographic, partition	(2469)
Pregnane	Spectrophotometric, X-ray	(1579)
Progesterone	Chromatographic, thin layer	(722)
	Colorimetric	(492)
Stanols	Chromatographic, thin layer	(328, 929)
	Colorimetric	(1511)
Sterols	Chromatographic, thin layer	(69, 923, 929)
	Colorimetric	(1056, 1356, 1511, 2523)
	Volumetric, ox.–red.	(2213)
Vasopressin	Chromatographic, thin layer	(604^B)

Table VIII. Sulfur-Containing Compounds

Compound or class	Method or subject	Reference
Benzothiadiazines	Chromatographic, thin layer	(*522*)
	Countercurrent distribution	(*2464*)
	Degradation kinetics	(*858*)
	Identification	(*1092*)
	Review, methods	(*1013*)
	Spectrophotometric, infrared	(*90*)
	Spectrophotometric, ultraviolet	(*90*)
	Stability	(*858, 2464, 2465, 2468*)
Phenothiazines	Chromatographic, gas	(*316, 628, 777, 778*)
	Chromatographic, ion exchange	(*2119*)
	Chromatographic, paper	(*1466*)
	Chromatographic, thin layer	(*1180, 1361, 1362, 1545, 1546, 1549 1865, 1997, 2182*)
	Colorimetric	(*988, 1714, 1798*)
	Extraction	(*414*)
	Fluorometric	(*1398*)
	Gravimetric	(*750, 2190*)
	Identification	(*221, 988, 1151, 1180, 2066, 2290*)
	Polarographic	(*1754*)
	Review, methods	(*129, 705, 2065*)
	Spectrophotometric, infrared	(*1580, 2413*)
	Spectrophotometric, NMR	(*2413*)
	Spectrophotometric, ultraviolet	(*135, 1704, 2065, 2413*)
	Spectrophotometric, visible	(*135*)
	Stability	(*379, 601, 602, 616, 778, 1643, 1997, 2364*)
	Volumetric, acid–base	(*83, 1533, 1589, 1640, 1641, 1642, 2065*)
	Volumetric, amperometric	(*160, 161, 1490*)
	Volumetric, complexometric	(*750*)
	Volumetric, conductometric	(*564*)
	Volumetric, coulometric	(*1404, 1405*)
	Volumetric, ox.–red.	(*170*)
Sulfonamides	Chromatographic, paper	(*746, 1322, 1825, 1992, 2287, 2354*)
	Chromatographic, thin layer	(*626, 672, 747, 1029, 1277, 1363, 1422, 1467, 1523, 1629, 1630, 1721, 1807, 1825, 2401, 2425*)
	Collaborative study, mixtures	(*1212*)
	Colorimetric	(*395, 410, 1060, 2286, 2388, 2397, 2401*)
	Crystallographic identification	(*1540*)
	Identification	(*247, 990, 1243, 1467, 1753*)
	Review methods	(*2, 1014, 1851*)
	Solubility	(*952, 1755, 1909*)
	Spectrophotometric, infrared	(*247*)
	Spectrophotometric, ultraviolet	(*457*)
	Stability	(*44, 308, 400, 1644, 1646*)
	Thermal analysis	(*348*)
	Volumetric, amperometic	(*159*)
	Volumetric, complexometric	(*1742, 1891*)
	Volumetric, coulometric	(*1749*)
	Volumetric, ox.–red.	(*93, 575, 886, 1231, 2452*)
	Volumetric, turbidimetric	(*2059*)
Sulfur (Miscellaneous)		
Benzothiepins	Spectrophotometric, ultraviolet	(*963*)
	Volumetric, ox.–red.	(*963*)
Cyclohexylsulfamates	Colorimetric	(*919*)
	Review, pharmaceutical applications	(*1425*)
2,3-Dimercapto-1-Propanol	Volumetric, coulometric	(*1089*)
Disulfides	Volumetric, complexometric	(*314*)
Isothiocyanates	Colorimetric	(*1132*)
	Volumetric, precipitation	(*481*)
6-Mercaptopurine	Chromatographic, paper	(*2406*)
Methionine	Volumetric, ox.–red.	(*915*)
Phosphorothioates	Chromatographic, gas	(*1033*)
Saccharin	Chromatographic, gas	(*769*)
Sodium Noramidopyrine methanesulfonate	Volumetric, precipitation	(*589*)
Sulfamoylureas	Colorimetric	(*2447*^{B})
Sulfides	Chromatographic, gas	(*1965*)
Sulfonic acids	Chromatographic, paper	(*1628*)
Sulfonylureas	Review, methods	(*183, 2414*)
	Volumetric, coulometric	(*1022, 1026*)
Sulfophthaleins	Chromatographic, adsorption	(*910*)
Sulfoxides	Polarographic	(*627*)
Sulfur	Gravimetric	(*1352, 1400*)
	Polarographic	(*2157*)
	Volumetric, ox.–red.	(*2168*)
	Volumetric, precipitation	(*1352, 2106*)
Thiadiazines	Stability	(*2309*)
	Volumetric, complexometric	(*2309*)
Thiazoles	Spectrophotometric, infrared	(*2054*)
	Spectrophotometric, ultraviolet	(*2054*)
	Volumetric, acid–base	(*1744*)
Thiazolidines	Volumetric, ox.–red.	(*736*)
Thioamides	Colorimetric	(*1784*^{B})
	Spectrophotometric, ultraviolet	(*1740*)
	Volumetric, ox.–red.	(*88*)
Thiobarbituric acids	Colorimetric	(*1516*)
	Chromatographic, paper	(*1516*)
	Polarographic	(*2253*)
	Review, mercury complexes	(*1725*)
	Review, reaction with aldehydes analytical applications	(*1414*)
	Stability	(*1516*)
	Volumetric, complexometric	(*662*)
Thioctic acid	Chromatographic, gas	(*924, 925*)
Thioglucose	Volumetric, complexometric	(*386*)
	Volumetric, ox.–red.	(*386*)
Thioimidazoles	Spectrophotometric, infrared	(*824*)
Thioxazolidones	Spectrophotometric, ultraviolet	(*774*)
Thioxolones	Polarographic	(*2158*)
Thiophenes	Volumetric, ox.–red.	(*1998*)
Thio-TEPA	Chromatographic, thin layer of ^{35}S-labeled	(*1440*)
Thiouracils	Identification	(*2015*)
	Volumetric, ox.–red.	(*912*)

Table IX. Vitamins

Compound or class	Method or subject	Reference
Oil soluble		
Vitamin A	Chromatographic, column	(*365*)
	Chromatographic, gas	(*526, 2471*)
	Chromatographic, paper	(*1063, 1770*)
	Chromatographic, thin layer	(*336, 391, 950, 979, 1068, 1222, 2330*)
	Colorimetric	(*524, 692, 986, 1210, 1222, 1254, 1661, 1769, 1872, 2294, 2329*)
	Extractive	(*497, 614, 1509*)
	Polarographic	(*1335*)
	Review, official methods	(*497, 498*)
	Spectrophotometric, infrared	(*1583, 2177*)
	Spectrophotometric, ultraviolet	(*986, 1420, 1509, 1575, 2436*)
	Stability	(*333, 833, 1062, 1069, 1116, 1364, 1777, 1822, 2221*)
Vitamin D	Chromatographic, column	(*111, 501, 1552, 2285, 2461*)
	Chromatographic, gas	(*2358*)
	Chromatographic, paper	(*501*)
	Chromatographic, thin layer	(*216, 226, 655, 828, 1446, 1521, 1552, 1625*)
	Colorimetric	(*111, 226, 501, 1256, 1521, 2310, 2461*)
	Extractive	(*364*)
	Fluorometric	(*363*)
	Identification	(*2310*)
	Microbiological	(*2228*)
	Polarographic	(*1333, 1334*)
	Review	(*1094, 1486*)
	Stability	(*364, 1094*)
	Spectrophotometric, ultraviolet	(*110, 472*)
Vitamin E	Chromatographic, gas	(*332, 1508, 2023*)
	Chromatographic, thin layer	(*1067, 2077, 2138, 2234*)
	Colorimetric	(*1067, 1375*^{B}*, 1610, 1952, 2294*)
	Polarographic	(*1122, 2249*)
	Stability	(*833, 2023*)
Vitamin K	Chromatographic, column	(*1301*)
	Chromatographic, gas	(*332, 1268*)
	Chromatographic, thin layer	(*548*)
	Stability	(*817*)
Water soluble		
B Complex	Chromatographic, ion exchange	(*1118, 1119*)
	Chromatographic, paper	(*738*)
	Chromatographic, thin layer	(*902, 1308*)
	Fluorometric	(*42*^{B}*, 1178, 1179*)
	Identification	(*31*)
	Review	(*1008*)
B$_1$, Thiamine	Chromatographic, paper	(*261, 1937*)
	Chromatographic, thin layer	(*468*)
	Colorimetric	(*25, 26, 476*^{B}*, 859, 1872*)
	Electrophoretic	(*353*)
	Fluorometric	(*476*^{B}*, 1745, 2311*)
	Identification	(*859, 2319*)
	Isotopic dilution	(*814, 815, 1553*)
	Polarographic	(*499, 567, 2250*)
	Review	(*1995*)
	Spectrophotometric, ultraviolet	(*1359*)
	Stability	(*67, 225, 510, 1216, 1777, 2205, 2443*)
	Volumetric, acid-base	(*500, 933*)
	Volumetric, precipitation	(*502*)
	Volumetric, ox.–red.	(*1691*)
Dihydrothiamine	Stability	(*1076, 1077*)
Oxythiamine	Colorimetric	(*476*^{B})
Thiamine pyrophosphate	Stability	(*2494*)
B$_2$, riboflavine	Chromatographic, column	(*2403*)
	Chromatographic, paper	(*2184*)
	Colorimetric	(*24, 465*)
	Complex formation	(*1948*)
	Electrophoretic	(*353*)
	Fluorometric	(*1369*)
	Identification	(*2319*)
	Stability	(*675, 2184*)
	Spectrophotometric, ultraviolet	(*2403*)
B$_6$, pyridoxine	Chromatographic, ion exchange	(*1117*)
	Chromatographic, paper	(*1596*)
	Colorimetric	(*29, 795, 1255, 1800*)
	Electrophoretic	(*353*)
	Identification	(*30, 2319*)
	Spectrophotometric, luminescence	(*132*)
	Spectrophotometric, ultraviolet	(*2008*)
	Stability	(*1777*)
	Volumetric, acid–base	(*933*)
B$_{12}$, Cyanocobalamine	Chromatographic, adsorption column	(*1115*)
	Chromatographic, ion exchange	(*775, 901, 1366, 1739*)
	Chromatographic, partition column	(*1791*)
	Chromatographic, thin layer	(*429, 888, 1746, 2380*)
	Colorimetric	(*292, 429, 514, 775, 1622, 2001*)
	Radiometric	(*318*)
	Radiometric, review	(*1847*)
	Spectrophotometric, ultraviolet	(*130, 148, 1366, 2490*)
	Stability	(*66, 67, 148, 234, 344, 897, 1633*)
	Volumetric, complexometric	(*553, 934*)
	Volumetric, ox.–red.	(*1399*)
B$_{15}$, pangamic acid	Polarographic	(*2324*)
Hydroxocobalamine	Chromatographic, paper	(*327*)
C, ascorbic acid	Chromatographic, disk	(*767*)
	Chromatographic, ion exchange	(*188*)
	Chromatographic, paper	(*1434*)
	Chromatographic, thin layer	(*272, 664*)
	Colorimetric	(*188, 447, 1083, 1494, 1501, 1715, 1872, 2099, 2201*^{B}*, 2293*)
	Identification	(*2319*)
	Spectrophotometric, ultraviolet	(*272*)
	Stability	(*768, 896, 1326, 1604, 1605, 1606, 1607, 1818, 2462, 2463*)
	Volumetric, acid-base	(*339*)
	Volumetric, complexometric	(*75, 1788*)

(*Continued*)

Table IX. Vitamins (*Continued*)

Compound or class	Method or subject	Reference
	Volumetric, ox.–red.	(215, 570, 887, 1494)
Folic acid	Colorimetric	(27)
	Spectrophotometric, ultraviolet	(1358)
	Stability	(1328, 1358)
	Volumetric, ox.–red.	(536)
General	Polarographic	(317)
	Review	(710)
	Spectrophotometric	(317)
	Volumetric, acid–base	(317)
Nicotinamide	Chromatographic, ion exchange	(361, 1117)
	Chromatographic, paper	(786)
	Colorimetric	(28, 396, 934, 1917)
	Electrophoretic	(1358)
	Gravimetric	(2513)
	Identification	(786, 938, 2513)
	Spectrophotometric, ultraviolet	(245, 1733)
	Stability	(610)
Nicotinic acid	Chromatographic, paper	(786)
	Colorimetric	(396)
	Identification	(786)
	Spectrophotometric, ultraviolet	(1701)
	Volumetric, acid–base	(1235)
	Volumetric, complexometric	(540)
Panthenol	Chromatographic, ion exchange	(1117)
	Colorimetric	(309, 310)
Pantothenol	Colorimetric	(311)
	Fluorometric	(1613)
Vitamin P	Identification	(2319)

Table X. Formulations, Mixtures, and General Methods

Compound or class	Method or subject	Reference
A. General Methods		
Acids	Chromatographic, paper	(1954)
	Thermal analysis	(2430)
	Volumetric, acid–base	(1168)
Acids, aryl glycolic	Spectrophotometric, ultraviolet	(349)
Acids, fatty	Chromatographic, paper	(2147)
Acetylenes	Spectrophotometric, ultraviolet	(2047)
Alcohols	Refractometric	(145)
Alcohols, fatty	Chromatographic, paper	(2148)
Alkylating agents	Colorimetric	(2211^{B})
Amines	Chromatographic, gas	(1238, 2357)
	Chromatographic, paper	(1826)
	Chromatographic, thin layer	(561^{B})
	Colorimetric	(440)
	Complexometric	(835)
	Extractive	(116, 907)
	Identity	(48, 63, 2481)
	Volumetric, acid–base	(189)
	Volumetric, complexometric	(689, 1657, 1658)
Amines, benzyl	Identity	(622)
Amines, primary	Volumetric	(1283)
Amines, quaternary	Chromatographic, ion exchange	(2264)
	Chromatographic, thin layer	(2408)
	Colorimetric	(491)
	Miscellaneous	(2243)
Analgesics	Chromatographic, ion exchange	(505, 2071)
	Chromatographic, thin layer	(521, 537^{B}, 565, 1547, 1922, 2503)
	Identity	(1912^{B})
Anesthetics	Chromatographic, gas	(668^{B}, 849, 1127, 1806)
	Review	(2407)
Anesthetics, local	Chromatographic, paper	(1105)
	Chromatographic, thin layer	(665)
	Identity	(1376, 1584, 1819, 2140, 2477)
Anthelmintics	Chromatographic, thin layer	(665)
Anthracene derivatives	Chromatographic, in crude drugs	(1305)
Anticholinergics	Adsorption, on antacids	(204)
Antibiotics	Chromatographic, thin layer	(1383)
	Review, physicochemical methods	(2094)
Antihistamines	Chromatographic, gas	(629)
	Chromatographic, thin layer	(609^{B}, 1471)
	Identity	(994, 995)
	Polarographic	(390)
Antimalarials	Chromatographic, paper	(1479)
Antioxidants	Chromatographic, paper	(1768)
	Spectrophotometric, ultraviolet	(1767)
Antipyretics	Chromatographic, gas	(818, 1074)
	Chromatographic, thin layer	(2503)
Antituberculars	Identity	(2029)
Aromatics	Chromatographic, gas	(920)
Ataractics	Polarographic	(911)
Barbiturates	Chromatographic, paper	(1408)
	Chromatographic, thin layer	(1886, 2013)
	Colorimetric	(2131)
	Identity	(1101)
	Miscellaneous	(155)
	Volumetric, complexometric	(1920)
Benzothiadiazines	Chromatographic, miscellaneous	(9)
Carbamic acid esters	Review	(12)
Citrates	Volumetric, acid–base	(412)
Diuretics	Chromatographic, thin layer	(691)
Dyes	Chromatographic, adsorption	(975, 2137)
	Chromatographic, ion exchange	(749)
	Chromatographic, paper	(1407, 1750)
	Chromatographic, thin layer	(84, 103, 2187)
	Identity	(103, 1407, 1656, 1659, 2137)
Ester	Chromatographic, thin layer	(64)
Ethoxy groups	Volumetric, ox.–red.	(2270)
Fats	Chromatographic, paper	(2147, 2148, 2150)
	Chromatographic, thin layer	(2381)
	Volumetric, ox.–red.	(1169, 1990)
General techniques	Activation analysis	(120)
	Ashing	(2092)
	Capillary luminescence	(2026)
	Catalytic analysis, review	(792)
	Chromatographic, adsorption	(1636)
	Chromatographic, gas	(668^{B}, 711, 849, 920)
	Chromatographic, ion exchange	(562)
	Chromatographic, paper	(10, 11, 999, 1209, 2068)
	Chromatographic, paper, review	(2472)
	Chromatographic, partition column	(374)

(Continued)

Table X. Formulations, Mixtures, and General Methods (*Continued*)

Compound or class	Method or subject	Reference
	Chromatographic, thin layer	*(665, 843, 1065, 1066, 1380, 1705, 1897, 2010, 2012, 2103, 2164*^{B}*, 2284, 2419, 2503)*
	Color reflectance	*(576, 1351)*
	Colorimetric	*(527, 1211, 1316, 1323, 1835, 2070)*
	Complexometric	*(834, 1042, 1172)*
	Dielectric constants	*(809)*
	Drug partitioning	*(511, 999, 1145, 1803)*
	Electrophoretic, paper	*(493)*
	Flame photometric	*(298, 1472)*
	Flask combustion	*(2105)*
	Fluorometric	*(325)*
	Identity, spot tests	*(1287, 1881, 2122, 2320, 2332, 2350, 2482, 2483, 2486)*
	Isotope dilution, review	*(1463)*
	Kinetic	*(807, 808)*
	Mass spectrometric	*(2222, 2223)*
	Microcrystallographic	*(1539, 1861, 1862)*
	Microcrystallographic, critical review	*(262)*
	Microcharacterization	*(263, 264)*
	Nuclear magnetic resonance	*(22)*
	Phosphorimetric	*(2444)*
	Polarimetric	*(939, 1761)*
	Polarographic	*(716, 839)*
	Polarographic, identity	*(529)*
	Radiometric	*(1154, 1463, 2259)*
	Radiometric, review	*(451)*
	Refractometric	*(703, 704, 720, 1309, 1310, 1796, 2196)*
	Solubility analysis	*(416, 2196)*
	Spectrophotometric, infrared	*(122, 1389, 1908)*
	Spectrophotometric, ultraviolet	*(265, 864, 1207, 1323, 1389, 1703, 1835, 2074, 2233)*
	Spectropolarimetric	*(1761)*
	Thermal analysis	*(479, 613, 949, 1061)*
	Volumetric, amperometric, review	*(167)*
	Volumetric, conductimetric	*(51, 52)*
	Volumetric, coulometric, review	*(1387, 1634, 1635)*
	Volumetric, ox.–red.	*(144, 569, 1519)*
	Volumetric, radiometric	*(2259)*
	Volumetric, thermometric	*(479, 480)*
	X-ray	*(667, 1618)*
Hydrophilics	Chromatographic, paper	*(2152)*
Hydroxyquinones	Chromatographic, thin layer	*(753)*
Hypnotics	Chromatographic, paper	*(2262)*
	Chromatographic, thin layer	*(665, 2263)*
	Electrophoretic, paper	*(2261)*
Iodides	Amperometric	*(3)*
Nitrogen	Colorimetric, automatic	*(2283)*
	Volumetric, ox.–red.	*(1964)*
Oils	Chromatographic, gas	*(1205, 1206)*
	Volumetric, ox.–red.	*(1989)*
Oils, essential	Volumetric, distillation	*(2128)*
Oils, umbelliferous	Chromatographic, thin layer	*(62, 854)*
Ointments	Chromatographic, paper	*(2147, 2148)*
Ophthalmic preparation	Miscellaneous	*(326)*
Phenols	Chromatographic, thin layer	*(753)*
Phenothiazines	Chromatographic, ion exchange	*(2119)*
	Chromatographic, thin layer	*(315, 1547)*
Preservatives	Chromatographic, gas	*(926, 1655)*
	Chromatographic, thin layer	*(422, 1705)*
Proteins	Chromatographic; polarographic	*(1027)*
Psychotropics	Chromatographic, thin layer	*(1152, 2245)*
	Identity	*(125*^{B}*, 1152, 2245)*
Purines	Chromatographic, thin layer	*(2503)*
Pyrazolones	Chromatographic, thin layer	*(787)*
Pyridine derivatives	Spectrophotometric, ultraviolet	*(1702)*
Pyrogens	Polarographic	*(584)*
Quinones	Chromatographic, thin layer	*(753)*
	Use as reagent	*(2480)*
Saluretics	Chromatographic, thin layer	*(9)*
Salves	Identity	*(2152, 2153)*
Sedatives	Chromatographic, gas	*(1459*^{B}*)*
Steroids	Chromatographic, thin layer	*(831, 1298, 1383, 1522, 2134)*
Stimulants	Chromatographic, paper	*(649)*
	Chromatographic, thin layer	*(1547)*
Sulfonamides	Chromatographic, thin layer	*(191, 747, 1029, 2503)*
	Critical review	*(1411*^{B}*)*
Surface-active agents	Review	*(175)*
Terpenes	Chromatographic, paper	*(2188)*
Vitamins	Chromatographic, thin layer	*(950, 1064, 1065, 1066, 1383)*
	Review	*(2141)*
Water	Dielectric	*(574)*
	Karl Fischer reagent	*(834)*
	Spectrophotometric, infrared	*(2123)*
Waxes	Identity	*(2149)*
B. Formulations, mixtures and miscellaneous		
Acedicon	Chromatographic	*(181*^{B}*)*
Acetaminophen	Chromatographic, partition column	*(1165)*
Acetylene	Chromatographic, gas	*(2084)*
Acetylsalicylic acid	Chromatographic, gas	*(1259)*
	Chromatographic, partition column	*(1165, 2288)*
	Chromatographic, thin layer	*(105)*
	Mixtures, miscellaneous	*(254)*
	Spectrophotometric	*(2040)*
	Spectrophotometric, ultraviolet	*(402)*
	Volumetric, acid–base	*(248, 973, 1275)*
Acetylsalicylic acid, aluminum salt	Moisture in, spectrophotometric, infrared	*(2123)*
Adenine	Polarographic	*(1385)*
Adenosin-5′-triphosphate	Chromatographic, column	*(1475)*
Allobarbitone	Complexometric	*(1920)*
Amethocaine	Volumetric, acid–base	*(485)*
p-Aminobenzoic acid	Colorimetric	*(625)*
	Extractive; colorimetric	*(92*^{B}*)*
Aminophylline	Chromatographic, ion-exchange	*(632)*
m-Aminophenol	Extractive; colorimetric	*(1470)*
Aminopyrine	Chromatographic, thin layer	*(1192, 2042)*
	Colorimetric	*(1592, 2041)*
	Spectrophotometric, infrared	*(1582)*
	Volumetric, acid–base	*(250, 319, 485, 1194)*
	Volumetric, ox.–red.	*(781)*
p-Aminosalicylate	Volumetric, ox.–red.	*(2048)*
Aminofurin	Spectrophotometric, ultraviolet	*(1672)*
Amphetamine	Chromatographic, partition column	*(1464)*

(*Continued*)

Table X. Formulations, Mixtures, and General Methods *(Continued)*

Compound or class	Method or subject	Reference
Amylocaine	Colorimetric	(*244, 246*)
Analgin	Volumetric, ox.–red.	(*781*)
Androisoxazole	Spectrophotometric, ultraviolet	(*164*)
Anisotropine	Monograph, proposed	(*989*)
Anthracene derivatives	Chromatographic	(*1305*)
	Chromatographic, adsorption column	(*2144*)
	Colorimetric	(*490*)
Antipyrine	Gravimetric	(*35*)
	Volumetric, acid–base	(*1194*)
Arbutin	Chromatographic, thin layer	(*1191*)
Armin	Spectrophotometric, ultraviolet	(*1736*)
Atropine	Phosphorimetric	(*2445*^{B})
	Volumetric, acid–base	(*2342*)
Barbital	Microcrystallographic	(*1289*)
Barbiturates	Volumetric, acid–base	(*203*)
Belladonna alkaloids	Chromatographic, partition column	(*1125*)
Bengal Red	Chromatographic, adsorption	(*974*)
Benzactyzine hydrochloride	Colorimetric	(*2039*)
Benzoic acids	Chromatographic, thin layer	(*1706*)
Benzalkonium chloride	Standard methods, review	(*760*)
Benzoylecgonine	Chromatographic, paper	(*1339*)
Bergapten	Colorimetric; spectrophotometric, ultraviolet	(*1672*)
Betaine	Chromatographic, thin layer	(*2160*)
Bile acids	Chromatographic, thin layer	(*813*)
Bisacodyl	Spectrophotometric, ultraviolet	(*1497*)
Bisadolal oxides	Chromatographic, thin layer	(*1940*)
Levo-*alpha*-Bisabolol	Chromatographic, thin layer	(*1940*)
Bismuth	Volumetric, amperometric	(*5*)
Boric acid	Volumetric, acid–base	(*485*)
Bovine plasma	Electrophoretic	(*1609*)
Brilliant Green	Polarographic	(*1360*)
Bucetin	Chromatographic, paper	(*918*)
Butylhyoscine bromide	Chromatographic, paper	(*473*)
	Gravimetric	(*89*)
	Spectrophotometric, ultraviolet	(*2055*)
Caffeine	Chromatographic, ion exchange	(*1028*)
	Chromatographic, paper	(*1209, 2221*)
	Chromatographic, partition column	(*375, 1165, 2288*)
	Chromatographic, thin layer	(*105, 2193*)
	Colorimetric	(*794*)
	Coulometric	(*1015, 1016, 1017, 1018*)
	Countercurrent, extractive	(*113*)
	Gravimetric	(*1394*)
	Identity	(*330*)
	Nuclear magnetic resonance	(*1259*)
	Spectrophotometric, infrared	(*230*)
	Spectrophotometric, ultraviolet	(*402*)
	Volumetric, acid–base	(*248, 700, 1275*)
	Volumetric, ox.–red.	(*35, 248, 413*)
Caffeine sodium benzoate	Spectrophotometric, ultraviolet	(*104*)
	Volumetric, acid–base	(*603*)
Caffeine sodium salicylate	Volumetric, acid–base	(*603*)
Calcium lactate	Colorimetric	(*295*)
Capsicum	Spectrophotometric	(*827*)
Carbon monoxide	Chromatographic, gas	(*2084*)
Carbon tetrachloride	Chromatographic, gas	(*719*)
Cascara anthracenes	Colorimetric	(*490*)
Cascara sagrada anthrones	Chromatographic, thin layer	(*1296*)
Cellulases	Analysis in enzyme mixtures	(*427*)
Ceresin	Fluorometric	(*556*)
Chamazulene	Chromatographic, thin layer	(*1940*)
Chloroform	Chromatographic, gas	(*719*)
	Volumetric, ox.–red.	(*2349*)
Chlorophyllin	Chromatographic, adsorption column	(*856*)
Chlorpheniramine	Chromatographic, partition column	(*909*)
Chlorpheniramine maleate	Colorimetric	(*2276*)
Chlorpromazine	Assay in mixture	(*100*)
	Phosphorimetric	(*2445*^{B})
Chymotrypsin	Colorimetric	(*1392*)
	Volumetric, acid–base	(*1878*)
Choline	Chromatographic, thin layer	(*2160*)
Cicutine	Colorimetric	(*2386*)
Cital	Volumetric, ox.–red.	(*696*)
Citric esters	Chromatographic, thin layer	(*962*)
Cocaine	Colorimetric	(*244, 246*)
	Phosphorimetric	(*2445*)
Codeine base	Volumetric, acid–base	(*2292*)
Codeine phosphate	Chromatographic	(*908*)
	Chromatographic, partition column	(*909*)
	Colorimetric	(*2040*)
	Countercurrent, extractive	(*113*)
	Volumetric, acid–base	(*973*)
Colamine cephalin	Chromatographic, thin layer	(*2402*)
Cortisones	Chromatographic; spectrophotometric	(*1918*)
Cysteine	Colorimetric	(*2136*)
Cyclamate sodium	Chromatographic, thin layer	(*1031, 1899*)
Cyclohexane sulfamic acid	Complexometric	(*1429*)
Dexamphetamine	Polarimetric	(*275*)
4,4′-Diacetoxydiphenyl-2-pyridylmethane	Spectrophotometric, ultraviolet	(*1497*)
Dibasol	Countercurrent, extractive	(*113*)
	Spectrophotometric, ultraviolet	(*1004*)
	Volumetric, acid–base	(*1670*)
Dicaine	Spectrophotometric, ultraviolet	(*1004*)
	Volumetric, acid–base	(*485*)
	Volumetric, mercurometric	(*2237*)
Dienestrol	Chromatographic, paper	(*2265*^{B})
Diethylpropion hydrochloride	Colorimetric	(*1896*)
Dihydrocodeinone	Chromatographic, paper	(*181*)
Dihydrocodeinone bitartrate	Chromatographic, spectrophotometric	(*908*)
Diiodofluorescein	Radiometric	(*1097*)
Dimethylaminoethyl diphenylhydroxyacetate hydrochloride	Colorimetric	(*2041*)
	Spectrophotometric, ultraviolet	(*313*)
Dimethylaminoethyl benzilate	Chromatographic, thin layer	(*2042*)
Diphenylhydantoin	Chromatographic, partition column	(*1367*)
	Spectrophotometric, ultraviolet	(*1929*)
Dionine	Countercurrent, extractive	(*113*)
	Volumetric, acid–base	(*1670*)
Dipyrone	Spectrophotometric, ultraviolet	(*2055*)
	Volumetric, ox.–red.	(*89*)
Doxylamine succinate	Sustained release study	(*1039*)
Dulcin	Chromatographic, thin layer	(*1031, 1899*)
	Volumetric, ox.–red.	(*1429*)

(Continued)

Table X. Formulations, Mixtures, and General Methods (*Continued*)

Compound or class	Method or subject	Reference
Ecgonine	Chromatographic, paper	(*1339*)
Ephedrine, and salts	Chromatographic, ion exchange	(*203*)
	Chromatographic, paper	(*466*)
	Chromatographic, partition column	(*1464*)
	Colorimetric	(*294*)
	Extractive	(*254*)
Epinephrine	Colorimetric	(*1896*)
Estr-4-en 17 *beta*-ol	Chromatographic, thin layer	(*669*)
Estrogen	Chromatographic, gas	(*1507*^{B})
Ethanol	Chromatographic, gas	(*1542*)
	Ebuliometric	(*2450*)
	Refractometric	(*2453*)
Ethoxybenzamide	Colorimetric	(*1593*)
Ethoxypolysiloxane	Stability, hydrolytic	(*566*)
Ethylaminobenzoate	Miscellaneous	(*254*)
Ethyl chloride	Chromatographic, gas	(*719*)
Ethylendiamine tetracetic acid, and salts	Complexometric	(*2109*)
Eucalyptus	Identity	(*2030*)
Euphylline	Volumetric	(*294*)
Fats	Peroxide value	(*1169*)
Ferrocal	Volumetric, coulometric	(*2089*)
Folia uvae ursi	Miscellaneous	(*1306*)
Folic acid	Colorimetric	(*2136*)
Frangula anthracenes	Colorimetric	(*490*)
Fuchsin	Chromatographic, ion exchange	(*2159*)
Fumaric acid	Identity, in ferrofumarate	(*1843*)
Furazolidone	Spectrophotometric	(*1627*)
Glyceryl trinitrate	Chromatographic, partition column	(*860*)
	Colorimetric	(*146*)
	Volumetric acid–base	(*1590*)
Glycyrrhizic acid	Spectrophotometric, ultraviolet	(*1736*)
Guaiacolsulfonate, potassium	Chromatographic, ion exchange	(*520*)
Hampi	Constituents of	(*659, 660*)
Hawthorne base	Chromatographic, paper	(*1526*)
Hemicellulases	Methods, discussion	(*427*)
Heparin	Standardization of	(*1615*)
Hetrazan	Colorimetric	(*2021*)
gamma Hexachlorocyclohexane	Polarographic	(*1492*)
Hexestrol	Chromatographic, paper	(*2265*^{B})
Hydrocortisone	Colorimetric	(*1639*)
ortho-Hydroxyquinoline-*meta*-sulfonic acid	Volumetric, acid–base	(*2344*)
Inositol	Colorimetric	(*242*)
Intrinsic factor	Radiometric	(*811*^{B})
Iodine	Volumetric, ox.–red.	(*1994*)
Iodochlorohydroxyquin	Colorimetric	(*409*)
Iron	Colorimetric, automatic	(*193*)
	Volumetric, ox.–red.	(*1964*)
Isoniazid	Colorimetric	(*2048*)
	Spectrophotometric, ultraviolet	(*531*)
Isonicotinic hydrazide	Chromatographic, ion exchange	(*1696*)
Isonicotinylisopropylhydrazine phosphate	Spectrophotometric, ultraviolet	(*796*)
Isopimpinellin	Colorimetric; spectrophotometric, ultraviolet	(*1672*)
Isopropylantipyrine	Miscellaneous	(*794*)
	Spectrophotometric, infrared	(*1582*)
Isopropylphenazone	Miscellaneous	(*794*)
Itobarbital	Chromatographic, partition column	(*2288*)
Lactic acid	Colorimetric	(*433*^{B})
Lanolin esters, sterols	Chromatographic, paper	(*2151*)
Lecithin	Chromatographic, thin layer	(*2402*)
Lidocaine	Colorimetric	(*483, 1221*)
Lipase	Turbidimetric	(*2043*)
Lisozime	Chromatographic	(*425*)
Liver hydrolysates	Chromatographic, paper	(*1012*)
Luminal	Volumetric, acid–base	(*295*)
Lysergic acid diethylamide	Chromatographic, thin layer	(*693*)
Marijuana	Chromatographic, thin layer	(*1160*)
	Colorimetric	(*766*)
	Review	(*765, 1964*)
Marsalid	Polarographic	(*267*)
Meclizine	Spectrophotometric, ultraviolet	(*1910*)
Melphalan	Fluorometric	(*372*^{B})
Menthol	Turbidimetric	(*2474*)
Mephenterminesulfate	Chromatographic, partition column	(*1464*)
Metaldehyde	Colorimetric	(*433*)
Methapyrilene	Colorimetric	(*1980*)
Methylethylaminophenylpropanol hydrochloride	Sustained release study	(*1039*)
Methylamphetamine hydrochloride	Chromatographic, partition	(*222*)
Methylcellulose	Chromatographic, paper	(*2148*)
Methyl-*N*-trifluoroacetyl derivatives	Chromatographic, gas	(*931*)
Methylephedrine	Identity	(*2423*)
Methylene blue	Gravimetric; polarographic	(*1360*)
N-Methyl-3-benzoyl-4-hydroxy-4-phenyl-piperidine hydrochloride	Volumetric, acid–base	(*158*)
Methyl-*para*-hydroxybenzoate	Chromatographic, thin layer	(*1706*)
Methylmethionine sulfonium derivatives	Colorimetric	(*1576*)
Molybdenum	Polarographic	(*1457*)
Moroxydine	Spectrophotometric	(*2039*)
Muscarine	Chromatographic, thin layer	(*2160*)
Naphazoline	Identity, micro	(*2423*)
Neon	Chromatographic, gas; in anesthetics	(*2084*)
Neostygmine bromide, sulfate	Colorimetric, spectrophotometric	(*1185*)
Niamid	Polarographic	(*267*)
Nicotine	Chromatographic, ion exchange	(*1536*)
	Distillation	(*2266*)
	Extractive	(*81*)
	Radiometric	(*2489*)
Nitrates, nitrites	Colorimetric	(*1741*)
Nitroglycerin	Chromatographic, partition column	(*860*)
	Colorimetric	(*146*)
	Volumetric, acid–base	(*1590*)
Nitropentone	Analysis, in mixtures	(*1590*)
Nitrous-oxide	Chromatographic, gas	(*1251*)
Norethynodrel	Colorimetric	(*373*)
Novalgin	Gravimetric	(*319*)
Novocaine	Chromatographic, paper	(*2238*)
	Spectrophotometric, ultraviolet	(*1004*)

(*Continued*)

Table X. Formulations, Mixtures, and General Methods (*Continued*)

Compound or class	Method or subject	Reference
Ointments	Chromatographic, paper	(*2153*)
	General	(*1176*)
17-Oxosteroids	Chromatographic, gas	(*1507*^{B})
Oxytetracycline hydrochloride	Colorimetric	(*1639*)
Pachycarpine	Identity	(*2511*)
Papain	Volumetric, acid–base	(*1878*)
Papaverine	Volumetric, acid–base	(*295, 377, 1495, 1670, 2342*)
Paracetamol	Spectrophotometric	(*289, 1163, 1724*)
Pentobarbital	Spectrophotometric; kinetic	(*351*)
Perphenazine	Chromatographic, paper	(*999*)
Persedon	Miscellaneous	(*794*)
Pervitin	Colorimetric	(*2386*)
Phenacetin	Chromatographic, ion exchange	(*794*)
	Chromatographic, paper	(*918*)
	Chromatographic, partition column	(*2288*)
	Chromatographic, thin layer	(*105*)
	Nuclear magnetic resonance	(*1259*)
	Spectrophotometric, ultraviolet	(*35, 402, 413, 2040*)
	Volumetric, acid–base	(*1275*)
	Volumetric, ox.–red.	(*248, 973*)
Phenazone	Volumetric, ox.–red.	(*413*)
Phendimetrazine; phenmetrazine	Volumetric, acid–base	(*642*)
Phenobarbital	Chromatographic, ion exchange	(*632*)
	Chromatographic, paper	(*999*)
	Chromatographic, partition column	(*1125, 1367, 2431*^{B})
	Chromatographic, thin layer	(*105, 1192*)
	Complexometric	(*1920*)
	Kinetic	(*351*)
	Phosphorimetric	(*2445*^{B})
	Spectrophotometric, ultraviolet	(*351, 905*^{B}*, 1929*)
	Volumetric, acid–base	(*35, 377, 413, 632, 1495*)
	Volumetric, argentometric	(*319*)
Phenothiazine; derivatives	Spectrophotometric, ultraviolet	(*2288*)
Phenol	Colorimetric	(*2214, 2485*)
Phenurone	Colorimetric	(*2189*)
Phenylbutazone	Volumetric, acid–base	(*250*)
Phenylephrine	Chromatographic, partition column	(*908, 909*)
	Chromatographic, thin layer	(*2269*)
	Methods, discussion	(*760*)
	Stability	(*2269*)
	Colorimetric	(*1901, 2389, 2393*)
Phenylpropanolamine hydrochloride	Chromatographic, partition column	(*1464*)
Phenylquinoline carboxylic acid	Analysis, in mixtures	(*254*)
Phenytoin	Chromatographic, partition column	(*2431*^{B})
	Extractive; spectrophotometric	(*905*^{B})
	Spectrophotometric, ultraviolet	(*2431*^{B})
Pholcodine	Chromatographic, paper	(*466*)
Phosphacol	Spectrophotometric	(*1736*)
Phthalic esters	Chromatographic, thin layer	(*962*)
Piperazine citrate	Colorimetric	(*2021*)
Platyphylline hydrotartrate	Countercurrent, extractive	(*113*)
Polyethylene glycol	Colorimetric	(*527*)
Polyisobutene	Spectrophotometric, infrared	(*1963*)
Poly(ethylene oxide) base	Effect on assays	(*1176*)
Polyvinylpyrrolidone	Chromatographic, paper; miscellaneous	(*2032*)
Potassium	Volumetric	(*378*)
guaiacolsulfonate	Volumetric, ox.–red.	(*294*)
Pregnanediol	Chromatographic, gas	(*1507*^{B})
Procaine hydrochloride	Colorimetric	(*625*)
	Phosphorimetric	(*2445*^{B})
	Volumetric, acid–base	(*2336, 2340*)
	Volumetric, ox.–red.	(*158*)
Promedol	Assay in mixture	(*100*)
Propylhydroxybenzoate	Chromatographic, thin layer	(*1706*)
Pyramidon	Chromatographic, partition column	(*375*)
	Countercurrent, extractive	(*113*)
	Volumetric, acid–base	(*2342, 2344*)
Pyrazolone derivatives	Chromatographic, paper	(*192*)
Pyrethrum	Method review	(*1442*)
Pyridoxal	Spot tests	(*150*)
Pyridoxal derivatives	Chromatographic, paper	(*799*)
	Chromatographic, thin layer	(*729*)
	Colorimetric	(*687*)
Pyridoxine	Spectrophotometric, ultraviolet	(*1910, 2389*)
Pyrrivinium pamoate	Chlorimetric	(*36*)
Quinine	Chromatographic, ion exchange	(*1028*)
	Chromatographic, thin layer	(*1192*)
Rose Bengal	Radiometric; miscellaneous	(*1097*)
Rhubarb anthracenes	Chromatographic, adsorption column	(*2144*)
Rutin	Colorimetric	(*937*)
Saccharin	Chromatographic, thin layer	(*1031, 1899*)
	Colorimetric	(*1429*)
Salicylamide	Chromatographic; spectrophotometric	(*1918*)
	Spectrophotometric, ultraviolet	(*402*)
Salicylic acid	Review, standard methods	(*760*)
	Spectrophotometric, ultraviolet	(*402*)
	Volumetric, acid–base	(*700*)
Salsolidine, salsoline	Volumetric, acid–base	(*1670*)
Senna leaves	Extractive; colorimetric	(*1853*)
	Spectrophotometric, ultraviolet	(*68*)
Sodium-4-aminosalicylate	Chromatographic, ion exchange	(*1696*)
Sodium benzoate	Volumetric, acid–base	(*700*)
Sodium iodide	Radiometric	(*2308*)
Sodium-*ortho*-iodohippurate	Radiometric	(*2308*)
Sodium theobromine	Volumetric, acid–base	(*700*)
Sorbic acid	Chromatographic, thin layer	(*1706*)
Spherophysine	Volumetric, ox.–red.	(*696*)
Stilbestrol	Chromatographic, paper	(*2265*^{B})
Strychnine	Chromatographic, ion exchange	(*1028*)
	Colorimetric	(*2292*)
Sulfadiazine	Chromatographic, paper	(*999*)
Sulprifen	Colorimetric	(*2393*)
Sulpyrine	Colorimetric	(*1591*)
	Equilibria, aqueous	(*1954*)
Sympathol	Colorimetric	(*2389, 2393*)
Tetracaine	Analysis, in mixtures	(*254*)
Tetrachlorethylene	Chromatographic, gas	(*719*)
Theobromine	Amperometric	(*255*)
	Chromatographic, paper	(*222*)
	Chromatographic, thin layer	(*2193*)
	Volumetric, acid–base	(*700*)
	Volumetric, coulometric	(*1018*)

(*Continued*)

Table X. Formulations, Mixtures, and General Methods *(Continued)*

Compound or class	Method or subject	Reference
Theobromine sodium salicylate	Volumetric, acid base	(*603*)
Theophylline	Chromatographic, ion exchange	(*632*)
	Chromatographic, paper	(*222*)
	Chromatographic, thin layer	(*2193*)
	Dissolution rate	(*848*)
	Volumetric, coulometric	(*1017, 1018*)
Theophylline aminoisobutanol	Dissolution rate	(*1039*)
Thyme oil, phenol in	Chromatographic; colorimetric	(*2214*)
Thiamine	Volumetric, ox.–red.	(*696*)
Trichlorethylene	Chromatographic, gas	(*719*)
Triethanolamine	Spectrophotometric, infrared	(*832*)
Triiodothyronine	Radiometric	(*2308*)
Trimeprazine	Chromatographic, paper	(*466*)
Trypsin	Colorimetric	(*1392*)
	Volumetric, acid–base	(*1878*)
Ureides	Colorimetric	(*2189*)
Validol	Turbidimetric	(*2474*)
Vaselines	Fluorometric	(*556*)
Veratrine	Chromatographic, ion exchange	(*1536*)
Xanthine alkaloids	Chromatographic, paper	(*796*)
Zinc oxide	Polarographic	(*1841*)

(*Continued from page 191 R*)

methods for organic compounds, and numerous drug references are cited. A book, "Newer Redox Titrants," (*172*) describes many of the newer and less familiar oxidation–reduction titrants. The "Encyclopedia of Industrial Chemical Analysis," contains various articles of interest, one example of which is Wimer's review of acid–base titrations (*2441*).

