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# PROCEEDINGS OF THE SIXTY-THIRD ANNUAL MEETING OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, 1949

The sixty-third annual meeting of the Association of Official Agricultural Chemists was held at the Shoreham Hotel, Washington, D. C., October 10, 11, and 12, 1949.

The meeting was called to order by the President, L. S. Walker, on the morning of October 10, at 10:00 o'clock.

## OFFICERS, COMMITTEES, REFEREES, AND ASSOCIATE REFEREES OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS FOR THE YEAR ENDING NOVEMBER 1950

### President

W. A. QUEEN, Food and Drug Administration, Washington, D. C.

### Vice-President

H. A. HALVORSON, Department of Agriculture, Dairy and Food, St. Paul, Minn.

### Secretary-Treasurer Emeritus

W. W. SKINNER, Kensington, Md.

### Secretary-Treasurer

HENRY A. LEPPER, U. S. Food and Drug Administration, Washington 25, D. C.

### Additional Members of the Executive Committee

W. B. WHITE, Washington, D. C.
H. J. FISHER, New Haven, Conn.
E. L. GRIFFIN, Washington, D. C.
L. S. WALKER, Burlington, Vt.

### Editorial Board

HENRY A. LEPPER, *General Chairman*

*Editorial Committee of The Journal*

W. B. WHITE (U. S. Food and Drug Administration, Washington 25, D. C.), *Chairman and Editor* (1955)*

E. L. GRIFFIN (1952) — J. B. SMITH (1953)
F. H. WILEY (1952) — A. H. ROBERTSON (1955)

KATHARINE RONSAVILLE, *Assistant Editor*

*Editorial Committee of Methods of Analysis*

H. J. FISHER (Agricultural Experiment Station, New Haven 4, Conn.) *Chairman*

J. WALTER SALE — E. L. GRIFFIN
F. H. WILEY — W. F. REINDOLLAR

MARIAN LAPP OTIS

* Figures in parenthesis refer to year in which appointment expires.

### Committees

*Committee to Confer with American Public Health Association on Standard Methods of Milk Analysis*

A. H. ROBERTSON (Director State Food Laboratory, Albany, N. Y.), *Chairman*
WILLIAM HORWITZ J. O. CLARKE

*Representatives on the Board of Governors of the Crop Protection Institute of the National Research Council*

W. H. MACINTIRE, Knoxville 16, Tenn.

## Committee on Recommendations of Referees†

W. F. REINDOLLAR (Bureau of Chemistry, State Department of Health, Baltimore 18, Md.), *Chairman*

#### SUBJECTS, REFEREES, AND ASSOCIATE REFEREES†

SUBCOMMITTEE A:

H. A. HALVORSON (1950), (Department of Agriculture, Dairy and Food, St. Paul, Minn.), *Chairman;* E. L. GRIFFIN (1952); and J. B. SMITH (1954).

FEEDING STUFFS:

*Referee:* M. P. Etheredge, Mississippi State College, State College, Miss.

MINERAL MIXED FEEDS (CALCIUM AND IODINE):
A. T. Perkins, Kansas State College, Manhattan, Kans.

LACTOSE IN MIXED FEEDS:
C. W. Sievert, American Dry Milk Institute, Chicago 1, Ill.

FAT IN FISH MEAL:
Maurice E. Stansby, Fish and Wildlife Service, Seattle 2, Wash.

ADULTERATION OF CONDENSED MILK PRODUCTS:
P. B. Curtis, Purdue University, Lafayette, Ind.

CRUDE FAT OR ETHER EXTRACT:
H. H. Hoffman, Department of Agriculture, Tallahassee, Fla.

MICROSCOPIC EXAMINATION:
J. A. Schrader, Agricultural Experiment Sta., Lexington 29, Ky.

FLUORINE:
D. M. Doty, Purdue University, Lafayette, Ind.

MINERAL CONSTITUENTS OF MIXED FEEDS:
J. L. St. John, Agricultural Experiment Station, Pullman, Wash.

CRUDE FIBER:
W. L. Hunter, Department of Agriculture, Sacramento 14, Calif.

PROTEIN EVALUATION IN FISH AND ANIMAL PRODUCTS:
Frank J. Kokoski, N. Y. Dept. Agriculture and Markets, Albany, N. Y.

SAMPLING AND ANALYSIS OF CONDENSED BUTTERMILK:
R. E. Bergman, State Department of Agriculture, St. Paul, Minn.

TANKAGE (HIDE, HOOF, HORN, AND HAIR CONTENT):
A. T. Perkins

SULFUR DRUGS IN FEEDS:
R. T. Merwin, Agricultural Experiment Station, New Haven, Conn.

† Referees appointed during the year for unassigned subjects will be announced in the *Journal*.

FERTILIZERS:

*Referee:* F. W. Quackenbush, Agricultural Experiment Station, Lafayette, Ind.

SAMPLING AND PREPARATION OF SAMPLE:

H. R. Allen, Agricultural Experiment Station, Lexington 29, Ky.

PHOSPHORIC ACID:

K. D. Jacob, Plant Industry Station, Beltsville, Md.

FREE WATER:

W. L. Hill, Plant Industry Station, Beltsville, Md.

NITROGEN:

M. P. Etheredge

MAGNESIUM AND MANGANESE:

John B. Smith, Agricultural Experiment Station, Kingston, R. I.

ACID- AND BASE-FORMING QUALITY:

E. W. Constable, State Department of Agriculture, Raleigh, N. C.

POTASH:

O. W. Ford, Purdue University, Lafayette, Ind.

SULFUR:

Gordon Hart, Department of Agriculture, Tallahassee, Fla.

COPPER AND ZINC:

H. J. Webb, A. and M. College of South Carolina, Clemson, S. C.

BORON:

G. N. Tyson, Pacific Coast Borax Co., 5 State St., Pasadena, Calif.

INERT MATERIALS:

K. G. Clarke, Division of Fertilizer and Agricultural Lime, Beltsville, Md.

ECONOMIC POISONS:

*Referee:* J. J. T. Graham, Production and Marketing Administration, Insecticide Division, Beltsville, Md.