Two books, "Dissociation Constants of Organic Acids in Aqueous Solution," by Kortum, Vogel, and Andrussow (*1161*), and "Dissociation Constants of Organic Bases in Aqueous Solution," by Perrin (*1673*), cite data for nearly 5000 compounds, including many of a pharmaceutical nature.

Polarographic analysis of medicinal substances was reviewed and considered in several papers (*441, 528, 752, 965, 1564*).

An investigation of the reliability of chemical analyses of biological materials in a number of French pharmaceutical laboratories was reported (*1079*^{B}).

Lachman (*1230*) reviewed stability in tablet dosage forms. The effect of auxiliary tablet components on the accuracy of analyses of active components was examined (*1860*), and the identification of unknown tablets with the help of systematic classification has been described by Mueller (*1484*).

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Rubber

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THIS REVIEW covers characterization and chemical analysis of rubber. While many methods, such as pyrolysis followed by gas chromatography, are appropriate for both rubbers and plastics, they are included here only when they have been applied to rubber. There are a few exceptions to this rule where the references are particularly interesting or may be obviously extended to rubber. Methods for the identification or determination of rubber and materials in rubber are included, but methods for the analysis of rubber chemicals before they are added to rubber are omitted.

The literature on chemical analysis which became available to the authors between September 1964, the end of the period covered by the last review in this series (*222*), and September 1966, is covered. In a few cases, articles cited in the previous review have been repeated because they have now been translated into English (*29, 70, 78*). In the section on polymer characterization by physical methods, a longer period of time is covered.

Literature searching was done by the mechanized information retrieval center operated by the University of Akron under contract with the Division of Rubber Chemistry of the American Chemical Society (*67*).

The abbreviations recommended in ASTM Designation D1418-65 have again been used (*9*). They are listed in Table I.

GENERAL INFORMATION

Working Group A of Technical Committee 45 of the International Organization for Standardization is compiling methods for analysis of rubber and rubber products which are involved in international trade. Recent progress toward agreement on methods for determination of extract, ash, carbon black, zinc oxide, copper, manganese, and iron was reported (*157*). A survey of polymer analysis was published in "Encyclopedia of Polymer Science and Technology" (*34*). Information on analysis can also be found in the article about the polymer of interest. Haslam and Willis' book deals with analysis of plastics but much of it is also applicable to rubber (*95*). A compilation of physical constants of rubbers has been published (*234*).

POLYMER IDENTIFICATION

Pyrolysis–Gas Chromatography. Differences of opinion about the most desirable conditions for pyrolysis continue to be reported. A study of pyrolysis techniques indicated that the fragments recombine if they are permitted to cool before being injected into the gas chromatograph (*79*). The method that seems to be least sensitive to experimental conditions is pyrolysis of a small sample at high temperature so that very rapid fragmentation to monomer occurs. Some workers found that temperatures above 500° C cause monomers to crack further, thereby destroying the characteristic features of the gas chromatogram (*128*). However, other investigators found that monomers are stable at 1050° C and concluded that it is the best temperature (*55*). It was pointed out that rapid removal of the initial breakdown products from the pyrolysis zone is necessary to prevent secondary fragmentation (*203*). Differences in this feature may be the reason for different viewpoints on optimum temperature. Most of the pyrolysis is done thermally. In addition to pyrolysis, there were preliminary reports on two other methods, electric discharge (*203*) and oxidative degradation (*193*). Neither of these has been thoroughly explored.

If the column effluent is split and then simultaneously examined by flame ionization and electron capture detectors, some surprising identifications can be made (*55*). For instance, emulsion SBR can be distinguished from solution SBR and IR can be distinguished from NR. Rubbers containing chlorine, nitrogen, sulfur, or oxygen have much more electron capture response than hydrocarbon rubbers.

Gas chromatography is more sensitive than infrared for detecting small amounts. This is useful for IIR (*79*).

Any method which uses pyrolysis as the initial step is unable to distinguish *cis-trans* structures in the original sample (*55, 79*). Therefore pyrolysis–gas chromatography cannot completely supplant infrared examination of the unpyrolyzed sample. The ability to distinguish mixtures of polystyrene and polyacrylonitrile from styrene-acrylonitrile copolymers was reported (*128*). The chromatograms of many polymers were reported (*33, 144*). The pyrolysis products of SBR, BR, IR, and EPM were identified (*239*).

Infrared. The long used Dinsmore and Smith method for degrading polymers by refluxing in 1,2-dichloro-

Table I. Abbreviations Recommended by ASTM (9)

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
VSi	Silicone elastomers having both methyl and vinyl containing groups on the polymer chain
IM	Polyisobutylene
EPM	Ethylene–propylene copolymer
EPDM	Terpolymer of ethylene, propylene, and a diene with the residual unsaturated portion of the diene in the side chain
CSM	Chloro-sulfonyl-polyethylene

benzene (*66*) was modified by bubbling air through during digestion (*194*). This makes the dissolution of polymer more rapid. The use of an airbrush to spray polymer solutions onto sodium chloride plates was described (*72*). The solutions are very dilute so the technique is applicable to polymers with limited solubility. The film can be built up to any desired thickness by repeated treatments. Solvent removal is complete under a heat lamp in 15 minutes. To save the cost of rock salt plates, films were cast on KBr disks (*100*). Samples were pyrolyzed and the products were trapped in CCl_4 or CS_2. Infrared spectra were used to identify NR, SBR, NBR, BR, CR, and blends (*205*).

Other Methods. A new approach to rapid polymer identification is examination of the ultraviolet spectrum of the gaseous pyrolysis products (*188*). No preliminary extraction is required and the main products are monomers, many of which have characteristic spectra in the 180–260 mμ region. As little as 5% NR or 3% SBR can be identified. Polysulfide rubber gives CS_2 and CSM gives SO_2. With spectrophotometers capable of going below 200 mμ now available, this method seems to be very interesting.

Some simple qualitative tests for rubber were described (*169*). CR is decomposed by boiling HNO_3 while polyvinyl chloride and CSM are not.

The analyst's task of distinguishing products of different manufacture can be made simpler by incorporating 0.02 to 0.05% of a fluorescent dye in a black stock (*103*). The dye is carried to the surface by wax and fluoresces under ultraviolet light. The only drawback is that the cost is about a cent per pound of rubber product.

Glass transition temperature can distinguish random, random-block, and stereo-block copolymers of ethylene and propylene (*145*).

POLYMER CHARACTERIZATION BY PHYSICAL METHODS

General. By design, the previous review article in this series did not dwell upon physical methods for the characterization of rubbers in terms of molecular weight, molecular weight distribution, transition behavior, etc. (*222*). The present review will cover some of these subject areas. The presentation is by no means comprehensive but is intended to point out some of the important problems in polymer characterization and the techniques which may be used to solve these problems. Since some of these topics have not been covered in recent articles in this series, the literature reviewed often covers a period of greater than the last two years.

In general, the analytical techniques to be considered are aimed at measurements on rubbers in the uncured state. The characterization of the properties of elastomers in the cured state is not considered. In a number of cases, the experimental techniques cited have been primarily aimed at the characterization of nonrubbery polymers, but such papers have been included when it is considered that the experimental techniques can be usefully applied to rubbers.

Among recently published books containing information of use in the physical analysis of rubber are those dealing with the molecular weight of polymers (*53*), molecular weight distribution (*182*), and light scattering (*139*). Other books discuss structure and property relationships in terms of molecular weight, microstructure, elasticity, and transition behavior (*146*) and recent advances in ebulliometry and fractionation (*184*). Still other books cover a variety of physical methods for polymer characterization including thermal analysis, fractionation, centrifugation, and elasto-osmometry (*118*), thermal analyses of polymers (*201*), and instrumental techniques as applied to plastics and rubbers (*232*). An extensive compilation of the properties of polymers has gathered into one source much useful information (*32*). Of particular interest are sections dealing with solid-state properties, solution properties, fractionation, and tables giving physical constants of BR, NR, SBR, IIR, and CR. Another recent book includes useful information on molecular weights, molecular weight distribution, branching, and transition behavior (*153*).

General reviews on elastomers have been published (*4, 5, 58*), including an extensive survey of nitrile rubber (*106*). Other review articles contain useful information on the characterization of polymers (*11, 54*). In more specific areas, publications are available on symposia dealing with thermal analysis of high polymers (*117*), analysis and fractionation of polymers (*155*), and the structure and properties of polymers (*212*).

Molecular Weights. During the period, a number of general papers have appeared dealing with various aspects of the established methods for determining the molecular weights and molecular weight distributions of polymers (*24, 25, 137, 160*). Major advances have been made in instrumentation of automatic membrane osmometers (*160*). The commercially available instruments are the Mechrolab design (*147*) and the Shell design (*185*). Variations of the latter instrument are produced by Hallikainen (*89*), Dohrmann (*68*), and Stabin (*202*). Each of these instruments has its advantages and disadvantages but in terms of speed, reproducibility, and validity of results, the automatic instruments represent a major improvement over previous designs. Their principal disadvantage is the necessity for using semipermeable membranes which may tend to be variable in their diffusion characteristics. However, the designs are such that the presence of an unsuitable membrane can readily be detected. Papers have also been published dealing with general aspects of osmometer design (*64*) and methods by which osmometers with semipermeable membranes can be employed to determine molecular weights of the order of magnitude of that of the solvent (*105*). Dynamic osmometry has been reported to be useful for polymers with molecular weights of 1200 and up (*76*). The preparation of very tight membranes useful down to molecular weights of 500 has been described (*221*).

In general, little new work has been reported on the use of cryoscopic and ebulliometric techniques to determine number average molecular weights. Despite the experimental difficulties, some advances have been made in ebulliometric techniques (*238*) with the useful upper limit of measurement extended to molecular weights of 170,000 (*87*). A cryoscopic method has been published showing measurements in the 4000 to 30,000 range (*166*).

An instrument reported to be useful for molecular weights up to 20,000 has also been described and is based on the sensitive measurement of temperature differences resulting from vapor transport from polymer solutions (*171*). Some applications of this instrument have been reported (*45, 168*).

Other papers dealing with the molecular weights of polymers include reports on sedimentation analysis of SBR (*107*) and *cis*-1,4-polybutadiene (*1*). The determination of molecular weights from sedimentation velocity (*138*), fluorescence spectrometry (*18*), and light scattering (*14*) has been reported. Richardson has illustrated the usefulness of the electron microscope for molecular weights of greater than one million (*183*). A review of the viscometric determination of molecular weight, including extensive experimental results, has appeared (*148*). For synthetic polyisoprene, a rather surprising dependency of density upon molecular weight has been found (*37*).

Molecular Weight Distribution. The last few years have produced major advances in methods for determining the molecular weight distribution of polymers. These advances have dealt with both experimental techniques and methods of validly treating the resultant experimental data. The high level of activity in these areas arises from a growing appreciation that molecular weight distribution is of considerable importance in governing the processing behavior of polymers and, to some extent, the physical characteristics of the finished product. In addition to the analysis of the molecular weight distribution of polymers, techniques have been improved to allow fractionation of polymers on a large scale in order to prepare a series of samples of different molecular weights.

A very useful review has been published by Schneider covering a variety of experimental techniques, together with a discussion of the associated theoretical aspects (*191*). A brief summary of the status of molecular weight distribution methods has also appeared with particular emphasis on applications in the rubber industry (*52*). In general, the classical methods of fractional precipitation from dilute solution or fractional elution of polymers have been replaced by more refined techniques some of which, however, depend upon the same scientific principles. One paper using the classical approach deals with the fractionation of Neoprene W and the changes brought about by mastication (*61*). Apparatus by which the molecular weight distribution of relatively monodisperse polymers may be determined by semiautomatic solvent extraction has been described (*92*). The fractionation of IM at high pressures has also been reported (*187*).

A considerable advance in the ease of determining molecular weight distribution has arisen from the use (*15*) of a chromatographic column from which the polymer is eluted through a temperature gradient by a liquid of gradually increasing solvent power. A number of papers have appeared discussing the principles of operation of the techniques and the effect of experimental conditions on the results obtained (*42, 115, 127, 173, 190, 192, 195*). The role played by the thermal gradient in increasing effectiveness of fractionation has been examined in detail (*192*). The application of the technique to a number of rubbers has been shown, including IM (*170*), CR (Neoprene WRT) and polyisoprene (high 3,4) (*177*), and polybutadienes of various structures (*59, 112, 177, 217*).

Improved experimental techniques for use with the experimental system include a programmed solvent dispenser which allows delivery of a binary solvent mixture of predetermined composition (*230*). Other improvements in instrumentation of solvent gradient and flow control have been proposed (*86*). In addition, experimental methods have been presented which allow a scale-up of the fractionation system so it may serve as both a preparative and an analytical tool (*42, 119, 176, 223*). It appears that this experimental approach may have general applicability to rubbers but in each application the effect of experimental variables on the results must be examined, in particular the possibility of rubber instability during fractionation (*59, 217*).

The experimental technique of gel permeation chromatography (GPC) promises to bring about a major improvement in the analytical methods available for characterizing molecular weight distribution. The possibility of using gel particles for the separation of polymer molecules according to size has been appreciated for a number of years (*178*) but, in general, the applications have been concerned with water soluble materials (*75*). It remained for Moore to point out the usefulness of hydrophobic gels when incorporated in a highly stable chromatographic system with a differential refractometer to monitor the eluent (*159*). The details of the principle of operation have not been well worked out but it appears that as the polymer solution moves through the column, the lower molecular weight polymer enters the pores of the gel while the higher molecular weights are excluded. Consequently, the high molecular weights follow a shorter path and are eluted first. A commercial instrument based on these principles is available (*140*) although other designs have been envisioned (*6*). Instrumentation and applications of GPC have recently been thoroughly reviewed (*46, 47*). The published applications of GPC to rubbers include measurements on butyl rubbers where the results did not agree well with those from precipitation chromatography (*83*). Adams and coworkers have made a thorough investigation of the GPC of polybutadiene including a discussion of the effect of experimental conditions upon the results obtained (*2*). Harmon also compared results from the GPC of *cis*-1,4-polybutadiene with those obtained by a coacervation technique (*91*). An extensive evaluation of the usefulness of GPC as applied to polyisobutenes has been made (*43*). In addition, many papers have been presented dealing with a variety of techniques and applications (*227, 228*). These seminars serve the very useful purpose of allowing early informal communication of progress by the various workers in the field. Reports on methods of treatment of data of this type have appeared (*19*), including a computer program for handling the experimental results (*174*). Papers have also appeared dealing with corrections for the limited resolving power of GPC, one of the major problems when attempts are made to characterize samples of relatively narrow molecular weight distribution (*101, 213–215*). The principle of operation of the system has also been discussed (*98, 149, 229*), including information on the effect of polymer concentration upon the experimental results (*44*). In addition to its usefulness as an analytical tool, GPC shows promise to be useful as a preparative unit but no information is available on the experimental performance of such a scaled-up version (*226*).

The work being reported on GPC results is expanding too rapidly for any adequate summary to be made in a short review of this type. However, the preceding discussion gives a general picture of progress to date. It is to be hoped that future work published will include further investigations on the calibration of the instrument and the quantitative significance of the experimental results obtained. Such information will serve to define the limits of usefulness to which the technique can be extended.

Progress in a number of other techniques for the determination of molecular weight distribution has been described. Results obtained on *cis*-1,4-polybutadiene and SBR with an ultracentrifuge have been found, after corrections for the effect of centrifugal pressure and solution concentration, to be in good agreement with elution chromatography results (*97*). Other applications of sedimentation analysis have been described (*1, 27, 107*). Density gradient centrifugation also appears to be useful for measurement of molecular weight distributions (*73, 74*). Polymer fractionation in a thermal diffusion column (*208*) and by zone refining (*135*) has been described. Another useful technique involving turbidity measurements has been published (*210*) together with improved instrumentation (*209*) and results obtained on an ethylene–propylene copolymer (*84*), with turbidity being automatically obtained as a function of temperature. Measurement of decrease in osmotic pressure by polymer diffusion in an automatic osmometer shows promise of being a useful tool for the measurement of distribution in low molecular weight polymers (*110*).

In the area of treatment of molecular weight distribution data, a numerical method has been proposed for the calculation of the distribution curve and distribution moments (*88*). A critical analysis of the significance of molecular weight distributions derived from fractionation data has been made including the effect of low and high molecular weight cut-off (*121–123*).

Branching. Another characteristic of rubbers which probably is of considerable importance in governing mechanical behavior is the frequency of branch points and the length of the side branches. The branching of synthetic polyisoprenes has been discussed (*175*), and reports have described the intrinsic viscosity (*156, 162*) and light scattering (*189*) behavior of branched molecules. Theoretical papers deal with the evaluation of the extent of branching and the molecular weight distributions of polydisperse polymers with random branching (*20, 200*).

Transition Behavior. Extensive work on the transition behavior of polymers continues to be reported but much of this effort is directed toward detailed examination of the morphology of highly crystalline materials. However, work has appeared on the first- and second-order transition behavior of rubbers and, in some cases, such measurement can serve as a sensitive analytical tool. An extensive review has reported on the transition behavior of polymers as related to chemical structure (*31*). The effect of pressure on the glass transition has been measured (*22*). A number of investigations have been made of the transition behavior of 1,4-polybutadienes, including a detailed dilatometric investigation of the melting behavior of *cis*-1,4-polybutadiene (*154*). Differential thermal analysis (DTA) measurements on *cis*-1,4-polybutadiene have shown the crystallization and melting temperature to be dependent upon the rate of cooling and heating (*56*). Under certain experimental conditions, two crystalline melting points could be detected.

A transition in *cis*-1,4-polybutadiene at about 60° C. has been detected by measurements of internal pressure (*23*) and coefficient of expansion (*172*) and may be related to the processing characteristics of this polymer. By x-ray measurements, the first-order transition behavior of 1,4-polybutadiene with *trans* contents from 71% to 94% has been reported (*21*). Transition measurements on 1,4-polybutadienes with a range of high *cis* contents have also been made (*142, 211*). For ethylene–propylene copolymers, Maurer has presented new data and summarized existing data on the glass transition as a function of composition, including the effect of nonrandom microstructure (*145*).

Further work on ethylene–propylene rubbers has been reported combining length–temperature, volume–temperature, and penetration techniques (*85*). With DTA, the first-order transition of ethylene–propylene copolymers has been examined and related to copolymer composition and degree of randomness (*16*). The glass transition behavior of various block copolymers has been reported, including the effect of overall composition and block size upon the torsion pendulum results (*10*). It has been shown that measurements of melting temperature and degree of crystallinity can be used to determine the average stereosequence length of copolymers (*3*). Among new experimental techniques for measuring transition behavior of rubbers are a thermo-mechanical method (*35*), a rolling ball spectrometer (*51*), and a new dilatometric apparatus (*141*).

Sequence Distribution. The wide range of copolymers that can be prepared with new catalyst systems has resulted in some detailed investigations of such copolymers, particularly with regard to possible nonrandom nature. Dilatometric measurements have proved useful for poly(propylene oxide) (*3*). The possibility of using solubility techniques to investigate irregularities in chemical composition has been discussed (*80*). Fractional crystallization shows promise for separating polymers which are heterogeneous with respect to ease of crystallization (*116*). This technique, combined with turbidimetric measurement of light transmission, has been applied to ethylene copolymers (*114*). The effects of molecular weight and molecular weight distribution were small compared to the effects of structural distribution. The latter were found to depend upon catalyst and experimental conditions. The possibility of using sedimentation velocity or density gradient centrifugation to detect differences in chemical structures has been reviewed (*99*). The chemical composition distribution of ethylene–propylene rubber has been discussed (*164*). A review has appeared on a number of specific applications of physical techniques such as adsorption spectra, NMR, thermodynamics of polymerization, and phase transition behavior, to the analysis of sequence distribution in copolymers (*94*).

Polymer Blends. Another area of increased activity involves the detailed analysis of polymer blends containing one or more rubber components. Of particular interest are measurements of polymer compatibility and, in the case of incompatible systems, the characterization of the heterogeneous structure. The glass transition behavior of rubber blends has been used as one criterion of compatibility, and results are presented for a number of systems (*17*), including a discussion of compatibility related to the solubility parameters of the components (*60*).

A series of photomicrographs resulting from electron microscope studies has been presented showing results for a number of rubber blends of potential technical importance (*120*). Phase contrast techniques and electron microscope studies of fracture surfaces have been applied to a number of rubber blends and the results discussed in terms of the properties of the separate components and the method of blending (*224*). A combination of swelling techniques and electron microscopy has also proved useful in studying rubber blends (*143*). Morris has applied rate of crystallization and glass transition measurements to an investigation of blends of *cis*-1,4-polybutadiene with NR and with SBR (*161*).

Other. Density gradient columns (*132*) offer a rapid and sensitive method of measuring polymer density, of particular use in the detection of small amounts of crystallinity.

POLYMER CHARACTERIZATION BY CHEMICAL AND SPECTROSCOPIC METHODS

General Information. Harwood reviewed the study of sequence distribution of copolymers by chemical degradation (*93*).

SBR. If styrene content is known from other measurements, refractive index can be used to find the 1,2- and 1,4-butadiene content (*206*). The results are in agreement with data determined by reaction with benzoyl peroxide.

BR. The absorption band at 911 cm^{-1} was used for 1,2-structure, 967 cm^{-1} for *trans*-1,4-, and 1658 cm^{-1} for *cis*-1,4-. The sum of the unsaturations agreed with chemical measurements (*237*). Binder extended his previous work by assigning the 740 cm^{-1} band to the hydrogen out-of-plane vibration of *cis*-1,4-structure (*26*).

The number average molecular weight of polybutadiene in the range 800 to 1800 was determined by combining spectroscopic techniques (*71*). NMR counts the number of terminal groups and, near infrared absorption at 1.636 microns, determines their concentration. Molecular weight is calculated from the two.

EPM. The "blockiness" was studied by examining the products of pyrolysis carried out in a hydrogen atmosphere and then passed over a hydrogenation catalyst (*218*). The mixture of saturated hydrocarbons is less complex than the original pyrolyzate. The skeletal configuration is related to polymer structure—e.g., propylene sequences yield 2,4-dimethylheptane. Alternating ethylene and propylene sequences give *n*-butane. Tail to tail propylene gives 2,5-dimethyl hexane, 3-methyl heptane, and 4-methyl octane.

The position of the infrared band in the 930- to 975-cm^{-1} region is related to the number of consecutive methylene groups on either side of a side methyl group (*165*).

EPDM. DCPD (dicyclopentadiene) is incorporated into the polymer through its 9,10-double bond, leaving the 1,2-double bond available for sulfur curing (*57*). The 3060-cm^{-1} band of the 9,10-double bond disappears during polymerization while the 3045-cm^{-1} band of the 1,2-double bond is preserved (*65*).

CR. Ozonolysis produced succinic acid. Absence of the 980-cm^{-1} vinyl and 910-cm^{-1} terminal methylene bands show that the structure is predominantly 1,4- (*12*). This was further studied by NMR measurements which can distinguish head to tail, head to head, and tail to tail addition (*77*).

Polyurethanes. Cured samples were solubilized by heating with dimethyl acetamide and *n*-butylamine (*163*). NMR was then used to measure the sum of biuret and allophanate. One linkage per 10,000 molecular weight can be determined with accuracy of 10 to 20%. In prepolymers the two types of linkage can be differentiated.

DETERMINATION OF POLYMERS IN POLYMER MIXTURES AND CONSTITUENTS IN COPOLYMERS

EPM. The absorbance ratio of the 2.28- and 2.31-micron bands is proportional to propylene content in the range 60 to 100% propylene, regardless of whether the sample is a random copolymer, block copolymer, or a mixture of homopolymers (*28*). Ratios of the 720- to 968-cm^{-1} bands and the 720- to 1163-cm^{-1} bands are proportional to the propylene content only for block copolymers or homopolymer mixtures (*28*). The methyl band at 1155 cm^{-1} is independent of block or random copolymer configuration as long as there is at least one methylene group on each side of the point of branching (*165*).

The use of the 1380-cm^{-1} methyl band has previously been limited to absorbance ratios with other bands or to polymers soluble in CCl_4. The intensity of the infrared absorption requires film samples to be very thin and there has been no practical way to measure film thickness. Now β-ray absorption has been used to make the thickness measurement (*219*). Accuracy is 1%.

Block copolymers can be analyzed to ±10% in the range 10 to 40% ethylene by proton magnetic resonance (*179*). The polymers are dissolved in diphenyl ether and the spectra are measured at 200° C. The method is also applicable to mixtures of homopolymers but not to random copolymers.

Glass transition temperature cannot be used for determination of ethylene–propylene ratio unless the microstructure is known (*85, 145*).

If EPM is pyrolyzed in a stream of hydrogen and the products are passed over a hydrogenation catalyst, all the fragments are hydrogenated. The composition was determined by passing the hydrogenated fragments into a gas chromatograph (*218*). Propylene content is proportional to the ratio of peak height of *n*-heptane to the sum of the peak heights of 2-methyl heptane and 4-methyl heptane. The calibration curve is independent of the sequence of monomers in the copolymer.

EPDM. One of the hydrogenated pyrolysis products from EPDM containing DCPD is cyclopentane (*218*). The ratio of the height of its gas chromatographic peak to that of *n*-butane is proportional to the amount of DCPD present. If the pyrolyzate is not hydrogenated, the height of the cyclopentene peak is linearly related to DCPD content.

The pyrolyzate may be caught in benzene to give a yellow solution whose absorbance at 436 mμ is a measure of DCPD (*90*).

The 3045-cm^{-1} band in the spectra of thin films was also used to determine DCPD (*65*). Results agree with values found by ICl addition.

NBR. The acrylonitrile content of NBR was measured by pyrolysis–gas chromatography using a thermal conductivity detector (*55*).

NR/SBR. Pyrolysis followed by gas chromatography gave linear calibration curves under conditions where monomers were produced (*128, 235*). The accuracy was ±2% (*235*). Accuracy of ±5% was achieved when the pyrolysis products were analyzed by means of the ratio of the infrared bands at 698 and 1380 cm^{-1}.

NR/BR. Pyrolysis–gas chromatography was also used for mixtures of these polymers (*55, 235*), in one case with accuracy of ±3%. Pyrolysis–infrared is accurate to ±5% using the ratio of the 965-cm^{-1} vinyl band of BR and the 1380-cm^{-1} methyl band of NR (*205*). As 1,2-addition, which leads to the vinyl structure, is only a minor part of BR, one must be sure to calibrate with BR made by the same method as that in the samples.

NR/NBR. Pyrolysis–infrared using the ratio of the 2260-cm^{-1} cyanide band and the 1380-cm^{-1} methyl band is accurate to ±5% (*205*).

NR/CR. The ratio of the 1220-cm^{-1} band from CR and the 1380-cm^{-1} methyl band from NR was used (*205*). Different calibration curves were required for cured and uncured samples.

SBR/BR. After degrading the sample in refluxing 1,2-dichlorobenzene, a film was cast. The ratio of the 737- and 1316-cm^{-1} bands is proportional to the BR content (*194*). The absorbance at 1316 cm^{-1} is proportional to film thickness. After pyrolysis, the ratio of the 698- and 1380-cm^{-1} bands was used with accuracy of ±5% (*205*). Pyrolysis followed by gas chromatography measurement of styrene and butadiene monomer gave results accurate to ±2% (*235*).

SBR/NBR. Pyrolysis–gas chromatography with thermal conductivity detection was used (*55*).

CR/NBR. Pyrolysis–gas chromatography with flame detection was used (*55*).

CR/ESM. After pyrolysis, the effluent from a gas chromatographic column was split 1:1 between flame ionization and electron capture detectors (*55*). The ratio of the heights of a flame ionization peak and an electron capture peak was used.

CR/IIR. The ratio of flame ionization and electron capture peaks is reproducibly related to polymer composition (*55*).

NR Grafts. The Weber test (*231*) used to identify NR has been put on a quantitative basis (*196*). The sample is treated with IBr, then phenol, and the resulting color is measured. The method was applied to mixtures and grafts with poly(methyl methacrylate) with accuracy of ±3%. Ungrafted NR was extracted by petroleum ether, poly(methyl methacrylate) by acetone, and polystyrene by ethyl acetate (*81*). The graft copolymer was taken as the material remaining after subtracting the homopolymers found. Accuracy was ±1% at the 40% level.

DETERMINATION OF RUBBER

Various rubbers can be determined by loss of weight under pyrolytic conditions (*104*). The cured, filled sample is acetone extracted, then heated at 520° C under nitrogen. The polymer determined by loss in weight is accurate to ±2% at the 56% level. The method is not applicable to CR which gives off HCl leaving a residue of carbon, which causes low results.

UNSATURATION

All of the new work reported in determination of unsaturation is concerned with EPDM, especially where the diene component is DCPD. The addition of ICl was found to be quantitative on EPDM made with DCPD tagged with carbon-14 (*90*). The amount of substitution is very small and can be neglected. EPDM containing 1,4-hexadiene and 1,7-octadiene also gave good results by ICl addition without correction for substitution (*216*). Where the diene is cyclooctadiene, there is considerable splitting out of HI, and a correction must be made. The amount of ICl taken up by EPM was found to be very low (*134*). The amount of unsaturation in DCPD polymers found by infrared agrees with that found by ICl addition and Br_2 addition (*57*). The 3045-cm^{-1} band was used to determine DCPD (*65*) while others prefer the ratio of the absorbances at 1600 and 4300 cm^{-1} (*90*). ICl addition was used to establish standards for calibration.

HALOGENS

Chlorine. Viscous IM was diluted in isooctane and burned in a Wickbold apparatus (*186*). Traces of chlorine from the samples were absorbed in $Hg(CNS)_2$. The thiocyanate which was displaced formed a complex with ferric iron which was measured colorimetrically. Precision was ±0.1 ppm in the range 3 to 10 ppm. Combustion products from a furnace or oxygen flask were absorbed in 6% H_2O_2 and titrated with $Hg(NO_3)_2$ to the diphenylcarbazole endpoint (*30*). Accuracy is ±0.5% in the 18 to 50% range.

Fluorine. Polymers containing fluorine were mixed with diethylene glycol and Na_2O_2 and decomposed in a Wurzschmitt bomb (*152*). The fluoride was converted to HF by passing the dissolved combustion products through an ion exchange resin in the H-form. The HF was then titrated with NaOH. The relative error was 2% when the sample contained 50% fluorine.

SULFUR AND SULFIDES

Samples were decomposed for determination of total sulfur by burning in a combustion furnace or oxygen flask (*30*). The oxides of sulfur were absorbed in 6% H_2O_2 which converts them to sulfate. The sulfate was then titrated with $Ba(NO_3)_2$ to the carboxyarsenazo endpoint. If Zn is present, it is masked by sodium 2,3-dimercaptopropanesulfonate.

Free sulfur was determined by the method of Brock and Osborne (*36*). It was found that only elemental sulfur reacts with triphenylphosphine and that polysulfide crosslinks do not react at 56° C (*225*). The copper spiral method for free sulfur agreed well with the polarographic determination (*41*).

Other workers found that triphenylphosphine removes sulfur from polysulfide crosslinks at 80° C, converting them to monosulfides (*158*). Sodium di-*n*-butyl phosphite reacts with polysulfides and disulfides, forming mercaptans or sulfides (*158*).

Mercaptans were determined by swelling the sample in acetic acid, adding a measured excess of $AgNO_3$, and back-titrating with KCl to an amperometric end point (*49*). The relative standard deviation is 5 to 7%.

Polysulfide crosslinks are reduced by $LiAlH_4$ according to:

$$R—S_x—R \rightarrow 2RSH + (X - 2)S^{-2}$$

The reaction mixture is treated with acid and H_2S is swept into cadmium acetate solution by a stream of nitrogen (*50*). The sulfide is measured by iodometric titration. The mixture remaining after removal of H_2S is filtered, and RSH is determined by amperometric titration of the filtrate with $AgNO_3$. In a slightly different approach, the acetone extract was titrated with KCN to determine free sulfur (*236*). Inorganic sulfide was released by treatment with HCl in ether. The H_2S was collected in cadmium acetate solution, where it was titrated potentiometrically with KIO_3. The extracted rubber sample was then treated with $LiAlH_4$, and the mixture was titrated potentiometrically with $AgNO_3$ to determine both sulfide and mercaptan from the two breaks in the curve. Monosulfide crosslinks were determined from the difference between total sulfur and the sum of all of the above.

The infrared band at 1600 cm^{-1} increased in intensity up to 20% sulfur when NR was cured with sulfur, with or without accelerators (*133*).

CURING AGENTS

Identification. Papers were published dealing with separation by paper (*199, 233*) or thin layer (*124*) chromatography. The early work on paper chromatography was reviewed (*13*). One scheme was described for separating the acetone extract by column chromatography (*180*). In general, identification after chromatographic separation is based on *R* values and color tests. If a positive identification is required, use of infrared or ultraviolet spectra and other physical properties is preferred (*13*). Once the material is separated the hard work is done, and examination of the spectra of the fractions is well worth the small extra effort required.

Determination. Sulfenamides and reaction products formed during vulcanization have been determined (*40, 41*). The benzene extract was separated on a silica gel column. Where possible the separated materials were converted to MBT (2-mercaptobenzothiazole) which was measured by means of its ultraviolet absorption. MBT is unique in that is has a band at 329 mμ with absorptivity of about 160 liters/gram cm. There are few materials with bands at this long wavelength and also few with such intense absorption. Where conversion to MBT was not feasible, polarographic measurement was used.

Another method for determining sulfenamides and their reaction products in vulcanizates is based on reduction to MBT by refluxing with $SnCl_2$ and HCl *48*). The MBT is then determined by amperometric titration with $AgNO_3$. Mixtures of sulfenamides with MBT, the zinc salt of MBT, or MBTS (benzothiazyl disulfide) were analyzed with a relative error of ±2%.

Methods for determination of MBT, MBTS, and sulfenamides, each one taken by itself, were compared (*108*). These workers concluded that iodimetric titration of MBT is preferable to conductometric titration with $AgNO_3$ or NaOH. However, they did not consider application to commercial stocks where other oxidizable materials, such as age resisters, might be present. The iodine produced when MBTS, sulfenamide, or benzimidazolyl disulfide is treated with KI and acid can be titrated with thiosulfate to determine the curing agent. Adaptations of iodimetric and iodometric titrations were used to analyze mixtures of MBT and MBTS, sulfenamide and MBT, or sulfenamide and MBTS (*109*).

MBT and its derivatives were determined by measuring the ultraviolet absorption of a thin film of the sample (*111*). This is applicable only to rubber without fillers. Polymer-bound materials were determined by analyzing extracted samples.

The dibenzoate ester of *p*-quinone dioxime in IIR was determined by measuring the absorption at 1754 cm^{-1} (*38*).

Diphenylguanidine was determined in the presence of *p*-phenylenediamine-type age resisters by a differentiating nonaqueous titration (*125*). If there are no complications due to the presence of extender oils, this is a simple method.

AGE RESISTERS

Identification. Separation by paper (*13, 233*), thin layer (*124*), or column (*180*) chromatography prior to the use of color tests or absorption spectra has been described for age resisters. Here, as for curing agents, the use of spectra is to be preferred.

Determination. Phenyl-2-naphthylamine and 2,6-di-*t*-butyl-*p*-cresol can be determined in uncured rubber or latex by gas chromatography (*96*). Solid rubbers are dissolved in $CHCl_3$ and latex is diluted with water to give solutions of low viscosity which can be injected into the chromatograph. The internal standard method is used. Relative standard deviation is 5% but recoveries are poor.

The method of Lorenz and Parks (*136*) was extended to *p*-phenylenediamine (*125*). The presence of extending oil or other basic materials interferes. *N*-2-propyl-*N'*-phenyl-*p*-phenylenediamine was determined by a similar method (*63*).

RESIDUAL MONOMERS

The strong ultraviolet absorption band at 250 mμ was used to determine styrene or α-methyl styrene in SBR latex (*70*). The sample was shaken with heptane to extract the monomer and as little as 0.1% was determined to ±5%.

Copolymers of styrene with butadiene and acrylonitrile were dissolved in dimethyl formamide. After adding toluene as internal standard, gas chromatography was used to determine 0.01 to 1% monomer (*198*).

CARBON BLACK

The possibility of saving time has prompted several investigations of pyrolysis methods. Carrying out the pyrolysis in a vacuum was reported to eliminate the need for corrections from concurrent runs on known samples (*181*), as required by ASTM method D1416 for SBR black masterbatches (*8*). The vacuum method has a standard deviation of 0.17% and accuracy of

±0.2%. The use of CO_2 as inert atmosphere was recommended for pyrolysis at 550° C (*78*). Relative error was ±1%. At 800° to 900° C, CO_2 can react with carbon black to form CO and cause low results (*104*). Therefore nitrogen was recommended for high temperature pyrolysis with errors of ±1% at the 25% level. Furnace black sometimes contains 1% volatiles, channel black as much as 6%, causing low results.

IM was decomposed with HNO_3 and xylene according to Kress's method (*126*). The organic matter was then extracted and the residue was dried. The weight loss upon ignition of the residue is the carbon black content (*29*). Fluorine-containing polymers were decomposed with tetrachloroethane, HNO_3, and dimethyl formamide before separating a residue of carbon black and ash which was finished as above (*152*).

When clay is present, errors can occur if the pyrolysis is done at 500°–600° C, and the final ignition at 800°–900° C because of incomplete dehydration of the clay at the lower temperature. In this case, it is better to measure the CO_2 evolved during ignition of the carbon black than to use the loss in weight (*104*).

METALS

Identification. Thin films of vulcanizates were cut with a microtome at −50° C. Infrared spectra of the sections were used to identify silica, clay, $CaCO_3$, and $MgCO_3$ (*204*).

Destruction of Organic Matter. Concentrated H_2SO_4 and 50% H_2O_2 rapidly decompose organic samples for subsequent determination of metals. Fe, Pb, Cd, and Zn, which tend to be lost in dry ashing due to the volatility of some of their compounds, are retained, and the blanks from reagents are low (*207*).

Total Ash. Ash content can be calculated from the weight of residue remaining after ignition of carbon black (*78, 104*). Porcelain crucibles must be avoided in ashing fluorine-containing polymers due to the volatility of SiF_4, which would cause low results (*152*).

EDTA Titration. In a rapid method for Zn, titration of a solution of the ash is carried out at pH 4.5. Ca, Mg, Al, Fe, and Ti are masked by fluoride and 2,4-pentanedione (*113*). This method is superior to titration with ferrocyanide.

Determination of Ca and Mg in the ash of *hevea brasiliensis* leaves was compared in four laboratories (*151*). One used a novel method of precipitating Ca with molybdate and tungstate (*150*). Mg can then be titrated directly in the filtrate. Ca can be titrated directly at pH 12 or calculated by difference from the titration of Ca plus Mg at pH 10. The other three laboratories did the latter two titrations, taking Mg by difference. The direct titration of Mg gave results significantly higher than the indirect determination.

Colorimetric Methods. The ASTM methods for copper in raw natural rubber have been revised (*7*). Zinc dibenzyldithiocarbamate is the reagent in the new referee method. It has the advantage that the copper complex forms in acid solution, thus neutralizing with ammonia is not necessary. Because the solution can be kept acidic, iron does not have to be masked with citrate. Interferences from other metals are low. Extraction is also more rapid. The new alternative method, which is being considered by ISO, uses zinc diethyldithiocarbamate as reagent. It, too, permits rapid extraction.

In an interlaboratory test program, Mn in *hevea brasiliensis* leaves was determined (*151*). There was no significant difference between oxidation to permanganate by potassium periodate or by sodium bismuthate.

X-Ray Diffraction. ZnO in vulcanizates is determined quantitatively and distinguished from zinc stearate (*225*).

Flame Photometry. The determination of Ca in leaves of *hevea brasiliensis* by EDTA titration and flame photometry was found to be in agreement (*62*). Flame photometry is preferred because it is faster.

Emission Spectrometry. Rubber is ashed in a carbon electrode. The electrode is placed in an AC arc to transfer Ti to the upper electrode. The upper electrode is then used for emission spectrometric determination of Ti (*82*). Standard deviation is ±5% in the range 16 to 50% TiO_2.

Radiochemical Methods. The Li in poly(butadienyl) lithium is exchanged with tritium from tritium-labeled alcohol. The sample is then reduced to a mixture of tritium and tritiated methane which is counted (*39*). The results are accurate to ±5% and agree with analysis by carbonation and acid number.

OTHER METHODS

The error of considering that all of the weight lost on heating or milling is due to water was pointed out. A preferred alternative is to pass dry nitrogen over the sample and to use a moisture monitor to measure the water picked up by the nitrogen (*129*). Precision is 20% at the 0.4% level.

The Baker-Mullen cell was modified by placing the trays in a cylinder made of screen (*167*). This prevents the gel from migrating.

Extender oil in EPDM was determined from the absorbance ratio of the 1620- and 4350-cm^{-1} bands of a polymer film (*69*). The accuracy is ±2% of the amount of oil present if a sample of the oil is available for empirical calibration. The sensitivity depends on the type of oil being used as the 1620-cm^{-1} band is due to aromatic structure.

The analysis of leaves of *hevea brasiliensis* for nitrogen and phosphorus was compared in four laboratories (*151*). Nitrogen was determined by Kjeldahl digestion with titration or colorimetric finish while phosphorus was measured colorimetrically as molybdovanadophosphoric acid or as heteropoly blue.

Starch was extracted from NR or SBR foam and hydrolyzed to glucose by HCl. Anthrone and H_2SO_4 were used in the colorimetric finish (*131*).

Vinyl side groups on VSi were treated with P_2O_5 and H_2O to liberate ethylene which was measured by gas chromatography (*102*).

The acetone extract of NR was separated by column chromatography, and the yellow pigment was identified as β-carotene (*220*).

Castor oil in *hevea* crumb was determined by gas chromatography (*130*).

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Solid and Gaseous Fuels

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THIS IS THE TENTH of a series of reviews on methods of sampling, analyzing, and testing solid mineral and gaseous hydrocarbon fuels. The period covered by this review is October 1964 to September 1966, inclusive, and it follows the general format of previous reviews.

SOLID MINERAL FUELS

This section is concerned with studies on methods of sampling, analyzing, testing, and evaluating coal, coke, and related materials. Most of the investigations reported are related to present standard methods and are proposed for the purpose of improving the accuracy or reducing the time of analysis. In the miscellaneous tests section are items of related interest to solid fuel technologists.

Sampling. The proposed ISO specification for coal sampling was tested by two trials by Crawford and Smith (*2A*). Sampling was done at stages between mine and power station; also included was coal transported by sea and rail. Keller (*4A*) made tests to determine the variance of sample preparation and the quantities of sample required for testing. A scheme for the determination of precision for various steps in sampling, sample preparation, and analysis was presented by Aresco and Orning (*1A*). The variances for each step are treated as a function of preparation, top size as sampled, and ash content. Sporbeck (*8A*) describes certain errors that are possible in sampling and indicates acceptable equipment. He introduces some novel instruments.

Tibbetts, Featherby, and Miller (*9A*) prepared guidelines for the design, selection, cost of installation, and justification for installing automatic coal-sampling devices.

In the analysis of coke the quantity of sample required depends on the specific test. Sorkin, Pedan, and Sulimova (*7A*) determined the minimum weight for each test. The validity of the weights of sample selected is based on the comparison of analyses made on individual pieces of coke with those from automatically collected samples per shift.

The preparation of the sample after collection is of equal importance. Riley (*5A*) discussed the Bond theory and described coarse crushing, intermediate crushing, and fine grinding.

Vibration grinding mills have certain advantages over ball mills, according to Smith (*6A*).

A new type of mill for crushing and reducing the quantity of sample, described by Zbraniborski (*10A*), consists of two vertical cylinders separated by a sieve. The upper cylinder has a rotary breaker and the lower one has a device to direct the crushed coal to the sides of the container.

Usually there are varying times of delay between the preparation of the laboratory sample and the analysis. DeSieghardt and Cole (*3A*) compared effects of air, nitrogen, and water on the storage of the laboratory sample.

Proximate Analysis. MOISTURE. Bull (*5B, 6B*) stated that the British Standards minimum-space oven for the weight loss method for moisture in brown coal was not satisfactory. Certain modifications in procedure would be required to make it acceptable.

The advantages and limitations of the British Standards Method 1016 for moisture in brown coal are discussed by Evans (*9B*).

McMillan (*28B*) reports that the ISO procedure for moisture in brown coals and lignites is by distillation with xylene or toluene and not by weight loss on heating.

The moisture in various grades of coal and coke can be determined by nuclear magnetic resonance according to Ladner (*25B*). The interferences due to mineral matter and particle size are described by Ladner and Wheatley (*26B*).

Continuous measurements of moisture in coal and coke with variations in application are described by Kobayashi (*19B*), Dresia and Fischotter (*8B*), Schuricht (*37B*), and Stewart and Hall (*41B*).

The measurement of moisture in coking coals by the dielectric constant method is described by Hornig (*15B*). Its application to brown coal in a briquetting plant is discussed by Kersting (*17B*).

Wilkinson's (*42B*) recent work on the moisture in the laboratory sample of coke showed that the 100° to 110° temperature should be raised to 300°.

Rapid or control methods for moisture have been studied by Nemec and Kas (*32B*). They compared methods based on hot air, infrared, and the reaction of moisture and calcium carbide. Ruschev and Tomova (*35B*) compared heating methods, infrared, xylene distillation, rise in temperature on addition of H_2SO_4, DTA, and conductance measurements.

Lorant and Pollak (*27B*) modified the acetyl chloride method for moisture in coal and obtained satisfactory results.

VOLATILE MATTER. The composition of the volatile matter of coke was determined by Bertling and Echterhoff (*2B*). A thermobalance was used at 880°. They found that the volatile matter evolved was related to the particle size of the sample. Occluded gases and moisture were released at 100° to 200° and the gases at 600° to 700° consisted of decomposition products of compounds formed by oxidation of coke.

Knauf (*18B*) found that the volatile matter of coke consisted of thermally unstable products of carbonization, products of decomposition of oxidation of compounds, and occluded N and CO_2.

Experiments by Peters and Bertling (*34B*) show the effect of rapid devolatilization of coals. The yield of volatile matter is higher at the more rapid rates of heating. There is less gas but much more tar and pitch produced at the rapid rate of heating.

Greenfield and Smith (*12B*) developed a method of consecutively determining volatile matter, fixed carbon, and ash in anthracite. The sample is heated at 925° in a N atmosphere. The volatile matter is passed over Co(II)-Co(III) oxide heated to 625°, the resulting CO_2 was absorbed in KOH and determined by change in conductance. The N in the combustion tube was changed to purified O to convert the fixed carbon into CO_2. It was determined quantitatively as for the volatile matter. The residue or ash remaining was weighed.

The effect of the interaction of siderite with the coal during the volatile matter determination was studied by Bardhan and Gupta (*1B*). Corrections were developed to adjust for errors in the analysis of washery products.

ASH. The rapid method of ashing coal was modified by Kozko and Ryukina (*23B*). Forced air circulation over the samples reduced the time of test considerably.

Kudela and Kedronova (*24B*) further improved Pozetto's rapid method of ash determination of coal by discontinuing heating as soon as the glowing of carbon ceases. Tolerances for various ash contents are proposed: up to 30% ash, 0.2%; 30 to 40% ash, 0.3%; and <70% ash, 0.9%.

The Sheffield high-temperature combustion furnace was adapted to ash determination by the British Coke Research Association (*4B*). Studies were made on weight of sample, time required for combustion, method of introducing sample, and composition of the combustion gas.

Gaberman (*10B*) reported that high-ash-content coking coals were permissible for smelting if the mineral matter was of compatible composition.

The bulk density of small coal was used by the Bretby Division of the National Coal Board (*31B*) to construct a meter to measure the ash content of coal.

Gorin (*11B*) used the densities of the coal and mineral matter to construct a nomograph and curves for the determination of ash content of coal.

The ash content of coal was determined with a pneumatic device by Jelinek (*16B*). The apparatus consisted of a series of two airtight vessels. A third cylinder with a piston applied pressure to the two vessels containing a coal sample in one vessel and a known substance in the other. The difference in pressures of the two vessels was read on a manometer and was related to the density or ash content.

Radiometric devices for the determination of ash content of coal on a belt conveyor are described by Dietzsch and Kiessling (*7B*) and Kobyakov (*20B*).

Beta rays were used by Borukhovich *et al.* (*3B*), Koch and Schleife (*22B*), and Schiller *et al.* (*36B*) in their studies of rapid nondestructive testing of coal.

Gamma-rays were employed by Hardt (*14B*), Koch, Richter, and Hempel (*21B*), and Stewart and Farrior (*40B*) in the instrument constructed for determining and recording the ash of a stream of coal.

The Cendrex analyzer or ash meter was studied in detail for interferences and optimum application by Hall, Macpherson, and Ruell (*13B*), Meerman (*29B*), Milner (*30B*), Padberg and Becker (*33B*), Siekmann (*38B*), and Stepanov and Gorelov (*39B*).

Ultimate Analysis. CARBON AND HYDROGEN. A semimicro method for solid fuels is described by Kapitaniak (*7C*). Its advantage is that less time is required to complete the test for C and H.

Pella (*10C*) reports on the source of errors of a microcombustion method for C and H in a variety of substances. The rate of combustion is controlled automatically.

Hydrogen in coal was determined radiometrically by Bockmann and Klusmann (*3C*) using β-rays. Certain corrections were required for coal.

NITROGEN. A semimicrodigestion method for N in coal was proposed by Okhapkina, Bykova, and Evstratova (*9C*). The coal is oxidized in a mixture of Cr_2O_3, $K_2S_2O_8$, and H_2SO_4 using Co_2O_3 as a catalyst. The NH_3 is distilled and titrated in a manner similar to the Kjeldahl method.

SULFUR. A rapid method for S in coal is reported by Baranowski *et al.* (*2C*). The coal is burned in an O-bomb and either of two titrimetric and one conductance method can be used to determine the SO_4 formed.

The bomb washings from the calorific value determinations are titrated with an alkali by Cross and Campau (*5C*) to estimate the S in coal. A constant correction was made for the nitric acid formed during combustion.

The S and Cl in bituminous shales were determined by the high temperature combustion method by Grinberg and Strel'kovskaya (*6C*). The procedure is applicable to the determination of Cl and S in amounts varying from 0.15 to several per cent.

Kolarova and Chekarlieva (*8C*) applied the Eschka, Strambi, and high-temperature combustion methods for S in lignite, brown, and hard coals. The Eschka method was more accurate than either of the other two methods.

The benzidine-HCl method for S in solid and semiliquid fuels is described by Ursu and Duinea (*12C*). A comparison of the proposal with the Eschka method indicated a considerable saving in time.

Speich investigated the distribution of S during the combustion of brown coal (*11C*). Stack gases, residual and fly ash were analyzed for S. Only 10 to 30% of the CaO was converted to $CaSO_4$. Retention of S by the ash depends on contact time of gas with ash, S content of coal, combustion temperature, particle size, and possibly the carbon content of fly ash.

Visapaa (*13C*) described the determination of S in coke by fluorescent x-ray spectroscopy. Standards were prepared from coke which had been analyzed by the Eschka method.

OXYGEN. The Unterzaucher method was modified by Abernethy and Gibson (*1C*) and Burns, Macara, and Swaine (*4C*) for the determination of O in coal. The chief difference between the two studies was the temperature of the reaction tube. In one case the platinized carbon was used at a temperature of 900° and in the other carbon was used at 1120°.

Inorganic Constituents in Coal and Coal Ash. In the standard procedures for determining ash in coal a number of minerals are altered. The carbonates are decomposed, the iron sulfides are converted to oxides, and the clays lose water of hydration. Considerable work has been done on identifying the minerals in coals in order to classify them on a mineral-matter-free basis.

MINERAL MATTER. Brown and Swaine (*5D*) describe the variations in mineral matter content for the various classes of Australian coals. Coking coals of New South Wales were studied by Brown, Belcher, and Callcott (*6D*), who reported the x-ray diffraction and chemical analyses show that montmorillonite, illite, kaolinite, and quartz are the main constituents. Hubacek and Lustigova (*14D*) analyzed a number of coals by low temperature ashing and acid extraction methods for the mineral content. Formulas were developed for converting ash content to mineral matter.

The Radmacher and Mohrhauer method for ash content and Brown's method for water of hydration were modified by Kanjilal *et al.* (*16D*) for the determination of mineral matter in Indian coals.

Several analytical techniques were used by Lissner and Goebel (*21D*) in their study of the organically bound metals and mineral matter in brown coal.

Ashes of oxidized coals contain more CaO and MgO and less SiO_2, Al_2O_3, and Fe_2O_3, according to Merkulov, Sinaiskii, and Sharlovskaya (*24D*).

Activated O produced in an electronic device was used by Gluskoter (*12D*) to oxidize the carbonaceous material in coal at an estimated temperature less than 200°. Except for some loss of S and Cl, most of the minerals were not changed during the ashing procedure.

Warne (*45D*) developed a DTA procedure for the rapid semiquantitative evaluation of mineral matter in coal.

ASH ANALYSIS. The accuracy of the British Standards method for the analysis of coal ash was checked by a round-robin study as reported by Flint (*11D*). There was no significant difference between the colorimetric and spectrochemical methods.

Radmacher and Schmitz (*31D*) report the adoption of German Standard DIN 51729. Different laboratories using alternative methods tested for SiO_2, Al_2O_3, TiO_2, Fe_2O_3, CaO, MgO, Na_2O, and K_2O.

A rapid method for the complete analysis of coal ash was reported by Roy and Gupta (*33D*). It is claimed that the ash is put into solution with a mixed acid solution. The time of test has been reduced to one-third of the previous method.

Vorlicek and Leitnerova (*44D*) described a rapid method for solid fuel ash which used gravimetric, titrometric, and colorimetric procedures.

Fedorovskaya, Khaskina, and Surina (*10D*) report an improvement for the direct determination of Al in solid fuel ash. They found that the trilonometric method with xylenol orange indicator was superior to the photocolorimetric method with aluminon.

The chemical oxidation of the carbonaceous matter of coal using $HClO_4$ and HIO_4 was reported by Spielhotz and Diehl (*38D*).

A method for the determination of Mg in the presence of Al is reported by Tardon and Balcarkova (*41D*). After a chemical procedure of putting the Mg into solution, the concentration is determined by atomic absorption spectrophotometry.

An accelerated method for the determination of CaO, MgO, Fe_2O_3, Na_2O, and K_2O in coal ash was developed by Obermiller and Freedman (*26D*). The ash is put into solution with $HClO_4$ and HF in place of the usual H_2SO_4 and HF. Lanthanum is added to reduce the interference of the Al in the atomic absorption spectrophotometrical measurement of the several concentrations.

X-ray fluorescence was used by Kiss (*18D*) in the determination of the inorganic matter in brown coal. The sample of coal is pressed into a wafer and the elements are determined without the usual ashing of the coal. Sulfur and Cl are also determined.

X-ray diffraction was used to determine the minerals in fly ash and other deposits from boilers using coal for fuel by Sinaiskii, Merkulov, and Sharlovskaya (*37D*), also by Chursina and Gutermakher (*7D*).

Martin, Mathur, and Morgan (*22D*) described nuclear techniques used in the examination of coal ash. Fast neutron scattering and activation methods were fairly accurate for C, O, Al, and Si, but for Fe and S special techniques were required. Hydrogen and the minor elements could not be determined.

Standard methods were used by Abernethy, Gibson, and Frederic (*1D*) in carrying out a survey for Cl, P, Na_2O, and K_2O contents in U. S. coals.

Trace Elements. The concentrations of minor and trace elements in coal were determined by Kessler, Malan, and Valeska (*17D*). A statistical treatment of the data showed regular changes due to depth on Na, K, Li, Rb, and Cs. These data with that for Pb, B, and Be can be used for control identification.

A semiquantitative spectrochemical method for analyzing coal ash was developed by Peterson and Zink (*30D*). Synthetic standards were prepared and used as references in looking for 68 elements.

Zubovic, Stadnichenko, and Sheffey (*47D*) studied the distribution of minor and trace elements in 12 beds in three states. Germanium is 10 times more abundant in the coal than in the rocks of the area.

A quantitative study for the Be content of Belgian, Dutch, French, and and German coals and coal products was made by Jedwab (*15D*). The bonding of the Be in the ash was very resistant to inorganic solvents.

Chlorine. The combustion bomb method was used by Gluskoter and Rees (*13D*) in their study of the Cl content of the Illinois No. 6 coal bed. A plot of the Cl concentrations on a map of the coal basin shows an increase in content from the edge to the center of the basin, and also with depth. Deviations from this statement are related to the Cl content of the ground water.

A combustion tube method for low concentrations of Cl was devised by Kunstmann and van Veijeren (*20D*). Their studies showed that Cl could be added to coke from the wet quenching process.

Gallium. Desyatskaya (*8D*) reviewed the published information on Ga and proposed a method for its determination in coal ash.

Germanium. According to Ratynskii (*32D*), Ge was introduced into coal by plants during peat formation, sorption with organic substances from surrounding solutions during coalification, and from circulating waters after coal formation.

A number of methods have been proposed by Medvedev and Akimova (*23D*), Minenko and Desyatskaya (*25D*), Pavlic (*27D*), Serebryakova and Bozhevol'nov (*34D*), and Szucs and Szava (*40D*) for the determination of Ge in coal or coal ash. There are some variations in the manner in which the Ge compounds are separated from the other elements—e.g., extraction or distillation.

The distribution of Ge and other trace elements along the profile of a coal bed is very difficult to explain, according to Pavlov and Fokina (*28D*).

Arnautov (*4D*) estimated the loss of Ge on heating to different temperatures. The loss was less than 5% if the temperature of heating was kept below 550°. A series of spectrographs was used to indicate changes in Ge content.

The Ge content of Spanish coals was determined spectrophotometrically by Perez (*29D*). The average Ge contents in lignitic, bituminous, and anthracitic ashes were 15.5, 36.0, and 8.5 ppm, respectively.

Veprek (*43D*) reported that on carbonization at 520° all the Ge remained in the coke.

Studies by Sharkov and Chekarlieva (*35D*) confirm the theory that the Ge is combined with the humic acids. The utilization of waste acids for production of Ge is a possibility.

The distribution of Ge due to different processes has been the subject of several studies by Adamenko and Yarvorskii (*2D*), Kranz, Krzyzaniak, and Gitter (*19D*), Shpirt, Yurovskii, and Sendul'skaya (*36D*), and Zahradnik *et al.* (*46D*).

Mercury. Mercury in coal was extracted with a saturated solution of Na_2S by Vasilevskaya, Shcherbakov, and Karakozova (*42D*). The extract was decomposed with HCl and $KMnO_4$ and finally the Hg was determined colorimetrically.

Phosphorus. Diering (*9D*) made a critical evaluation of three methods used to determine P in coal. One method gave very unsatisfactory results and is being further examined.

Scandium. The occurrence and distribution of Sc in Australian coals were studied by Swaine (*39D*), who examined bituminous and brown coals, vitrains, cokes, boiler deposits, and fly ash. The amount of Sc in the coal substance was variable, with the highest concentration of 600 ppm in the ash of certain vitrains.

Uranium. Angelova *et al.* (*3D*) studied the distribution of U in Bulgarian fuels using the hydrosulfite-phosphate-vanadate method. Included in this work were peat, lignite, brown and bituminous coals, anthracite, and bituminous shale. Some of the shales contained 51.5 grams per ton.

Laboratory Coking Tests. The Gieseler plastometer was used by Zashkvara, Semisalova, and Cherkasskaya (*23E*) to determine the effect of oxidation on the plasticity of coal. It was found that the initial softening temperature increased and the maximum plasticity decreased with an increase in the oxidation temperature.

Totsev (*20E*) compared the performance of the Sapozhnikov apparatus, Audibert-Arnu dilatometer, Gieseler plastometer, and Brabender plastograph on 17 coals and six mixtures of coals.

Allen (*2E*) reported that the depth of immersion of the retort of the Gieseler plastometer had a significant effect on the maximum fluidity. It had no effect on the initial softening, fusion, maximum fluidity, and solidification temperatures.

Studies of the thickness of the plastic layer by Kovalik, Wolfson, and Fischler (*7E*) show a relation to the Gieseler maximum fluidity, plastic temperature range, free-swelling index, and other plastic properties. Only a general relationship was noted between plasticity and coking properties of coal.

A special centrifuge was used by Emtsev (*4E*) to separate the plastic mass of coal at 475°. The two fractions differed in chemical and physical properties and prove the existence of a fluid mass that affects the coking of coals.

The fluidity of coal determined by an extrusion process was used by Geguchadze (*5E*) to evaluate coals and coal mixtures. These tests show that the fluidity indexes of the mixtures were additive.

Volkov, Emtsev, and Ovchinnikova (*21E*) have shown that the rate of heating affects the structure of the plastic mass and the resulting coke.

Speranskaya and Emtsev (*16E*) have determined the effect of temperature on the formation of the plastic mass. The physical and chemical processes were examined by determining the changes in density of coal heated under pressure. At 427° to 433°, the properties of the plastic mass and the resulting coke were optimal.

The composition of coal affects the sintering process. Taits and Sen (*19E*) used coals of different ranks to show the effect of composition on sintering. Particle size also has a decided effect.

The Audibert-Arnu dilatometer was evaluated by Schreiber (*14E*). He presented the principle, development, and application of the apparatus. Effects of particle size, weight, moisture content, volume, specific gravity, and compression are discussed.

Lazarov and Angelova (*9E*) determined the dilatation of a number of coals with the Audibert-Arnu apparatus. They report that the dilatation depended on permeability and rate of gas evolution. The dilatation of two- and three-component mixtures was lower than that expected from an additive law.

"Cementation coefficient" is a new parameter proposed for the evaluation of coking coal. Kunstmann and Kerkovius (*8E*) have developed a new test similar to agglutinating tests of yesteryear. The binding capacity of a coking coal is determined by heating at 850° with an excess of powdered coke, so that only small conglomerates are formed. After cooling, the residue is sieved, with light knocking of the sieve, until a constant amount passes a 0.03-mm sieve. Cementation coefficient = [(g residue + last fraction of passed material)/g coal applied] × 10.

CARBONIZATION. A review of the technology of coal carbonization by Russell (*13E*) includes a history, description of processes, coal used, coke ovens, chemical recovery, economics, and low-temperature carbonization.

The relationships among the coal properties, conditions of coking, and quality of coke have been studied in detail by Beck, Echterhoff, and Simonis (*3E*).

The use of the 400-kg test oven to predict coke quality, production capacity, heat required, pressure of coking, product yield for industrial-scale ovens is described by Loison and Foch (*10E*).

Porter and Ellman (*12E*) describe the laboratory carbonization of lignite at atmospheric pressure in controlled atmospheres of N, recycle carbonization gas, or steam. Different methods are used to determine the activity of the char.

A procedure is given by Taits *et al.* (*18E*) to produce solid metallurgical coke or smokeless fuel from gas coals mixed with long flame or slightly coked coals. The material is first briquetted with a binder and then heat-treated.

Laboratory apparatus using electrically heated sand in place of glowing semicoke was used in the rapid carbonization of coal at 400° to 600° by Novak, Ludvik, and Nadenik (*11E*). This procedure gave lower tar and CO_2 yield, but a higher H content in the gas.

Zhurovski and Karaneshev (*24E*) produced experimental results that show that expansion pressure is increased by a faster coking rate; also harder coke is produced with the higher expansion pressure.

The strength of lump coke was determined by the impact method at elevated temperatures by Suzuki and Otsuki (*17E*). The strength of ordinary coke (50 to 100 mm) is 97 at 500° and 93 at 1000°.

A correlation of ASTM and Micum coke testing procedures was determined by Walters, Birge, and Wolfson (*22E*), based on the results of 18 cokes tested by both methods. Equations were developed to convert the results by one method to those of the other method.

A small 14-kg capacity laboratory test oven for the determination of the coking properties of coals was constructed by Sen Gupta and Das Gupta (*15E*). The coke produced was tested in two small-scale Micum drums, one circular and the other hexagonal. The circular design was more satisfactory and the results could be related with those from full-scale equipment.

Agroskin and Goncharov (*1E*) developed a procedure for determining the specific heat of coals during carbonization up to 1000°. The method of test is based on the principle of diathermal casings.

Radioactive isotopes were used by Haberski (*6E*) to study the processes involved in the plastic and carbonization stages.

Standard Methods. NATIONAL STANDARDS. The United States of America Standards Institute, formerly known as American Standards Association, usually represents the United States of America at international standardization meetings. There is very close cooperation between this organization and the American Society for Testing Materials. The ASTM through voluntary working groups consisting of producers, consumers, and general interest develop standards and specifications for coal and coke (*1F*). ASTM Committee D-5 has the responsibility of reviewing and keeping current all standards and specifications concerned with coal and coke.

INTERNATIONAL STANDARDS. Technical Committee 27 on Solid Mineral Fuels of ISO has been very active in the development of standards for international trade in solid fuels. Most standards required for the evaluation of hard coals have reached the ISO Recommendation or Draft ISO Recommendation stage. Some standards special for brown coal and lignite are still in the development stage. Table I gives the status of the work for hard coals through 1965.

Miscellaneous. Three methods were compared by Allen *et al.* (*1G*) for the determination of CO_2 in coal. The gravimetric method was accurate but required considerable time, the manometric method was rapid but unsatisfactory for the range of coals tested, and the titrometric was rapid and accurate.

Cudmore (*10G*) states the unmodified B.S. manometric method unsatisfactory. He recommends the Burns modification for Australian bituminous coals.

The grindability of coal was determined in a special hammer mill by Kiselgof, Chelishchev, and Lifshits (*19G*). The results compared favorably with those by the ball mill and Hardgrove methods.

Neuroth (*24G*) described a special test mill for measuring mechanical and thermal stresses. The loading of the drying gas can be varied independently.

A rapid method for the determination of moisture-holding capacity of coals, proposed by Hinz (*14G*), is reported to overcome certain objections to the rapid forced air method.

The free-swelling index determination as used in the U.S.A. is described by Young and Zawadzki (*35G*). They enumerate factors that affect the test and give means for eliminating errors.

The determination of pyrite or the iron sulfide in coal has received considerable attention. Consequently, a number of modifications of existing standard methods have been proposed. Belcher and Callcott (*5G*) have indicated serious errors in the B.S. method. Brown *et al.* (*6G*) have pointed out techniques to overcome the indicated deficiencies. Mayland (*23G*) is critical of most standard methods and gives a procedure to measure the inability of the HNO_3 to penetrate the coal particle. Volokhova (*33G*) proposed a rapid method for pyrite in lignite, and Masciantonio (*21G*) describes a method of removing pyrite from coal with molten caustic. A method for the determination of the forms of sulfur including the HCl-soluble sulfides was proposed by Strnad (*31G*).

The physical properties of coals are being studied as changes in utilization occur. English and Hiorns (*12G*) have devised a method for measuring the energy of breaking, expressed as the energy required to produce a unit of new surface. Cooling (*9G*) reviewed the various methods of test for hardness of solids and studied the hardness of coal macerals, maceral groups, minerals in coal, and associated strata.

Considerable advances have been made in the determination of the reflectance of coal and the application of the results. Rapid and automatic identification equipment has been reported by Alpern and Busso (*2G*), Pickhardt and Robock (*25G*), and Soos (*30G*).

The electrical resistance of solid anthracite was reported by Baker (*4G*) and for coke by Hoy and Maehre (*15G*). Descriptions of the equipment were given.

Podbel'skii (*26G*) revised the national standard for the determination of the true specific gravity of coal. The use of water with a surfactant (OP-7, or OP-10) was recommended in place of MeOH.

Jellinek (*16G*) described a pneumatic device for measuring the apparent specific gravity of coal.

An evaluation of several methods used to determine the true and apparent specific gravity of coke was made by Kudela, Perman, and Kuncova (*20G*).

Shemeryankin, Tsynovnikov, and Rytchenko (*29G*) report a study of the determination of apparent specific gravity of coke in a large vessel such as a railroad car.

The measurement of the surface area of finely pulverized coal by several different methods was reported by Walker and Kini (*34G*). Sorption of CO_2 at 298°K appeared to be the most reliable method.

Reactivity studies on coal and coke are being reported in greater numbers. The many variables in the test are starting points for modifications by the researchers. Representative reports have been made by Bagghi (*3G*), Kessler and Dockalova (*17G*), Massart (*22G*), and Syunyaev, Akhmetov, and Gimaev (*32G*).

Studies on the fusibility of coal ash have been made by Carabogdan (*7G*), Carabogdan, Singer, and Panoiu (*8G*), Kirsch (*18G*), and Rees (*28G*). Some of the variables studied are atmosphere of test, effect of composition of ash, interaction of mineral during preparation of ash and during test, and acid to base ratio.

Gebert, Koplin, and Steiner (*13G*) describe a method for the quantitative determination of gases contained in coal.

Table I. Status of Work of ISO Technical Committee 27

1. ISO Recommendations

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
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

2. Draft ISO Recommendations

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
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
DR865	Determination of the yields of tar, water, gas and coke residue by low temperature distillation of brown coal and lignite

The handling of lignite in subfreezing regions is a considerable problem. Ellman, Belter, and Dockter (*11G*) report a method of freeze-proofing lignite that has been adopted commercially. The method consists of adding 10 to 15% lignite fines dried to 10 to 15% moisture to the lignite as mined.

The utilization of minerals and ashes from solid fuels was reported by Ratynskii and Yurovskii (*27G*). Various elements have been recovered, pyrite used for production of H_2SO_4, and ashes used for building materials and fertilizer.

GASEOUS FUELS

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, liquefied petroleum gas, sludge gas, manufactured gas, natural gas, producer gas, synthesis gas, water gas, or other gases that can be used as fuels.

Reviews. Fundamental developments in gas analysis have been reviewed by Hobbs (*9H*) and modern methods of gas analysis by Scholler (*17H*). Classic, physicochemical, and chromatographic methods of gas analysis and their applications were reviewed by Huenerbein and Adamski (*10H*). Apparatus for gas chromatography in the field was also discussed.

Andrzejak and Gilewicz (*1H*) have reviewed gas liquid adsorption chromatography, adsorbents, apparatus, and carrier gases in respect to the analysis of gases obtained from pyrolysis and cracking in the petroleum industry. Heller (*8H*) has reviewed the use of gas chromatography in the gas industry, while Dietz (*4H*) has prepared a review of some applications of GC to hydrocarbon analysis and fuel gases. A review of GC in the industrial analysis of petroleum and gases has been prepared by Tagiev (*19H*). Fundamentals in the field of GC have been reviewed by Dal Nogare and Juvet (*3H*). Jeffrey and Kipping (*12H*) are the authors of the first text that deals specifically with gas analysis by gas chromatography. The practical, rather than the theoretical, aspects are considered, and the volume contains an extensive subject index.

Spaggiari and Turtura (*18H*) reviewed the problems associated with the analysis of town gas, the application of modern physical-chemical techniques, such as infrared spectroscopy, polarography, and gas chromatography. Guerin (*7H*) has also reviewed the application of physical methods to gas analysis. The petroleum review by Gambrill (*6H*) contains 1171 references; a great number refer to petroleum gases.

Lahmann (*13H*) reviewed the methods for determining CS_2, mercaptans,

H_2S, and SO_2, and Donchenko (*5H*) reviewed the analytical methods for the determination of sulfur compounds in gases from petroleum processing.

Apparatus necessary for obtaining representative gas samples under all possible temperature and pressure conditions have been reviewed by LeMoan (*14H*). Nencetti (*15H*) reviewed the sampling and analysis of gases from natural vapor sources.

The various types of automatic gas analyzers were reviewed by Janak (*11H*), and the use of the gas analyzer in the chemical industry was discussed by Nomura and Nukada (*16H*).

Applications of gas analysis, reactions, dew points, water contents, infrared analysis, and direct resistance measurements to the control of carburizing gases were reviewed by Chatterjee-Fischer (*2H*).

Gas Chromatography. New developments in the analysis of gaseous fuels continue to increase in the field of gas chromatography. Table II contains, in summary form, data pertinent to applications that generally are unique in some aspect of application, procedure, apparatus, or column packing.

Two new detectors that may have application to the analysis of gaseous fuels are the adaptation of a Clapp oscillator by Winefordner, Williams, and Miller (*45J*) and a high-frequency discharge detector (*23J*) which is characterized by the small volume and the absence of contact between the sample and any part carrying a high potential.

Jacobs (*16J*) utilized a single open column, flame ionization detector, and temperature programming from −55° to 140° C for the rapid determination of C_1 to C_{10} hydrocarbons in quantities as low as 1 ppm of each hydrocarbon. In all, 85 compounds were separated. Harris and Habgood (*14J*) have compiled a bibliography of programmed temperature gas chromatography for the period 1952–61.