TETRA ETHYL PYROPHOSPHATE:

S. A. Hall, Production and Marketing Adm., Insecticide Div., Beltsville, Md.

HERBICIDES:

A. B. Heagy, Md. Inspection and Regulatory Service, College Park, Md.

RODENTICIDES:

J. B. La Clair, Department of Agriculture, Sacramento 14, Calif.

BENZENEHEXACHLORIDE:

C. V. Bowen, Bur. Entomology and Plant Quarantine, Beltsville, Md.

ORGANIC THIOCYANATES:

H. A. Rooney, Department of Agriculture, Sacramento 14, Calif.

DIMETHYL DITHIO CARBAMATES:

J. D. Patterson, Department of Agriculture, Salem, Oreg.

DDT:

E. E. Fleck, Bur. Entomology and Plant Quarantine, Beltsville, Md.

OIL EMULSIONS:

Lloyd Keirstead, Agricultural Experiment Sta., New Haven, Conn.

PARATHION:

F. I. Edwards, Production and Marketing Adm., Insecticide Div., Beltsville, Md.

SODIUM FLUOROACETATE:

---

CHLORDANE AND TOXAPHENE:

T. H. Harris, Production and Marketing Adm., Insecticide Div. Beltsville, Md.

QUATERNARY AMMONIUM COMPOUNDS:
R. L. Caswell, Production and Marketing Adm., Insecticide Div., Beltsville, Md.
COAL-TAR DISINFECTANTS:
W. A. Affens, Production and Marketing Adm., Insecticide Div., Beltsville, Md.

DISINFECTANTS:
*Referee:* L. S. Stuart, Production and Marketing Adm., Insecticide Div., Beltsville, Md.

PLANTS:
*Referee:* E. J. Miller, Agricultural Experiment Station, East Lansing, Mich.
SAMPLING:
E. J. Miller
IODINE AND BORON:
L. K. Wood, Agricultural Experiment Station, Lexington 29, Ky.
SUGAR:
Kenneth T. Williams, Western Regional Research Lab., Albany, Calif.
ZINC:
E. J. Benne, Agricultural Experiment Station, East Lansing, Mich.
COPPER AND COBALT:
Kenneth C. Beeson, U. S. Plant, Soil, and Nutritional Lab., Ithaca, N. Y.
CAROTENE:
E. J. Benne
SODIUM:
Ray L. Shirley, Agricultural Experiment Sta., East Lansing, Mich.
CELLULOSE AND LIGNIN:

PECTIN:

STARCH:
Carroll L. Hoffpauir, Southern Regional Research Lab., New Orleans, La.

SOILS AND LIMING MATERIALS:
*Referee:* W. H. MacIntire, Agricultural Experiment Station, Knoxville 16, Tenn.
HYDROGEN-ION CONCENTRATION OF SOILS:
Lannes E. Davis, Div. of Soils, Calif. Agr. Expt. Sta., Davis, Calif.
BORON AND FLUORINE
L. K. Wood
ZINC AND COPPER:
W. L. Lott, U. S. Bur. Plant Industry, Soils, and Agr. Eng., Raleigh, N. C.
EXCHANGEABLE CALCIUM AND MAGNESIUM:
W. M. Shaw, Agricultural Experiment Station, Knoxville 16, Tenn.
EXCHANGEABLE HYDROGEN:
W. M. Shaw
EXCHANGEABLE POTASSIUM:
J. F. Reed, N. C. State College of Agr. and Eng., Raleigh, N. C.
PHOSPHORUS:

STANDARD SOLUTIONS:
*Referee:* H. G. Underwood, Food and Drug Administration, Washington, D. C.

VITAMINS:

*Referee:* Chester D. Tolle, Food and Drug Administration, Washington 25, D.C.

VITAMIN A:

J. B. Wilkie, Food and Drug Administration, Washington 25, D. C.

VITAMIN A IN ANIMAL FOODS:

Maxwell L. Cooley, General Mills, Inc., Larraine Div., Rossford, Ohio

VITAMIN C IN MILK:

W. L. Hall, Food and Drug Administration, Washington 25, D. C.

VITAMIN D—POULTRY:

Leo Friedman, Food and Drug Administration, Washington 25, D. C.

NICOTINIC ACID:

J. P. Sweeney, Food and Drug Administration, Washington 25, D. C.

CAROTENE:

F. W. Quackenbush

PANTOTHENIC ACID:

H. W. Loy, Jr.

FOLIC ACID:

Laura Flynn, College of Agriculture, University of Missouri, Columbia, Mo.

VITAMIN $B_{12}$:

Carl H. Krieger, Wisconsin Alumni Foundation, Madison, Wis.

SUBCOMMITTEE B:

G. R. CLARK (1950), (Food and Drug Administration, Washington 25, D. C.), *Chairman;* F. H. WILEY (1952), and HARRY J. FISHER (1954).

RADIOACTIVITY:

*Referee:* L. Costrell, National Bureau of Standards, Washington 25, D. C.

SPECTROGRAPHIC METHODS

*Referee:* W. T. Mathis, Connecticut Agricultural Expt. Station, New Haven, Conn.

VEGETABLE DRUGS AND THEIR DERIVATIVES:

*Referee:* P. S. Jorgensen, Food and Drug Administration, San Francisco, Calif.

THEOBROMINE AND PHENOBARBITAL:

Daniel Banes, Food and Drug Administration, Washington 25, D. C.

AMINOPYRINE, EPHEDRINE, AND PHENOBARBITAL:

H. C. Heim, School of Pharmacy, University of Colorado, Boulder, Colo.

QUININE AND STRYCHNINE:

D. [illegible]. Miller, Food and Drug Administration, Buffalo 3, N. Y.

RUTIN IN TABLETS:

A. Turner, Eastern Regional Research Lab., U.S.D.A., Philadelphia, Pa.

SYNTHETIC DRUGS:

*Referee:* F. C. Sinton, Food and Drug Administration, New York 14, N. Y.