Berezkin, Mysak, and Polak (*4J*) determined the concentration of olefins in a gas by placing a 2-cm tube, filled with silica gel saturated with concentrated H_2SO_4, between two detectors and observing the out-of-balance of the signal from the detectors. Roberts and Ward (*33J*), in discussing the applications of modern analytical techniques in the gas

Table II. Gas Chromatographic Methods

Type of Gas or Compounds Separated	Column(s)	Carrier Gas	Miscellaneous	Ref.
Natural gas	I. Activated C II. Silica gel III. Diazoamyl phthalate	. . . .	Column temp. 20–30° C	(*43J*)
Natural gas	I. 30% quinoline on firebrick or 25% α-bromonaphthalene on firebrick II. Mol. sieve	H H	II used to determine N, O, and CH_4	(*32J*)
Fuel gas	I. 13× mol. sieve, 6 ft II. Type H activated alumina deactivated with 20% NaOH, 10 ft long III. Silica gel, 6 ft long IV. Type H activated alumina deactivated with 40% NaOH, 12 in. long V. Mol. sieve, 6 ft long	H H H H N	Column temp. 32° C I. Separates O, Ar, N, CH_4, and CO II. Separates C_2–C_5 hydrocarbon III. Separates CO_2, C_2H_6, and C_2H_4 IV. Separates benzene, toluene, and C_6 hydrocarbons V. Separates hydrogen Micro ionization cross-section detector	(*5J*)
Odorant and S compounds in natural gas	I. 10% β,β′-oxydipropionitrile and 25% dodecanethiol on Chromosorb P II. 5% silicone oil 550 and 5% 1-dodecanethiol on Chromosorb P III. Same as II	Ar	Ionization detector. 12 mercaptans and sulfides were separated from interfering hydrocarbons, some in concentrations as low as 0.001 grain S/100 cu ft gas	(*2J*)
Cracked light petroleum gas	I. Activated C, 4 m long II. Silica gel, 2 m long	CO_2	Orsat used to determine O and CO_2. Volume of desorbed gases measured with azotometer filled with NaOH solution. Temp. of II, 180–190° C	(*10J*)
Furnace gas (H, Ar, O, N, CO, CO_2, CH_4, C_2H_4, and C_2H_6)	I. 15% PEG 1500 on 60–80 mesh Chromosorb (2 ft long) and 1 ft silica gel II. Mol. sieve 5A, 40–60 mesh, 4 ft long III. 60–70 mesh water-washed mol. sieve	He	I and II at 45° C and III at 0° C	(*6J*)
Blast furnace gas (N, O, H, CH_4, CO, and CO_2)	I. Mol. sieve II. Silica gel or activated C	He		(*27J*)
Blast furnace gas	I. Activated C II. 10× mol. sieve	Ar		(*36J*)
Flue gas (O, N, CO, and CO_2)	Mol. sieve 5A exchange with Ca^{+2}	. . .	Temp. programmed from 20–400° C	(*38J*)
Coke-oven gas (aromatic hydrocarbons)	13% Tween 80 on Rysorb BLK	. . .	Poly (ethylene adipate), paraffin oil, dioctyl phthalate, and Tween 60 also used as liquid phases	(*17J*)

(*Continued*)

Table II. Gas Chromatographic Methods (*Continued*)

Type of Gas or Compounds Separated	Column(s)	Carrier Gas	Miscellaneous	Ref.
Naphthalene in coke-oven gas	Apiezol L or asphalt on Rysorb BLK support	N	Column at 200° C. Five other substrates investigated. Homologs of naphthalene can be determined simultaneously	*(18J)*
Naphthalene and methylnaphthalene in town gas	Di-*n*-decyl phthalate, 1 m long	He	Column heated to 160° C	*(20J)*
Impurities in ethylenic and benzene fractions of coke (oven) gas	I. 40% dibutyl phthalate on firebrick, 900 cm long II. 25% polyethylene glycol 400 on firebrick, 300 cm long III. 66% solution of $AgNO_3$ in ethylene glycol on firebrick, 600 cm long	Air	III. Necessary to resolve butylene from isobutylene	*(22J)*
Impurities in acetylene	Activated C	. . .	Impurities separated, CO, CH_4, CO_2, and C_2H_4	*(37J)*
Impurities in acetylene	Silica gel, 3 m long	H or Air	Impurities separated: H, O, N, CO, CO_2, CH_4, propadiene, butadiene, methylacetylene, ethylacetylene, vinylacetylene, dimethylacetylene, diacetylene, and C_6H_6. Column operated at 20° and 80° C	*(21J)*
Impurities in ethylene	1:2 solution of $AgNO_3$ and *p*-xylyl cyanide on 60–80 mesh Chromasorb	. . .	*m*-Xylyl cyanide, 1-naphthylacetonitrile, 3,4 - dimethylphenylacetonitrile, andoleol nitrile also used with $AgNO_3$	*(46J)*
Impurities in ethylene, (C_1-C_4) hydrocarbons)	I. 8% $NaHCO_3$ on alumina, 300 cm long II. 20% diisodecyl phthalate on firebrick, 200 cm long	N or He	I. Programmed from 20° to 70° C II. At room temperature	*(1J)*
O, N, and lower hydrocarbons	5×, 10×, and 13× zeolites	. . .	Separation of C_2H_4 and C_3H_8 was facilitated when moisture was absorbed on column	*(19J)*
Lower hydrocarbons and fixed gases	I. Mol. sieve II. Silica gel III. Liquid - phase partition column	. . .	CO and CH_4 separated on I, C_2H_6, C_2H_4, and CO_2 separated on II, and higher hydrocarbons on III	*(24J)*
C_4 and lighter hydrocarbons	Dinitriles of $NC(CH_2)_nCN$ general formula, where *n* varies from 4 to 8 as liquid phase	He	Unsaturated hydrocarbons delayed	*(26J)*
Saturated and unsaturated C_1-C_4 hydrocarbons	Glauconite, 210 cm long	He	Column temp. 20–110° C	*(11J)*
C_1-C_4 hydrocarbons	37% DBM and 63% ODPN on capillary column	. . .	Other liquid phases also investigated	*(31J)*
C_1-C_4 hydrocarbons	Zeolite X exchanged with Li^+, Na^+, K^+, Mg^{+2}, Ca^{+2}, Ba^{+2}, or Ag^+	. . .		*(13J)*
C_1-C_4 hydrocarbons	I. Alumina, 300 cm long II. 25% triethylene glycol butyrate or 20% dibutyl phthalate on diatomite	Air	Column temp., 38° C	*(7J)*
C_1-C_4 saturated and C_4 unsaturated hydrocarbons	Corning porous glass beads, 50–80 mesh (code 7930), 6 ft long	. . .	H flame detector. Normal C_5-C_{14} hydrocarbons separated by temp. programming to 250° C	*(29J)*
C_1-C_4 hydrocarbons and H, O, N, CO, CO_2, and H_2S	I. Hexamethylphosphoramide II. Silica gel	He	Columns at room temp. Either or both columns can be used as desired	*(9J)*
C_1-C_5 hydrocarbons	I. 20% *n*-heptadecane on Celite, 1.24 m long II. 15% *n*-hexadecane or squalene, 14 cm long	. . .	Flame ionization detector	*(44J)*
C_1-C_5 hydrocarbons, and O, N, CO, CO_2, and H_2S	I. 30% bis(2 - methoxyethyl) adipate on firebrick, 24 ft long II. Mol. sieve 5A, 6 ft long III. 1% Flexol 8N8 on firebrick, 5 ft long	. . .	Btu can be determined up to 1100° C to within ±1.5%. Thermal conductivity and flame ionization detector	*(39J)*
C_2-C_4 hydrocarbons	2% KOH and 8% adiponitrile on 0.25–0.5 mm TZK-M, 584 cm long	CO_2, N, Air or He	Column temp., 40° C Flame ionization detector	*(8J)*
C_2-C_8 hydrocarbons	60-80 mesh alumina, 6 ft long	N	Temp. programmed at 21° C/min to 300° C	*(15J)*

(*Continued*)

Table II. Gas Chromatographic Methods (*Continued*)

Type of Gas or Compounds Separated	Column(s)	Carrier Gas	Miscellaneous	Ref.
H, O, N, CO, CH_4, C_2H_6, and C_2H_9 hydrocarbons	I. Mol. sieve, 1 m long II. 20% triethylene glycol butyrate on firebrick, 8 m long	H, Ar, or He	I at 40° C and II at 40–45° C quinoline, β,β′-dipropionitrile, and propylene glycol also tried on II	(*28J*)
H, CH_4, CO_2, and air	SKT activated C on 0.5–1.0 mm fraction, 2 m long	He	Column temp., 200–290° C	(*42J*)
H, N, CH_4, CO, and CO_2	Mol. sieve, 110 cm long	Ar	Column at 25° C until all components except CO_2 are eluted; temp. then raised to 350° C to desorb CO_2	(*35J*)
Gas mixtures containing H, N, CO, and CH_4	Activated C, grain size, 0.5–1.0 mm	CO_2	CO_2 removed from effluent by absorption in alkaline sol. CO_2, C_nH_m, and C_2H_6 determined by standard methods	
CO_2, C_2H_4, C_3H_6, and $Me_2C{:}CH_2$	10% $C_6H_4(CO_2C_9H_{19})_2$ in ligroine on C (SKT), 30 cm long	H	Column temp., 166° C	(*3J*)
H, O, N, NO, CO, N_2O, CO_2, C_2H_6, C_2H_4, C_2H_2, and NO_2-(N_2O_4)	I. 0.5% Carbowax 1500 on silanized glass beads, 1 ft long II. 40% Me_2SO on Gas Chrom RZ, 20 ft long III. 13× mol. sieve, 8 ft long	He	I. at −76° C II. at 25° C III. at 25° C Two thermal conductivity detectors	(*41J*)
CH_4, C_2H_6, C_3H_8, and isobutane	10% octadecane on Chromosorb	H	Improved resolution of light hydrocarbons	(*25J*)
O, N, CH_4, NO, C_3H_8, C_4H_{10}, CO, N_2O, and CO_2	I. Silica gel impregnated with 10% triethanolamine II. Mol. sieve 5A, 4 m long	He		(*30J*)
CO, O, C_2H_4, C_2H_6, C_4H_{10}, 1-butene, *trans*-2-butene, *cis*-2-butene, and ethylene oxide	I. bis-[2-(2-methoxyethoxy)] ethyl ether on firebrick, 12 ft long II. Silica gel, 9 ft long	. . .	Apparatus uses no stopcock grease and permits operation at elevated temperatures	(*34J*)

industry, conclude that all the constituents of a fuel gas may be detected by analytical methods based on vapor-phase chromatography with comparative rapidity and an accuracy sufficient for most purposes.

Sulfur Compounds. Mason (*10K*) investigated the methylene blue colorimetric method for determination of H_2S in natural gas at concentrations of <0.3 grain per 100 cu ft. Traces of compounds frequently found in natural gas, such as mercaptans, organic sulfides, and disulfides, thiophane, ethanolamines, and ethylene glycol, did not interfere with the analysis. A C-steel cylinder, lined with a baked phenolic resin, was found to be superior to stainless steel containers pretreated with HNO_3 or Mylar bags for the storage of samples.

Connelly and Wagner (*3K*) discuss the application of a process polarograph for continuously measuring ppm of H_2S, by using 0.75 *N* NH_4OH as the support electrolyte and a drop time of 0.1 to 0.4 second for increased diffusion current. Sampling, reagent flow, Hg flow and recovery, and sample degassing are performed automatically.

Risk and Murray (*13K*) describe an apparatus and procedure for determining H_2S and SO_2 (25 to 2500 ppm) in a process stream. Equipment consists of a modified ultraviolet analyzer, a sample-handling system, and a catalytic furnace capable of oxidizing H_2S quantitatively to SO_2. SO_2 is measured in both streams, the reference stream in which the H_2S remains unchanged, and the sample stream in which it has been oxidized to SO_2, by ultraviolet absorption, and the difference in SO_2 in the two streams is proportional to the H_2S concentration in the sample.

The Bethge method for the oxidation of S^- with KIO_3 in alkaline medium was modified by Bartouska and Cerny (*1K*) by using 9×10^{-4} *N* KIO_3 in an equivalent ratio, $IO_3^- : S^- = 3:2$ to determine very small amounts of H_2S. Sensitivity was found to be 5 μg of S per ml of solution with an error of ±0.5%.

An apparatus for the continuous automatic determination of H_2S is described by Oucharenko, Korosteleva, and Misharina (*11K*) and is based on the measurement of electrical conductivity of the sorbent [0.025 to 0.1*N* aqueous solution of $Zn(OAc)$], which is proportional to the amount of H_2S absorbed.

Geyer, Doerffel, and Hoebold (*6K*) used a double-beam photometer to determine small amounts of COS in gases by infrared spectrometry. CO_2 and hydrocarbons do not interfere but CO absorbs near the band used (2054 or 2072 cm^{-1}), and it is necessary to compensate the reference band with a mixture of Cu_2O, H_2SO_4, and 2-naphthol.

Carbon disulfide in carbonization gas was determined by Vlckova (*15K*) by the color reaction of dithiocarbamates with Cu ions. Carbon dioxide interferes but mercaptans and thiophene do not. Carbonyl sulfide, after conversion to H_2S, is determined by the methylene blue method.

Erdos and Bares (*4K*) describe a method of determining SO_2 that is based on the conductimetric determination of H_2SO_4, formed by the absorption of SO_2 in a H_2O_2 solution. The determination has an accuracy of ±0.1% when the quantity of SO_2 is 2 to 5 mg per sample.

Fukui (*5K*) determined the total S oxides and SO_3 in stack gases by absorption in H_2O_2 and iso-PrOH, respectively, and titrated with $Ba(OAc)_2$ containing $Pb(OAc)_2$ by using Arsenazo III as an indicator. Arsenazo III gave a clear end point and is believed to be superior to thorin.

Popov and Grushetskya (*12K*) modified the method of Goksoeyr and Ross for determining SO_3 in flue gases by passing the gas through a water-cooled condenser and collecting the condensed vapors from the wall.

Jaworski and Chromniak (*8K*) describe a procedure to determine microgram quantities of sulfur in gases by the use of Raney nickel. NiS is formed and decomposed with HCl, and the resulting H_2S absorbed in aqueous NaOH and titrated with $Hg(AcO)_2$, using dithizone as an indicator. The relative error was $< 8\%$ when 0.05 to 2 mg S equivalents of CS_2, MeSH, and thiophene were tested.

Organically bound sulfur in coke-oven gas was determined by Struck (*14K*) by passing the gas over Pt gauze at 940° C to reduce all the S compounds to H_2S. The H_2S was then determined volumetrically with Hg acetate or colorimetrically with $(NH_4)_2MoO_4$. Thiophene in coke-oven gas was determined by Kavan (*9K*), using gas chromatography. Four liquid phases were evaluated and 12% polyethylene glycol adipate on Rysorb Blk gave optimal results.

Brinkmann (*2K*) determined SO_2 in the presence of CS_2, COS, H_2S, CO_2, and inert gases by gas chromatography utilizing a 2-meter column (6 mm diameter), packed with Diaphorit (0.4 to 0.5 mm) containing 5% phthalic anhydride and 20% Me silicone oil MO 1000. Hydrogen was the carrier gas and the separation temperature was 30° C.

Hodges and Matson (*7K*) achieved a gas chromatographic separation of CO_2, COS, H_2S, CS_2, and SO_2, using 1-foot columns containing Diatoport S or Teflon with benzyl Cellosolve, tricresyl phosphate, carbowax 400, or Dow Corning 200/500 grease as the liquid phase and a 25° to 75° C temperature program.

Trace Component Analysis. Vagin (*6L*) determined 10^{-8} to $10^{-9}\%$ of low boiling saturated and unsaturated hydrocarbons in a mixture by concentrating the gas mixture in a refrigerated column containing 20% triethylene glycol on Inzen brick. Separation was accomplished on a 3.4-meter column of 20% dimethylsulfolane on Chromosorb and an ionization detector was used. The theoretical basis of chromatographic methods used in the preliminary concentration of the components is discussed.

Traces of O and CO in propylene were determined by Mirzayanov and Berezkin (*4L*) by utilizing the frontal displacement concentration of poorly adsorbed contaminants in a narrow zone preceding the main component (C_3H_6). Sensitivity was $5 \times 10^{-4}\%$ with a 6% error for O and 4% for CO.

Novak, Vasak, and Janak (*5L*) concentrated trace impurities in the air and other gases in a packed tube and then analyzed the concentrate by gas chromatography. Two columns were used for the separation: The first was 170 cm long and was packed with 25% Apiezon L on Celite 545; the second, for polar substances, was an 85-cm column containing 9.4 grams of polyethylene glycol. The mass of the components in the volume unit of the sample could be estimated from the mass of the sorbed components, the sorbent mass, and the phase equilibrium under the sampling conditions. This method of frontal chromatography, which achieved the concentration equilibrium between the gas and liquid phase, allowed analysis of the unknown volume of gas and selective concentration of constituents.

Halpert, Madsen, and Foley (*2L*) described a gas phase polarograph for the determination of oxygen in gas at low temperatures and pressures. Operating conditions are −20° to 80° C and 10 mm absolute Hg pressure to atmospheric for oxygen concentrations of 0.001 to 100% with an accuracy of 1%.

Sulfur, halogens, and phosphorus in trace quantities were determined by Kainz, Schoeller, and Scheidl (*3L*) by combustion in pure oxygen with a jet burner. Sample flow rates must be uniform. Sulfur oxides were adsorbed on Ag wool at 650° C, and the resultant Ag_2SO_4 was determined argentimetrically. Chlorine was also determined argentimetrically. Bromine and iodine were titrated iodometrically.

Dantsig (*1L*) analyzed gases containing $10^{-4}\%$ impurities by thermal shock chromatography. The apparatus consists of a single straight stainless steel column fitted with a movable ring in a manner so that both ring and column can be heated electrically. The ring was slowly moved along the column, heating a zone 30–70° C higher than the rest of the column. This action concentrated the adsorbed bands and improved separation. C_3H_5 and C_4H_8 in a 1 to 1750 ratio were separated on a 700-mm long column filled with alumina.

Dew Point, Water Vapor, and Suspended Matter. Condensation was determined for H and CH_4 streams saturated with C_3H_8 and C_4H_{10} in the liquid form at ice-bath temperatures by Ahland (*1M*). Pressures ranged from 3.5 to 67 atm., and the dew point was measured with a UGC instrument. Condensation points were calculated by the NGAA method and by that of Orticek. Both methods gave calculated values differing from determined values; this was due in part to extrapolation of data.

Jindra (*4M*) determined that the dew point of city gas is dependent on temperature, pressure, water, and tar content and related these variables in a series of diagrams to simplify calculations.

Ehnert (*2M*) describes a method of determining suspended matter in gas by using a matted glass filter in a water-jacketed sampling tube. The jacket is kept at a temperature about 50° C above that of the gas to avoid deposition of naphthalene and water. Gas flow rate through the filter is 1500 liters per hour, and the gas sample volume is chosen so that 50 to 200 mg of suspended material is collected on the filter. Results are given for suspended matter in coke-oven gas and agree with tar removal by an electrostatic precipitator.

Liebetrau, Daehne, and Mohnke (*5M*) utilized the reaction of moisture with CaH_2 to form calcium hydroxide. The moisture determination was made by the change in electrical conductivity in an adsorption cell filled with 0.01*M* HCl.

Glass and Moore (*3M*) have patented a device for water vapor determination in gases, which operates by determining the electrical energy necessary to electrolyze the moisture in the gas. Gas containing water vapor is run through the cell at a constant rate for a prescribed time and absorbed, and the quantity of H_2O is equated to the electrical energy necessary for electrolysis. Sample gas is bypassed during electrolysis and an inert gas substituted at the same pressure and rate of flow.

Samarskii and Stupak (*6M*) patented a device for determining the moisture content of gases by the dew point. The device consists of a mirror, a cooler, thermocouple, illuminator, and a photosensitive element. The addition of a control mirror and another photosensitive element in the measuring circuit permits differential measurement.

Calorific Value. Thomas (*2N*) describes the work which has led to the acceptance by the British Standards Institution of a bomb method for the calorimetry of fuel gases. The theoretical accuracy of the bomb method compares favorably with estimates for a Boys instrument, and when a resistance thermometer is used, the theoretical accuracy is considerably better than that which may be achieved with a Boys calorimeter.

Dauer (*1N*) discusses the inherent error in the determination of the calorific value of combustion gas by means of the Junkers calorimeter. It is suggested that the error can be determined by an exact balance of matter and heat.

Sampling. Silicone grease and Molycote were found by Knoezinger and Kudla (*1P*) to be unsatisfactory as lubricants in connections between a gas-sampling device at elevated temperatures and a chromatographic column. They recommend a 4-mm thick Teflon ring between steel disks up to 300° C.

O'Keeffe and Ortman (*2P*) describe the apparatus and method of preparing gases with known quantities of impurities, in trace amounts, by utilizing the permeation of gases through plastic tubing.

Saltzman and Clemons (*3P*) describe a quantitative dilution device that provides a convenient means of diluting gaseous samples as much as 3000 to 1. Applications are in the field of the ionization detectors, which require special techniques, and many are not suited for measurements over extremely wide ranges of concentration.

An automatic vapor sampling device for samples for gas chromatographic analysis from flow systems that contain components that condense at room temperature is described by Sze'kely and Traply (*4P*).

Orsat Analysis. Andress (*1Q*) claims that quiet, explosion-free combustion of the H, CO, and CH_4 remaining after technical absorption gas analysis can be achieved by first admitting oxygen to a loop-shaped combustion chamber and preheating the Pt wire. The gas is then slowly leaked in the rapid oxygen stream caused by thermal convection.

Jobahazi (*2Q*) evaluated CO absorbents used in classical gas analyses and concludes that a two-pipet system, consisting of a H_2SO_4-Cu_2O suspension and an ammoniacal CU^+ solution is the most quantitative.

Physical Properties. The change with temperature of such properties as vapor pressure, heat of vaporization, liquid and vapor heat capacities, liquid density, viscosity, and surface tension in the temperature range −200° to 160° C for methane, ethane, propane, and butane are presented by Gallant (*1R*). The same properties are presented for the C_2 to C_4 monoolefins (*2R*), C_2 to C_4 alkynes (*3R*), and C_3 to C_4 diolefins (*4R*). Vapor heat capacity for the monoolefins, diolefins, and alkynes are given for the temperature range from 0° to 1000° C.

Green (*5R*) reviewed the structure, physical and thermodynamic properties, flammability and explosibility, chemical properties and reactions, manufacture, analysis, toxicity, and uses of CO.

Automatic Analyzers. Boreham and Armstrong (*2S*) describe some of the techniques for determination of a wide range of elements and compounds encountered in the gas and petroleum industries.

Various instruments have been patented for determining concentrations of a specific compound or element in gas streams: low concentrations of oxygen (*1S*, *4S*, *5S*), CO_2, SO_2, SO_3, and moisture (*9S*), hydrogen (*3S*, *6S*), and O_3 and SO_2 (*10S*).

Kohli (*8S*) discusses automatic gas analyzers with diagrams, analysis of gas mixtures by thermal conductivity and infrared absorption, and O by paramagnetic and electrochemical methods. Teske (*11S*) discusses the apparatus and electrochemical basis for the continuous measurement of SO_2, H_2S, N_2H_4, O, O_3, and H in flue gas, natural gas, sewage, and in the atmosphere, and compares this type of analysis with other methods. Janda and Hrudka (*7S*) describe an instrument for the continuous polarographic determination of H_2S in gases that utilizes an axially bored carbon electrode. Holes in the periphery of the electrode allow passage of the gas to the electrolyte, which has a pH of <6.

Standards. The 1966 book of ASTM standards, part 19, for Gaseous Fuels, Coal and Coke (*2T*) lists several methods of analysis approved as standards or tentative standards.

Standards approved in 1965 were Analysis of Commercial Butane-Butylene Mixtures by Gas Chromatograph (D 1717-65) and Analysis of Liquefied Petroleum (LP) Gases and Propylene Concentrates by Gas Chromatography (D 2163-65). Tentative status is accorded Test for Hydrogen Sulfide and Mercaptan Sulfur in Natural Gas (Cadmium Sulfate–Iodometric Titration Method) (D 2385-65T) and Test for Hydrogen Sulfide in Liquefied Petroleum (LP) Gases (Lead Acetate Method) (D 2420-65T). A proposed method of determining Moisture in Liquefied Petroleum Gases by the Valve Freeze Method is also included.

Part 18, of the 1966 ASTM Standards, Petroleum (*1T*), lists an approved method for determining Residues in Liquefied Petroleum (LP) Gases (D 2158-65) and a tentative method for the Interconversion of the Analysis of C_5 and Lighter Hydrocarbons to Gas-Volume, Liquid Volume, or Weight Basis (D 2421-65T).

Miscellaneous. Epsztein and Zakowski (*2U*) determined naphthalene in town gas by adsorption in $HCONMe_2$ and a polarographic determination in a solution of Bu_4NH in $HCONME_2$. To determine C_6H_6, PhMe, and xylenes in the gas, the gas is passed through concentrated H_2SO_4 (to remove olefins) and the aromatic hydrocarbons are absorbed in nitrating acid. The resultant dinitro compounds are diluted with H_2O and polarographed.

Spaggiari and Turtura (*5U*) compare the conventional picric acid method with infrared analysis of naphthalene in town gas.

Vlckova and Base (*6U*) describe a colorimetric procedure for the determination of phenol in fuel gas. Phenol was absorbed in 0.1*N* K_2CO_3, treated with 4-aminoactipyrine in the presence of $K_3Fe(CN)_6$ at pH 10.0. The K_2CO_3 solution was decomposed with 25% H_3PO_4 and distilled, the color reaction carried out, and color density determined with a photocolorimeter with a blue filter.

Dempsey (*1U*) has devised a computer program to determine dry natural gas viscosity as a function of pressure, density, and temperature. Larson (*4U*) developed a computer program to convert gas analysis data from units of volume-per cent to molar quantities. Landwehr, Boesch, and Wolverton (*3U*) cover the wide range of factors to be considered when data from gas stream analyzers are integrated into a computer system and stress that analyzer data are important factors in computer control of processes.

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Water Analysis

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THIS TWELFTH REVIEW of the literature of analytical chemistry applied to water analysis covers a period from October 1964 through September 1966. The present review follows the plan of the previous reviews, the last of which appeared in ANALYTICAL CHEMISTRY for April 1965 (*20*).

A review of the literature on water pollution control which includes a section on analytical methods is published annually by a Research Committee of the Water Pollution Control Federation. The 1964 review of the literature (*18*) includes 118 references to analytical methods for determining organic carbon, pesticides, phenolic materials, general organics, cations and anions, various forms of nitrogen, and dissolved oxygen. Ninety-three references are included in their 1965 review (*7*) and cover topics like dissolved gases, organic acids, phenols, total carbon, isolation-concentration, and others.

Other reviews on water analysis have been published during the past two years. Kitano and Nasu (*13*) prepared a review with 495 references. A review on various methods of water analysis was published by Mitchell (*16*). The review contained 51 references. A review of 80 references for determining chlorinity (salinity) in sea water was prepared by Bruevich (*2*). Coutinet (*8*) published a review, with appropriate details, of methods for determining potassium, sodium, calcium, magnesium, carbonate, bicarbonate, chloride, and sulfate in water.

A handbook of analytical procedures for water analysis was prepared by Bush and Higgins (*3*). The handbook also contained sections on radiochemical analysis, determination of uranium in various media, and procedures for determining major and minor constituents of silica rocks and ores. Ceausescu (*4*) proposed the use of a number of analytical methods, which were taken from standard methods of various countries, for the control of water quality in supply centers. Twenty-eight references are given. Detailed analytical methods as applied to the analysis of hydrothermal fluids were described by Ritchie (*19*).

Husmann and Malz (*12*) discussed the use of photometry, gas chromatography, polarography, and paramagnetic oxygen measurements as applied to new methods of analysis of water and waste water. New or improved methods for determining silica, phosphate, oxygen, chloride, and organic compounds in boiler feed and condensate waters were described by Dzysyuk *et al.* (*10*). Ballezo (*1*) discussed, in general, analytical methods used in water analysis and then proposed new methods for determining chloride, bicarbonate, hardness, iron, and fluoride. Instrumental methods of analysis for determining microgram and submicrogram quantities of various elements in water were reported by Monnier (*17*). A review on the use of spectrophotometry in water analysis was prepared by Weber (*21*). Eight references are listed. Ceausescu (*5*) discussed the use of ion exchange in water analysis in a review of 97 references covering the period from 1927 to 1962.

Automated procedures, using an *AutoAnalyzer* for routine analysis of water samples, were reported by Henriksen and Samdal (*11*). Semiautomated methods for determining free chlorine, silicic acid, phosphoric acid, and hardness in water have been described by Leithe (*15*).

The advantages of neutron activation as applied to the analysis of water are discussed by Cerrai (*6*). Landstrom and Wenner (*14*) described procedures for determining a number of elements in ground water by neutron activation.

A review of procedures for determining the alkali metals and uranium in sea water by stable isotope dilution analysis was prepared by Dobkowski (*9*).

ALKALI METALS

Flame photometry is still used as the principal means of determining the alkali metals in water. Rozanova (*16A*) checked the applicability of flame photometry for determining sodium and potassium in mineral waters. The sample was introduced into the flame in the form of an aerosol. No problems occurred for samples low in total mineral content and containing sodium and potassium concentrations less than 20 mg per liter. Dilutions were used for samples containing high concentrations of minerals. Potassium in brines was also determined by the cobalt nitrite method, but results were 9 to 20% higher than results by flame photometry.

A portable flame photometer was used by Zatsepa (*20A*) for determining sodium and potassium in various types of natural water. A sensitivity of 0.5 mg per liter was obtained for potassium between 1 and 20 mg per liter, and for sodium between 1 and 12 mg per liter. The maximum relative error was 7% for sodium and 15.4% for potassium.

A direct flame photometric analysis of sodium and potassium in aqueous extracts was recommended by Azovtsev (*1A*), instead of a calculation based on the gravimetric determination of sulfates.

Hosokawa *et al.* (*6A*) determined sodium and potassium in sea water with a filter-type flame photometer using lithium as an internal standard. Lithium is added to a diluted sea water sample, the sample aspirated, and the transmittance adjusted to 100% using a lithium interference filter. The filter is then replaced with a potassium interference filter and the transmittance of the sample measured. Sodium is determined in a similar manner.

A flame photometric method for determining lithium in the parts per billion range after concentration by evaporation was reported by Joensson (*7A*). The intensity of the lithium emission was increased by adding a 20% mixture of acetone–amyl alcohol (4:1) to the samples. A special absorption filter was used to eliminate interference by stray light from sodium. Aluminum was used to suppress interferences from alkaline earth oxides. Ion exchange followed by flame photometry was used by Sulcek, Povondra, and Stangl (*18A*) for determining lithium in mineral waters. A sample of water containing between 5 and 1000 μg of lithium and less than 10 meq total cations was acidified with hydrochloric acid. The carbon dioxide formed was then removed either by heating or by passing a stream of nitrogen through the sample. The sample was then passed through a strong acid cation-exchange resin, the lithium eluted with a mixture of $0.5M$ hydrochloric acid in 80% methanol, and lithium determined by flame photometry.

Collins (*4A*) described a method for detecting less than 0.01 mg of rubidium or cesium per liter in oil-field waters. Samples which contain very low concentration of rubidium and cesium are first concentrated by either evaporation or ion exchange. The samples are then transferred to separatory funnels, volumes adjusted to 100 ml with water, sodium tetraphenylboron is added, and cesium and rubidium tetraphenylborates are extracted with nitroethane. The cesium and rubidium are then determined by flame photometry.

Podobnik, Dular, and Korosin (*14A*) described a flame photometric method for determining lithium, sodium, potassium, calcium, magnesium, and strontium in natural and mineral waters. Interfering anions were removed by ion exchange. Lithium, potassium, and strontium were determined by standard addition, and sodium, calcium, and magnesium determined by dilution. Detection limits of 0.0006, 0.0002, and 0.0008 ppm were reported, respectively, for lithium, sodium, and potassium. Flame photometry was also used by Pulido *et al.* (*15A*) for determining the alkali metals and the alkaline earths. A sample volume of 500 or 1000 ml was passed through Dowex 50 W-X resin, and the cations eluted with 0.2, 0.7, 1.2, and 2.0N hydrochloric acid. The order of elution with some overlapping was reported as follows: lithium and sodium, potassium, rubidium and cesium, and finally calcium and strontium. Khitrov and Kotlyarov (*8A*) described a flame photometric system which they used to determine sodium, potassium, cesium, rubidium, calcium, and strontium in sea water.

Normand (*12A*) compared atomic absorption techniques with colorimetric, flame emission, and activation methods for determining lithium, sodium, potassium, and magnesium in ice cores. Good results were obtained but some drawbacks were evident. Meshkova, Zelyukova, and Poluektov (*9A*) developed an atomic absorption method for determining rubidium in sea water. With the use of scale expansion the sensitivity was 0.02 μg of rubidium per ml. The sensitivity is also increased by adding 5% butanol. Lithium, strontium, magnesium, barium, calcium, iron, and uranium do not interfere. Rubidium ionization is suppressed by the addition of 1 mg of potassium salt per ml.

Truesdell, Jones and VanDenburgh (*19A*) analyzed a number of waters for sodium using a sodium-sensitive glass electrode. The sodium in the waters ranged from 10 to 130,000 ppm. The method was compared with a flame photometric method and over two thirds of the results were within 5% of each other. The glass electrode method allowed for a rapid and accurate field measurement of sodium. Nogina and Kobyak (*11A*) recommended an indirect complexometric method for determining sodium in water which is ideal for use under field conditions. Sodium is first precipitated with zinc uranyl acetate, the precipitate washed, and redissolved in hot water. Ammonium carbonate is added to keep the uranium in solution, and the solution is then titrated with Complexon III after adding ammonium hydroxide and Eriochrome Black T.

Bauman and Tagliatti (*3A*) reported that the method of determining potassium as the tetraphenylborate is equivalent to the flame photometric method. They recommended it for routine control purposes. The method is cheaper and is suitable for micro- and macrodeterminations. An indirect titrimetric method for determining potassium in sea water was described by Murakami (*10A*). Potassium is precipitated with sodium tetraphenylborate and the excess tetraphenylborate titrated with benzalkonium chloride. A number of different indicators at various pH's were used.

Pencheva and Stoyanova (*13A*) described a spectrographic method for determining low concentrations of rubidium and cesium in natural waters. Rubidium, cesium, and potassium are precipitated with sodium tetraphenylborate, and the precipitate separated and dried. The residue is mixed with potassium sulfate and lithium carbonate in a ratio of 2:50:48, packed into an electrode and arced. Standards are taken through the procedure and treated exactly as the samples. The limit of detection in the initial specimen was in the order of 10^{-7}% rubidium and 10^{-6}% cesium. Parallel results obtained with flame photometry were always higher by 8% for rubidium and 9 to 10% for cesium. Potassium did not interfere. Shestov, Shurubor, and Tver'e (*17A*) concentrated potassium, lithium, rubidium, zinc, and manganese in natural waters with silica gel and then determined the elements by flame spectroscopic analysis. A mixture of fused silicon dioxide and sodium carbonate is dissolved in 100 ml of sample, the sample is acidified, and the precipitate separated and dried. The residue is then powdered and fired in carbon electrodes.

Micro- and semimicroanalysis of mineral waters for sodium and potassium was investigated by Ghimicescu, Musteata-Ghimicescu, and Dumbrava (*5A*). Sodium was determined as the triple Mg–UO_2–Na acetate and potassium determined as the Na–K–cobaltinitrite. Bartalis (*2A*) used ascending and circular paper chromatography for detecting sodium, potassium, magnesium, and calcium in mineral water.

HARDNESS, ALKALINE EARTH METALS

A review listing 20 references on the history of the determination of the hardness of water was published by Snelders (*30B*). Luppi and Herce (*16B*) reviewed the EDTA methods for the determination of total and permanent hardness of water. A statistical appraisal of the manganometric and complexometric methods for calcium, and of the 8-hydroxyquinoline and complexometric methods for magnesium was reported by Kotkowski and Wierzchlejska (*15B*). They stated that the complexometric methods are faster, more precise, and cheaper than the other methods. Sedlacek and Dusek (*28B*) investigated various indicators for use in the chelatometric determination of calcium in water. Best results were achieved with glyoxalbis(2-hydroxyanil), Fluorexon mixed with phenolphthalein, and a combination of glyoxalbis(2-hydroxy-5-chloroanil) with methylene blue. Murexide was not so suitable.

Kleber and Franke (*13B*) used a EDTA complexometric titration method to determine hardness in water. Blaedel and Laessig (*4B*) developed a continuous potentiometric titrator with direct readout for determining concentrations of calcium and magnesium as low as $10^{-5}M$. A block diagram of the titrator is given. The continuous methodology and techniques described in their paper permit titrations at concentrations where conventional colorimetric and electrometric methods of EDTA are not applicable. The method has been applied to the determination of hardness in stream and lake waters. A simultaneous complexometric determination of calcium and magnesium in water was reported by Handa (*10B*). Total hardness due to calcium and magnesium is determined by adding a few drops of murexide indicator to a 25-ml sample and titrating with EDTA to a deep purple color. Buffer is added near the end point. A known amount of standard hard water or additional amount of water sample is then added to the above titrated sample, sodium hydroxide added, and the sample again titrated with EDTA. Calcium hardness is then calculated and magnesium is determined by the difference in the value of total hardness and calcium.

Marti and Herrero (*17B*) proposed an ion exchange separation of calcium and magnesium prior to their determination with EDTA. A sample of water is passed through a column of Zerolit 225 in the Na form. The magnesium is eluted separately with 1M ammonium acetate and the calcium removed by elution with 1M ammonium lactate. The magnesium is then titrated with Na-EDTA using Eriochrome Black T as indicator, and the calcium titrated similarly using murexide as indicator. Corless (*6B*) described an ion exchange technique for determining calcium in sea water. Calcium is separated from magnesium and strontium using Dowex 50W-X8 resin. Calcium-45 is used as a tracer to monitor calcium elution. The calcium is then titrated spectrophotometrically with EDTA using murexide as indicator.

Ceausescu (*5B*) determined total water hardness by titrating a 100-ml sample with 0.1N potassium stearate using a methyl orange–phenolphthalein indicator. One milliliter of titrant corresponds to 0.1 meq total hardness.

Meshcheryakov (*18B*) proposed a trilonometric method for determining calcium, calcium plus magnesium, and sulfate in water. A sample is first acidified with hydrochloric acid, boiled to remove carbon dioxide, and then cooled. The sum of calcium and magnesium is determined first by titration with trilon using chromogen indicator. Calcium is then determined separately using murexide indicator. To determine sulfate, it is precipitated with barium chloride and then the excess barium, calcium, and magnesium are titrated with trilon using chromogen indicator.

Roueche and Monnier (*26B*) described a rapid and direct amperometric method for determining calcium and magnesium in water. A sample is evaporated to dryness, the residue dissolved with concentrated hydrochloric acid and the solution diluted to 50 ml with water. A 2-ml sample is then buffered and the calcium titrated with EDTA until a sharp rise in current is observed.

Ruf (*27B*) described a flame photometric method for determining calcium in water. Aluminum, iron, and titanium interferences are removed by precipitation with ammonium hydroxide–ammonium chloride solution. Sulfate and phosphate are removed by anion exchange. Pavlik (*23B*) determined calcium in water by flame photometry. Sulfate, phosphate, and carbonate, which interfere, are removed prior to analysis by ion exchange (Dowex 2-X8). Results obtained by this method agreed with results obtained by volumetric analysis. To determine calcium in atmospheric water, Gorbenko, Krasuskaya, and Kuchkina (*8B*) first extracted traces of calcium with a 4:1 mixture of 0.01% azo-azoxy BN reagent in carbon tetrachloride and butyl phosphate. The calcium was then reextracted with 0.1*N* hydrochloric acid and determined spectrophotometrically with glyoxybis (2-hydroxyanil).

A spectrophotometric method for determining low concentrations of magnesium in natural waters based on the development of the blue color formed between magnesium and Azovan Blue dye was reported by Rai, Pande, and Tripathi (*24B*). Beer's law is obeyed over the range of from 0 to 30 mg of magnesium per liter. Interferences from a number of other elements are negligible.

Konopac (*14B*) described a polarographic method for determining strontium and barium in the presence of an excess of calcium in mineral spring waters. Calcium is removed by ion exchange and the stronium and barium are determined directly by recording the polarographic curve in a nitrogen atmosphere, beginning from −1.7 volts *vs.* Hg pool in the same solution. A polarographic method for determining magnesium and calcium in water was reported by Gutman (*9B*).

Isotopic dilution for determining water hardness was described by Takanaka *et al.* (*32B*). The method is readily applicable for calcium concentrations greater than 150 mg per liter, but is somewhat difficult for lower concentrations.

Myasnikov and Shumaev (*21B*) determined strontium in water directly by flame photometry. The sample and standards, which contained from 0 to 20 mg of strontium per liter were sprayed into the flame of an acetylene burner and the strontium in the sample was determined from a working curve. The detection limit was 0.05 mg per liter while the error of the determination was ±4 to 6%. An ion exchange flame photometric method was used by Jonsson (*12B*) to determine strontium. Anions and alkali metals which might interfere were first separated from the sample by an ion exchange operation. Strontium was then determined flame photometrically after adding butanol to sample to increase the intensity of the strontium emission. The method permitted the detection of a 0.1 μg of strontium per ml with a standard deviation of 3.1%. Aleksandrov, Shermet'ev, and Zakharenko (*2B*) described a simple standard addition–flame photometric method for determining strontium in waters from oil and gas deposits. Strontium is first determined directly on the water sample and then an amount of strontium nearly equivalent is added to one of two aliquots of sample and a similar amount of distilled water added to the other. Both samples are then analyzed for strontium and the actual strontium content of the sample determined by a given formula. The method permitted the determination of a 0.1 mg of strontium per liter with a relative error of 10%. Standard addition was also used by Osmolovskaya (*22B*) as a rapid means of determining strontium in sea water. Strontium was determined by measuring the intensity at 4607 Å and subtracting background intensity measured at 4540 Å. The mean deviation was found to be ±3 μg/ml.

Several spectrographic procedures for determining strontium in water have been reported. To determine strontium spectrographically, Sednev, Starobinets, and Akulovich (*29B*) first separated strontium from calcium and magnesium by ion exchange. Thirty to 50 liters of water are passed through sulfopolystyrene cation exchangers containing 0.16% divinylbenzene, and the strontium is eluted from the resin with Complexon III. Mints and Bylinkina (*20B*) boiled a 500 ml to 1000 ml sample to dryness, ashed the residue with nitric acid, treated it with sulfuric acid, and then powdered the sample. The powdered sample was arced and the spectra were recorded. The strontium line at 4607 Å and calcium line at 4878 Å were measured photometrically. Analytical sensitivity in respect to the solid residue was 0.01%. Ichikuni (*11B*) determined strontium spectrographically after separating the alkaline earth oxides gravimetrically. Only 100 to 1000 ml of water is necessary, whereas 30 to 60 liters are necessary by other methods; and the difference between chemical and spectrographic results is very small. To determine strontium in surface waters spectrographically using carbon electrodes, Dojlido and Jedlewska (*7B*) evaporated the samples to $^1/_{100}$ of their initial volume before measurements were made. The strontium content of the samples, which were analyzed, ranged from 0.27 to 0.65 mg per liter. A simple spectrographic method for determining strontium, calcium, and magnesium in natural waters was described by Berman (*3B*). A spark discharge between a plate-like and a hemispheric electrode dipped into the sample or standard was used. The standards also contained sodium and potassium in concentrations comparable to the sample. The Cu 3247.5 Å line was used as an internal standard. The results agreed with flame photometric and chemical analysis.

Rao, Khan, and Kamath (*25B*) used activation analysis to determine stable strontium in sea water. Strontium is quantitatively coprecipitated on calcium oxalate, the precipitate irradiated, $^{87}Sr^m$ separated by radiochemical methods, and activity counted by a NaI(Tl) detector and compared with a standard treated in the same manner. Afanas'eva and Tsikunov (*1B*) surveyed the existing methods for the separation of strontium from sea water. They proposed a simplified modification of one of the methods. Complete details are given.

Miller and Komarova (*19B*) described a highly sensitive method for determining 0.03 to 3 μg of beryllium per liter in natural waters. The beryllium is coprecipitated on aluminum hydroxide and the precipitate analyzed for beryllium spectrographically, or determined fluorometrically using morin which is ideal for field assaying. Solov'ev and Bozhevol'nov (*31B*) determined beryllium in waste water with dibenzoyl-methane by measuring the phosphorescence spectrum of the treated sample at the temperature of liquid nitrogen and in a like manner a similar sample with the addition of a known amount of beryllium. The amount of beryllium in the sample is then calculated.

IRON, MANGANESE, CHROMIUM, AND ALUMINUM

Sturla (*32C*) studied the 1,10-phenanthroline, 4,7-diphenyl-1,10-phenanthrolineisoamyl alcohol extraction and the 2,4,6-tripyridyl-*s*-triazine–nitrobenzene extraction methods for determining reactive and total iron in industrial water. Detailed procedures, reagents, and equipment are described. The first method, listed above, has an accuracy of 0.02 ppm in the 0.1 to 2.0-ppm range. The second method has an accuracy of 0.05 ppb in the 0.1- to 100-ppb range. The third method has an accuracy of 0.2 ppb in the 10- to 100-ppb range. O'Connor, Komolrit, and Engelbrecht (*24C*) evaluated the *o*-phenanthroline method for ferrous iron and found that it gives high values for ferrous iron when the samples have a low ratio of ferrous to ferric iron, that acidification and buffering of natural water samples result in an increase in the observed ferrous iron concentration, and that color of complex is not stable and intensity of color increases with time.

Gorbenko *et al.* (*11C*) detected 2 μg of iron per liter with bathophenanthroline with a relative error of less than 10% in atmospheric precipitation. Iron is reduced with hydroxylamine–hydrochloric acid, complexed with bathophenanthroline, and the complex is extracted with nitrobenzene. The iron–bathophenanthroline complex is then determined colorimetrically. A rapid colorimetric method for determining iron in water was described by Fadrus and Maly (*8C*). Dilute hydrochloric acid was added to a 50-ml sample, the sample boiled until there is approximately 40 ml left, and cooled. Hydroxylamine, *o*-phenanthroline, and sodium acetate were added, and the solution was diluted to 50 ml. Comparison was then made with standards containing 0 to 2 mg of iron per liter. Fresenius and Schneider (*10C*) reported the use of 2,2′-dipyridyl for determining ferrous and total iron in water. An aliquot of sample is buffered to pH 5 to 6, 2,2′-dipyridyl added, and the absorbance of the colored ferrous complex measured spectrophotometrically at 546 mμ. Total iron is determined after reduction of ferric iron to ferrous iron with ascorbic acid at pH 2 to 3. A spectrophotometric method based on the blue-colored complex formed between ferrous iron and 2,4,6-tripyridyl-*s*-triazine at pH 3.8 to 5.0 was used by Nakashima and Sakai (*22C*). The method is sensitive to 0.005-ppm iron. Tanno and Nakashima (*33C*) determined iron in boiler water with 2,4,6-tripyridyl-*s*-triazine and an automatic recording analyzer. The analysis requires 30 minutes and the range of iron that can be determined is between 30 and 150 ppb.

A double-extraction procedure for determining ferric iron in turbid and colored waters was reported by Gregorowicz and Suwinska (*13C*). Iron is extracted with ethyl acetate from a neutralized solution containing ammonium acetate and sodium benzoate and then re-extracted into water acidified with hydrochloric acid. The solution is buffered, Variamine Blue hydrochloride added, and the absorbance of the iron complex measured at 580 mμ. Sono *et al.* (*31C*) used solvent extraction to determine ultramicro quantities of iron in water. The ferrous–1,10-phenanthroline complex reacts with sodium dioctyl sulfosuccinate to form an electrically neutral compound which is extracted into chloroform. The absorbance of the extract is measured at 515 mμ. Up to 24 ppb of iron obeys Beer's law. In the determination of 3 ppb of iron, the 90% confidence limit is 0.67 ppb.

A kinetic method for determining iron in natural water was described by Birmantas and Jasinskiene (*1C*). The reaction takes place at pH 2 to 3 in the presence of Pyrocatechol Violet and hydrogen peroxide. The absorbance of the solution is measured at 413 mμ and the iron content compared to standards. The determination of iron by means of a luminescent catalytic reaction in the presence of stilbexon was investigated by Bozhelol'nov and Kreingol'd (*3C*). Stilbexon is oxidized by hydrogen peroxide, and the resulting decrease of fluorescence measured. A number of other elements studied caused no interference. Sakurai (*27C*) determined iron by a continuous coulometric detection method. A sample of water is passed through a Jones reducer and the ferrous iron, which is formed, is determined coulometrically with electrolytically produced bromine.

Shidlovskaya-Ovchinnikova (*29C*) reported that several methods have been proposed for determining suspended iron in fresh waters. He recommended determining total and dissolved iron and then calculating suspended iron by difference. Shapiro (*28C*) reviewed the methods of analysis of ferrous iron in natural waters and discussed the sources of error.

A method for determining manganese and iron in one aliquot of sample was described by Kajiwara and Goto (*15C*). Manganese and iron are complexed with formaldoxime and the absorbance of the colored complex is measured at 470 mμ. Hydroxylamine and EDTA are then added to the solution and the absorbance is again measured at the same wavelength. The latter measures only the manganese complex. Iron is determined by difference. Nickel and cobalt severely interfere.

Ingols, Esterman, and Enginun (*14C*) prepared a review of methods for determining manganese in water. Morgan and Stumm (*20C*) stated that methods used for determining manganese in water should distinguish states of oxidation and physical states, and be capable of determining concentration, less than 50 μg per liter. They investigated several methods and developed a spectrophotometric method using *o*-tolidine for quantitatively distinguishing manganous ion from total manganese. They also mentioned that the *o*-tolidine and formaldoxime methods, with the use of a membrane filtering technique, have potential for determining the occurrence and distribution of various forms of manganese.

Several catalytic methods for determining manganese have been reported during the past two years. The method reported by Gottschalk (*12C*) is based on the oxidation of manganous ion to permanganate with persulfate at a temperature of 99 ± 1° C. The reaction is catalyzed by silver. The working curve covers the range between 0.006 and 0.55 mg. Substances precipitated by silver ion or oxidized by persulfate interfere unless excess reagents are added. Manganous ion catalyzes the oxidation of $Et_2NC_6H_5$ by potassium iodate to a yellow dye, and this reaction was used by Oradovskii (*25C*) to determine manganese in natural waters. The optimum pH is 7 and the optimum temperature for the reaction is 18.6° C. Iron interferes, but is masked with a phosphate–citrate buffer. Between 0.0005 and 0.01 μg of manganese per cubic centimeter in the sample can be determined.

Blank and Voronkova (*2C*) determined manganous ion by its catalytic effect on the oxidation of Eriochrome Black T by hydrogen peroxide. The reaction is carried out at pH 10 to 10.5 and at a temperature of 18° to 20° C. The excess Eriochrome Black T remaining is extracted with *n*-amyl alcohol and its absorbance measured. As little as 5×10^{-9} gram of manganese in 30 to 150 ml of sample can be detected.

Matejak, Jurkiewicz, and Pietrow (*18C*) reported that chloride interferes when manganese is being determined by the ammonium persulfate method, but that the interference is eliminated by the addition of mercuric nitrate. A spectrophotometric method for determining manganese in water with acetaldoxime was described by Kajiwara and Goto (*16C*). Acetaldoxime and potassium hydroxide are added to a sample containing less than 4 ppm of manganese, the sample diluted to 25 ml, and after 20 minutes the absorbance of the complex measured. Calcium and magnesium interfere. Sikorowska and Dozanska (*30C*) compared the mercuric nitrate and mercuric sulfate methods for determining manganese in water and sewage. The latter method was most suitable. Mokievskaya (*19C*) described procedures for determining the different forms of manganese in sea water.

A polarographic method for determining nickel and manganese in ocean water was described by Tikhonov and Shalimov (*34C*). The sample is filtered, nickel and manganese carbamates are formed, the complexes extracted with chloroform, and evaporated to dryness. The residue is dissolved with hydrochloric acid, buffered with ammonium hydroxide–ammonium chloride solution, and a polarogram made.

To determine chromium in sea water, Chuecas and Riley (*4C*) coprecipitated chromium with ferric hydroxide, separated chromium from the iron by De-Acidite FF anion exchange resin, and measured the absorbance of the chromium–diphenylcarbazide complex. Details of the procedure are given. Chromium-51 was used as a tracer to determine the recovery of chromium. Kovalenko and Petrashen (*17C*) determined chromium in water by complexing Cr^{+6} with diphenylcarbazide–β-naphthalene sulfonate, extracting the complex with isoamyl alcohol, and measuring the absorbance of the complex at 545 mμ. An interference study was made and the maximum allowable amounts of other elements are given. Bromine water in the presence of sodium carbonate is used to oxidize any Cr^{+3} to Cr^{+6}.

Delaughter (*6C*) described an atomic absorption method for determining chromium and molybdenum in brines. Chromium is complexed with diphenylthiocarbazone and the complex is extracted with methyl isobutyl ketone. Molybdenum is extracted with the same ketone after it is complexed with dithiol. With an 800-gram sample of brine 1 ppb of either element can be detected.

An amperometric method for determining chromate in waste water was reported by Dietz and Schwabe (*7C*). Chloride concentrations greater than 50 mg per liter interfere. Iron should first be eliminated. The advantages and provisions of a continuous amperometric determination of chromate are given.

De Angelis, Chiacchierini, and D'Ascenzo (*5C*) developed gravimetric and spectrophotometric methods for determining chromium in water. Isocinchomeronic acid and Cr^{+3} react at pH 4.5 to form a gray-blue precipitate that can be used to determine chromium gravimetrically. However, if the precipitate is dissolved slowly in water, a red-violet solution is formed which can be measured spectrophotometrically. The latter method is useful in the range of from 2 to 30 mg chromium per 50 ml.

A radio release procedure was developed by Richter and Gillespie (*26C*) for determining dichromate ion in natural waters. A sample is acidified, and then stirred with a paddle of irradiated silver gauze. Silver ions are stoichiometrically produced in the sample and the activity of the silver ions in solution determined by scintillation counting. The activity is directly proportional to the dichromate present. The limit of sensitivity is 10 μg of chromium per liter.

A spectrophotometric method for the determination of microgram quantities of aluminum in water was described by Fishman (*9C*). Aluminum solutions buffered to pH 6.0 form, with Eriochrome Cyanine RC, a red lake which exhibits maximum absorbance at 535 mμ. Aluminum concentrations as low as 0.01 mg per liter can be determined. Ascorbic acid inhibits the interference by iron. Interferences by fluoride and silica are eliminated by evaporation of the sample with concentrated sulfuric acid.

An extraction method for determining aluminum, chromium, copper, iron, manganese, molybdenum, and nickel spectrophotometrically in pure water was reported by Motojima and Ishiwatari (*21C*). Details of the procedure are given. With this method 3 ppb of Al, Cr, and Mn and 5 ppb of Cu, Fe, Mo, and Ni can be detected. Nowicka-Jankowska and Golkowska (*23C*) described methods which are used to determine trace amounts of iron, aluminum, and silica in reactor water. Bathophenanthroline or 2,2′-bipyridine is used for iron. Aluminum is determined by the hematoxylin method, and silica by the molybdenum blue method. Wheat (*35C*) used atomic absorption to determine Fe, Cr, Ni, Co, Mn, and Cu in water. Techniques used to extend detection limits were extension of the path length, multiple passes through the flame, fuel-rich flames, different fuels, and increased sample uptake by mechanical pumping.

COPPER, ZINC, LEAD, CADMIUM, NICKEL, COBALT, BISMUTH, AND ANTIMONY

Bilikova and Bilik (*4D*), Bilikova (*2D*), and Bilikova and Zyka (*5D*) evaluated several methods for determining copper and zinc in natural waters. The dicupral and oxaldihydrazide methods yielded the best results for copper, whereas the results obtained by the cupral method were less satisfactory. A modified zincon method utilizing ion exchange was best for zinc, and in a later publication Bilikova (*3D*) described a zincon method. A sample containing less than 0.6 μg of zinc per ml is passed through Dowex 2-X8 resin and the zinc eluted with 0.1*N* potassium nitrate. Borate buffer and a zincon solution are then added to an aliquot of eluate, the solution is diluted to 50-ml volume, and zinc is determined spectrophotometrically at 620 to 625 mμ. Up to 1000 μg of Fe, Mn, Cd, Hg, Pb, Cu, Cr, Co, and Ni do not interfere.

Pavlik (*28D*) determined copper in water by concentration of the copper on a strongly acidic cation exchanger, elution of the copper with 5*N* calcium chloride solution, and determination of the copper spectrophotometrically in the solution of lead cupral in chloroform at a wavelength of 425 mμ. The sensitivity is 0.001 mg of copper per liter.

Sono *et al.* (*34D*) described a solvent extraction procedure for determining ultramicroquantities of copper. Copper is extracted from a 400-ml sample with cadmium diethyldithiocarbamate–chloroform solution. The absorbance of the complex is measured at 435 mμ. Beer's law is obeyed from 0 to 10 ppb. Citric acid is added to the sample to prevent ferrous iron interference. Morimoto and Hirakoba (*26D*) used thiosemicarbazide to determine between 0.5 and 20 ppm copper(II) in water. A stable blue complex is formed between copper and the thiosemicarbazide in acid solution. The absorbance of the complex is measured at 365 mμ. Ferric iron also forms a complex with the reagent and must first be reduced. Corbett (*9D*) investigated the complex formed between copper and 3-methyl-5-hydroxy-5-(D-arabino - tetrahydroxybutyl) thiozolidine-2-thiore (MHATT). The complex forms at pH 4.5 to 7.0 and shows maximum absorption at 432 mμ. The reaction is highly selective and the organic reagent has negligible absorption at 432 mμ. He used the method to determine copper in water. Casassa (*6D*) described a semiquantitative method for detecting 0.06 to 1 ppm copper in drinking water. The method is based on the white precipitate formed when 2,3-dimercaptopropanol is oxidized in 1*M* sodium hydroxide by oxygen or peroxide reagents. The reaction is catalyzed by copper. Ions which are precipitated by hydroxide are first removed by filtration.

Tanno (*35D*) described a spectrophotometric method for determining traces of copper in boiler water. Copper forms a colored complex with 2-carboxy-2′-hydroxy-5′-sulfoformazylbenzene which shows maximum absorbance at 600 mμ. Beer's law is obeyed up to 50 ppb. Tanno (*36D*) then described an automatic recording analyzer which utilized the above method. Results obtained agreed with results obtained by the manual method.

Magee and Rahman (*23D*) determined copper in sea water by atomic absorption. Copper is chelated with ammonium pyrrolidine dithiocarbamate, extracted with ethyl acetate, and then aspirated in the flame. The detection limit is about 2.5 μg of copper per liter. Cummins and Woods (*10D*) developed an atomic absorption method using the copper resonance line at 3248 Å and an air–coal gas flame to determine copper in artificial sea water. Sodium, magnesium, and chlorine had little effect on the copper absorption.

Giambastiani, Filho, and Lima (*15D*) used radioactivation analysis for determining ppb levels of copper and chlorine in high-purity water.

Hickey and Overbeck (*19D*) developed a chelometric titration method for determining zinc in municipal and industrial waters. Diethylenetriaminepentaacetic acid (DTPA) is used as a titrant and is 200 times better than EDTA in its effective chelation of zinc over calcium. Up to 1000 ppm of calcium as calcium carbonate can be tolerated. Concentrations as low as 0.2 ppm zinc can be determined. Dithizone is used as the indicator. Isopropanol is added to the sample to keep the dithizone in solution. The interference effects of other substances have been investigated. A highly sensitive direct determination of zinc in sea water using a cathode-ray polarograph operating for anodic polarography was reported by Macchi (*22D*). Good reproducibility was reported at the 1 μg/liter level. The relative standard deviation was about 4.5%. The method is rapid, specific, and also relatively free from contamination from reagents because no chemical treatment of the sample is required.

Chakrabarti, Robinson, and West (*7D*) studied the various factors that affect the determination of lead by atomic absorption and then described a method for the determination of lead in water. To a 25- to 250-ml sample of water containing from 1 to 10 μg of lead, add 4 ml of saturated aqueous potassium iodide solution and enough concentrated hydrochloric acid to make 5% (v/v) of the total volume; extract the lead with methyl isopropyl ketone and measure the absorption of the lead in the extract at 2170 Å.

Nangniot (*27D*) described a method for determing lead, down to $10^{-7}M$ in water, using differential oscillopolarography. The preferred medium was a mixture of $0.1M$ tartaric acid and $0.5M$ ammonium acetate adjusted to pH 5.0 with nitric acid. An automatically operating single-sweep polarograph using anodic stripping for determining as little as $2 \times 10^{-8}M$ lead in water was designed by Ito and Musha (*20D*). The sample is mixed with the electrolytic solution to make the solution $0.1N$ in respect to HCl and 0.01% in respect to gelatin. The solution is then deaerated with nitrogen and passed into the cell.

To determine small quantities of cadmium in water, Kuyumdzhieva (*21D*) first removed copper, silver, and mercury by extraction with dithizone-chloroform at pH 2, raised the pH to 5, and extracted cobalt and nickel with dimethylglyoxime-chloroform. The pH is then raised with 10% sodium hydroxide and the cadmium is extracted as cadmium dithizonate with carbon tetrachloride and determined colorimetrically with a green filter at 520 mμ. A dithizone procedure for determining cadmium was also described by Mal'kov, Kosyreva, and Fedoseeva (*24D*). The red cadmium complex is compared to a scale of cadmium standards prepared by the same procedure.

A modified dimethylglyoxime method was used by Forster and Zeitlin (*11D*) to determine nickel in sea water. Nickel is precipitated from 2 liters of water with sodium carbonate, the precipitate dissolved, and the nickel separated by extraction with dimethylglyoxime-chloroform solution at a pH of 8.0. Nickel is then re-extracted into an aqueous phase, oxidized to Ni^{+4} with bromine water, and determined colorimetrically as the nickel dimethylglyoxime complex. The average recovery of nickel determined in this manner is 90%. To compensate for this loss the calibration curve, 0 to 12 ppb nickel, is constructed by adding known amounts of nickel to sea water. Forster and Zeitlin (*13D*) modified the above method. The nickel after being extracted with dimethylglyoxime-chloroform solution is re-extracted into the aqueous phase with 1:1 hydrochloric acid. The solution is then made basic with concentrated ammonia, quinoxaline-2,3-dithiol reagent is added, and the absorbance is measured at 520 mμ. Miller and Johannes (*25D*) studied the factors affecting the coprecipitation of nickel and cobalt with cadmium sulfide in the presence of sodium diethyldithiocarbamate. They stated that the method is applicable to the determination of trace concentrations of nickel and cobalt in natural waters.

A detailed procedure for determining cobalt in water spectrophotometrically was described by Golobov and Vakhrameeva (*16D*). Cobalt is complexed with pyridylazoresorcinol at a pH of 6.8 to 8.2 in the presence of ammonia, sodium citrate, and saturated Trilon B, and the absorbance of the complex measured at 510 mμ. The sensitivity is 0.01 μg per ml with an accuracy of 2 to 5%. Rozhanskaya (*31D*) determined cobalt in sea water by digesting a liter of sea water with hydrochloric acid. The cobalt after digestion is complexed with β-nitroso-α-naphthol, extracted with toluene, and the absorbance of the complex measured at 530 mμ. The hydrochloric acid digestion gave a 98.9% recovery of cobalt using cobalt-60 as a tracer. The sensitivity is 1 μg of cobalt per liter. Forster and Zeitlin (*12D*) determined cobalt in sea water by precipitation of the cobalt from 10 liters of water with sodium carbonate. The precipitate is dissolved with hydrochloric acid and the cobalt extracted with dithizone–carbon tetrachloride after the pH is raised to 8.5. At this point citrate is added to complex the iron. The extract is then dried, treated with perchloric acid–sulfuric acid mixture, and redissolved. The colored cobalt complex is developed with nitroso-R, and the absorbance of the cobalt complex measured at 415 mμ. Standards are prepared by adding known amounts of cobalt to sea water.

A spectrophotometric method for determining 0.1 μg of antimony per liter in sea water was described by Portmann and Riley (*30D*). Antimony is concentrated from 5 liters of water by coprecipitation with hydrous manganese dioxide and separated from manganese, iron, and other interfering elements by extraction with methyl isobutyl ketone from a solution $5M$ with respect to sulfuric acid and $0.01M$ with respect to iodide. The cobalt is then back extracted with $0.4M$ hydrochloric acid and subsequently determined photometrically with rhodamine B. Antimony-125 is used as a tracer to determine the chemical yield, which amounted to approximately 80%. Solov'eva and Arzamastsev (*33D*) determined antimony in water with Crystal Violet. The method involves the formation in a strongly acidic solution of a complex compound of Crystal Violet with $SbCl_6^-$. The complex is then extracted with toluene and the absorbance of the complex measured in a spectrophotometer. Sheveleva and Buidakova (*32D*) used brilliant green to determine antimony in waste waters. The complex is extracted with toluene and analyzed colorimetrically.

A spectrophotometric method was described by Portmann and Riley (*29D*) which determines 0.025 to 1.5 μg of bismuth per liter in natural waters, particularly sea water. Ten liters of sample are acidified with hydrochloric acid and passed through an anion exchange resin. The bismuth is eluted with nitric acid and determined photometrically with dithizone. The average chemical yield, using a radioactive tracer, was approximately 85%.

Several polarographic procedures have been reported for the elements in this group. Ghelberg and Puscasiu (*14D*) determined lead, cadmium, and manganese to within 0.015, 0.005 and 0.005 mg per liter, respectively, by using a $0.1N$ hydrochloric acid electrolyte. A 1500-ml sample is evaporated to dryness and the residue redissolved with acid prior to polarographic analysis. Copper, zinc, and cadmium in the presence of each other were analyzed in drinking water by Grebenshchikova (*17D*) with an ammonium chloride inert electrolyte. Lead was determined with hydrochloric acid and calcium chloride inert electrolytes. The sensitivity of the methods was 10^{-3} to $10^{-4}M$. Virf and Makai (*37D*) used polarography to determine copper, zinc, and cobalt in some mineral waters. The elements are concentrated from a 500- to 1000-ml sample of water. Polarographic analysis, using standard addition, was chosen by Visintin, Monteriolo, and

Giuseppi (*38D*) on the basis of specificity and sensitivity. They analyzed lead, cadmium, tin, and zinc in potable waters. Weiss and Fidler (*39D*) reported on oscillopolarographic determination of 10^{-5} to $10^{-6}M$ concentrations of lead, cadmium, copper, nickel, and zinc in mine waters after preliminary separation of the individual elements by extraction or ion exchange.

Biechler (*1D*) described an atomic absorption procedure for determining copper, lead, zinc, cadmium, nickel, and iron in industrial waste waters. A 500- or 1000-ml sample is passed through Dowex A-1 resin and the above metals are eluted with $8M$ nitric acid. The acid solutions were diluted to $4M$ before analysis because the burner capillaries were attacked by nitric acid solutions more concentrated than $6M$. With a liter sample, copper, cadmium, and zinc could be determined to 5 ppb and lead and nickel to 50 ppb. With a 500-ml sample, iron could be determined to 50 ppb.

A photometric method for determining microgram amounts of copper, zinc, and lead in natural waters with xylenol orange was described by Gurkina and Igoshin (*18D*). The determination is carried out after preliminary extraction of the three elements with dithizone. Copper is separated from zinc and lead by extraction with 0.005% dithizone in carbon tetrachloride in a $0.01N$ acid medium and determined as the copper–xylenol orange complex after re-extraction with $6N$ hydrochloric acid. Lead and zinc are then extracted as the dithizonates and re-extracted with hydrochloric acid. Zinc is determined on one portion of the sample with xylenol orange after lead is masked with sodium thiosulfate, and lead is determined on another portion with xylenol orange after zinc is masked with potassium ferrocyanide. Chuiko, D'yachenko, and Shpikula (*8D*) determined copper, nickel, and zinc in water colorimetrically after concentration of the elements by coprecipitation. Detailed procedures are given.

SILVER, GOLD, AND MERCURY

Warburton and Maher (*7E*) and Warburton (*6E*) made measurements on the concentration of silver in precipitation samples in areas where cloud seeding with silver iodide had occurred. Details are presented on the experimental process involved in the concentration of silver, formation of silver iodide colloid, and effects of storage. Samples were pumped through Amberlite CG 120(H) resin columns at a rate of 15 ml per minute, and the silver was eluted from the columns with $0.05M$ barium nitrate solution at a rate of 0.5 ml per minute. Potassium iodide solution (10^{-4} gram per ml) was then added to form a colloidal suspension of silver iodide which was detected by its ice-nucleating properties in a 10-liter cold chamber at about −15° C. Standards of $^{110}\mathrm{AgNO_3}$ were used to determine the loss of silver ions on the walls of the container. The lower limit of detection was 4×10^{-8} gram of silver collected on the ion exchange column. Kyuregyan and Eksuzyan (*4E*) used a dithizone method to detect 1 μg of silver per ml in waters.

A spectrographic method for the determination of gold in natural waters was described by Borovitskii, Miller, and Shemyakin (*2E*). The pH of a 3-liter sample is adjusted to between 5 and 6. Saturated calcium chloride, mercuric nitrate, and ascorbic acid solutions are added to the sample. Gold ion is reduced to gold metal by the ascorbic acid, and the gold coprecipitates with mercurous chloride which is formed in the presence of excess chloride ion. The sample is left to stand overnight, the transparent solution siphoned off, and the mercurous chloride suspension filtered through an ashless filter. The filter is dried at room temperature and slowly ashed in a muffle furnace by gradually increasing the temperature to approximately 500° C. The ash is then fired in a spectrograph and the intensity of the gold line at 2375.95 Å compared with standards. The sensitivity of the determination from a 3-liter sample is 0.01 μg per liter. Silver, thallium, arsenic, tin, antimony, and tellurium can also be determined simultaneously.

Oka, Kato, and Sasaki (*5E*) determined gold in sea water by neutron activation analysis. A 100-ml sample is evaporated to dryness, the residue dissolved in 50 ml of $3M$ hydrobromic acid, the gold extracted with ethyl acetate, and the extract evaporated to dryness. The residue is transferred to a quartz tube with a small amount of aqua regia, and the sample irradiated along with a blank and standards in a neutron flux of 2×10^{13} neutrons/sq cm per second for 72–130 hours. After the samples are allowed to cool for a week, gold carrier and hydrobromic acid are added and the gold is extracted with ethyl acetate. Gold is then reduced with hydroquinone and the gold-198, which is formed during activation, is counted. A quick method of detecting gold in sea water by activation analysis with thermal flux was also described by Cappadona (*3E*).

To determine mercury in natural waters, Beisova, Generalova, and Fesenko (*1E*) acidified a sample of water with sulfuric acid to destroy the stable organic mercury compounds and then oxidized any mercurous ions present to mercuric ions with permanganate. After cooling, excess permanganate is destroyed with hydroxylamine and mercury is determined with dithizone.

VANADIUM, TANTALUM, SCANDIUM, ZIRCONIUM, AND URANIUM

Chan and Riley (*4F*) described the combination of an almost specific ion exchange process with spectrophotometry to provide a simple procedure for the determination of vanadium in sea and other types of water. Vanadium from 3 liters of water is quantitatively coprecipitated with ferric hydroxide in the pH range of 4.0 to 7.5. The precipitate is dissolved and passed through Zeo-Karb 225 ion exchange resin. The vanadium is then eluted with 0.3% hydrogen peroxide solution. Iron and a number of other elements are not eluted by the hydrogen peroxide. After elution, vanadium is determined spectrophotometrically with diaminobenzidine. Vanadium reacts with 8-hydroxyquinoline to form a colored dye which can be extracted with chloroform and was the basis of a method by Mulikovskaya (*10F*) to determine vanadium in natural water. Vanadium is coprecipitated from a 500- to 1000-ml sample of water with ferric hydroxide, fused with sodium carbonate, and extracted with hot water. The vanadium 8-hydroxyquinoline dye is formed and extracted into chloroform and compared with standards.

Sharova and Chufarova (*16F*) reported that EDE-10P, basic ion exchange resin, concentrated small amounts of vanadium from waste waters which contained copper, magnesium, manganese, calcium, aluminum, iron, phosphorus, titanium, and silica. These ions are not adsorbed by the resin in the chloride form. Vanadium is then eluted with $1N$ hydrochloric acid which in turn restores the resin.

Gillespie and Richter (*6F*) developed a radio-release technique to determine vanadium(V) in water. The sample is acidified to pH 3 or less with either phosphoric or sulfuric acid, and the sample passed through a column containing radioactive metallic silver-110^{m}–110. The vanadate ion oxidizes the silver, and silver ions are released into the solution where their concentration is measured by Geiger or scintillation counting. The sensitivity is 0.1 ppm of vanadium. Chloride interferes but is complexed with 1,3-diethyl-2-thiourea prior to analysis. Iron also interferes and is likewise complexed before analysis with EDTA and fluoride. Lower valances of vanadium can be determined if they are first oxidized to vanadate. Chlorine gas is suited for this purpose.

Pets and Aleskovskii (*12F*) and Pets, Aleskovskii, and Miller (*13F*, *14F*) described methods for determining tantalum in natural and waste waters. Tantalum is determined after preliminary concentration by carrier precipitation with calcium carbonate or cadmium sulfide. A small amount of ferric salts increases significantly the precipitation of tantalum. Tantalum is then determined colorimetrically with Rhodamine-6Zh. The radioactive isotope tantalum-182 was used to determine the degree of precipitation. A thermal neutron activation method was developed by Hamaguchi *et al.* (*7F*) for the determination of tantalum in sea water. Tantalum is coprecipitated with ferric hydroxide from 5 to 10 liters of water and the precipitate is air-dried and irradiated for 12 days at neutron fluxes of approximately 3×10^{11} neutrons per sq cm per second. Solvent extraction using HF–H_2SO_4–isopropylacetone is used to separate tantalum-182. The activity is then measured on a β-proportional counter. Approximately 0.1 μg of tantalum can be detected. Belopol'skii, Gumbar, and Popov (*2F*) developed a method for separating traces of scandium in natural sulfate waters; however, zinc and copper interfere and must be removed. They found that scandium could be separated from these two elements by coprecipitating scandium with ferric hydroxide with a 20% solution of pyridine at pH 6.5. If the precipitation is repeated three times, 96 to 98% of the scandium is recovered.

A method for determining trace quantities of zirconium in sea water was reported by Shigematsu *et al.* (*17F*). Zirconium is coprecipitated with aluminum hydroxide from a large volume of sea water. The precipitate is filtered and dissolved in hydrochloric acid, and zirconium is extracted with TTA in benzene. The organic phase is evaporated to dryness and the organic material destroyed with perchloric acid; zirconium is dissolved in hydrochloric acid and determined fluorometrically with morin. Zirconium recovery is about 95%. A number of sea water samples which were analyzed contained from 0.011 to 0.041 μg of zirconium per liter. An automated procedure to determine zirconium in water in the range of 0.5 to 100 ppb has been developed by Demmitt (*5F*). It is based on a differential fluorescence technique which employs morin as the indicator in a hydrochloric acid medium. The fluorescence is measured before and after addition of Na_2EDTA to the solution. Further tests and evaluations are still being conducted.

A simple extraction method for field determination of uranium was described by Chamberlain (*3F*). Sodium chloride is added to reduce the solubility of carbon tetrachloride in water, and Na_2-EDTA to complex interfering elements. Uranium is then complexed with diphenylpropanedione and pyridine, and carbon tetrachloride is added to dissolve and extract the complex. The color of the sample is compared with standards. Nemodruk and Deberdeeva (*11F*) determined uranium in water spectrophotometrically with Arsenazo III. Uranium is precipitated from a liter of water on ferric hydroxide. The precipitate is dissolved and uranium extracted with a solution of butyl phosphate in toluene. The uranium is back-extracted with water and Arsenazo III, and the absorbance of the complex measured at 656 mμ. Kuznetsov *et al.* (*8F*) used organic precipitants in the determination of uranium in natural water.

A method was described by Barker *et al.* (*1F*) for the determination of low concentrations of uranium in water based on the fluorescence of uranium in a pad prepared by fusion of the dried solids from the water sample with a flux of 10% sodium fluoride, 45.5% sodium carbonate, and 45.5% potassium carbonate. This flux permits use of a low fusion temperature and yields pads which are easily removed from platinum dishes for fluorescence measurements. Uranium concentrations of less than 1 μg per liter can be determined in a sample of 10 ml or less. Liu and Chang (*9F*) stated that fluorescimetric measurement is the most sensitive method for determining traces of uranium in waste and natural water, and described a method for its determination.

Physicochemical methods, including ultrafiltration, dialysis, adsorption on ion exchange agents, and electrodialysis, were used by Serebryakova (*15F*) for determining the forms of uranium occurring in subsurface waters.

BORON AND SELENIUM

Glebovich (*3G*) developed a colorimetric method for determining boron in natural waters with H-resorcinol. Complexon III is added to complex interferring cations. H-resorcinol is added to sample and standards and the solutions are left in the dark overnight and then the color of the sample is compared with the standards. With a 5-ml sample the sensitivity is 1 μg of boron. If a 100-ml sample is used and the reagents increased proportionally, the sensitivity is increased. A number of water samples analyzed by this method agreed with results obtained by the carmine method.

Vasilevskaya and Lenskaya (*8G*) determined boron in water with salicylic acid and Crystal Violet. The sensitivity of the method is 0.01 μg of boron per ml. The water sample is neutralized with acid or alkali to pH 6 and passed through a column of KU-2 cation exchange resin. The solution is then neutralized to pH 5 or 6 with alkali, salicylic acid added, and the solution evaporated to dryness to remove excess salicylic acid. Crystal Violet and 0.01M hydrochloric acid are added, the solution is transferred to a separatory funnel, and the complex is extracted with benzene. The absorbance of the extract is measured at 585 mμ. A spectrophotometric procedure for routine analysis of boron in water was reported by Saxena, Baser, and Durge (*5G*). A sample of water is evaporated to dryness and the residue dissolved in 1 ml of water and mixed with turmeric–oxalic acid reagent. The mixture is evaporated to dryness, the residue treated with a known volume of ethanol and filtered, and the intensity of the dye measured at 540 mμ. Semenov (*6G*) described a methylene blue extraction method to determine boric acid in sewage and reservoir water. The limit of detection is 0.05 mg of boron per liter.

Sidel'nikova (*7G*) proposed a modified 3,3'-diaminobenzidine method for determining selenium in natural waters. Selenium, iron, and lead are precipitated with ammonium hydroxide, the precipitate is dissolved with hydrochloric acid, and the selenium is separated from the iron and lead by KV-2 ion exchange resin. Trilon B is added to the eluate to mask other multivalent cations, followed by a solution of 3,3'-diaminobenzidine. After 40 to 50 minutes, the pH is adjusted to 7 to 8 and the yellow selenium complex extracted with toluene. Subsequently, the complex is analyzed colorimetrically. Preliminary tests revealed that the detection limit is approximately 5×10^{-6} gram per liter.

A similar method is described by Chau and Riley (*2G*) for determining selenium in sea water. Isotope dilution is used to correct results for the small losses occurring during the analysis. For sea water which contains approximately 0.4 to 0.5 μg of selenium per liter, a standard deviation of 0.03 μg per liter was obtained. Morette and Divin (*4G*) used a 3,3'-diaminobenzidine extraction method to determine selenium in water in which the selenium varied from zero to 74 μg per liter.

A ring-oven technique was used by Biswas and Dey (*1G*) for determining selenium in water. Selenium is extracted from sea water by distillation with bromine and hydrobromic acid. The separation of selenium from arsenic is achieved with the ring oven by using a magnesia mixture that precipitated arsenic as ammonium magnesium arsenate and it is retained at the center of the filter paper. Washing with 50% aqueous ethanol caused the selenium to migrate to the ring zone. The selenium ring is developed with a 5% aqueous solution of thiourea and the selenium determined quantitatively by ring colorimetry. It was possible to determine selenium in water at a level as low as 0.23 ppm.

Methods were described by Zanoni (*9G*) to determine selenium in water.

CHLORIDE, BROMIDE, AND IODIDE

Gromoglasov, Subbotina, and Komarova (*8H*) described a diphenylcarbazone method for determining chloride concentrations between 0.1 and 0.5 mg per liter in water of high purity. The determination requires constant temperature, pH 2.8 to 3.2, 200 μg mercuric ion, and 2 mg diphenylcarbazone per 100 ml. The absorbance is measured at 540 mμ. Bystritskii, Aleskovskii, and Degtyarenko (*5H*) also used diphenylcarbazone for chloride and found that color development is affected by temperature. After diphenylcarbazone is added, the solution is held in a thermostat for 1 hour at 24° C. Mercuric nitrate is added, the sample brought to volume, and the solution again held at 24° C for 40 minutes before the absorbance is measured.

A rapid method for determining chloride in water by liquid detector tubes was developed by Kobayashi and Takeno (*9H*). A tube approximately 2.0 mm in diameter is filled with a known amount of a mixture of potassium chromate, silver nitrate, and silica gel. To determine chloride, 2 ml of sample are injected into the column with a syringe over a period of 5 minutes. The amount of chloride is determined by measuring the length of the discolored zone in the tube. Between 5 and 40 ppm chloride can be determined within a relative error of ±10%. Results obtained by this method agreed with results obtained by the silver nitrate turbidimetry and mercuric thiocyanate methods.

Dyrssen and Jagner (*6H*) determined the chlorinity of sea water by potentiometric titration with 0.1*M* silver nitrate. Polished silver rods were used as electrodes and the cell was as follows: Ag/diluted sea water/0.1*M* KNO_3/0.1*M* $AgNO_3$/Ag. Aleskovskii, Bardin, and Bystritskii (*1H*) described a potentiometric chloride method with a sensitivity of 1 μg of chloride per liter. Silver chloride electrodes are used. One electrode is immersed in an electrolyte free from chloride and the other electrode is immersed in the sample. The emf is then measured. Two concentration cells for the potentiometric determination of chloride in sea water were described and discussed by Koske (*10H*). The first cell is Ag/AgCl,NaCl(c″)/M/NaCl(c′), AgCl/Ag in which the electrodes are immersed in sodium chloride solutions of different concentrations and separated by a polyethylene membrane. In the second cell, Ag/AgCl,NaCl(c)/M^+/NaCl/(c″)/M^-/NaCl(c′),AgCl/Ag the electrolyte solutions are separated by an ion exchange membrane.

An automatic potentiometric method for determining chloride in sea water was developed by Shimanova and Ponomarev (*16H*). A silver and a calomel electrode are placed in a beaker containing the sample. The sample is then titrated and at the equivalence point the automated system closes the buret valve.

To determine chloride in water from nuclear power plants, Maley (*11H*) designed a flame photometer which records the chloride concentration by measuring the sodium content. Complete details of the apparatus and operation are described.

An indirect spectrophotometric method for determining bromide in natural water with a standard deviation of 2 μg for bromide concentrations of approximately 40 μg per ml was described by Elbeih and El-Sirafy (*7H*). Bromide is oxidized to bromine in acid solution with acetic acid and hydrogen peroxide. The temperature of the reaction is kept between 40° and 50° C. A known amount of Chromotrope 2B is added and the bromine allowed to bleach the color for 30 minutes. The reaction is then stopped by buffering the solution to pH 5. The absorbance of the residual color is measured at 511 mμ. The color is stable for several days. Most elements which occur naturally in water can be tolerated.

A review with 38 references on the determination of iodide in water was prepared by Pavlova and Shishkina (*13H*). The sodium thiosulfate method was the preferred method to determine iodide.

An indirect catalytic method for determining as little as 0.005 μg iodide in water was developed by Mano (*12H*). Iodide catalyzes the reduction of ceric salts by arsenite in acid solution. The reaction can be stopped by adding excess ferrous iron which immediately reduces the free Ce^{+4}. The ferric iron which is found is then determined colorimetrically with thiocyanate. Another feature of the method is the addition of sodium chloride to the reaction which increases the catalytic effect of iodide and also inhibits silver and mercury interference. Yonehara (*18H*) described a catalytic method for determining iodide and iodate in sea water. Both ions show the same effect on the color fading of a solution of ferric thiocyanate. To determine iodide and iodate, total iodine is first run, and then iodide is extracted with carbon tetrachloride from a separate sample after it is oxidized to free iodine. Iodate is then determined and iodide calculated by difference.

Bobek and Kolczak (*4H*) adapted the alkaline method to determine iodide in water. Two samples are taken for analysis. A known amount of iodide is added to one sample and an equal volume of water to the other. Sodium arsenite is added and the samples equilibrated at 25° C for 10 minutes at which time cerric sulfate is added. The absorbance of the resulting solutions is measured at 435 mμ. Ramanauskas (*14H*) stated that tetramethyldiaminodiphenylmethane is oxidized by iodine set free from iodide with chloramine B to a blue quinoid derivative. The absorbance of the blue quinoid is measured at 584 mμ or compared to copper sulfate standards. He used this reaction to determine iodide in natural waters. Chloride and up to 0.5 mg of bromide per liter do not interfere.