METHYLENE BLUE:

H. O. Moraw, Food and Drug Administration, Chicago 7, Ill.

SULFANILAMIDE DERIVATIVES:

H. W. Conroy, Food and Drug Administration, Kansas City 6, Mo.

PROPADRINE HYDROCHLORIDE:

R. D. Stanley, Food and Drug Administration, Chicago 7, Ill.

CARBROMAL:
R. Hyatt, Food and Drug Administration, Cincinnati 2, Ohio
BUTACAINE SULFATE:
L. H. Welsh, Food and Drug Administration, Washington 25, D. C.
SPECTROPHOTOMETRIC METHODS:
J. Carol, Food and Drug Administration, Washington 25, D. C.
PROPYL THIOURACIL:
Gordon Smith, Food and Drug Administration, New York 14, N. Y.
PYRIBENZAMINE AND BENADRYL:
H. C. Heim
SYNTHETIC ESTROGENS:
Daniel Banes, Food and Drug Administration, Washington 25, D. C.

MISCELLANEOUS DRUGS:
*Referee:* Iman Schurman, Food and Drug Administration, Chicago 7, Ill.
MICROSCOPIC TESTS FOR ALKALOIDS AND SYNTHETICS:
W. V. Eisenberg, Food and Drug Administration, Washington 25, D. C.
MERCURY COMPOUNDS:
R. L. Herd, Food and Drug Administration, St. Louis, 1 Mo.
ORGANIC IODIDES AND SEPARATION OF HALOGENS:
V. E. Stewart, State Department of Agriculture, Tallahassee, Fla.
ALKALI METALS:
H. F. O'Keefe, Food and Drug Administration, Chicago 7, Ill.
GLYCOLS AND RELATED COMPOUNDS:
Harry Isacoff, Food and Drug Administration, New York 14, N. Y.
PRESERVATIVES AND BACTERIOSTATIC AGENTS IN AMPUL SOLUTIONS:
C. N. Jones, Food and Drug Administration, New York 14, N. Y.
ESTRONE AND ESTRADIOL:
E. O. Haenni, Food and Drug Administration, Washington 25, D. C.
METHYL ALCOHOL:
J. F. Guymon, Agr. Expt. Sta., College of Agriculture, Davis, Calif.

COSMETICS:
*Referee:* G. R. Clark, Food and Drug Administration, Washington 25, D. C.
COSMETIC CREAMS:
C. F. Bruening, Food and Drug Administration, Chicago 7, Ill.
COSMETIC POWDERS:

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COSMETIC SKIN LOTIONS:
H. R. Bond, Food and Drug Administration, Kansas City 6, Mo.
DEODORANTS AND ANTI-PERSPIRANTS:
H. Kramer, Food and Drug Administration, Baltimore 2, Md.
HAIR DYES AND RINSES:
S. W. Newburger
MASCARA, EYEBROW PENCILS, AND EYE SHADOW:
Paul W. Jewel, Max Factor and Company, Hollywood, Calif.
SUN TAN PREPARATIONS:
E. Hoshall, Food and Drug Administration, Baltimore 2, Md.

COAL-TAR COLORS:

*Referee:* K. A. Freeman, Food and Drug Administration, Washington 25, D. C.

INTERMEDIATES IN TRIPHENYL-METHANE DYES:

A. T. Schram, National Aniline Division, P.O. Box 975, Buffalo 5, N. Y.

ETHER EXTRACT IN COAL-TAR COLORS:

S. S. Forrest, Food and Drug Administration, Washington 25, D. C.

HALOGENS IN HALOGENATED FLUORESCEINS:

J. H. Jones, Food and Drug Administration, Washington 25, D. C.

IDENTIFICATION OF CERTIFIED COAL-TAR COLORS:

Rachel Sclar, Food and Drug Administration, Washington 25, D. C.

VOLATILE AMINE INTERMEDIATES IN COAL-TAR COLORS:

J. O. Millham, Food and Drug Administration, Washington 25, D. C.

NON-VOLATILE UNSULFONATED AMINE INTERMEDIATES IN COAL-TAR COLORS:

L. S. Harrow, Food and Drug Administration, Washington 25, D. C.

SULFONATED AMINE INTERMEDIATES IN COAL-TAR COLORS:

N. Ettlestein, Food and Drug Administration, Washington 25, D. C.

UNSULFONATED PHENOLIC INTERMEDIATES IN COAL-TAR COLORS:

H. Holtzman, Ansbacher-Siegle Corp., Rose Bank, Staten Island, N. Y.

SULFONATED PHENOLIC INTERMEDIATES IN COAL-TAR COLORS:

W. C. Bainbridge, H. Kohnstamm Company, Brooklyn 31, N. Y.

INTERMEDIATES DERIVED FROM PHTHALIC ACID:

C. Graichen, Food and Drug Administration, Washington 25, D. C.

MIXTURES OF COAL-TAR COLORS FOR DRUG AND COSMETIC USE:

W. C. Bainbridge

LAKES AND PIGMENTS:

C. Graichen

SPECTROPHOTOMETRIC TESTING OF COAL-TAR COLORS:

J. H. Jones

SUBSIDIARY DYES IN D&C COLORS:

L. Koch, H. Kohnstamm and Company, Brooklyn 31, N. Y.

HEAVY METALS IN COAL-TAR COLORS:

C. Stein, Food and Drug Administration, Washington 25, D. C.

ARSENIC IN COAL-TAR COLORS:

L. S. Harrow

SUBSIDIARY DYES IN FD&C COLORS:

M. Dolinsky, Food and Drug Administration, Washington 25, D. C.

HYGROSCOPIC PROPERTIES OF COAL-TAR COLORS:

C. Stein

BOILING RANGE OF PSEUDO-CUMIDINE XYLIDINE IN CERTIFIED COAL-TAR COLORS:

L. S. Harrow

COLORING MATTERS IN FOODS:

*Referee:* C. F. Jablonski, Food and Drug Administration, New York 14, N. Y.