A simple, radiometric procedure for determining microgram quantities of iodide in natural water was developed by Richter (*15H*). The principle of the procedure is as follows: If a benzene solution of radioiodine of known specific activity is shaken with a sample of water containing iodide, the radioactivity will distribute itself in both phases in direct proportion to the mass of iodine or iodide in the individual phases. Measuring the radioactivity of each phase and knowing the quantity in the benzene phase permits calculation of the iodide in the aqueous sample. The reaction is specific for iodide and iodine species in the absence of oxidizing or reducing agents and precipitating or complexing agents. The lower limit of detection with this method is 2 μg of iodide per liter.

A potentiometric method for determining iodide in water was described by Atanasiu, Lazar, and Constantinescu (*2H*). Iodide is first concentrated by electrolysis in which it is oxidized to iodate. After electrolysis the iodate is reduced to iodine and concentrated by ion exchange (Dowex 1). The iodine is eluted with sodium sulfite and determined potentiometrically.

A polarographic method for determining iodide was reported by Yazhemskaya and Shtukovskaya (*17H*). Iodide is concentrated by evaporation; the solution is made basic with potassium carbonate and evaporated to dryness. Organic material is then removed by heating at 400° C. The residue is dissolved in water, iodide oxidized with HBrO, and the excess of this reagent and oxygen removed by the addition of sodium thiosulfate. Iodide is then determined polarographically in gelatin solution using a dropping mercury electrode.

A spectrographic method for determining iodide, bromide, and chloride in natural waters was developed by Belobragina and Miller (*3H*). Details of the procedure are given.

FLUORIDE

Cox and Dirks (*4J*) compared the cerrous-alizarin-Complexon and the zirconium-Eriochrome Cyanine methods for determining fluoride in fluorinated drinking water and stated that the alizarin method seemed simpler to use. The zirconyl chloride–sodium alizarinsulfonate and zirconyl chloride–Eriochrome Cyanine R methods and the effects of sulfate and bicarbonate on these methods were studied by Debiard and Dupraz (*5J*). The latter method gave better results; was simple, rapid, and sensitive; and did not require distillation to isolate fluoride. Cooke, Dixon, and Sawyer (*3J*) investigated a number of fluoride methods and found that most suffer from aluminum interference. They then described two improved methods. The first was a modification of the visual method of Megregian and Maier. A color disk is available which offers improved color discrimination over the range of 0.5 to 1.3 ppm fluoride and much greater accuracy in the 1.0- to 1.3-ppm range. Color development was reduced from 1 hour to approximately 20 minutes. The second method was a spectrophotometric method based on the bleaching of the zirconium Solochrome Cyanine R complex by fluoride ions. Distillation in most cases was not necessary. Ruzicka and Mrklas (*12J*) compared a number of well known visual fluoride methods and found none precise for waters with high concentrations of aluminum.

Baeumler (*1J*) for several years has used the SPADNS method for determining fluoride in waters where the phosphate content is low and no aluminum is present. In the absence of these interfering ions the method works well.

Palin (*10J*) devised a simple method to eliminate aluminum interference in existing direct colorimetric fluoride methods. The sample is first treated with a weakly acidic cation exchange resin. The reaction between fluoride, cerous ion, and alizarin complexon was employed by Jeffery and Williams (*7J*) to determine fluoride in precipitation waters. Interfering elements are first separated by cation exchange or by distillation from perchloric acid solution. The results obtained agreed well with results obtained by a method involving distillation from perchloric acid and subsequent titration with thorium nitrate solution using alizarin red S as indicator.

Zolotavin and Kazakova (*14J*) also used the reaction of fluoride with alizarin complexon and cerous ion to determine fluoride in natural and waste waters. Iron and aluminum interference is eliminated by cation exchange. The absorbance of the final solution is measured spectrophotometrically at 617 mμ.

A similar alizarin complexon method for determining fluoride in sea water was described by Murakami and Uesugi (*9J*). Lanthanum ion is used instead of cerous ion. The absorbance of the color solution is measured at 610 mμ. Beer's law is obeyed up to 1.0 ppm fluoride. The tolerance limits for other elements normally found in sea water are given.

A spectrophotometric method to determine fluoride in mineral waters with zirconium–Pyrocatechol Violet complex was described by Ruzicka (*11J*). Fluoride is steam distilled with perchloric acid. The distillate is then acidified with hydrochloric acid, mixed with zirconium reagent and Pyrocatechol Violet, and the absorbance of the resulting solution measured at 580 mμ.

Zolotavin and Kazakova (*13J*) determined fluoride in natural and waste waters by ion exchange and titration. A 100-ml sample is acidified with hydrochloric acid and the sulfate precipitated with barium chloride. The filtrate is then passed through a KU-2 cation exchange resin. Calcium chloride and sodium alizarinsulfonate are added to the eluate and the solution is acidified with hydrochloric acid and titrated with thorium nitrate which has been standardized against known concentrations of fluoride. Ghimicescu, Musteata-Ghimicescu, and Dumbrava (*6J*) used thorium nitrate titration to determine fluoride in mineral waters. The fluoride is initially steam distilled with perchloric acid.

An *Auto*Analyzer was used by Chan and Riley (*2J*) to determine fluoride in sea water and other natural waters. A flow diagram of the system is shown. The spectrophotometric procedure used with the above system is based on the blue complex formed in the presence of fluoride when the sample is treated with a buffered reagent containing the lanthanum chelate of alizarin fluorine blue. Satisfactory results were obtained in the range 0 to 1.5 μg of fluoride per ml.

A consecutive determination of sulfate and fluoride in water was described by Li and Huang (*8J*). Sulfate was precipitated from a sample with lead nitrate, filtered, redissolved, and the lead titrated with EDTA using dimethylphenol orange as indicator. The filtrate was passed through a cation exchange resin, and the fluoride in the eluate determined colorimetrically with thorium–dimethylphenol orange.

SULFATE, SULFITE, AND SULFIDE

Kleber and Franke (*8K*) discussed the disadvantages of determining sulfate in water by EDTA methods and recommended the conventional gravimetric method. Koval'tsov and Konovalov (*9K*) prepared a review of the volumetric and physical-chemical methods for determining sulfate in water. The object of the review was to find suitable methods for determining sulfate with automatic analyzers. Turbidimetric and nephelometric methods were suitable. Kanwar and Chopra (*7K*) compared a rapid volumetric EDTA method with gravimetric and turbidimetric methods for determining sulfate in irrigation waters. The results obtained by the EDTA method were intermediate between the other two methods.

Page and Spurlock (*14K*) investigated three indirect titrimetric procedures to determine sulfate in sea water. The cations are first removed by ion exchange. Sulfate is then precipitated with barium chloride and the excess barium in the filtrate is titrated with one of the following: EDTA–NH_4OH reagent or disodium EDTA, disodium EGTA, or standard sodium hydroxide. In the latter method the cation-free sea water is partially evaporated and sulfate is precipitated with benzidine hydrochloride. The benzidine sulfate is redissolved and titrated with sodium hydroxide. Results by the sodium hydroxide and EDTA–NH_4OH methods agreed fairly well with results obtained by the conventional gravimetric method.

Guerello (*4K*) also determined sulfate indirectly by precipitating sulfate with a known excess of barium and titrating with EDTA. Calcium and magnesium interfere but can be complexed with cyanide and triethanolamine. Kral (*10K*) critically reviewed the volumetric sulfate method and recommended that sulfate be precipitated with excess barium chloride and excess barium titrated with Chelaton III using Eriochrome Black T as indicator. This gives the total calcium, magnesium, and excess barium in the sample. Therefore, another sample is titrated for calcium and magnesium, and sulfate is determined by difference. Bicarbonate and free carbon dioxide are removed by boiling an acidified sample. Phosphate interferes and is removed by precipitation as silver phosphate. Heavy metals are masked with potassium cyanide and aluminum with triethanolamine.

A complexometric process for determining sulfate in drinking water by adding excess barium chloride and titrating the excess barium was also reported by Hoevers (*6K*). Riego (*16K*) described an indirect complexometric method to determine sulfate in sea water. Chloride, phosphate, and carbonate are precipitated with silver nitrate using potassium chromate as indicator. The precipitate is filtered and potassium chromate in the filtrate is reduced to chromic salt to avoid later precipitation of lead chromate. Lead acetate is then added, and the lead sulfate precipitate which is formed is mixed with EDTA, ammonium hydroxide, and ammonium hydroxide–ammonium chloride buffer. When the lead sulfate precipitate is dissolved, the solution is titrated to a violet end point with zinc chloride using Eriochrome Black T indicator. A formula is given to determine the amount of sulfate present.

Lehocky (*12K*) determined sulfate directly in water, after removal of interfering cations by ion exchange, by titration with lead nitrate using solid dithizone as indicator. In the concentration range 5 to 400 mg of sulfate per liter, the precision is 2 to 3%.

Ion exchange followed by titration with barium chloride was used by Handa (*5K*) to determine sulfate in water. Disodium tetrahydroxyquinone indicator (THQ) and a few drops of an inert dye (xylene cyanol FF, indigo carmine, or methylene blue) are used to detect the end point, which is difficult to see with only the indicator (THQ). The end point with the dye is the change in color from an olive green to a gray violet. A cation exchange method for determining sulfate in ground water was reported by Lees (*11K*).

Pavlovich, Oshchepkova, and Solov'eva (*15K*) developed a rapid method to determine free sulfuric acid in mine water. Ferrous iron is oxidized to ferric iron with hydrogen peroxide and then complexed with ammonium oxalate. Sulfuric acid is then titrated with $0.1N$ sodium hydroxide using methyl red indicator. The total free sulfuric acid is the sum determined by titration and the amount consumed by the oxidation of ferrous iron. A formula is given to calculate the latter. Ferrous iron is determined separately.

Babkin, Veber, and Averbukh (*1K*) described a photometric method to determine sulfate in water. Barium sulfate suspension and an acid solution of barium chromate are added to a sample. The sample is mixed, and a 40% solution of sodium acetate is added. The precipitate is then centrifuged and the absorbance of the chromate in the filtrate measured with a photocolorimeter. At the 75 mg of sulfate per liter level, the precision is ±1 mg per liter. Calcium, magnesium, and bicarbonate do not interfere. Sykulska and Drazkiewicz (*18K*) used barium rhodizonate to determine sulfate colorimetrically in purified water. Various techniques of concentration were used.

A review of the methods for determining sulfide compounds in water and sewage was published by Dojlido (*3K*). Nusbaum (*13K*) modified the standard colorimetric method for determining sulfide in water and waste water. The stability and reproducibility of reagents are improved by the use of *p*-aminodimethylaniline oxalate as the colorimetric reagent and $Na_2S \cdot 3H_2O$ for preparation of standard solutions. A direct potentiometric method for determining hydrogen sulfide in mineral waters with an accuracy of 7 to 10% was described by Shpeizer and Zaidman (*17K*). Chloride, sulfate, sulfite, carbonate, benzene, phenols, and resins did not interfere. Details are given for preparing the electrodes.

Indirect procedures for determining mixtures of sulfide, sulfate, and sulfite in mineral waters were reported by Burriel-Marti and Alvarez-Herrero (*2K*). To determine sulfide, an excess of $0.1M$ copper nitrate is added to precipitate sulfide and the excess copper in the filtrate is titrated with EDTA using murexide as indicator. Sulfate is determined after an acidified aliquot of water is boiled to remove sulfide and sulfur dioxide. The sulfate is precipitated with excess barium chloride, and the excess barium in the filtrate is titrated with EDTA. To determine sulfite, another aliquot is treated with bromine to oxidize all the sulfur to sulfate, and total sulfate is determined as above. Sulfite is then determined by difference.

SILICA, PHOSPHORUS, AND ARSENIC

Matveev and Bashmakova (*16L*) used the yellow molybdosilicate complex to determine silicon in waters with low mineralization. The color is compared with standard potassium chromate solutions. The samples can also be analyzed colorimetrically if the yellow complex is reduced to the blue form with 1 - amino - 2 - naphthol - 4 - sulfonic acid. Grasshoff (*10L*) described a yellow molybdosilicate method for determining dissolved reactive silica in sea water. A sample of water is filtered, monochloroacetic acid and molybdate reagent added, the sample stored for 6 hours or more, and the absorbance of the complex measured at 390 mμ. Fresenius and Schneider (*9L*) determined silica in mineral water by the molybdosilicate method after converting polymeric silicic acid to monomeric silicic acid. Polymeric silicic acid on treatment with hydrofluoric acid yields silicon fluoride which with boric acid gives monomeric silicic acid. Novoselov and Simolin (*21L*) used a *p*-methylaminophenol sulfate–sodium sulfite mixture to reduce the yellow molybdosilicate complex. The reduced blue solution is determined spectrophotometrically. The method was used to determine silicates in sea water.

A simple extraction procedure for determining silica in sea water was described by Schink (*25L*). Ammonium molybdate–sulfuric acid reagent and sulfuric acid were added to an aliquot of sample. The sample was mixed, transferred to a separatory funnel, and the complex extracted with ethyl acetate. The absorbance of the organic phase was then measured at 3350 Å. Turbid waters did not have to be filtered prior to analysis. Kanie (*14L*) used ion exchange to separate silica in high purity water, and then determined silica by the molybdenum blue method. An aliquot of sample, hydrofluoric acid, and amberlite IRA 402 (OH-type) are shaken together for 30 minutes in a polyethylene separatory funnel. The resin is filtered through a nylon cloth, and silica is desorbed with a saturated solution of boric acid.

Webber and Wilson (*31L*) used the molybdate blue method of Morrison and Wilson (*19L*) to determine silica in boiler water. Tests were performed to confirm the suitability of the method in the presence of phosphate and other substances. The phosphate effect was small for silica concentrations exceeding 0.1 ppm. A method for determining total silica in water was reported by Kostrikin, Shtern, and Dzysyuk (*15L*). A sample of water was evaporated in a platinum dish, fused with sodium carbonate, and the residue dissolved in hot water. The solution was transferred to a volumetric flask, sulfuric acid was added, it was brought to volume with water, and silica was determined by the molybdenum blue method.

Wilson (*32L*) described a continuous automatic method to determine reactive silica in boiler water with the molybdate blue procedure. A Technicon *Auto-Analyzer* was investigated in respect to response time, calibration curve, precision, stability of response, and effect of phosphate. The instrument performed reliably and appeared to be suitable for continuous analysis.

Brewer and Riley (*2L*) determined silicate in natural waters in the range 0 to 4 mg per liter with a Technicon *Auto*Analyzer. Again, the method is based on the reduction of silicomolybdic acid to the molybdenum blue complex. Oxalic acid is used to prevent interference from phosphate. Tanno (*28L*) also described an automatic recording analyzer based on the molybdenum blue method for the determination of silica concentrations greater than 20 ppb.

Jones (*13L*) compared several methods of determining inorganic phosphate in sea water and recommended the method of Murphy and Riley (*20L*) as a standard procedure for estimating phosphate in sea water. The method involves adding a measured amount of a single-solution reagent to an aliquot of the sample and, after 10 minutes, determining the absorbance of the intense blue complex. The reagent is prepared by dissolving appropriate amounts of ammonium molybdate, ascorbic acid, and antimonyl potassium tartrate in dilute sulfuric acid. This reagent solution is unstable and must be prepared fresh.

Fishman and Skougstad (*7L*) described a simplification of the method of Murphy and Riley and adapted the procedure to the field determination of phosphate in natural waters through the preparation of a set of permanent color standards against which samples may be compared, and a single-solution reagent prepared immediately before use by dissolving a measured amount of a mixture of dry chemicals consisting of ammonium molybdate, ascorbic acid, and antimonyl potassium tartrate in dilute sulfuric acid. Watanabe and Olsen (*30L*) reported that the method of Murphy and Riley is accurate for determining phosphate in water or sodium bicarbonate aqueous soil extracts. The color of the complex developed with ascorbic acid is stable for 24 hours whereas that developed by stannous chloride is liable to interference by organic matter in soil extracts.

Chan and Riley (*3L*) used a Technicon *Auto*Analyzer to determine 0 to 90 μg of phosphate in spiked sea water by the method of Murphy and Riley. Twenty samples can be analyzed in an hour.

Ambuehl and Schmid (*1L*, *26L*) described a molybdenum blue procedure for determining small amounts of phosphate and total phosphate in inland lake water. Stannous chloride is used as the reducing agent. Data on accuracy are given. Hartmann and Stenzel (*11L*) determined phosphate in drinking water by a molybdenum blue method. Polyphosphate is first hydrolyzed with sulfuric acid at 95° C. The molybdate reagent consisting of ammonium molybdate, hydrazine sulfate, and sulfuric acid is then added and the solution heated for 35 minutes at 95° C. The absorbance of the blue color is measured at 750 mμ. Edwards, Molof, and Schneeman (*6L*) used ascorbic acid as the reducing agent in the molybdenum blue method to determine orthophosphate in fresh and saline waters. They also found that 5% alcohol in the color development mixture extended the linear color development range and eliminated precipitation in sea water. Ninety-nine percent color development occurred within 4 minutes. Umnova (*29L*) also used ascorbic acid as a reducing agent in the molybdenum blue method to determine phosphate in industrial waste water. Total phosphate is first hydrolyzed by boiling a sample with potassium permanganate and sulfuric acid.

A spectrophotometric method for determining phosphate in water based on the reaction of phosphate and vanadate-molybdate reagent was described by Moeller (*17L*). A sample of water is evaporated to dryness and calcined for 1 hour at 450° to 550°. The sample is evaporated twice more with water and hydrochloric acid, and then dissolved with water and hydrochloric acid. The solution is diluted to a known volume passed through an anion exchange resin, and vanadate-molybdenum reagent added to a portion of the eluate. After 1 hour the absorbance of the solution is measured at 366 mμ. Silicic acid removal is not complete and the phosphate values are high by 0.05 ppm. Proft (*24L*) also used a vanadate-molybdenum reagent to determine phosphate in water and waste water. All forms of phosphate are first brought into solution by evaporating an aliquot of sample on a hot plate with perchloric and nitric acids. The sample is then cooled, transferred to a volumetric flask, and vanadate-molybdenum reagent is added; the mixture is diluted to the mark and phosphate determined spectrophotometrically.

Won (*33L*) described a spectrophotometric method to determine orthophosphate in natural waters. An aliquot of sample containing from 0.3 to 1.2 μg of phosphate is transferred to a separatory funnel, and ammonium molybdate and perchloric acid are added. After at least 5 minutes a known volume of butyl acetate is added and the mixture shaken. A portion of the organic phase is transferred to a flask, and stannous chloride in 5.5*N* hydrochloric acid and copper sulfate in 5*N* perchloric acid are added. The mixture is heated, mixed, cooled, and ammonium thiocyanate added. The absorbance of the organic phase is then measured at 460 mμ.

A phosphomolybdate extraction procedure utilizing benzeneisobutanol was used by Morkowski (*18L*) to differentiate the various forms of phosphorus in water and waste water. Details of the procedures are discussed.

An automatic method for determining orthophosphate and acid-hydrolyzable phosphates in fresh and saline waters with the Technicon *Auto*Analyzer was described by Henriksen (*12L*). The method is based on the extraction of phosphomolybdate with isobutanol and color development with stannous chloride. A schematic and details of the procedure are given. The lowest detectable concentration of phosphorus achieved with this method is 1 μg of phosphate per liter.

Cosoveanu (*4L*) preferred determining phosphate in boiler water with ammonium molybdate, sodium sulfite, and hydroquinone rather than by the conventional reduction of phosphomolybdic complex with stannous chloride or metallic tin. Pavlik (*22L*) described methods for determining phosphate in natural waters. For the concentration range 0.05 to 1 mg per liter a molybdenum-tungstate reagent is added directly to the sample, the complex reduced, and the solution analyzed colorimetrically at 700 mμ. For the concentration range 0.001 to 0.1 mg per liter, phosphate is concentrated by anion exchange (Dowex 2-X8) and then determined colorimetrically.

Fresenius and Schneider (*8L*) determined arsenic in water with silver diethyldithiocarbamate. A sample is acidified with sulfuric acid, As^{+5} reduced to As^{+3} with potassium iodide and stannous chloride, and the As^{+3} converted to AsH_3 in the presence of copper-coated zinc. Arsine is then absorbed by silver diethyldithiocarbamate in pyridine, and the absorbance of the red color measured at 510 mμ.

Conversion of As^{+5} to As^{+3} with ascorbic acid and cocrystallization of As^{+3} with thionalide in a 0.05*N* sulfuric acid medium was proposed by Portmann and Riley (*23L*) for the recovery of microgram amounts of arsenic from sea water. Arsenic is then determined by measuring the absorbance of the molybdenum blue complex by a modification of the single-solution method of Murphy and Riley (*20L*).

A spectrophotometric method for determining trace amounts of arsenate, arsenite, and phosphate in natural waters was described by Sugawara and Kanamori (*27L*). Phosphate and arsenate are extracted as molybdophosphate and molybdoarsenate with different solvents. Arsenite is then oxidized and extracted. The respective fractions are then treated with sodium hydroxide to liberate molybdenum, which is then measured spectrophotometrically as the thiocyanate.

Davidyuk (*5L*) proposed a new polarographic method for determining arsenic in natural waters. Details of the procedure are given.

NITRATE, NITRITE, AND NITROGEN COMPOUNDS

A statistical comparison of the brucine, chromotropic acid, and phenoldisulfonic acid methods for determining nitrate in water was conducted by Jurupe Ch. (*17M*). No significant difference between the methods was detected. However, the brucine method was recommended when a rapid analysis is desired, and when the sample is low in nitrate and high in chloride. But for better precision when time is available, the phenoldisulfonic acid and chromotropic acid methods were suggested in order of preference.

Waters (*27M*) compared a number of methods for determining nitrate in water and found that a considerable lack of information existed on the range, precision, and accuracy of present methods. The brucine and indigo carmine methods for nitrate in water were evaluated by Fadrus and Maly (*10M*) with regard to precision and reliability.

West and Ramachandran (*28M*) described a chromotropic acid method which will tolerate over 4000 mg of chloride per liter, and which eliminates other sources of interference. Murai *et al.* (*23M*) modified the traditional aniline procedure to establish a simple and reliable method for determining nitrate in the presence of nitrite. Grasshoff (*13M*) determined nitrate in salt and potable waters by reducing nitrate to nitrite by a cadmium amalgam in a Jones reductor, followed by diazotization with sulfanilamide and coupling with 1-naphthylethylenediamine. The absorbance was measured at 543 mμ. No interferences were observed from other ions in salt water.

Ostrowski and Nowak (*24M*) determined nitrate in sea water by a colorimetric reaction utilizing diphenylbenzidine. The use of both diphenylamine and diphenylbenzidine was investigated by Hsiung (*16M*) in an effort to improve the sensitivity and accuracy of the nitrate determination. A simplified method using a mixture of the two reagents was developed, and gave more accurate values for nitrate in sea water samples than either reagent alone. It may be applied in the field as well as in the laboratory.

A sensitive method for determining nitrate in water by reaction with 2,6-xylenol and extraction into toluene was described by Andrews (*1M*). The absorbance is measured spectrophotometrically at a wavelength of 432 mμ. Interferences from nitrite and chloride are eliminated by the addition of sulfamic acid and mercuric sulfate, respectively. A standard deviation of ±0.013 mg of nitrate nitrogen per liter was obtained in recovery tests on natural waters. Bloomfield, Guyon, and Murmann (*4M*, *5M*) discussed two similar methods for determining nitrate in water based on the interference which nitrate exhibits in the formation of colored complexes of heptavalent rhenium with α-furil dioxime and *syn*-phenyl-2-pyridyl ketoxime in acid solution in the presence of stannous chloride. The effects of a large number of diverse ions were tested. Most common ions do not interfere.

An automatic method for determining nitrate in sea water using a Technicon *Auto*Analyzer was reported by Brewer and Riley (*8M*). The sample was mixed with an EDTA reagent (pH 6.5–7.0) and passed through a tube of cadmium filings. The resultant nitrite was determined by diazotization with sulfanilamide and coupling with *N*-naphthylethylenediamine according to the method of Bendschneider and Robinson (*3M*). Allowance is made for the presence of nitrite in the sample. About 30 samples per hour can be analyzed. The method has a coefficient of variation of 1.6% at the 200 μg per liter concentration level. Henriksen (*14M*) described an automatic method for determining nitrate and nitrite in fresh and saline waters using a Technicon *Auto*Analyzer. The *Auto*Analyzer system was shown schematically. The method is based upon the diazotization procedure originally published by Mullin and Riley (*22M*) in which hydrazine is used to reduce nitrate to nitrite in the presence of a copper catalyst, followed by diazotization with sulfanilic acid and coupling with 1-naphthylamine. Increasing the temperature from 20° to 70° decreases the time required for reduction of nitrate to nitrite from 24 hours to 15 minutes.

A study of the nitron method for the gravimetric determination of nitrate in sewage effluents was made by Quon and Lagvankar (*25M*).

Dey (*9M*) determined nitrite in water colorimetrically by a procedure involving diazotization with *p*-aminobenzoic acid and coupling with *N*-naphthylethylenediamine dihydrochloride in the pH range of 1.7 to 2.2. The color intensity is a linear function of concentration in the 0.005 to 0.15 ppm nitrite range.

Richards and Kletsch (*26M*) determined ammonia and labile amino compounds in fresh and sea waters by oxidation to nitrite with sodium hypochlorite in strongly basic solution. The resulting nitrite is diazotized with sulfanilamide, coupled with *N*-naphthylethylenediamine and analyzed spectrophotometrically at a wavelength of 540 mμ. In the procedure reported by Konnov (*18M*), the pH of the sample is adjusted with a borate buffer and the ammonia distilled under reduced pressure. This is followed by addition of sodium hypobromite, after which the color is developed with α-naphthylazo-β-naphthol-3,6-disulfonic acid. A standard curve is prepared by using ammonium chloride. As little as 1 μg of nitrogen per liter can be detected.

A method for determining total nitrogen in diluted water solutions was proposed by Gertner and Grdinic (*11M*). Nitrogen is freed for analysis by decomposing the organic matter in sulfuric acid solution containing potassium persulfate and hydrogen perioxide, using silver as the catalyst. The method takes less than 2 hours and works irrespective of the manner in which nitrogen is bound in the organic molecule.

Gonter and Schmitt (*12M*) reported a procedure for determining cyanides in effluents from coke oven and blast furnace operations. A reflux distillation procedure employing cuprous chloride as a catalyst was used for removing interfering materials and for conversion of the cyanides into sodium cyanide. The cyanide was then determined by titration or colorimetrically. Several electrode systems were suitable for the electrometric titration of sodium cyanide in alkaline solution. The benzidine-pyridine and the pyridine-pyrazolone colorimetric methods were evaluated independently by 10 laboratories. The results obtained indicated that more precise results can be obtained by using the benzidine-pyridine procedure.

Hissel and Cadot-Dethier (*15M*) isolated free cyanides from complex cyanides and thiocyanates with Amberlite IRA-400 in the hydroxyl form. After elution with 1% potassium nitrate, free cyanide was measured colorimetrically with pyridine-pyrazolone reagent. Boye (*6M*) used a method for determining cyanides in waste water from gas works and coke oven plants by a modification of the method reported by Boye and Scholz (*7M*). The sample was acidified with citric acid and the evolved HCN absorbed in sodium hydroxide solution. The cyanide was then determined colorimetrically with pyridine and benzidine, or by titration with silver nitrate. The modifications consisted of clarifying the effluent with trisodium phosphate, precipitation of sulfides with lead carbonate, and sweeping out the apparatus with air instead of nitrogen. Bahensky (*2M*) described a rapid colorimetric method for estimating cyanides in waste waters using chloramine T and 1-phenyl-3-methyl-5-pyrazolone.

Lur'e and Panova (*19M*) used both sulfanilic and barbituric acids to determine cyanides in waste waters. Ferrocyanides in concentrations of less than 10 mg per liter did not interfere. The detection limit was approximately 0.01 mg per liter. The decomposition of ferro- and ferricyanides which occurs during the determination of free cyanides by distillation methods was studied by Mrkva (*20M*). By distillation, HCN is freed from these compounds at pH 7. The amount of decomposition depends upon the pH value, method of distillation, amount distilled, and concentration of the complex cyanides. The distillation methods were unsuitable for determining free cyanides in samples containing ferro- and ferricyanides.

Mrkva and Mica (*21M*) developed a method for the determination of thiocyanates in phenol waters. An excess of lead carbonate powder is added to the sample to precipitate sulfides. After filtration, the pH is adjusted and the sample heated with a germanium sulfate–ammonium sulfate solution to precipitate cyanides. The sample is again filtered, decolorized with sodium persulfate, and precipitated by boiling with cupric sulfate. The cuprous thiocyanate precipitate thus formed is filtered and washed. Nitric acid and ferric ammonium sulfate are added to redissolve the precipitate and the red solution is titrated with 0.05*N* silver nitrate until it is colorless.

OXYGEN DEMAND

A critical review of the various methods for the determination of biochemical oxygen demand (BOD) in river water and sewage was published by Niemitz (*11N*). Young and Clark (*16N*) prepared a review with 12 references on the history of the BOD test.

Wolfner (*15N*) determined BOD values in effluents by using dichromate oxidation. The sensitivity of the method was increased by adjusting the amounts of the reagents used to the expected BOD. Filatova (*5N*) reported that 5-day BOD determinations on biologically purified effluents in which nitrification has begun give high results because nitrogen-containing compounds are oxidized. To avoid this difficulty, the methylene blue method is recommended over the one involving pasteurization. Initial values of BOD in rivers strongly contaminated with sewage were determined by Sedzikowski (*13N*).

A barometric vibration method for BOD was described by Burchard (*1N*). It consisted of measuring the change in pressure of the atmosphere in a closed 903-ml vessel containing the sewage, which is vibrated to provide continued mixing and contact with the air. Results were comparable after 5 days' vibration with volumetric BOD measurements.

An improved apparatus for determining BOD of polluted water was reported by Young, Garner, and Clark (*17N*). A standard 1-liter narrow mouth reagent bottle is used as the reaction flask. Claimed advantages are reduced cost, simplicity of operation, and obviation of a large head space which was a source of error with varying external barometric pressure. A detailed description and scale drawings are given. A rapid polarographic technique for BOD determinations was reported by Krishnamurty (*10N*).

Fitzgerald (*6N*) reported a study on the measurement of BOD on several species of algae, alone, and in association with bacteria under 5 days of incubation in the dark at a temperature of 20°. The object was to determine if algae have an effect on BOD measurements under standard conditions. Eighteen references were cited.

A procedure for measuring low chemical oxygen demand (COD) by the dichromate method in the presence of high chloride concentrations was described by Cripps and Jenkins (*3N*). The method relies on the formation of undissociated mercuric chloride which is only slightly oxidized under standard COD conditions. The limitations of the Cripps and Jenkins method were studied by Burns and Marshall (*2N*), and a revised procedure was developed to correct for sodium chloride concentrations as high as 3%. The improved method is reported to give the most accurate COD measurements presently possible.

Pitter and Se-Kvan-Son (*12N*) compared the dichromate and iodate methods for determining COD for different boiling times with and without silver sulfate as a catalyst. The dichromate method with silver sulfate gave higher results than the iodate method. Without silver sulfate, both methods gave comparable results. The dichromate method was easier to carry out and responds best to the organic pollution of sewage and industrial wastes. El-Dib and Ramadan (*4N*) investigated dichromate as compared to ceric sulfate for COD and concluded that dichromate is far superior, especially if silver ion is used as a catalyst.

Gaudy and Ramanathan (*7N*) reported a colorimetric adaptation of the standard dichromate procedure. Dichromate is added to the sample as an oxidant, and the excess determined colorimetrically at 440 mμ. The method is rapid and yields results comparable to the standard titration technique. Hankus (*8N*) and Jakubowska (*9N*) each investigated several methods for determining the oxidizability of various waters. Both investigators agree to the superiority of the dichromate method.

A titrimetric method for oxygen demand using chloramine-T was described by Steinmetz and Losonczy (*14N*). Chloramine-T and HCl are added to the sample, which is then kept in the dark for 30 minutes. Potassium iodide is added, the sample left in the dark for 5 minutes, and the excess chloramine-T titrated with standard sodium thiosulfate solution. Five types of waters were tested and the results compared with those obtained using the permanganate method.

OXYGEN AND OTHER GASES

A routine method for the determination of oxygen in water or in gas mixtures was developed by Takahashi, Sakurai, and Sakamoto (*44P*). The sample solution or gas was passed through a Jones reductor and the liberated Zn^{+2} was measured polarographically. An acetate buffer (pH 6) was mixed with the continuously flowing sample solution and used as the supporting electrolyte. The calibration curve was linear in the concentration range of 0.1 to 7.0 mg of oxygen per liter in water under the optimum conditions of pH, flow rate, and column length. A Pt/NaCl/AgCl/Ag cell for determining dissolved oxygen with a mean accuracy of 2% was reported by Khaidarov (*18P*). The cell is covered with a polyethylene membrane and yields a current which is proportional to the oxygen concentration. The principal limitations are the difficulty of temperature compensation and the fact that the platinum cathode must be polished and the instrument periodically standarized. Shol'ts and Ostrovskii (*36P*) described an improved polarographic cell for the quantitative determination of dissolved oxygen which features ease of construction and handling. The basic model was that of Ostrovksii and Gel'man (*27P*) where thermostating was obtained with a flow of liquid in the cell and mixing was done by a magnetic stirrer placed above a film covering the electrodes; the current fluctuations due to temperature variations were almost completely eliminated with a semiconductive thermoresistance. Duxbury (*6P*) reported the use of a galvanic-type cell for use in field determinations of oxygen. It is a modification of the Mancy, Westgarth, and Okun (*22P*) instrument, and its basic construction and calibration are given. The use of a lead anode and silver cathode eliminated the oxidizing procedures required with platinum electrodes.

A simple, all glass apparatus was constructed by Slechtova and Novacek (*38P*) for determining dissolved oxygen in waste waters. The device consisted of two cells and operated according to Henry's law. Results compared favorably with results obtained by the Winkler method using permanganate.

A coulometric technique for determining dissolved oxygen in natural waters was reported by Healy (*13P*). Burkert (*3P*) described a recording apparatus for the determination of oxygen in boiler feed water. It depends on the reaction $O_2 + 4NO + 2H_2O = 4HNO_2$ and measures the change in conductivity brought about by this reaction. Kabanova and Zalogina (*16P*) used the method of Wright and Lindsay (*53P*) for determining trace amounts of oxygen in water. The method is based on the change in specific conductance of the sample after passage through a thallium column. The change is proportional to the dissolved oxygen content. Oxygen concentrations between 10 and 200 μg per liter can be analyzed.

Gillespie and Richter (*11P*) constructed an apparatus for the determination of as little as 1 part-per-trillion dissolved oxygen in sea water, streams, ponds, and drinking water. The analyzer consists of a column of ^{204}Tl (half life 3.6 years, 0.75-mev B, ≈2% electron capture) deposited on copper turnings. Water to be analyzed passes through the column forming Tl^+ ions, and simultaneously the ^{204}Tl is released and detected by a Geiger-Mueller tube. The counting rate is a direct measure of the dissolved oxygen concentration. A battery powered and transistorized portable instrument has been constructed.

Toedt (*46P*) reviewed and discussed difficulties which arise in the continuous electrochemical determination of oxygen in impure water. The major limitations are the deposition of calcium carbonate and the formation of foam on the electrode surface. Two improvements of known methods are given. In the first method the analyzed water is continuously acidified to maintain a pH which prevents the deposition of calcium carbonate. The second method is based on the fact that deposition of calcium and magnesium carbonates vanishes when the current is switched off for some time. An apparatus was constructed which makes possible the continuous determination of oxygen by periodic interruptions of the current. With measurement intervals of 15 seconds and pauses of 45 seconds, the correct and constant values of oxygen content were obtained even in water having a high content of calcium and magnesium carbonates and even over periods of several days.

A review of the various colorimetric methods for determining dissolved oxygen in boiler feed water was given by Shtern (*37P*).

Ungureanu (*47P*) reported two photometric methods for determining dissolved oxygen in water: One is based on the oxidation of the leuco-indigo compound (intense yellow) in alkaline solution into the indigo carmine compound (intense blue), and the other utilizing the oxidation of Ce^{+3} in alkaline media to Ce^{+4} by using *o*-toluidine as indicator. The first method was more rapid than the second, but was more prone to errors because of the lower stability of the leuco-indigo as compared to that of the Ce^{+3} salts.

Sorgatz and Dubois (*41P*) used a cerous chloride photometric method for determining oxygen in condensate and feed water. A special bottle is used to exclude errors in sampling and in performing photometric determinations.

Ivanitskaya and Mostofin (*15P*) determined low concentrations of dissolved oxygen in water of thermoelectric stations by colorimetric analysis with Safranine T, whose leuco compounds are nearly colorless and do not form intermediates during oxidation. A sampling device and an apparatus for reducing Safranine with zinc amalgam were described.

Ostrowski, Jasinska, and Zolendziowska (*28P*) reported the use of *N*,*N'*-bis-(2 - hydroxypropyl) - *o* - phenylenediamine for determining dissolved oxygen in water. Manganous ion (added as MnCl$_2$) in basic solution is precipitated as Mn(OH)$_2$ and promptly oxidized by oxygen in the solution to Mn^{+3}. *N*,*N'*-Bis(2-hydroxypropyl) - *o* - phenylenediamine in 25% HCl is then added and the color which develops measured at 533 mμ. Beer's law is followed in the range of 0 to 5 ppm, and the method is claimed to be faster and to give better precision in this range than the Winkler titration.

Ozone between 0.02 and 1.00 mg per liter in water in the presence of other oxidants was determined by Koppe and Muhle (*19P*). At pH 2 in phosphoric acid–dihydrogen phosphate media, ozone oxidizes *N*,*N*,*N'*,*N'*-tetramethyl-*p*,*p'* - diaminodiphenylmethane and *N*,*N*,*N'*,*N'* - tetramethyl - *p*,*p'* - diaminotriphenylmethane to the corresponding fluorene which is extracted into chloroform and measured at 492 mμ. A small amount of permanganate eliminates interference from MnO$_2$, Cl$_2$, and H$_2$O$_2$.

Several investigators suggested modifications of the Winkler method. Malik (*20P*) used a redox titration with 1,10-phenanthroline as indicator for greater convenience and reliability as compared to the classic method. Felicetta and Kendall (*8P*) reported a "rapid permanganate" modification of the Winkler test and galvanic cell methods for determining dissolved oxygen in spent sulfite liquor.

However, the modified Winkler procedure was not satisfactory because of a tendency to yield low results. Rabinovich and Sherman (*33P*) described a Winkler modification for determining dissolved oxygen concentrations in small biological cultures and sludge solutions.

Carpenter (*4P*, *5P*) examined the potential errors in the various techniques used for the Winkler method and developed a new technique for improved accuracy. A special titration apparatus is illustrated. Errors in sampling for dissolved oxygen were studied by Montgomery and Cockburn (*24P*). The effects of sample handling were evaluated by Porges (*32P*), and precautions and recommendations were given for the field treatment of samples to provide a more accurate and reliable analysis.

Ross (*35P*) described a method which is under consideration as a British standard for determining oxygen in fresh or saline waters. The method involves the sodium thiosulfate titration of the iodine released after the addition of alkaline iodide reagent, manganous sulfate, and sulfuric or phosphoric acid. The complexometric method of Malik (*21P*) utilized the oxidation of manganous ion to the manganic ion, and titration of the deep violet complex formed between manganic ion and ethylenediaminetetraacetate with standard ferric alum solution. The end point may be detected visually or potentiometrically.

Park and Catalborno (*30P*) determined dissolved oxygen in sea water using a modification of the gas chromatographic method of Swinnerton, Linnenbom, and Cheek (*42P*). Argon is used as the carrier gas. Gaunt and Shanks (*9P*) reported a chromatographic method for the microdetermination of oxygen in boiler feed water using hydrogen as the carrier gas. The calibration curve is linear with respect to peak height or peak area over the 0.000- to 0.015-ppm range.

In discussing the determination of trace concentrations of oxidizing and reducing compounds in liquids and gases, Teske (*45P*) described an apparatus and the electrochemical basis for the continuous measurement of sulfur dioxide, hydrogen sulfide, hydrazine, oxygen, ozone, and hydrogen in flue gas, natural gas, boiler make-up water, sewage, and the atmosphere. A comparison was made with other methods. West (*51P*) reported a mass spectrographic method for determining dissolved gases in water in the 5- to 30-ppb range. Methods and apparatus diagrams were given by Vignet *et al.* (*48P*) for the determination of total gases dissolved in water at a pressure of approximately 200 kg per sq cm.

An amperometric titration using sodium arsenite for determining free residual chlorine was described by Ghelberg (*10P*). The current indicator was a cell containing the Au/Cl water, KCl, AgCl/Ag electrode system immersed in the water. A dual polarizable electrode apparatus consisting of two platinum electrodes, a mercury cell, a microammeter, a resistor to fix the potential, and an a.c./d.c. stirring motor was used by Morrow (*25P*) for the phenylarsene oxide titration of residual chlorine. Precision, sensitivity, and interference levels are similar to those for conventional amperometric titrations, but more rapid titrations are possible. The unit may also be battery operated for field use. Two coulometric titration methods were described by Takahashi, Sakurai, and Namiki (*43P*). A dead-stop end point detection method was used and results were superior to those obtained by the potentiometic titration method.

Williams (*52P*) used *o*-tolidine for measuring residual chlorine in swimming bath waters; however, Palin (*29P*) stated a preference for the titrimetric version of the diethyl-*p*-phenylenediamine method. A colorimetric method for determining free chlorine in polluted waters based on the bleaching action of chlorine on methyl orange was developed by Solls and Larson (*40P*). Absorbance is measured at 510 mμ. Results agreed well with those obtained by the amperometric titration method. Webber and Wheeler (*50P*) reported a modification of the absorptiometric method of Asmus and Garschagen (*1P*) for determining chlorine in water.

Methods for determining free chlorine in water were evaluated by Nicolson (*26P*) on the basis of reproducibility, specificity, sensitivity, limit of detection, recovery of chlorine, effect of temperature, stability of reagents, simplicity, and convenience. Of the eight methods studied, the procedure based on the reaction of *N*,*N*-diethyl-*p*-phenylenediamine with chlorine to form a colored oxidation product was judged best in the presence of combined chlorine, if the color is measured immediately. In the absence of combined chlorine, a method based on the Zincke-Koenig reaction in which an alkali metal cyanide reacts with chlorine to form cyanogen chloride, which in turn reacts with an amine (barbituric acid) in pyridine to form an intensely colored solution, was selected as the best laboratory method. Titrimetric procedures were less reproducible than spectrophotometric procedures. Thirty-eight references are listed.

Kegel (*17P*) pointed out that errors in the titrimetric determination of free carbon dioxide in water are caused by the presence of calcium bicarbonate. A new method was developed based on the measurement of pH before and after the addition of a small amount of inorganic acid. Haesselbarth (*12P*) evaluated the relative errors of the Kegel procedure and calculated the systematic and accidental errors involved in the acidimetric titration of carbon dioxide in water. Comparative investigations showed good agreement between the two methods after slight improvements in the Kegel procedure in the concentration range of 0.5 to 1.0 meq of alkalinity per liter and up to 20 mg of free carbon dioxide per liter.

Rao (*34P*) used a conductivity technique for determining total carbon dioxide in dilute tropical waters in the 40 to 100 mg per liter concentration range. Park, Kennedy, and Dobson (*31P*) investigated the gas chromatographic and pH-alkalinity methods for measuring total carbon dioxide in sea water and found that both methods give comparaable results. An infrared gas analyzer was used by Waterman (*49P*) for determining carbon dioxide in surface ocean water.

Matveev and Zavodonov (*23P*) determined carbon dioxide in snow and ice by allowing a weighed sample to melt in a stoppered flask with barium hydroxide and phenolphthalein indicator. After 6 to 10 hours, aliquots are titrated with standard hydrochloric acid solution. Directions for determining the changes in carbon dioxide concentration from changes in pH were given by Beyers *et al.* (*2P*). The method involved titration with gaseous carbon dioxide using distilled water as carrier to obtain curves relating pH value to carbon dioxide concentration. Detailed instructions were given for preparing the carbon dioxide saturated water, for titrating the water sample, and for computing the total changes in carbon dioxide content.

Herrmann (*14P*) reported a proposed ASTM method for determining carbon dioxide in water-formed deposits. The sample is treated with acid and the loss in weight is taken as carbon dioxide.

An indirect method for determining dissolved hydrogen based on the reaction between hydrogen and oxygen in the presence of a platinum catalyst was described by Faber and Brand (*7P*). Two identical samples are collected. A dissolved oxygen determination is performed on the first sample. A platinum catalyst emulsion is added to the second sample and the dissolved oxygen determined. The decrease in the oxygen content is a measure of the dissolved hydrogen in the sample. In samples having a deficiency of oxygen, a known amount of hydrogen peroxide is added to the second sample to facilitate the oxidation. A simple method for determining hydrogen in condensed steam was reported by Smit and Ehrenburg (*39P*). The oxygen-free sample is divided into two branches, each containing a platinum electrode. A known hydrogen concentration is introduced into one branch, and the unknown concentration is determined from the potential difference.

DETERGENTS

Three reviews of determinations of detergents in water and sewage were reported. Heinerth (*2Q*) described 21 references on nonionic surfactants, mostly on colorimetric methods, not one of which gives an entirely satisfactory method. A thin layer chromatographic method was judged best because the constitution of the ethoxylate under analysis does not have to be known *a priori*. However, this complicated method is not suitable for routine determinations. Krygielowa and Szmalowa (*5Q*) reviewed 40 references on anionic detergents, and Pitter and Hyankova (*8Q*) reviewed 24 references.

Several investigators described methods for the determination of anionic detergents in water with Testa (*10Q*, *11Q*) reporting two automatic ones. Both methods involve the extraction in chloroform of the colored complex formed between the detergent and methylene blue and the spectrophotometric reading of the chloroform coloration at 660 mμ. Samples should contain less than 12 ppm of detergent. Sodergren (*9Q*) used an automatic method for the determination of anionic detergents in water. Peintre and Romens (*7Q*) determined traces of anionic detergents in water by fixing them on Amberlite IRA 68 (OH form). The exchanger was eluted with a mixture of acetone and $0.1N$ sodium hydroxide and the detergent–methylene blue complex was measured spectrophotometrically.

Panowitz and Renn (*6Q*) outlined a modification of the Longwell-Maniece method for the determination of anionic detergents. The essential steps of chloroform handling are carried out in a large glass hypodermic syringe. The device consists of a 50-ml Luer-loc-type syringe, mounted horizontally in a Teflon cradle, and coupled to a 60-rpm CCW gear motor. By using a calibrated syringe, a controlled volume of reagents can be drawn into the reacting system. Another modification of the Longwell-Maniece method, described by Kobayashi, Kanno, and Takuma (*4Q*), reduced the interference of anionic detergents. To a 50-ml water sample containing 0.02 to 0.14 mg of ABS, 25 ml of hydrochloric acid was added and boiled for 1 hour with a water-cooling condenser. After neutralization with sodium hydroxide to the phenolphthalein indicator end point, the Longwell-Maniece method was used. Kambara and Hasebe (*3Q*) determined alkylbenzenesulfonate in water by solvent extraction–alternating current polarography. The range of the method covers from 0.07 to 0.028 mg ABS per 10 ml chloroform, and the time required for a single determination is about 25 minutes.

Arpino (*1Q*) discussed some analytical techniques, particularly the colorimetric and spectrophotometric ones, for the determination of surface active agents. Wayman and Miesch (*12Q*) compared two field methods using a modified methylene blue technique and the dye Toluidine Blue O as the complexing agent *vs.* a laboratory method using methylene blue as the complexing agent. The precision decreases in the order: methylene blue laboratory method, modified methylene blue field method, and the dye Toluidine Blue O field method. Significant bias was detected only in the latter.

PESTICIDES AND HERBICIDES

Van Valin and Kallman (*11R*) investigated the use of activated carbon for the adsorption of chlorinated hydrocarbons from dilute solutions. Warnick and Gaufin (*12R*) used electron capture gas chromatography to obtain reproducible results and measurements of both organic phosphorus and chlorinated hydrocarbon pesticide concentrations of less than 1 ppb in water. Using a flame ionization or electron capture detector unit, May, Hindin, and Dunstan (*8R*) obtained gas chromatographic analyses of pesticides in water. For pesticides containing chloride, a microcoulometric detection unit was used to avoid interference. Most water samples had chlorinated hydrocarbon contents in the range 1 to 10 ppt.

Lamar, Goerlitz, and Law (*7R*) described the advantages, disadvantages, interferences, and application of an electron capture gas chromatographic procedure for the identification and measurement of chlorinated organic pesticides in water. Aldrin, dieldrin, *o,p*-DDT, *p,p*-DDT, endrin, and lindane were studied. Concentrations in the parts-per-trillion range can be determined.

Baker (*2R*) reported on a preliminary study of freezing combined with flame-ionization detection chromatography for concentrating and determining phenols, cresols, and chlorophenols in water. The freeze technique is rapid and may be done with simple, relatively inexpensive equipment. Contaminants are not subject to chemical modification as they would be in distillation or extractive procedures. At a critical dissolved-solids content, spontaneous ice crystallization precludes further separation.

Sanderson and Ceresia (*9R*) described a continuous liquid-liquid extractor that can determine chlorinated aromatic pesticides in parts per billion. It has an efficiency of 95%, is gravity fed, easily controlled, and is safe to operate overnight.

Calderbank and Yuen (*3R*) used a combination ion exchange and spectrophotometric method to determine paraquat residues in food crops and water. Sensitivity is 0.01 ppm.

Atabaev and Khasanov (*1R*) reported a determination of small amounts of aldrin in water. The method is based on the reaction of aldrin with phenyl azide. The resulting aldrin phenyldihydrotriazole, when treated with diazotized 2,4-dinitroaniline in an acid medium gave a pink color that was measured colorimetrically. The intensity of the color and its stability were improved by the addition of 0.50 ml of sulfuric acid (2:1).

In an examination of surface waters and sewage effluents for organochlorine pesticides, Holden and Marsden (*5R*) obtained a concentration factor of 800 by extraction of a 1-liter stream sample with 50 ml of redistilled hexane followed by evaporation to less than 1 ml. The extract was analyzed by gas–liquid chromatography in an all-glass apparatus equipped with electron capture detectors. Dieldrin and DDE were the two residues most commonly found. Their minimum detectable concentration was 0.5 gram per 10^{12} grams of water.

Katz (*6R*) reported determinations of several substituted urea herbicides in surface waters. The herbicides are extracted in chloroform and hydrolyzed under reflux conditions with 6*N* hydrochloric acid. The resulting aniline is diazotized and condensed with *N*-(1-napththyl)ethylenediamine to form a magenta dye, which is extracted with normal butyl alcohol and measured colorimetrically at 555 mμ. Levels as low as 0.02 ppm of linuron, diuron, and neburon, 0.03 ppm of monuron, and 0.04 ppm of fenuron can be determined. Faust and Hunter (*4R*) described chemical methods, ion exchange chromatography, gas–liquid chromatography, and steam distillation methods for the determination of aquatic herbicides. Sigworth (*10R*) noted that coagulation studies with alum and iron salts indicate that these methods do not remove sufficient quantities of herbicides and pesticides from water. Activated carbon is suggested as a means of purification.

ORGANICS

Methods for the determination of organic materials in water are listed for four broad groups: acids, organic carbon, phenols, and miscellaneous.

Starikova and Yablokova (*82S*) determined amino acids in sea water by preparing chromatograms on No. 4 paper. Spots were cut out, extracted with methyl alcohol, and determined at 540 mμ. Error is ±4%. Optimum amount of amino acid for the determination is 2.5 to 12.5 μg. Maistrenko (*60S*) developed a method for the determination of α-amino acids in river waters. After a 0.5- to 1-liter sample was concentrated to 1 or 2 ml and treated with citrate buffer to a pH 2.5 to 4.0, 50 mg of ninhydrin and 3 drops of 10% isopropyl alcohol were added. This mixture was transferred to a Parnas apparatus with 10 ml of water and heated 10 minutes. Ten milliliters of 30% sodium hydroxide were added to the mixture and ammonia was distilled and determined. The method was used with and without preliminary hydrolysis. In waste water analysis Deyl and Rosmus (*20S*) used centrifugal paper chromatography to determine aliphatic acids as sodium or ammonium salts, acid hydroxamates, and 2,4-dinitrobenzyl esters; phenols as arloxyacetic acids; and amino acids.

Verigo and Mamontova (*89S*) described a potentiometric method for determining 10 to 100 mg of free butyric acid per liter in waste water containing high quantities of sodium chloride and hydrochloric acid. Fifty to 200 ml of sample were neutralized with 20% sodium hydroxide, acidified with concentrated phosphoric acid, and the butyric acid was steam distilled into a 200-ml flask. The distillate was boiled 30 minutes under a reflux condenser, cooled to room temperature, and titrated with 0.01*N* potassium hydroxide potentiometrically using normal glass and standard calomel electrodes. Relative error is 5 to 10%.

Stradomskaya and Goncharova (*83S*) reported the use of chromatography on silica gel for separate determinations of butyric, propionic, and acetic acids in natural water. The procedure consists of adding the sample to a silica gel column, eluting with three column volumes of 1% alcohol in $CHCl_3$, and adding thymol blue indicator dropwise to collected fractions. Butyric acid is eluted first, followed by propionic acid; 2% butyl alcohol in $CHCl_3$ elutes acetic acid. Recoveries are: butyric acid, 95 to 103%; propionic acid, 94 to 112%; and acetic acid, 116 to 118%. Lur'e, Alferova, and Bondareva (*57S*) used silica gel in the determination of low molecular weight fatty acids in waste waters. Silica gel was mixed with nearly its own weight in water and with benzene and placed in a column. Dry silica gel, 0.5 gram, was placed on top of this layer. The sample of waste water was neutralized to phenolphthalein, filtered, and evaporated to dryness on a water bath. The residue was dissolved in *N* sulfuric acid and then 1 ml of this solution containing 15 to 30 mg of acids placed at the top of the column. Kapronic and valeric acids were eluted with benzene, butyric acid with 2% butyl alcohol in benzene, propionic acid with 5% butyl alcohol–benzene, acetic acid with 20% butyl alcohol–benzene, and formic acid with 30% butyl alcohol–benzene. The effluents were divided into fractions of 2 ml, diluted with 2 ml of neutralized ethyl alcohol, and titrated with 0.02*N* alcoholic potassium hydroxide with phenolphthalein as indicator. Relative error was approximately 3.5%.

Several researchers described methods for determining humic acid in water. Obenaus (*68S*) determined humic acid by titration with potassium permanganate. Chalupa (*15S*) reported a procedure in which 250 ml of sample containing 5 ml of concentrated sulfuric acid was shaken with 25 ml of amyl alcohol for 30 seconds. The extract was separated and then shaken for 10 seconds with 0.5% sodium hydroxide. Ten minutes later, the alkaline layer was filtered and its absorbance measured at 420 mμ.

Lamar and Goerlitz (*54S*) used gas, paper, and column chromatography and infrared spectrometry to study the characteristics of organic acids in naturally colored surface water samples. The acids were removed from the water by either continuous extraction with *n*-butanol or by vacuum evaporation at 50°. The study indicated the presence of 20 or more carboxylic acids, of which 13 were identified. Approximately 10% of the acids were volatile or could be made volatile for gas chromatographic analysis. Concentrations of the gas chromatographically volatile acids ranged from trace quantities to about 590 ppb. Paper chromatography indicated a mixture of many similar or related compounds but no distinct separations were obtained. Many fractions eluted by column chromatography provided similar infrared spectra. Infrared analysis of column chromatographic fractions indicated that hydroxy carboxylic acids were predominant and included olefinic, aromatic, hydroxy, and carboxylic groups.

Faehnrich and Soukup (*25S*) reported on the ultraviolet absorbance characteristics of organic substances in some river waters. Most of these waters showed distinct minimum absorbance at 260 mμ and maximum values at 280 mμ. Equations are given for calculating the partial absorbance modules of lignin and humus in water samples.

Dement'eva and Vasilevskaya (*18S*) used gas–liquid chromatography to determine C_1 to C_5 fatty acids in water. Fluoroplast-4 powder treated with a solution of stearic acid and SKT (silicone) rubber in a mixture of ethyl alcohol and benzene was used as the packing material. An internal standard and correction factor are employed when the water content is high. The time of analyses was 25 to 30 minutes, error 0.1 to 1.0%.

Sedlacek (*76S*) modified Montgomery's colorimetric method for the determination of fatty acids. To a 2-ml sample of filtrate obtained by coagulation and filtration of sludge waters were added 5 ml of acidic ethylene glycol. After 10 minutes' heating on a water bath and cooling to room temperature, 2 ml of a 10% solution of hydroxylamine–hydrochloric acid and 8 ml of 4.5*N* sodium hydroxide were added. The solution was then mixed with 20 ml of ferric chloride solution and diluted to 100 ml. After 10 minutes, the absorbance of the solution was measured. The method is more precise and simple than the original method and that based on distillation.

Using a modification of the Barker and Summerson method for the determination of lactic acid, Barnes and Finlayson (*5S*) estimated concentrations of lactic acid in sea water solutions and homogenates. The modification consisted of removing bromide ion, which interferes with the determination of lactic acid in sea water, by precipitation as thallous bromide before proceeding with the Barker-Summerson method as indicated by Umbreit, Burris, and Stauffer (*87S*).

Dudova (*22S*) evaluated titrimetric and turbidimetric determinations of naphthenic acids in underground waters. Both methods are unsatisfactory because fatty, humic, and other organic acids are also included in the results. Bykova (*11S*) used the capillary-luminescent method to determine naphthenic acids in water. Naphthenic acids were extracted from 150 ml of analyzed water with 15 ml of chloroform at pH 3 and transferred to a paper strip 50 × 0.8 cm by a capillary lift of extraction accompanied by evaporation of solvent. The optimal concentration of organic substances corresponds to the zone of capillary extraction of less than 10 cm. The paper was transferred to a test tube containing 3 to 4 ml of nonluminescent sodium ester and then the same contacted end of the paper was immersed into the sodium ester, closed hermetically, and chromatographed for 25 to 30 hours. After the paper was dried in ultraviolet light, the boundaries of zones and the luminiscent color of naphthenic acids were determined. Contents of naphthenic acids were determined from a standard curve. The method is more convenient and sensitive than the turbidimetric method. Reproducibility is satisfactory.

Potentiometric determination of organic acids in natural waters was reported by several investigators. Semenov, Bryzgalo, and Datsko (*77S*) concentrated organic acids by passing 100 to 250 cc of water through a column containing either the cation exchanger KU-2 or the anion exchangers AN-22 and AV-17. The acids are eluted with 100 cc of $2N$ ammonium carbonate, the eluate is treated with 2 cc of sodium hydroxide, and the solution is evaporated to dryness on a water bath. The residue is dissolved in 0.5 cc of water, treated with thymol blue indicator, neutralized to pink using N hydrochloric acid, transferred into a centrifuge tube, diluted to 1 cc with water, and heated on a boiling water bath for 1 minute. The organic acids are centrifuged with 2 cc of ether and the extract is added to 0.5 cc of butyl alcohol; this solution is titrated potentiometrically with BuONa in butyl alcohol. Bykova (*12S*) used steam distillation followed by potentiometric titration with $0.01N$ barium hydroxide to determine organic acids in underground waters. Corrections were introduced for the control and for the degree of mineralization of the water. Several organic acids were identified from the shape of the titration curve. Phenols evaporated with steam can be determined in an aliquot of the distillate. Bryzgalo and Semenov (*10S*) studied possible errors in the determination of volatile and nonvolatile organic acids in natural and contaminated waters. Volatile acids were distilled from a water sample acidified with phosphoric acid. The acids were concentrated using anion exchangers AV-17 or Dowex 1X4 and determined by potentiometric titration in nonaqueous media. The same sample was analyzed again to determine the total amount of volatile and nonvolatile acids. Distillation in a vacuum at 15° and cooling the vapor in liquid air prevented analysis errors.

Pohland and Dickson (*72S*) used column chromatography to determine volatile acids in waste water. The acids of a filtered sample of sludge are absorbed on 100-mesh silicic acid in a Gooch or fritted glass crucible. The acids are eluted with a mixture of chloroform, normal butyl alcohol, and sodium bisulfate solution. The quantity of eluted acids is determined by titration with $0.02N$ sodium hydroxide in absolute methyl alcohol. Recovery of the acids is 96.6 ± 3.5%. Andrews, Thomas, and Pearson (*2S*) applied gas chromatography to the determination of volatile acids in sewage sludge. Goncharova and Stradomskaya (*35S*) reviewed 53 references on methods for the separate determination of organic acids in natural waters.

Several investigators reported determinations of organic carbon in water. Tuerkoelmez (*85S*) compared the results of the determination of organics by potassium permanganate and potassium dichromate consumption at different concentrations as a function of the boiling time, and by determining the carbon content by the wet incineration method and the persulfate process. Experiments were made using many organic chemical compounds and samples of industrial waste water. The wet incineration process gave the best results. The persulfate method is not suitable for industrial waste waters containing volatile organic components. An average of only 30 to 40% of the organics is determined by the potassium permanganate method, whereas 90 to 96% of the total organic matter content is determined by the potassium dichromate consumption. Kramig and Schaffer (*51S*) devised a technique for the rapid determination of organic pollution. A sample collected in a polluted stream is sent through a combustion tube with a flow of oxygen to convert all the carbon to carbon dioxide. The carbon dioxide is then determined by infrared spectrometry. Height of peaks obtained is compared with peaks obtained with known amounts of organic materials. Depending upon sample size and instrument sensitivity, as little as 0.1 mg of carbon per liter of solution can be measured. Because total carbon is measured, inorganic carbon must be eliminated. Schaffer *et al.* (*74S*) found that the infrared carbon analyzer used to measure small concentrations of carbon in water could be adapted to the measurement of carbon in a variety of waste samples, particularly those taken at various stages throughout waste treatment processes. Measurements were obtained 15 minutes to 1 hour after sample pickup. Data clearly show correlation of total organic carbon measurements with BOD and chemical oxygen demand.

In a study of the potassium dichromate method for total organic material in water, Bolberitz and Hegyessy (*8S*) found that extending the reaction time significantly raised the characteristic decomposition of the dichromate; however, increasing the temperature to 190° caused virtually no decomposition. Of the catalysts, only silver sulfate was satisfactory.

Brodovskaya (*9S*) noted that in the preparation of underground water samples for the determination of organic carbon, large amounts of an acidic volatile component are lost. He found that this component can be retained by neutralizing the sample with borax before boiling it down.

Van Hall and Stenger (*88S*) developed a method for the rapid determination of total carbon in aqueous solutions in concentrations equal to or less than 2 mg per liter. The method involves combustion of a microsample in oxygen in a heated tube followed by measurement of the carbon dioxide produced. The latter is accomplished by the use of a nondispersive infrared analyzer sensitized specifically for carbon dioxide. The signal from the analyzer is recorded on a strip chart. Another combustion method for the determination of total carbon in water was reported by West (*92S*). As little as 0.004% by weight of carbon in a 10-μl sample was determined. A rate of 10 to 12 samples per hour was achieved by the injection of a liquid sample from a syringe onto a hot copper oxide bed that was continuously swept by the carrier gas. The relative standard deviation was 3.1% at the 0.3 wt % carbon level and 8.2% at 0.004 wt % carbon level.

Organic carbon in sea water received the attention of several investigators. Oppenheimer, Corcoran, and Van Arman (*69S*) described a sensitive and simple method for the determination of particulate or total organic carbon by wet combustion, gas collection, and chromatographic analysis of the carbon dioxide and monoxide gases liberated. The method was calibrated with glucose, was accurate to 1%, and had a lower limit of sensitivity of 0.05 mg of carbon per sample.

Two teams of investigators described similar methods for the determination of dissolved organic carbon in sea water. In both methods 5-ml samples were freed of inorganic carbonate and oxidation of the organic carbon was carried out in sealed glass ampuls. Fredericks and Hood (*29S*) flushed the resulting carbon dioxide gas through a gas chromatograph with helium as the carrier gas. The signal was recorded on a strip chart recorder. Chromatographic analysis time was approximately 11 minutes per sample with a precision of ±0.1 mg of carbon per liter. The organic content of the sample was determined by measurement of the peak area using an appropriate carbon dioxide calibration curve. Menzel and Vaccaro (*64S*) passed the carbon dioxide gas through a nondispersive infrared analyzer with nitrogen as the carrier gas. The signal output was recorded. By using appropriate calibration curves, the carbon content can be determined from the height of the peak. Approximately 100 samples can be analyzed per day with a precision of ±0.1 mg per liter.

A comparison method is also described for the determination of particulate carbon by high temperature combustion after concentration of the sample on a glass fiber filter. The precision of this method is +10 μg of carbon in a range of 0 to 500 μg of carbon. Approximately six samples can be analyzed in an hour. Szekielda and Kreg (*84S*) described an apparatus that permits the determination of particulate organically bound carbon in sea water within 5 to 6 minutes aboard ship. Sea water is passed through a glass filter and the dried filter and retained organic matter are burned in oxygen at 550° in a combustion train. The carbon dioxide is finally absorbed in 0.01N sodium hydroxide and titrated conductometrically. Calibration curves are made with known compounds.

Kreg and Szekielda (*52S*) used a special microscale apparatus to determine organic carbon in sea water. A 5-ml sample was added to the apparatus and oxidized by a hot solution of silver dichromate–potassium dichromate–sulfuric acid. The evolved gases were carried with oxygen through decomposed silver permanganate at 550° for complete oxidation to carbon dioxide, dried with magnesium perchlorate, freed of oxides of nitrogen by special manganese dioxide, and measured by the decrease in the conductance of 0.01N sodium hydroxide by using a recorder to follow the conductance. For 10 to 30 μg of carbon the error is 0 to 2 μg. A single analysis requires 20 minutes.

The determination of oil products in water is outlined in several papers. Bykov and Lakeeva (*13S*) used a modified pycnometric method to determine oil concentrations in waste water. By this modified method, a sample of water is extracted three times with 20 ml of carbon tetrachloride; the extract is dried 1 hour with 10 grams of calcium chloride and finally filtered. After evaporation to 7 ml on a water bath, the extract is placed in a 10-ml pycnometer, diluted to 10 ml with carbon tetrachloride, and allowed to stand 20 to 30 minutes at 20° ± 1°. A formula is given to calculate the quantity of oil products. Nagibina and Ershova (*66S*) noted that determination of petroleum products in industrial water by extraction, evaporation of the solvent, and weighing the residue can give different results depending on whether ethylene oxide, petroleum ether, or benzene is used. Deviations were smallest where the effluents contained only petroleum distillates and nitrobenzene but were quite large with effluents from synthetic fatty acid and alkylphenol plants.

Fastabend (*26S*, *27S*) described two methods for the determination of oil in water by infrared spectrometry. One paper gave a general method. The other reported that mineral oil contamination of ground and surface waters can be determined rapidly with adequate accuracy by taking a single extraction of 1 liter of water with 50 cc of carbon tetrachloride, taking the infrared spectrum, and measuring the absorbance of the CH bands between 3 and 3.5 mμ. The mean error is about ±9.2% for 30 ppm.

Ladendorf (*53S*) discussed the disadvantages of determining the content of different oils in waste water by the Robert Taft Center–developed method and by a German standard method. A universally usable method based on the density of an extract of a waste water was developed. The accuracy and the reproducibility are better than those of the infrared method. A disadvantage is that concentrations less than 0.3 ppm cannot be determined. Giebler, Koppe, and Kempf (*32S*) described a gravimetric method for determination of oils and fatty materials in water. By this method, a mixture consisting of 10 ml of 10% complexon III solution, 500 ml of the water to be treated (vacuum-distillation residue of 1 to 100 mg), 50 grams of sodium chloride, and carbon tetrachloride equivalent to 1% of the water sample is shaken for 1 hour in a funnel wet on the inside with 10 ml of sodium hydroxide solution. The mixture is allowed to stand for 15 minutes and then the lower layer is transferred to a centrifuge tube. After centrifugation for 5 minutes at 3500 rpm, the lower phase is transferred quantitatively to a filter and vacuum distilled at 20° under definite time and pressure conditions. Accuracy of the method is 15% of the carbon tetrachloride–extractable material. The same investigators (*33S*) described a thin-layer chromatographic determination of mineral oil in water. Chromatograms are developed on plates, dried 5 minutes in air, and then sprayed uniformly with 7 ml of 1% phosphomolybdic acid in *tert*-butyl alcohol. Fuel and lubricating oils give blue-gray spots. The amount of oil is estimated by comparison with chromatograms for known amounts of oil. The method permits determination of 0.02 mg of mineral oil per liter of water. Golubeva (*34S*) outlined a determination of petroleum and petroleum products in water. The water is acidified with sulfuric acid and saturated with sodium chloride and then extracted with diethyl ether. After 5 hours drying over sodium sulfate or calcium chloride, the diethyl ether is evaporated in a tared special flask on a 45° to 50° water bath. Colorimetric, nephelometric, or luminescence methods may be used to complete the analysis.

Several investigators reported chromatographic and instrumental determinations of phenols and related materials in water: Baker (*4S*) and Scholz (*75S*), gas chromatography; Goren-Strul, Kleijn, and Mostaert (*36S*), gas–liquid chromatography; Bidlo (*7S*), Koch (*48S*), and Lur'e and Nikolaeva (*58S*), paper chromatography; Dyatlovitskaya and Maktaz (*23S*), thin layer chromatography; Skrynnikova, Govorova, and Matveeva (*80S*), coulometric and colorimetric; Babkin and Spitsin (*3S*), Barskii and Noskov (*6S*), Chalupa and Dvorakova (*16S*), Csanady (*17S*), Goto, Kawahara, and Sato (*37S*), Herrig (*41S*), Parfenov, Shaginyan, and Ardzhanov (*71S*), and Zege, Shenderovich, and Stoma (*100S*), colorimetric. Fleszar (*28S*) described a method for the determination of nitrochlorobenzenes in water. A 2-liter sample is prepared by shaking with approximately 0.03 gram of activated carbon for 60 to 80 minutes. Then the sample is filtered under vacuum, the carbon is extracted with 15 ml of dimethyl ketone, kept at its boiling point for 90 minutes, cooled, and washed three times with a 1:1 dimethyl ketone–water mixture. Washings and extractions are combined with 5 ml of reagent solution (0.2M $C_5H_5N \cdot HCl$ in C_4H_5N mixed 2:3 with 7% barium chloride and diluted with water to 50 ml) are added. Five milliliters of solution are treated with nitrogen to remove oxygen and scrubbed with an alkaline solution of pyrogallol and dimethyl ketone; the polarograph curve is recorded from 0.2 to 0.9 volt at an applied potential of 4 volts *vs.* an internal mercury electrode. To the analyzed solution is added 0.5 ml of standard containing 0.02 gram of nitrochlorobenzene per liter in 1:1 dimethyl ketone–water mixture and the process is repeated.

Lewis (*55S*) described a continuous

phenol analyzer. The sample is converted to sodium phenolate, which is continuously decanted in a separatory funnel, filtered through $^1/_8$-inch Raschig rings, and cleaned through glass wool. Hydrochloric acid is added to reform the phenols before the sample enters a 2.5-cm cell. A collector rod transmits the light from a hydrogen lamp to a chopper mirror which, with corner mirrors, alternately passes the light rays through the sample and reference cells. A relay lens forms the light on a photomultiplier tube. Chemical and interference filters absorb unwanted radiation. The difference in intensities striking the photomultiplier is proportional to the phenol content. Electronic adjustments automatically reset the amplifier to zero when periodically calibrating.

From a study to find a rapid and accurate method for the determination of pentachlorophenol in water, Uede, Nagai, and Osafune (*86S*) reported that Numata's 4-aminoantipyrine method and the colorimetric method following oxidation with nitric acid are the best methods to use. From his survey of the different methods to determine phenol and detergents in waste water, Malz (*61S*) reported that the photometric methods and the spectrometric methods using *p*-nitroaniline and 4-aminoantipyrine are the most convenient ones for phenols within the concentration range 0.001 to 100 ppm. For detergents, only the anionic ones that react with methylene blue can be determined accurately.

Melpolder *et al.* (*63S*) developed a method for the determination of nonionic detergents in water and sewage; it involves passage through an ion exchange resin to remove any ionic material, passage through a carbon column (a little deactivated) to remove organic material, and foam fractionation. The method gives 80% recovery of nonionic material from river water, more than 90% recovery from sewage effluents, and between 86 and 100% recovery from crude sewage. However, the method is time-consuming and a day is required to complete a single determination.

Higashiura (*42S*) described a determination of para-substituted phenols in waste water. Phenols other than the para-substituted ones react with 4-aminoantipyrine to form dyes. Thus, a mixture of phenols was first reacted with this chemical and then passed through an ion exchange column containing the OH form of Dowex 1-X8. The formed dyes from the unsubstituted phenols pass through the column but the para-substituted phenols remain within. Elution with methyl alcohol plus normal sodium chloride (1:1) and methyl alcohol plus 1.7*N* acetic acid (1:1) removes the para substituted phenols which are diazotized with sulfanilic acid and determined photometrically at 495 mμ *p*-Cresol, 2,4-xylenol, 3,4-xylenol, and *p*-ethylphenol were determined by this method.

Palaty and Deylova (*70S*) noted that when 4-aminoantipyrine and *p*-aminodimethylaniline are used in the determination of phenols in water, certain substances, if present, interfere. The substances include aniline, sodium sulfide, sodium cyanide, sodium sulfite, sodium thiosulfate, and ammonium thiocyanate. The investigators recommend distillation of an acidified sample with heavy metal salt.

Dozanska and Sikorowska (*21S*) compared the 4-aminoantipyrine and 2,6-dibromoquinone chlorimide methods for determination of phenols in water and sewage. Fujii and Shimamine (*30S*) applied the 4-aminoantipyrine method to the determination of phenol in drinking water.

Kondo and Murakami (*49S*) found that a mixed solvent containing 30 to 60% acetophenone in benzene gives greater than 95% extraction of phenols in water.

In addition to the methods for the determination of organic acids, organic carbon, and phenols, numerous methods were reported for a variety of organic materials. Nemtsova, Semenov, and Datsko (*67S*) described a spectrophotometric determination of volatile amines at 410 mμ. Kalalova (*45S*) reported a determination of hexamethylenetetramine in the presence of formaldehyde in waste waters. The total formaldehyde is determined in 10 ml of sample that is refluxed with 20 ml of 1% sulfuric acid at 60° for 30 minutes, cooled, and diluted to 50 to 100 ml. To a 10-ml. aliquot, 1 ml of concentrated hydrochloric acid and 5 ml of Schiff's base are added. After 1 hour the color is measured with a green filter. To determine hexamethylenetetramine, 10 ml of sample are refluxed with 2 ml of 1% sodium hydroxide and 0.2 ml of 1.2% hydrogen peroxide at 60° for 30 minutes to remove the free formaldehyde. The hexamethylenetetramine is then oxidized with 20 ml of 1% sulfuric acid for another 30 minutes and determined in concentrations 0 to 100 mg per liter.

Konenko and Yarysh (*50S*) outlined a determination of residual concentration of 3-(*p*-chlorophenyl)-1,1-dimethylurea in reservoir waters after their treatment with monuron. A 100- to 200-ml sample and 50 to 100 ml of 30% sodium hydroxide are boiled 1 hour under reflux, after which 80 to 85 ml are distilled into 5 ml of 6*N* hydrochloric acid and made up to 100 ml; 50 ml are treated with 1 ml of 1% sodium nitrite, 1 ml. of 10% sulfamic acid, and 1 ml of 2% *N*-(1-naphthyl)ethylenediaminehydrochloride. The violet color is measured spectrophotometrically. A standard solution of *p*-chloraniline is treated similarly.

Masek (*62S*) reviewed sources of carcinogenic polycyclic hydrocarbons in waters and the methods of detection and separation of these substances.

Yurko and Volkova (*95S*) determined acrolein content of waste water. Filtered water is treated with ammonia to remove iron salts and then mixed with hydrochloric acid and a 96% alcohol solution; 19.5 ml solution is treated with 2 ml of a 0.2% tryptophane solution in hydrochloric acid at 45° to 50° for 15 to 20 minutes, and the determination is made colorimetrically.

Adamovsky (*1S*) described a benzene determination based on the desorption of benzene from waste water by nitrogen as carrier gas, which is then passed into a nitration mixture; the resulting *m*-dinitrobenzene is determined polarographically.

Shabunin and Lavrenchuk (*79S*) determined microamounts of chlortetracycline in waste water by a method based on the fluorescence of its decomposition products. A 20-ml sample is brought to pH 8.5 to 9 and diluted to 25 ml. Its fluorescence is then compared with that of a similarly prepared standard of approximately the same concentration in a LOF-54 fluorometer. Methyl alcohol, isopropyl alcohol, butyl alcohol, phenols, diethyl ether, dimethyl ketone, carbon tetrachloride, and cyanides do not interfere.

Zamyslova (*97S–99S*) described methods for determining specific substances in water. These included tetranitromethane, dimethylformamide, and hydroperoxide of isopropylbenzene.

Klinger (*47S*) noted that a disadvantage of the amperometric method for the determination of collectors in the flotation of pulp and organic substances in waste water due to anodic oxidation and deposition of butylxanthate and xylenol on graphite electrodes can be minimized by mechanical cleaning of electrodes with nylon fiber brushing devices or by removing the oxidized products from electrodes through periodic alternating polarization of electrodes. A concentration of 10^{-4} to $10^{-6}M$ organic materials in pulps can be determine amperometrically with these cleaned electrodes.

DePablo (*19S*) determined total glycoluril in swimming pool water. The determination includes the addition of EDTA and sodium arsenite solutions followed by a mixed reagent consisting of sodium hydroxide, potassium ferricyanide, and sodium nitroprusside. After 20 minutes, the absorbance is determined at 540 mμ. Time, reagent preparation, and additions are critical. The precision is less than 0.1 mg per liter at the 8 mg glycoluril per liter level and less than 0.2 mg per liter at 26 mg of glycoluril.

Vorob'eva (*90S*) determined hexadecanol in surface water. The material is extracted in benzene; the extract is made up to 15 ml, dried with 1 gram of sodium sulfate, and a 3-ml aliquot is mixed with 1 ml of specially prepared benzene solution. After 20 minutes at 65° to 70°, 1 ml of 1*N* sodium hydroxide is added, the mixture shaken 1 to 2 minutes, and the color of the benzene layer after separation and drying with sodium sulfate is compared with that of standards by using a blue filter in the colorimeter. Accuracy with 0.06 to 1 mg of hexadecanol per liter is ±6.4% of the determined amount.

To determine hydrazine in waters containing organic matter, Kalinina (*46S*) added a few crystals of sodium azide to remove nitrite ions from the sample and a few drops of 2*N* hydrochloric acid to neutralize the sample. The sample is then mixed and two 50-ml aliquots are taken for analysis. Iodine is added dropwise to one, and the solution reduced again with 0.1*N* sodium sulfite to remove the hydrazine. After 0.8 ml of 10% *p*-dimethylaminobenzaldehyde in 2*N* hydrochloric acid is added to both aliquots, which are kept for 30 minutes in the dark, the light absorption of the two aliquots is measured by using a blue filter and the first aliquot as a blank.

Lur'e and Nikolaeva (*59S*) described a determination of small amounts of hydroquinone in solutions and waste water. To 5 ml of sample is added 1 ml of acetate buffer, pH = 5.75; 1 ml 0.004*N* ferric chloride containing 1.5 ml of hydrochloric acid per liter; and 0.5 ml of 0.5% 1,10-phenanthroline containing 2 ml of hydrochloric acid per liter. This is diluted to 20 ml with water. The absorption is determined at 508 mμ against a blank after standing 1 hour. If the water contains resorcinol and pyrocatechol, the sample is treated with lead acetate whose pH is adjusted to 5.75 to 5.85 with 0.1*N* sodium hydroxide 10 minutes later, and then 0.25 ml of freshly prepared 2% sulfite ion is added. The determination is continued as above but the absorbance is measured only after 30 minutes' standing.

Zadera (*96S*) outlined a colorimetric method for the determination of lignin substances in waste waters. To 100 ml of sample, 4 ml of 10% acetic acid are added and the absorbance is determined; 2 ml. of fresh 10% sodium nitrite are then added, and after 20 to 120 minutes the absorbance is determined. The precision of the method is 10%.

Loehr and Higgins (*56S*) reported that the wet extraction of lipids in waste water, sewage sludge, and effluents is more efficient than the Soxhlet method where the filter does not retain all the insoluble lipids of the water. A series of 10 samples can be determined in 4 hours. The wet extraction method can extract motor oil, tristearin, and sodium oleate up to concentrations of 6500, 7400, and 9200 mg per liter, respectively, with the same efficiency as at lower concentrations.

Yazhemskaya (*93S*) reported that naphthalene in water can be determined by extracting with heptane and measuring the absorbance of the extract at 220 mμ; or air can be passed through a 200-ml sample on a boiling water bath and then through two absorbers containing ethyl alcohol and the absorbance of the ethyl alcohol at 220 mμ can be compared to that of similar solutions prepared using water containing known amounts of naphthalene.