SUBCOMMITTEE C:

J. O. CLARKE (1950), (Food and Drug Administration, Washington 25, D. C., *Chairman;* P. A. CLIFFORD (1952), and A. H. ROBERTSON (1954).

PROCESSED VEGETABLE PRODUCTS:

*Referee:* L. M. Beacham, Food and Drug Administration, Washington 25, D. C.

QUALITY FACTORS:

R. D. Lovejoy, Food and Drug Administration, Washington 25, D. C.

MOISTURE IN DRIED VEGETABLES:

B. Makover, Western Regional Research Laboratory, Albany 6, Calif.

CATALASE IN FROZEN VEGETABLES::

B. M. Gutterman, Food and Drug Administration, Washington 25, D. C.

PEROXIDASE IN FROZEN VEGETABLES:

M. A. Joslyn, College of Agr., Univ. of Calif., Berkeley 4, Calif.

FILL OF CONTAINER METHODS (FOODS, DRUGS, AND COSMETICS):

*Referee:* Sumner C. Rowe, Food and Drug Administration, Washington 25, D. C.

COFFEE AND TEA:

*Referee:* S. T. Colamaria, Food and Drug Administration, Boston, Mass.

CHLOROGENIC ACID IN COFFEE:

E. H. Grant, Food and Drug Administration, Boston, Mass.

DAIRY PRODUCTS:

*Referee:* W. Horwitz, Food and Drug Administration, Minneapolis 1, Minn.

PHOSPHATASE TEST IN DAIRY PRODUCTS:

W. Horwitz

ASH IN MILK AND EVAPORATED MILK:

Guy G. Frary, State Chemical Laboratory, Vermillion, S. Dak.

SAMPLING, FAT, AND MOISTURE IN HARD CHEESES:

W. Horwitz

ACIDITY OF MILK:

Guy G. Frary

PREPARATION OF BUTTER SAMPLES:

A. L. Weber, Food and Drug Administration, New York 14, N. Y.

TESTS FOR RECONSTITUTED MILK:

---

SERUM TESTS:

Henry J. Hoffman, Minnesota Dept. of Agriculture, St. Paul, Minn.

FAT IN DAIRY PRODUCTS:

Ernest O. Herreid, Illinois Agricultural Expt. Sta., Urbana, Ill.

SAMPLING, AND PREPARATION OF SAMPLE, OF SOFT CHEESES:

Sam Perlmutter, Food and Drug Administration, Minneapolis, Minn.

FROZEN DESSERTS:

H. M. Boggs, Food and Drug Administration, Philadelphia, Pa.

LACTIC ACID:

L. C. Mitchell, Food and Drug Administration, Minneapolis, Minn.

EGGS AND EGG PRODUCTS:

*Referee:* F. J. McNall, Food and Drug Administration, Cincinnati 2, Ohio
ADDED GLYCEROL:
George E. Keppel, Food and Drug Administration, Minneapolis 1, Minn.
AMMONIA NITROGEN:
E. B. Boyce, State Dept. Public Health, Boston 33, Mass.

DECOMPOSITION AND FILTH IN FOODS (CHEMICAL INDICES):

*Referee:* W. I. Patterson, Food and Drug Administration, Washington 25, D. C.
FISH PRODUCTS:
Fred Hillig, Food and Drug Administration, Washington 25, D. C.
SHELLFISH:
R. E. Duggan, Food and Drug Administration, New Orleans 16, La.
ANIMAL FECAL MATTER (CHEMICAL INDICES):
R. E. Duggan
PINEAPPLE (DECOMPOSITION, CARBOHYDRATE):
J. F. Weeks, Jr., Food and Drug Administration, New Orleans, La.
PINEAPPLE BLACKHEART:
W. O. Winkler, Food and Drug Administration, Washington 25, D. C.
STRAWBERRIES (DECOMPOSITION):
H. P. Bennett, Food and Drug Administration, New Orleans, La.
TOMATOES (SUCCINIC ACID):
H. VanDame, Food and Drug Administration, Cincinnati 2, Ohio
URIC ACID IN CEREAL PRODUCTS:
Helen Barry, Food and Drug Administration, New Orleans, La.
URIC ACID IN FRUIT PRODUCTS:
Doris Tilden, Food and Drug Administration, San Francisco, Calif.
URIC ACID IN NUTS:
H. M. Bollinger, Food and Drug Administration, Los Angeles, Calif.
FRUITS (GALACTURONIC ACID):
P. A. Mills, Food and Drug Administration, San Francisco, Calif.
FISH (INDOLE):
R. W. Williams, Food and Drug Administration, San Francisco, Calif.

GELATINE, DESSERT PREPARATIONS, AND MIXES:

*Referee:* Sumner C. Rowe, Food and Drug Administration, Washington 25, D. C.
GELATINE AND GELATINE DESSERTS (COMPOSITION):
Joseph H. Cohen, General Foods Corporation, Woburn, Mass.

FISH AND OTHER MARINE PRODUCTS:

*Referee:* A. M. Allison, Food and Drug Administration, Boston 10, Mass.
TOTAL SOLIDS AND ETHER EXTRACT:
Menno D. Voth, Food and Drug Administration, Boston 10, Mass.

GUMS IN FOODS:

*Referee*: F. Leslie Hart, Food and Drug Administration, Los Angeles 15, Calif.

CHEESE:

M. J. Gnagy, Food and Drug Administration, Los Angeles, Calif.

FROZEN DESSERTS:

F. Leslie Hart

CACAO PRODUCTS:

Flora G. Mendelsohn, Food and Drug Adm., Los Angeles 15, Calif.

CATSUP AND RELATED TOMATO PRODUCTS:

E. W. Coulter, Food and Drug Administration, Chicago 7, Ill.

STARCHY SALAD DRESSINGS:

Sutton Redfern, Fleischman Lab., 810 Grand Concourse, New York 51, N. Y.