Hegi, Fischer, and Maerki (*40S*) described a chromatographic method to determine 10 μg or more of nicotine in water. Details of the procedure are given.

Ryckman, Burbank, and Edgerley (*73S*) outlined advantages of new collection, concentration, and characterization procedures for the evaluation of organic pollutants. Myrick and Ryckman (*65S*) designed and used a technique to obtain significant quantities of organic refractories from a large volume of highly turbid natural water. Sedimentation and diatomite filtration were used as pretreatment processes. Activated carbon adsorption filters in series were used under both basic and acidic conditions to concentrate these dissolved agents.

Yurko and Volkova (*94S*) developed a colorimetric method for the determination of phthalic anhydride in waste water. Hopkins and Head (*43S*) reported that concentrations as low as 0.01 ppm of pyrethins in public water supplies may be extracted with light petroleum ether and determined either spectrophotometrically or by gas-liquid chromatography. The spectrophotometric method may be used quantitatively over a range of 7 to 100 μg. The gas-liquid chromatography method gives good response down to 0.1 μg of total pyrethins.

Gavrilescu (*31S*) reported that various experimental data show that glucide substances occur in both fresh and salt water. These substances are a result of algae and phytoplankton. The results verify the validity of the Puetter theory. Ivleva, Semenov, and Datsko (*44S*) used *p*-aminohippuric acid and a spectrophotometer to determine reducing sugars in natural water. The average error is 10 to 15% and the sensitivity of the determination is 20 μg of sugars. Semenov, Ivleva, and Datsko (*78S*) found that the optimum condition for hydrolyses of reducing sugars in natural waters preparatory to their determination is heating the sample for 12 hours at 100° in 1*N* sulfuric acid.

Sokolov (*81S*) noted that when waste water is acidified to pH of less than 2, an acidic, water-insoluble styromal forms; thus, the formed precipitate is stabilized by a gelatin solution and the styromal is determined neophelometrically.

Carlucci and Silbernagel (*14S*) described a method using carbon-14 uptake by the diatom *Cyclotella nana* for the bioassay of vitamin B_{12} in sea water.

Gottauf (*38S*) outlined an improved heat-space technique for gas chromatographic determinations of 2-pentanone, hexanal, isoamyl alcohol, limonenene, hexyl acetate, PrSSPr, and Me_3N. The lower limit of detection is $<10^{-8}\%$ with a relative standard deviation of ±0.05.

Weiss, Johnson, and Kwon (*91S*) reported that organic components of natural waters can be chromatographed on suitable liquid phase columns (poly *m*-phenylene ether, silicone, polyethylene glycol) with helium as a carrier and at an optimal temperature of the column of 150°. Use of a hydrogen flame detector established that 0.1-μl samples were sufficient for routine analyses.

Harder and Holden (*39S*) described two methods for the determination of methane gas in ground water.

Ettinger (*24S*) reviewed developments in the detection of trace organic contaminants.

RADIOACTIVITY AND ISOTOPIC ANALYSIS

Loveridge, Gordon, and Weaver (*46T*) determined actinium-227 in effluent by coprecipitation with ferric hydroxide. If algae are present, the precipitate is dissolved in acid and oxidized with nitric acid and perchloric acid before adjusting the acidity to pH 1.6 and extracting the actinium with bis(2-ethylhexyl)phosphoric acid in heptane. The actinium is back extracted into 1.5*N* hydrochloric acid, and the solution passed through an anion exchange column to remove traces of lead, bismuth, and polonium. The eluate containing actinium is evaporated. After drying overnight and allowing any actinium-228 to decay, the residue is dissolved in dilute acid and fed into a reverse phase partition chromatography column to separate actinium from rare earths and from its own daughters. The eluate is evaporated and the residue dissolved in dilute acid and counted. Radiochemical recoveries range from 80 to 95%.

Tyrrell, Demetsopoullos, and Collinson (*72T*) used a nuclear emulsion to determine the alpha activity of drinking water. Solutions were concentrated by evaporation and adsorbed on 50-micron nuclear emulsions. After suitable exposure and development, the emulsions were scanned for alpha tracks.

Kotlyarov and Khitrov (*41T*) described a 4-π counter for measuring beta activity in sea-going laboratories. The performance of the instrument was tested by measuring strontium-90 and cerium-144 in ocean water and beryllium at the ocean's bottom. Characteristics of the counter include a background of 1.5 to 2.6 cpm and an efficiency of 60 to 80%. Other methods for determining beta activity in water were described by Haberer (*29T*), Yakimov (*77T*), and

Henke (*33T*). The latter two investigators also described methods for measuring alpha and gamma activity. Malvicini (*48T*) reported that total alpha and beta activity can be measured directly and continuously on a flowing stream by a gas-filled proportional counter connected to a recorder. Gamma activity is measured with a scintillation counter.

Crosby and Chatters (*17T*) advocated the use of anion-exchange resins for stripping water of carbon dioxide for carbon-14 determination. Equipment that uses anion exchangers and that can be adapted to pump-site sampling, in-well sampling, or sampling of surface waters was developed. Analytical results, by means of which resin techniques and standard methods are compared, proved to be within the normal errors expected in the carbon-14 method.

Methods for the determination of radioactive cesium in rainwater include one by Fonseca *et al.* (*22T*) that uses ammonium molybdophosphate as an ion exchanger; one by Derecki, Geisler, and Jaworowski (*18T*) that is a modification of the preceding method; one by Benes (*9T*) that counts the beta activity of cesium-137 dipicrylaminate; and a method by Johnson *et al.* (*36T*). Other determinations of cesium-137 include one by Shipman and Mueller (*65T*) that uses permanganate as a precipitant; one by Folsom, Pillai, and Finnin (*21T*) that uses the pulse height spectrum of the internal conversion electrons acting upon detectors to determine cesium-137 in sea water; and one by Boni (*13T*) that uses $K_2CoFe(CN)_6$ as the ion exchanger to absorb cesium from neutral, concentrated hydrochloric acid, concentrated nitric acid, and highly salted (sodium nitrate) sodium hydroxide solutions.

In a determination of radiocerium in biological matter and sea water by Hampson (*32T*), cerium-141 and cerium-144 are coprecipitated with iron or titanium (as hydroxides) and are purified by solvent extraction with bis(2-ethylhexyl) hydrogen phosphate in *n*-heptane. Cerium-144 is measured by counting the beta irradiation from the praseodymium-144 daughter; aluminum sheet is used to absorb the beta irradiation from cerium-141.

Hahn and Marsh (*30T*) described a determination of cobalt-60 that involves stripping cobalt from an organic layer and gamma counting it in a deep well-type scintillation counter. For exchangeable cobalt in sea water, Viswanathan *et al.* (*74T*) recommended coprecipitation and determination by activation analysis.

In a determination of iodine-131, Brutovsky *et al.* (*15T*) extracted the iodine in carbon tetrachloride, precipitated the iodide as silver iodide, and dissolved the precipitate in sodium cyanide. As little as 10^{-11} curies of iodine-131 per liter can be determined by this method. Fairman and Sedlet (*20T*) determined iodine-131 in milk, water, and other materials by ion exchange on silver chloride.

Kautsky and Schmitt (*38T*) described a determination of iron-55 and iron-59 in sea waters. Iron along with added carrier elements (inactive iron, manganese, zirconium, zinc, cobalt, cerium, and yttrium) was precipitated at pH 10.2 from acidified sea water samples by the addition of sodium hydroxide. Then, through a series of precipitations, iron was separated and fired to the magnetic iron oxide form. As little as 0.45×10^{-12} curies of iron-55 per liter could be determined by radiation measurement of the magnetic iron oxide.

Tamas and Opauszky (*71T*) used the reactions of equilibrium between water and carbon dioxide at room temperature and at 900° C to determine the oxygen-18 content of small water samples. The method is more reproducible at room temperature than at 900° C but its long time of exchange (24 hours) is a disadvantage. A mass spectrometer was used to analyze the isotopes. Vetshtein (*73T*) also used mass spectrometric analysis to determine isotope oxygen composition of water. The method consists of the determination of oxygen evolved from water by an improved persulfate method instead of carbon dioxide used in the old method. The method gives reproducible results with an accuracy of $\pm 0.02\%$.

Hahn and Ross (*31T*) described a method for the determination of radioactive phosphorus in water. Phosphorus is concentrated with aluminum hydroxide, the precipitate dissolved in perchloric acid, and a manganese dioxide scavenging step performed to remove radioniobium. Phosphorus is then separated from other activities by extraction of the phosphomolybdate complex into a butanol–chloroform mixture, back extracted into ammonia and precipitated as magnesium ammonium phosphate, which is weighed and beta-counted.

Rushing (*61T*) applied the coprecipitation of polonium-210 with tellurium by stannous chloride as precipitant to the determination of polonium-210 in water. The doubly precipitated tellurium-210 polonium precipitate is alpha-counted on 0.22-micron membrane filters. A shortcut single precipitation method is also given. The methods have sufficient sensitivity to permit their use in the determination of lead-210 in water by polonium-210 ingrowth.

Determinations of radium in water were reported by three different teams of investigators. Barker and Johnson (*5T*) determined radium by coprecipitating it with barium sulfate and comparing the alpha activity of the precipitate to a standard radium solution. Polynkova, Titova, and Novikov (*56T*) mixed saturated calcium chloride solution and aqueous solutions containing 0.48 gram radium per liter at various pH's and added sodium carbonate until calcium carbonate began to precipitate. Carbon dioxide is removed by a stream of air, and then activated carbon and sodium carbonate are added to precipitate calcium carbonate and radium. Optimum pH of precipitation for various activated carbon materials is given. In another determination of radium using activated carbon, Drozhzhin, Lazarev, and Nikolaev (*19T*) described a method for determining radium without advance chemical separation. A sample of water is acidified, air bubbled through it to remove any radon present, and then the container is sealed. The sample is allowed to age and the accumulated radon is adsorbed on activated charcoal by bubbling nitrogen through the sample. The radon is finally desorbed from the charcoal and run through an ionization or scintillator counter. The initial radium concentration of the water is then calculated. Radium concentrations ranging from 10^{-13} to 10^{-11} gram per liter can be determined by the method.

Spirin, Belle, and Chimina (*67T*) described a method of measuring radon content in water as well as the decomposition products radium B and radium C that are in equilibrium with radon. The measuring system consisting of 20 Geiger-Mueller counters is able to measure samples of a volume down to 0.5 liter. The sensitivity is 1.3×10^{-10} curies.

Skonieczny (*66T*) reported methods for the determination of radioactive ruthenium and niobium in water and sewage. Iwashima (*34T*) reviewed 230 references on the separation and determination of radioactive ruthenium in natural water.

Basu (*7T*) described a method for the estimation of sulfur-35 in sea water. Sulfur is oxidized by boiling the sample with hydrogen peroxide in ammoniacal solution. Through a series of precipitations purified barium sulfate is formed which is then beta-counted to determine the concentration of sulfur-35. Sensitivity of the method is 0.005 $\mu\mu$c sulfur-35 per milliliter of sea water.

Several methods of determining strontium-90 in natural waters were reported. In a method for determining strontium in sea water, described by Azhazha (*4T*), strontium is separated from calcium through precipitation with sodium carbonate in the presence of disodium EDTA and nitric acid, which removes the calcium by dissolving calcium nitrate only.

Petit and Kienberger (*55T*) determined strontium-90 in drinking water at levels equal or greater than 5 dpm per ml by adding strontium carrier, evaporating the sample to dryness, and leaching the residue with 1*N* hydrochloric acid. The leachate was scavenged with ferric hydroxide and strontium oxalate precipitated in solution of ammonium oxalate and ammonium chloride. The precipitate was metathesized to strontium nitrate in concentrated nitric acid, dissolved in water, and reprecipitated. Finally strontium oxalate was precipitated and counted in a proportional counter. An absorption curve was run to correct for yttrium-90 and strontium-90.

Ralkova (*58T*) described a rapid control method for the determination of strontium-90 in drinking water. The method is based on the coprecipitation of trivalent cations, including yttrium-90, with ferric hydroxide. Strontium-90 remains in the filtrate assisted by calcium carrier. According to Aksel'rod (*2T*), the determination of strontium-90 background activity in water basins requires concentration of at least 100 liters of water. Concentrated hydrochloric acid added directly to the water sample aids desorption of strontium-90 from suspended solids and prevents its adsorption on the container walls. To determine strontium-90 in river waters, strontium-90 and calcium must be separated with nitric acid. Before the nitrate separation of strontium and calcium, the trivalent ions of iron and aluminum must be separated by precipitation with ammonium hydroxide.

Molt (*51T*) reported that the nitric acid method for determining strontium-90 in water is improved by separating strontium as the nitrate from calcium nitrate. This is accomplished by boiling the nitrate precipitate in anhydrous amyl alcohol in which the soluble calcium nitrate is separated from the insoluble strontium nitrate.

Jeanmaire, Patti, and Bullier (*35T*) and Talvitie and Demint (*70T*) used ion exchange and precipitation techniques to determine strontium-90 in water. Sereda and Bobovinkova (*63T*) suggested a simple method for large-scale observation of the strontium-90 content of fresh water reservoirs. Addition of ammonium carbonate and ammonium hydroxide to the sample precipitate calcium salts. The method permits the precipitation of carbonates, while uranium and potassium remain in the settling basin. Strontium-89, strontium-90, cerium-141, zirconium-95, niobium-95, barium-140, etc. are precipitated with calcium carbonates. Precipitation is followed by the ordinary radiochemical process of dissolution in acid and rare earth separation on iron hydroxide. Yttrium daughter products are precipitated later as yttrium oxalate. Relative error in strontium separation from yttrium is ±5%.

A more improved method permits the precipitation of strontium and calcium directly upon separation from other radioactive impurities and measurement of the beta spectrum of the carbonate precipitate after 2 to 15 days. The accumulated yttrium-90 verifies strontium-90 content. Shipman (*64T*) determined strontium-90 in sea water after concentration by manganese dioxide.

Several methods for determining strontium-90 in water, but along with cesium-137, were reported. Golutvina *et al.* (*25T*) noted that in the electrodialysis of natural water in a glass electrodialyzer with a platinum mesh anode and a platinum mesh cathode contained within a cellophane membrane cell concentrating strontium-89, strontium-90, and cesium-137 in the cathode cell was very slow; much faster concentration was obtained when a circular platinum wire anode surrounding the cathode cell was used. Determination of strontium-90 and cesium-137 in rain water was outlined by Govaerts, Guillaume, and Walch (*26T*). A liter of sample, to which strontium nitrate and cesium nitrate carriers have been added, is acidified, filtered, and concentrated to 100 ml. Two 40-ml aliquots are removed. Strontium sulfate is precipitated from one, and cesium cobalt nitrite from the other. The precipitates are counted automatically at 10-minute intervals for 5 hours in a thin window 2-π Geiger counter.

Kautsky (*37T*) reviewed work to 1961 on the checking of radioactivity in sea water and described methods that he used for the determination of strontium-90 and cesium-137 in sea water. Strontium was precipitated as carbonate and freed from calcium by nitric acid separation. After 14 days, strontium-90 was determined by separation and measurement of the yttrium-90 formed. After carbonate and potassium cobaltate precipitation of cesium and its subsequent conversion to iodobismuthate, the cesium-137 was determined by measurement of its beta radiation. Patin (*54T*) reviewed 26 references on the determination of strontium-90 and cesium-137 content of sea water.

Aksel'rod and Lyalikov (*3T*) described a photometric determination of thorium in natural waters using Arsenazo III. Details of the procedure are given. The complex is measured at 665 mμ using a red filter. Extraction of thorium from dilute solutions with gelatin tannate was described by Keshishyan, Andreev, and Danilov (*39T*). Addition of 1% tannin to a solution containing 1,500 μg thorium per liter did not result in precipitation between pH 1 and 12; however, addition of 1% gelatin to the same solution precipitated a gelatin-tannate complex that occluded different amounts of thorium. The maximum amount of thorium precipitated occurred at pH 3 to 4.

Determination of thorium in tap water and sea water was reported by Akaishi (*1T*). Thorium was precipitated with sodium hydroxide at pH 9 in the presence of calcium and phosphoric acid. The precipitate was dissolved in 1*N* nitric acid and ethyl alcohol and then passed through an anion exchange column to separate calcium, magnesium, and phosphate ions. Thorium was eluted with 8*N* hydrochloric acid and determined by Neothorin spectrophotometry. The method has a sensitivity of 0.5 μg of thorium.

Several investigators reported determinations of tritium in natural waters. Bibron (*11T*) described two methods that use a low-background coincidence counter. In the first method, tritium is concentrated by electrolysis in aqueous form and then introduced into a water-miscible scintillation medium. In the second method, tritium in the sample is converted to benzene, which serves as the scintillation solvent for an organic scintillation medium. Lal and Athavale (*44T*) used a nonelectrolytic enrichment method that permits an accurate determination of tritium/hydrogen ratios exceeding 10^{-17}. A factor of 20 enrichment suffices to measure up to 25 years of "age" levels of tritium in ground waters.

Modes and Koch (*50T*) used a proportional counter to determine the absolute activity of tritiated water. Tritiated water was reduced to $^3H_2$ in a sealed bomb, and the $^3H_2$ was carried with propane into a cylindrical counting tube. Specific activity values were 1% higher than those obtained microcalorimetrically and 2% lower than those from a steam ionization chamber. Nishiwaki and Kawai (*52T*) outlined a routine method of monitoring tritium in natural waters. The tritium concentration of samples is enriched electrolytically from a 6-liter volume to a final volume of 5 ml or less. The radioactivity of the enriched tritium is measured in a liquid scintillation counter.

Takahashi (*69T*) described a measurement of tritium at natural levels in water. The distilled sample and sodium hydroxide are put into an electrolytic cell containing nickel and stainless steel electrodes. After the volume has been reduced to one twelfth of its initial size, the tritium concentration is 8 to 10 times as great as that in the initial volume. Hydrogen is then generated by exposing magnesium chips to the sample vapor at 570° to 600° C and the counting tube filled. Zel'venskii *et al.* (*80T*) showed that the maximum degree of enrichment in tritium and oxygen-18 is obtained under total reflux at an average column temperature of 52.5° C and a pressure of 100 mm of mercury (in the upper section).

Determinations of deuterium and heavy water in natural water were reported by several investigators. Bastick, Baverez, and Castagne (*6T*) separated H_2–HD–D_2 gases formed by the reduction of water–heavy water mixtures with iron by passing them through a chromatographic column packed with aluminum oxide impregnated with ferric chloride as the stationary phase and using helium as the carrier gas at the temperature of liquid nitrogen. The maximum error of determination was ±0.05.

Applications of infrared spectrometry to the measurement of deuterium and heavy water were described by Bayly, Stevens, and Thurston (*8T*); Bosselaar (*14T*); Fydelor and Rawson (*23T*); and Knezevic (*40T*). Lazzarini (*45T*) reported good agreement between heavy water concentrations determined by means of a vapor pressure osmometer and calculated values. Gandara, Gispert, and Rojas (*24T*) studied relationship between heavy water concentrations and the variation in density *vs.* temperature of water.

Techniques for the determination of fallout activities in natural waters were discussed by several investigators. After concentrating radioactive species by electrodeposition, Richter and Wright (*59T*) used a nonselective stripping agent and standard chemical separation procedures to determine thallium, cadmium, ruthenium, silver, cesium, selenium, tantalum, strontium, and cadmium plus ruthenium. Wallace (*75T*) outlined separation and purification procedures for strontium, calcium, barium, lead, polonium, and beryllium, and radiometric methods for strontium-89, strontium-90, cesium-137, cerium-141, cerium-144, barium-140, and the naturally occurring lead-210, polonium-210, and beryllium 7. Yamaoka *et al.* (*78T*) used both the ion exchange method and gamma ray spectrometry to determine radionuclides in rain water and also investigated methods for the determination of small amounts of uranium-237 and uranium-238. Wood and Richards (*76T*) modified and extended a sequential analysis scheme for the radiochemical determination of fallout nuclides in drinking water and rain water. In the same sample they were able to determine manganese-54, yttrium-91, promethium-147, and zirconium-95 as well as the more commonly determined barium, cesium, cerium, and strontium fallout nuclides. Malvicini (*47T*) reviewed the methods of control and measurement of radioactivity in waste waters and presented detailed determinations of strontium and cesium.

Rovinskii (*60T*) derived an equation that permits the calculation of the concentration of a radioactive contaminant in the water of stationary reservoirs at any moment after the introduction of the contaminant. Its validity was verified experimentally in lakes having a surface of 4.5 and 11.3 km². Lai and Goya (*43T*) prepared a compendium of radiochemical procedures for the following fission-product nuclides: cesium-137, strontium-89, strontium-90, yttrium-91, cerium-141, cerium-144, barium-140, zirconium-95, ruthenium-103, ruthenium-106, molybdenum-99, and iodine-131. All procedures use either solvent extraction, precipitation, or ion exchange chromatography.

Methods for the determination of low levels of radioactivity were presented by several investigators. Kranz and Weise (*42T*) used a proportional counter to measure low levels of radioactivity in river waters. In a continuous measurement of low levels of activity in water, Quirk (*57T*) used a stream of hot air to evaporate a fine spray of water. The resulting solids were collected on a filter, positioned under a detector, and counted. According to Grune *et al.* (*27T*), factors that must be considered in selecting an ion exchange resin for concentrating low-level concentrations of nuclides include ionic radius, activity of ion, valence, hydration of resin and ion, diffusion rate through the resin, flow rate through the column, and temperature. Resin cross-linkage, mesh size, capacity for exchange, and stability are also important.

Grune *et al.* (*28T*) also described a method of wet ashing ion exchange resins by the use of concentrated perchloric, nitric, and sulfuric acids, followed by measurement of activity of the ashed resins. A dry-ashing procedure was studied and found to be less practical than direct dry ashing in a muffle furnace. Elution was considered impractical as a rapid radio-assay technique. Schonfeld, Kibbey, and Davis (*62T*) tested γ-ray spectrum resolution for use in studies of processes concerned with the decontamination of waste water containing only 10^2 to 10^5 μμc of radionuclides per liter. Tests with two-multichannel spectrometers and associated data analyses by the method of least squares were made with solutions of cobalt-60, strontium-85, cerium-141, and cerium-144 added as tracers to water containing strontium-90, zirconium-95, niobium-95, ruthenium-106, cesium-134, and cesium-137. Some of the solutions were concentrated by evaporation by factors up to 20 to evaluate this feature for improving accuracy of analyses. For comparison, the solutions were also analyzed by standard radiochemical methods.

Agreement between the two methods was excellent for cobalt-60 and strontium-85 concentrations down to about 500 μμc per liter (scatter of ±5 to ±20%). Agreement between the two methods was good for cesium-137 at activities down to 3,000 to 4,000 μμc per liter; comparative values from the two methods differed by factors of 5 to 10 for cerium-144, except at activities above 5,000 μμc per liter. More dilute solutions were successfully analyzed only when first concentrated by evaporation. Because of large statistical fluctuations at ruthenium-106 concentrations as high as 1,000 to 2,000 μμc per liter, no particular significance can be given to the data for this nuclide.

Stefanov *et al.* (*68T*) removed uranium, thorium, and radium from solution by adsorption on KU-2 resin at pH 4 to 6. Uranium can be separated from thorium and radium by extraction of the resin with 3.6% oxalic acid; thorium can be removed by treatment of the resin with 5% oxalic acid; and thorium and radium can be removed by treatment of the resin with 20% hydrochloric acid or acetic acid.

Minarik (*49T*) showed that γ-spectrometry is suitable for the identification of some natural isotopes in surface waters. Yule (*79T*) used a sodium iodide (thallium) detector to find limits of determination of 72 elements for whole blood, urine, milk, tap water, distilled demineralized water, and polyethylene vials. Craft, Erb, and Hill (*16T*) outlined detailed procedures for chemical separation prior to counting of strontium, barium, yttrium, cesium, iodine, total rare earth activity, cerium, ruthenium, zirconium, and niobium.

Blaga, Blaga, and Chifu (*12T*) devised a variant of the droplet method for determining the isotopic composition of water. A micropipet was incorporated into the apparatus so that the drop volume was determined directly. A mechanical control system allowed very fine adjustment of the water level in the micropipet capillary. The micropipet is easily removed and washed under the best conditions. Pascalau, Blaga, and Blaga (*53T*) described an apparatus that automatically records falling time in the isotopic analysis of water by the falling drop method. Benes, Drasil, and Petrlik (*10T*) described a nuclear emulsion for use in the autoradiographic determination of low alpha activities in water.

MISCELLANEOUS

Marten (*36U*) described automatic colorimetric, Kjeldahl digestion, turbidimetric, solvent extraction, and distillation techniques. Using activation analysis, Grimanis *et al.* (*23U*) determined concentrations of silver, gold, arsenic, barium, bromide, chloride, copper, iodide, potassium, manganese, sodium, rhenium, vanadium, and zinc in lakes; Ikeda, Kondo, and Yamashita (*28U*) determined the concentrations of the rare earth elements in hot spring waters; and Wyttenbach (*58U*) determined concentrations of barium, nickel, copper, uranium, rubidium, and cesium in ground-, river-, and mineral waters.

Various investigators reported analytical methods for determination of alkalinity and pH of water. Herce and Luppi (*26U*) reviewed methods for determination of alkalinity. Smith and Hood (*50U*) recommended a sea water buffer, tris(hydroxymethyl)-aminomethane, having a dissociation constant of 1.202×10^{-6}, as a replacement for distilled water buffers to eliminate drift in pH measurements. Temperature effects were determined to be ±0.005 pH units for ±0.17° errors. Gauna (*22U*) reported that the Berl-Lunge-D'Ans method for the acidimetric determination of calcium bicarbonate in presence of sodium bicarbonate in natural waters containing calcium and magnesium chlorides and sulfates is valid only for waters in which other salts of alkaline earth metals are not present.

In analysis of carbonate brines containing carbon dioxide in the range 0.5 to 9.0% by potentiometric titration and by manometric measurements of total carbon dioxide evolved upon acid treatment, Rettig and Jones (*43U*) found that a plot of the potentiometric data showed inflection points indicating that the carbonate and bicarbonate end points could differ significantly from the pH values of 8.2 and 4.5 used in routine water analysis. Zavodnov (*60U*) presented equations for use in the calculation of carbon dioxide and pH of underground waters from the carbonate equilibrium. Results obtained by this method compare favorably with experimental ones. According to Barker (*4U*), modern high-pressure boilers have a phosphate concentration between 10 and 25 ppm, which puts the theoretical equivalence point in the range pH 5.4 to 5.6.

Three synthetic phosphate solutions, containing a known amount of sodium hydroxide were titrated to a pH of 5.4 and also 4.5. The titration to pH 5.4 gave the nominal phosphate concentration whereas the results at pH 4.5 were too high. Wilms, Van Haute, and Rand (*57U*) reported a simple method for calculating adjustments needed to obtain equilibrium of water. The method involves solving two equations given in two unknowns and assumes that the only basic ions present are bicarbonate, carbonate, and hydroxide.

Beisova, Solomin, and Fesenko (*5U*) compared sodium hydroxide titration of aluminum- and iron-containing waters with potentiometric titrations. The results of the sodium hydroxide titration are 0.3 to 6.2% high compared with the potentiometric titrations, and they resemble more nearly the sum of aluminum plus ferric iron plus hydrogen ions.

Several investigators described apparatus used in the determinations of chemical and physical characteristics of water. Shvaishtein (*46U*) proposed an apparatus for measuring sea ice salinity where the precision need not exceed 0.1 to 0.05% with the use of the transistorized salinometer CM-6M. It consists of a high frequency transistorized voltage converter and a measuring scheme consisting of a microamperometer, rectifier, voltage divider, and filter. The detector of the instrument is a plastic vessel with two graphite electrodes in its walls. The measurement is based on the principle of the determination of electrolytic conductance of solutions. Konovalov and Semenov (*30U*) described an apparatus that reduces the time of extraction of trace elements and permits the use of greater volumes of water. Research and development work in progress on a portable self-contained water quality monitor was discussed by Testerman *et al.* (*52U*).

Cozzi (*14U*) designed a flexible, portable water analysis apparatus for laboratory and field use. Provision is made for the determination of the redox potential, pH, temperature, and conductance; for polarometric and amperometric measurements, including the determination of oxygen; and for colorimetric, turbidimetric, fluorometric, and nephelometric measurements. Natural and industrial waters and aqueous solutions of inorganic and organic substances can be analyzed. Mobile laboratories for analysis of water were described by Brondi *et al.* (*11U*) and by Haesselbarth and Langer (*24U*).

In geochemical studies, Larionov, Alekseeva, and Rivman (*34U*) proved the efficiency of using various analytical methods for analysis of ground waters. For waters having a mineral content less than 0.5 gram per liter, they recommended coprecipitation of metals with cadmium sulfide in addition to chemical and spectral analysis of dry residue. For samples having a mineral content greater than 5 grams per liter, they recommended chemical analysis.

Allemand (*2U*) applied the laser technique to emission spectroscopy. A sample is placed on the stage of a microscope and is brought visually into focus. Then the microscope is used to focus a laser beam onto the sample. The luminous flux from the laser evaporates the sample, and the vapors emitted by the sample pass through an electric field and produce a discharge that excites a luminous emission strong enough for analysis by a spectrograph.

Atomic absorption analysis of natural and industrial waters is being used more widely. Elements that have been determined include: lithium, sodium, magnesium, potassium, calcium, manganese, iron, cobalt, nickel, copper, zinc, rubidium, strontium, and barium—Billings and Harriss (*7U*), Wheat (*56U*), West and Herrin (*55U*), Fabricand *et al.* (*19U*), Kristiansen (*32U*), Platte and Marcy (*41U*), Fishman (*20U*), Fishman and Downs (*21U*), Slavin (*49U*), and Strasheim and Kruger (*51U*).

Correction to standard conditions of color measurements of natural water were discussed by Singley, Harris, and Maulding (*47U*). They adopted pH 3, a representative value of finished waters, as a reference standard and devised a nomograph that allows ready determination of water color at the standard pH 8.3. Using a quartz spectrophotometer in the wavelength range 335 to 800 mμ, Petrov (*40U*) compared the platinum-cobalt and imitation scales for the determination of color in natural water. The imitation scale gave spectral curves similar to that of the platinum-cobalt scale but with a characteristic bend in the range 400 to 500 mμ and a nearly constant transmission coefficient between 425 and 465 mμ. Curves of the imitation scale reached their limiting value at 540 to 600 mμ, whereas curves of the platinum-cobalt scale reached the limit at 480 to 580 mμ. A combination in aqueous solution of ferric chloride and cobalt chloride gave a spectral curve closer to that of Ladoga Lake water than either of the two scales examined.

Determinations of specific conductance and dissolved-solids content of water received the attention of several investigators. Duran-Lopez (*15U*) devised a nomogram for the rapid determination of specific conductance and dissolved-solids content. Shimanova and Ponomarev (*45U*) developed and tested an apparatus for determining the salt composition of sea water by measuring the electrical conductance. Accuracy was 0.005 ppt for ocean water and 0.01 to 0.02% for sea water. Using an inductively coupled conductance indicator, Reeburgh (*42U*) measured the absolute specific conductance of solutions prepared from a sample of Red Sea water. The conductance was measured at 2° temperature intervals over the range 1 to 35° C and at 1% chlorinity intervals from 16 to 22%. The precision of individual measurements was 0.001 mmho per cm in conductance, 0.002% in chlorinity, and 0.001° in temperature.

A new type of plastic hydrometer for the rapid determination of total salts in ground water in the field is described by Yao, Chu, and Yu (*59U*). The accuracy is approximately 0.2 gram per liter. The effect of photosynthesis and respiration on the electrical conductance of sea water was studied by Park and Curl (*39U*). They found a 0.06% decrease in conductance after 65 hours of illumination and a 0.03% increase after a 49-hour dark period as a result of photosynthesis and respiration of the diatom *Skeletonema costatum* in sea water enriched with nutrients. There were also changes in pH, total chlorophyll, and oxygen concentration.

Roennefahrt (*44U*) discussed the theoretical basis of redox-potential measurement and cited examples of applications in the determination of metals and other ions in effluents.

Techniques for sampling of water were discussed by several investigators. Veshchezerov (*54U*) reported that a piston nonwash sampler can be used to collect water samples for the determination of carbon dioxide and hydrogen sulfide provided that the component to be determined can be bound chemically and converted to a stable compound at the moment of sampling. An appropriate reagent of barium hydroxide and cadmium acetate is poured into the sampler under the movable piston before letting it down into the borehole. The reagent is loaded with a special buret with a valve; the sample is poured from the sampler with a special funnel. This sampling method can be used for the determination of ferrous iron, ferric iron, and other unstable and volatile components.

Cherry (*13U*) described a portable sampler for collecting water samples from specific zones in uncased or screened wells. The sampler consists of two inflatable boots, one mounted above the submersible pump and the other below it. The sampler is portable and easily repositioned without removing it from the well. Auxiliary instruments for measuring temperature, specific conductance, or other chemical or physical characteristics of the ground water can be inserted between the boots.

Edeline and Heuze (*17U*) described two tools for collecting hydrocarbons from surface films. The simplest consists of loose-weave filter paper disks placed flat on the surface to remove the hydrocarbon film. The second consists of a copper cylinder 7 cm high and 17.5 cm in diameter, a handle, and lateral orifice of 5 mm. The inside of the cylinder is lined with chromatographic paper and is wetted with distilled water. The sampler is submerged to within 1 cm of the top edge of the paper. One or two drops of concentrated commercial detergent are added at the center of the intercepted circle. When the hydrocarbon film has receded to a 1- or 2-cm annulus at the periphery of the cylinder, the sampler is slowly withdrawn.

Brusilovskii and Kanakina (*12U*) suggested that differences in the cation-anion sum of ordinary methods of analyzing high-acid waters are due to specific features of the composition of these waters where the hydrogen ion concentration is comparable to the concentration of other components. They present a detailed discussion of calculations that attempt to eliminate the difference in the cation-anion sum. Dyrssen (*16U*) used the methods of Gran I and Gran II to determine equivalence points in potentiometric titrations of sea water. In an analysis of waste water, Borchert (*9U*) used rapid colorimetric and volumetric methods for the quantitative determination of hexavalent chromium, oxidizable cyanides, and chloride in aqueous plant effluents. Experiments by Belyaev (*6U*) with sea water showed that the values closest to the time concentration of trace heavy metals can be obtained from the frequency curve of the probability "d" of distribution. Use of statistical processing permits one to decrease the error to a few per cent and simultaneously to decrease considerably the number of repeated analyses.

Suspended matter in water was the subject of several papers. Electron micrographs enabled Ishii and Ishikawa (*29U*) to identify certain constituents of suspended solids in sea water. Eden (*18U*) summarized a progress report of an analytical panel charged with a comprehensive study of the measurement of turbidity in water. Results obtained by eight laboratories using their own methods to analyze a prepared sample indicated enormous variability between laboratories.

A series of experiments indicated that the apparent reasons for variability between laboratories was inherent in the range of instruments, methods, and operators and could not be eliminated by complete standardization. Black and Hannah (*8U*) used the Brice-Phoenix Absolute Light Scattering Photometer and the Photovolt Lumetron, Model 450, with a nephelometry attachment to measure the light-scattering properties of 17 processed waters. Both instruments were calibrated with a standard fuller's earth suspension. Intensities of scattered light per unit volume of water were 80 to 400 times greater at 15° from the incident beam than at 90°. Because of the different relative scattering intensities from the samples at any given angle, the absolute photometers with high-angular resolution will not give turbidities in close agreement with those measured by the photocell instruments, which integrate the light scattered over a wide angle. With the absolute photometer, relative changes in turbidity equivalent to 0.001 turbidity unit can be measured in low turbidity waters.

Baalsrud and Henriksen (*3U*) noted that suspended matter in water can be determined by light absorption and light scattering methods. The physical properties of settling, particle size, and filtering, and the chemical properties are determined through inorganic and organic methods of analysis. No one method provides a complete description of the suspended matter. Tettamanzi (*53U*) described an apparatus that reduces the time of a suspended solids in water determination from approximately 8 to 2 hours. Hannah and Dwyer (*25U*) used a Perkin-Elmer Model 521 with single and triple reflection ATR accessories to obtain infrared spectra of colloidal trace contaminants in water.

Several investigators used the spectrograph to determine trace elements in water. Aidarov and Gribanova (*1U*) used the ISP-22 and KS-55 spectrographs to determine concentrations of lithium, rubidium, cesium, thallium, strontium, and boron in salts, brines, and silts. Using a direct-reading spectrochemical procedure and the rotating-disk, high-voltage spark technique, Kopp and Kroner (*31U*) determined the following minor elements: silver, aluminum, arsenic, boron, barium, beryllium, cadmium, cobalt, copper, iron, molybdenum, manganese, nickel, phosphorus, lead, strontium, vanadium, and zinc. By using background as the internal standard, concentrations in the order of 0.01 to 100 mg per liter in the processed sample are determined in a matter of minutes by converting the counts from a sequential readout system on a drum calculator. Recoveries of the elements from known solutions ranged from 80 to 113% at the 90% confidence level.

Marti and Herrero (*37U*) reported the spectrochemical determination of 38 elements in the concentration range 0.001 to 1% in mineral waters. Morris (*38U*) described the use of gallium oxide in spectrographic procedures. With gallium oxide as a carrier, beryllium oxide was excited by a dc arc. A gas mixture of 80% argon and 20% oxygen was used to determine iron, chromium, nickel, manganese, lead, magnesium, copper, calcium, silicon, and aluminum to 1 ppm. Impurities in distilled and deionized water were determined with gallium oxide as a collector. Zyrin *et al.* (*61U*) recommended a spectrographic analysis in which the concentration is determined by darkening of analytical lines on a photographic spectrum. Concentrations of approximately 30 trace elements in soils, plants, and water can be determined by this analysis. The sensitivity of the method is generally between 5 and 10 mg per kg of material except for zinc for which the sensitivity is much lower.

By means of spectrochemical analysis, Boswell and Brooks (*10U*) determined the organic aqueous distributions (% extraction) for extractions at 25° with cyclohexanone and cyclohexanol. The two solvents were presaturated with the same *M* of hydrochloric acid that the aqueous phase had. The average coefficients of variation for several elements were given. Cyclohexanone is more efficient but less selective than cyclohexanol. A simple technique for *in situ* extraction of many trace elements in sea water was described by Lal, Arnold, and Somayajulu (*33U*). The method is based on ferric hydroxide adsorption. A matrix of finely dispersed ferric hydroxide on a natural sponge or fibrous jute framework is towed through the sea. Silicon, titanium, aluminum, and beryllium (present in sea water in concentrations of 10^{-4} to 10^{-9} grams per liter) were extracted from the equivalent of some hundreds of tons of coastal waters. The radioactive nuclides beryllium-7 and silicon-32 were measured in the extract.

Hueckstedt (*27U*) obtained a sensitive redox indicator from the following reagents: (*a*) 10 ml commercial 0.025*M* ferroin solution diluted to 100 ml. with water and (*b*) 3 ml commercial 0.1*N* cerium (IV) sulfate solution mixed with 30 ml 25% sulfuric acid diluted to 100 ml with water. These solutions can be stored for at least 2 months, and exclusion of atmospheric oxygen was found to be unnecessary. For the determination of traces of reducing compounds, 0.5 ml of each solution was accurately measured into a white dish, mixed with water, and the test solution added slowly. The end point was marked by the appearance of a bluish red color that turned pure red after standing 30 to 60 seconds.

In studies of light-dependent quality changes in stored water samples, Slack and Fisher (*48U*) found no significant difference among samples after dark-light storage in polyethylene bottles of three degrees of transparency. After exposure to alternate light and dark periods, the pH ranged from 9.27 to 10.17. In most samples the carbonate concentration ranged from 7 to 13 ppm, and free carbon dioxide and nitrate were not detected. During the period of dark storage, pH decreased 2 units to the range 7.15 to 8.02, and free carbon dioxide and bicarbonate concentrations increased greatly. Nitrate and sulfate increased in all samples. Phosphate concentration was greater in the illuminated samples than in the continuously dark samples, but the magnesium concentration was greater in the continuously dark samples.

Margara (*35U*) described a simple apparatus for the evaporation of several liters of dilute mineral waters for the determination of the residue and of the principal cations. Standard methods for total hardness, dry residue, alkali metals, sulfate, and chloride were outlined. Bicarbonate was determined by acidimetric titration of the calcium, sodium, and potassium carbonates and of the $x\mathrm{MgCO_3}\cdot y\mathrm{Mg(OH)_2}\cdot z\mathrm{H_2O}$ formed during evaporation of the sample. The ratio x/y does not affect the determination of bicarbonate.

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