MEAT AND MEAT PRODUCTS:

*Referee:* Roger M. Mehurin, Meat Inspection Div., Bur. of Animal Ind., Washington 25, D. C.

SOYBEAN FLOUR IN MEAT PRODUCTS:

O. L. Bennett, Meat Inspection Division, Bureau of Animal Industry, Washington 25, D. C.

DEFATTED MILK SOLIDS IN MEAT PRODUCTS:

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CREATIN IN MEAT PRODUCTS:

J. M. McCoy, Meat Inspection Division, Bureau of Animal Industry, Washington 25, D. C.

HORSEMEAT IN GROUND MEAT:

C. E. Hynds, State Food Laboratory, Albany, N. Y.

STARCH IN MEAT PRODUCTS:

R. A. Chapman, Dept. National Health and Welfare, Ottawa, Can.

METALS, OTHER ELEMENTS, AND RESIDUES IN FOODS:

*Referee:* H. J. Wichmann, Food and Drug Administration, Washington 25, D. C.

COPPER:

R. E. Henry, Continental Can Company, Inc., Chicago 39, Ill.

ZINC:

O. R. Alexander, American Can Company, Maywood, Ill.

MERCURY:

A. K. Klein

DDT AS SPRAY RESIDUE ON FOODS:

R. H. Carter, Bureau of Entomology and Plant Quarantine, Beltsville, Md.

INSECTICIDES IN CANNED FOODS:

W. A. Britton, Beechnut Packing Co., Canajoharie, N. Y.

PARATHION:

P. A. Clifford, Food and Drug Administration, Washington 25, D. C.

SODIUM FLUOROACETATE (1080):

L. L. Ramsey, Food and Drug Administration, Washington 25, D. C.

METHOXYCHLOR:

E. Laug, Food and Drug Administration, Washington 25, D. C.

MICROBIOLOGICAL METHODS:

*Referee:* G. G. Slocum, Food and Drug Administration, Washington 25, D. C.

CANNED MEATS:

M. L. Laing, Armour & Company, Chicago 9, Ill.

CANNED ACID FOODS:

A. P. Dunningan, Food and Drug Administration, Washington 25, D. C.

CANNED VEGETABLES:

C. W. Bohrer, Natl. Canners Assn., Washington, D. C.

EGGS AND EGG PRODUCTS:

M. T. Bartram, Food and Drug Administration, Washington 25, D. C.

NUTS AND NUT PRODUCTS:

William R. North, Food and Drug Administration, Washington 25, D. C.

FROZEN FRUITS AND VEGETABLES:

H. E. Goresline, Production and Marketing Administration, Poultry Division, Washington 25, D. C.

SUGAR:

E. J. Cameron, Natl. Canners Assn., Washington, D. C.

CANNED FISHERY PRODUCTS:

L. R. Shelton, Jr., Food and Drug Administration, Washington 25, D. C.

MICROCHEMICAL METHODS:

*Referee:* C. O. Willits, Eastern Regional Research Lab., Philadelphia, Pa.

ELEMENTAL ANALYSIS:

C. L. Ogg, Eastern Regional Research Laboratory, Philadelphia, Pa.

NUTS AND NUT PRODUCTS:

*Referee:* A. M. Henry, Food and Drug Administration, Atlanta 3, Ga.

OILS, FATS, AND WAXES:

*Referee:* J. Fitelson, Food and Drug Administration, New York 14, N. Y.

SPECTROPHOTOMETRIC METHODS:

Gardner Kirsten, Food and Drug Administration, New York 14, N. Y.

PEANUT OIL:

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ANTIOXIDANTS:

S. Kahan, Food and Drug Administration, New York 14, N. Y.

COAL-TAR COLORS IN OILS:

Marie Offutt, Food and Drug Administration, New York, N. Y.

SPICES AND OTHER CONDIMENTS:

*Referee:* ———————

VINEGAR:

G. A. Michael, Dept. Public Health, State House, Boston 33, Mass.

VOLATILE OIL IN SPICES:

N. A. Carson, Food and Drug Administration, St. Louis, Mo.

SUGAR, ASH, AND PUNGENT PRINCIPLES IN MUSTARDS:

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PREPARATION OF SAMPLE, AND FAT IN MAYONNAISE AND SALAD DRESSING:

Juanita E. Breit, Food and Drug Administration, Cincinnati 2, Ohio

SEEDS AND STEMS IN GROUND CHILI:

A. N. Prater, Gentry, Inc., Los Angeles 54, Calif.

ENZYMES:

*Referee:* J. W. Cook, Food and Drug Administration, San Francisco, Calif.

HYDROCYANIC ACID GLUCOSIDES:

W. O. Winkler, Food and Drug Administration, Washington 25, D. C.

SUBCOMMITTEE D:

KENNETH L. MILSTEAD (1950) (Food and Drug Administration, Cincinnati, Ohio), *Chairman;* J. Walter Sale (1952); and C. S. Ferguson (1954)

ALCOHOLIC BEVERAGES:

*Referee:* J. Walter Sale, Food and Drug Administration, Washington 25, D. C.

MALT BEVERAGES, SIRUPS, EXTRACTS, AND BREWING MATERIALS:

Robert I. Tenney, Wahl-Henius Inst., 64 E. Lake St., Chicago, Ill.

HOPS:

D. E. Bullis, Oregon State College, Corvallis, Oreg.

INORGANIC ELEMENTS IN BEER:

R. E. Henry, Continental Can Company, Inc., Chicago, Ill.

COLOR AND TURBIDITY IN BEER:

B. H. Nissen, Anheuser-Busch, Inc., St. Louis, Mo.

DISTILLED SPIRITS:

G. F. Beyer, Bureau of Internal Revenue, Washington 25, D. C.

CHROMATOGRAPHIC ABSORPTION:

Alex P. Mathers, Bur. Internal Revenue, Washington 25, D. C.

CARAMEL IN ALCOHOLIC BEVERAGES:

Peter Valaer

CORDIALS AND LIQUEURS:

John B. Wilson, Food and Drug Administration, Washington 25, D. C.

METHANOL:

J. F. Guymon, Agr. Expt. Station, College of Agriculture, Davis, Calif.

CACAO PRODUCTS:

*Referee:* W. O. Winkler, Food and Drug Administration, Washington 25, D.C.

LECITHIN:

J. H. Bornmann, Food and Drug Administration, Chicago 7, Ill.

MALT SOLIDS:

E. W. Meyers, Hershey Chocolate Company, Hershey, Pa.

PECTIC ACID:

W. O. Winkler

CACAO INGREDIENTS:

W. O. Winkler

LACTOSE:

Donald G. Mitchell, Walter Baker Co., Dorchester 24, Mass.

FAT:

Carl Stone, Food and Drug Administration, Cincinnati, Ohio

CEREAL FOODS:

*Referee:* V. E. Munsey, Food and Drug Administration, Washington 25, D. C.

STARCH IN RAW AND COOKED CEREALS:

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FAT ACIDITY IN GRAIN, FLOUR, CORN MEAL, AND WHOLE WHEAT FLOUR:

Lawrence Zeleny, Agricultural Research Center, Beltsville, Md.

MILK SOLIDS AND BUTTERFAT IN BREAD:
V. E. Munsey
PROTEOLYTIC ACTIVITY OF FLOUR:
Byron S. Miller, Federal Hard Wheat Quality Lab., Manhattan, Kans.
SOYBEAN FLOUR:
T. C. Law, 16 Baker St., Atlanta, Ga.
BAKED PRODUCTS (MOISTURE, ASH, PROTEIN, FAT, AND CRUDE FIBER):
R. P. Smith, National Biscuit Co., 449 W. 14th St., New York, N. Y.
MOISTURE IN SELF-RISING FLOUR, PANCAKE, WAFFLE, AND DOUGHNUT MIXES:
Frank H. Collins, Food and Drug Administration, Cincinnati, Ohio
BROMATES IN FLOUR:
W. L. Rainey, Commander-Larabee Milling Co., Minneapolis, Minn.
UNSAPONIFIABLE MATTER AND STEROLS IN NOODLES AND BAKERY PRODUCTS:
V. E. Munsey
ALBUMEN IN NOODLES AND MACARONI PRODUCTS:
V. E. Munsey

EXTRANEOUS MATERIALS IN FOODS AND DRUGS:
*Referee:* K. L. Harris, Food and Drug Administration, Washington 25, D. C.
DRUGS, SPICES, AND MISCELLANEOUS MATERIALS:
W. V. Eisenberg, Food and Drug Administration, Washington 25, D. C.
DAIRY AND EGG PRODUCTS:
K. L. Harris
NUT PRODUCTS:
Maryvee G. Yakowitz, Food and Drug Administration, Washington 25, D. C.
BAKED PRODUCTS, CEREALS, AND CONFECTIONERY:
J. F. Nicholson, Food and Drug Administration, Washington 25, D. C.
BEVERAGE MATERIALS:
F. A. Hodges, Food and Drug Administration, Washington 25, D. C.
FRUIT PRODUCTS:
W. G. Helsel, Food and Drug Administration, Washington 25, D. C.
VEGETABLE PRODUCTS:
F. R. Smith, Food and Drug Administration, Washington 25, D. C.
SEDIMENT TESTS (MILK AND CREAM):
C. R. Joiner, Food and Drug Administration, St. Louis 1, Mo.

FLAVORS AND NON-ALCOHOLIC BEVERAGES:
*Referee:* John B. Wilson, Food and Drug Administration, Washington 25, D.C.
BETA-IONONE:
John B. Wilson
LEMON OILS AND EXTRACTS:
John B. Wilson
ORGANIC SOLVENTS IN FLAVORS:
R. D. Stanley, Food and Drug Administration, Chicago 7, Ill.
EMULSION FLAVORS
John B. Wilson
MAPLE FLAVOR CONCENTRATES AND IMITATIONS:
J. H. Bornmann, Food and Drug Administration, Chicago 7, Ill.
DIACETYL:
John B. Wilson
VANILLA EXTRACTS AND IMITATIONS:
L. Ensminger, Food and Drug Administration, Cincinnati, Ohio

FRUITS AND FRUIT PRODUCTS:

*Referee:* R. A. Osborn, Food and Drug Administration, Washington 25, D. C.

FRUIT ACIDS:

L. W. Ferris, Food and Drug Administration, Buffalo, N. Y.

FRUIT AND SUGAR IN FROZEN FRUIT:

R. A. Osborn

VOLATILE ACIDS (CHROMATOGRAPHIC SEPARATION):

C. G. Hatmaker, Food and Drug Administration, Washington 25, D. C.

PRESERVATIVES AND ARTIFICIAL SWEETENERS:

*Referee:* Margarethe Oakley, State Department of Health, Baltimore 18, Md.

FORMIC ACID:

M. L. Dow, Food and Drug Administration, St. Louis, Mo.

QUARTERNARY AMMONIUM COMPOUNDS:

John B. Wilson, Food and Drug Administration, Washington 25, D. C.

MONOCHLORACETIC ACID:

John B. Wilson

FORMALDEHYDE:

Howard Bennett, Food and Drug Administration, New Orleans, La.

MOLD-INHIBITORS, PROPIONATES:

L. H. McRoberts, Food and Drug Administration, San Francisco 2, Calif.

THIOUREA:

W. O. Winkler, Food and Drug Administration, Washington 25, D. C.

ARTIFICIAL SWEETENERS:

William S. Cox, Food and Drug Administration, Atlanta, Ga.

SUGARS AND SUGAR PRODUCTS:

*Referee:* C. F. Snyder, National Bureau of Standards, Washington 25, D. C.

DRYING METHODS:

Lester D. Hammond, National Bureau of Standards, Washington 25, D. C

DENSIMETRIC AND REFRACTOMETRIC METHODS:

C. F. Snyder

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COLOR AND TURBIDITY IN SUGAR PRODUCTS:

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## PRESIDENT'S ADDRESS

# THE HISTORY OF FERTILIZER AND FEED CONTROL IN VERMONT

By L. S. WALKER (Agricultural Experiment Station, Burlington. Vt.)

It is a pleasure and an honor to appear before one of the outstanding organizations in this country as well as the world. The Association of Official Agricultural Chemists has a history of which we must all be proud, and no one can question the importance of the work it is doing to improve the methods of analysis of the many products represented. In looking over the many editions of our "Methods of Analysis," I am amazed at the progress and the importance of our work. Every chemical laboratory, to be up to date, should have the latest copy of these methods on their shelves. It is amazing, also, to think of many hours of labor of love spent by officers, committees, referees, associate referees, and collaborators in this valuable contribution to science. I wish to thank each and every one of you, especially our secretary, Mr. Lepper, for the work done this past year.

For nearly 45 years my work has been confined chiefly to commercial fertilizer and feed control. During that time many changes have taken place. The composition of these commodities has changed considerably. By-products have come and gone and new materials have been placed on the market and found available. The chemist has had to be on the lookout for materials of low grade and keep a check on their use. Let me quote from Bulletin 323 (1930), "A Half Century of Fertilizer Control in Vermont," an article by J. L. Hills: "A new industry was born in the middle of the last century. There embarked, therein, some who were well-informed and some who were not; some who were well-intentioned and some who were not. The ignorant and the unscrupulous sold worthless goods, buyers became distrustful, and honest manufacturers disadvantaged. Neither by color, odor, texture, nor by any other ready means could a buyer determine the value of the wares offered him, and there was no handy way whereby the worth-while could be differentiated from the worthless. Field tests were costly and time consuming and the results secured were untrustworthy. It was easy to fool the farmer. Obviously, this situation was such that the establishment of some adequate form of State control and inspection was required. The situation in Vermont in respect to the sale of these commodities 60 years ago, now almost 80, was not a happy one. Thus, for example, we find that Jonathan Lawrence, the father of the Grange in Vermont, in his first report to the State Board of Agriculture (1872) stated that "some brands (of commercial fertilizer) have given

good satisfaction one year and the next they were comparatively worthless, while other brands have never given satisfaction." The Secretary of the Board, in the same volume, pointed out that "the field has been a fruitful one to the knaves and the markets filled with many so-called fertilizers which proved worthless—barely worth the barrels in which the stuff is packed." At the same time, however, he states that most of the brands found on sale contained fair amounts of nitrogen and available phosphoric acid (potash was rarely used in those days). He further pointed out that since "the trade has grown under the patronage of farmers alone —(who) continue to pay out money in sums larger and larger every year— the facts prove that so much as has been skillfully and honestly manufactured must have been very good and very profitable." He then went on to declare that almost incredible ignorance on the part of manufacturers has been noted in some instances but that, on the other hand, "more or less mismanagement in application and use occurred on the part of the farmers—" a stricture which holds even unto this day.

I quote these paragraphs because it is interesting to know what was going on more than a half century ago. Other States, no doubt, have had similar experience in the establishment of their control laws.

In 1863 the late Dr. Samuel W. Johnson published the analysis of samples of commercial fertilizer. Also, the late Dr. Charles A. Goessman, professor of chemistry at the Massachusetts Agricultural College, followed along the same line. These departments were rather crudely organized, but at the same time, a start was made which eventually led to an efficient law to control the sale of fertilizers. Other States from time to time have followed, and now practically all have fertilizer laws on their statute books and can testify to the value of fertilizer control work in this country. The progress made has been an outstanding contribution to agriculture. The Indians put a fish under a hill of corn. Natural manures and night soils were used. Then animal by-products, followed by chemical fertilizer largely with bone and tankage base, and chemical fertilizers carried organic nitrogen of unknown quality. The alkaline and neutral permanganate methods were developed to determine this quality. Now organics have nearly disappeared from the market and the organic part of complete fertilizer is a mere trace.

Farmers were instructed to maintain the percentage of organics in soils by plowing under green crops. We have the agronomist to thank for this progress.

The manufacture of commercial feeding stuffs is of comparatively recent origin. About 60 years ago farmers began to feed grains in addition to pasture feeding. In 1888 it was the habit of the farmer to go to the feed bin and take out a measure of a mixture consisting of equal parts, by weight, of corn meal and wheat bran. This mixture was considered a standard ration with which to compare others, as it represented then the

common feed. In 1899 a feed called "Quaker Dairy Feed" was manufactured by Quaker Oats Co. It contained about 13 per cent of protein and consisted of cottonseed, linseed, gluten meal or feed, and probably some oats by-products. In 1900 F. B. Howe, a Burlington, Vermont, man, made a dairy feed containing 26 per cent of protein. About the same time Blatchford Calf Meal appeared, and this is still on sale. In 1902 feeds mixed with molasses appeared. It was called "Nutrene" and was a product of a New Orleans house, guaranteed to contain 17 to 18 per cent of protein and composed of molasses and sugar compounds with wheat, corn, and oat products, and other highly nitrogenous materials. In 1905 "Union Grains"—said by some to be the Adam of the trade of high grade proprietary feeds—was offered, then Ralston Purina feeds were sold in Vermont, apparently for the first time. The commercial feed business grew and it was estimated that Vermont alone used over 800,000 tons of commercial feeding stuffs. All this time the farmer was supposed to be protected. Farmers' organizations were formed for educational purposes. Then in 1898 the State legislature had passed a law controlling the sale of commercial feeding stuffs. It was much like the Maine State law, which was the first in the country. This had about as many teeth as the proverbial "biddie" on the farm. It was difficult to enforce and the revenue came from a tonnage tax. The manufacturers didn't like it and would not cooperate. Thus, there was much unlicensed goods on the market. In 1902 the tonnage tax was repealed and a $500 enforcement appropriation went into effect. Amendments were made, but the $500 was far too small to administer a $10,000,000 business, to which the feed trade had grown.

In 1923, with the help of the representative from the American Feed Manufacturers' Association, the present law was enacted. This law, as you know, provided a brand tax which enabled the establishment of a laboratory, and sufficient help to enforce it in an adequate manner.

How different is the picture we look upon now, The manufacturers have put many hours of thought in the manufacture of their goods. They run experimental farms—many of which rival the research work done by the various Agricultural Experiment Stations of the country; and they employ expert research men on their own farms. They are alert as to what is being done by the various State experiment stations and do not hesitate to send their experts to study what is being accomplished, and are always ready to improve their products when deemed advisable. Some manufacturers have appropriated funds to experiment stations for certain research work. This spirit of cooperation is highly commenable.

Before I close, please accept my appreciation for the honor of being your presiding officer during this past year. In truth, I have had little to do. Our efficient secretary is really the administrator of our Association, and he should receive the reward for a job well done.

I close with a quotation from a Vermont bulletin, No. 324, published in

1930, entitled "A Third of a Century of Feeding Stuff Inspection." One of the largest manufacturers in the country recently placed the following advertisement in a leading agricultural paper. It speaks well for the trade as a whole.

"In this hurrying world of ours it is easy to more or less take for granted great public services that vitally affect the well being of great numbers of people. Usually such services are esteemed by the individual—seldom is appreciation made manifest. We believe we speak for the farmer and the public generally when we say, in complete sincerity: 'More Power to the Agricultural Colleges, Experiment Stations, and Extension Workers.'"

"No rehearsal is necessary of the countless benefits which have resulted from the organized efforts and scientific research of the men and institutions who work ceaselessly for a more prosperous agriculture. Today's progress in the farm field is linked too closely with the outstanding achievements of colleges and experiment stations to call for long paragraphs of specific examples."

"For our part we gratefully acknowledge the debt which we believe every progressive feed manufacturer—and every owner of farm animals—owes to the pioneers in research, and the practical feeding specialists who have supplanted ideas with facts and vague theories with definite practices of known value. May the good work go on, and may we all acknowledge freely the ever broadening benefits of this great public service."

### ORDER OF PUBLICATION

The reports of the committees presented on the last day of the annual meeting are given at the beginning of the proceedings, not in their chronological order. This arrangement will assist the referees, associate referees, and collaborators in planning and developing their year's work. The remainder of the proceedings will follow in their usual order.

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## Third Day

## WEDNESDAY—AFTERNOON SESSION

### REPORT OF THE EDITORIAL BOARD

By H. A. LEPPER, *Chairman*

The forthcoming publication of the 7th edition of *Methods of Analysis* will gradually bring to a close the sale and distribution of the most successful edition the Association has published. The sixth edition was the largest in size and sales of any of those previously issued. It contained 932 pages and up to date 12,470 copies have been sold. This is mute testimony to the position our Association holds as a leader in the development and establishment of reliable, accurate, and precise analytical methods. The Association is fortunate in having the acceptance by a majority of the members of the Committee on Revision of Methods for the sixth edition, for service on the seventh. Dr. Fisher, who is again chairman of that committee, will present its report. A vacancy occurs on the Editorial Committee of the *Journal* this year. The Executive Committee has approved the reappointment of W. B. White as Editor of the *Journal*, and the appointment of Dr. A. H. Robertson as a member of the editorial committee, for the customary term of six years. The financial status of the *Journal* was given in the Secretary's report. Dr. White will present the Editor's report.

It is recommended that the Committee on Revision of Methods be authorized to make such editorial and format changes in the manuscript for printing as are necessary in the preparation of the book as a complete, integrated unit.

Approved.

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# REPORT OF THE EDITORIAL COMMITTEE OF THE *JOURNAL*

By W. B. WHITE, *Editor and Chairman*
(Food and Drug Administration, Federal Security Agency, Washington 25, D. C.)

It is gratifying to announce that the printing delays that vexed us last year are no longer a problem. Our first three issues were sent out on time, and there is nothing to indicate that the November issue will be delayed.

Volume 31 carried 811 pages (last year's report, through a clerical error, reported 800 pages for the first three issues, instead of 700). Volume 32 will carry about 815 pages.

Contributed papers were of exceptional value and scope. There will be 37 this year, and in addition 2 notes and 8 book reviews.

Subscriptions during the year were 1862 against 1800 last year: an increase but by no means enough to take us out of the red. We wish again to urge all members to point out to educational institutions, and to others who should know about it, the value of the *Journal* as a source of up-to-date, rigidly tested analytical methods for a wide variety of materials. The educational value of the *Journal* is by no means confined to students who plan to go into regulatory work; all those who work, or plan to work, with any of the wide variety of materials covered would find the *Journal* of inestimable value.

While there has been some publicity of the *Journal* at a few meetings where there was promise that it would bear fruit, we feel that there is opportunity for much more in this direction. The publicity committee reports that suggestions for suitable advertising media and lists of prospective subscribers have not yet been forthcoming. We again urge that every member give some oral constructive thought to our problem and give the Committee his suggestions. Send them to W. A. Queen, H. A. Lepper, or W. B. White, all of the U. S. Food and Drug Administration, Washington 25, D. C.

We wish again to thank our reviewers for their invaluable service, and our contributors for their unfailing courtesy in considering editorial suggestions.

Approved.

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# REPORT OF COMMITTEE ON REVISION OF METHODS

H. J. FISHER, *Chairman*

The Committee submits the following report on the progress of work on the 1950 edition of our *Methods of Analysis.*

The Committee has met twice, once on March 2 and once during this

meeting. At the March meeting general questions in regard to the form and content of the new edition were discussed and decided. Some criticism had been received that the system of numbering methods that was used for the first time in the 6th edition was confusing, because it was not a true decimal system, i.e., Method **12.11** did not follow Method **12.1.** The Committee considered two possibilities: (1), adding extra zeros so that the above cited method numbers would be **12.011** and **12.001**, respectively, which would make them true decimals; and (2), retaining the present system of numbering but using a colon instead of a decimal point to avoid confusion with true decimal numbering. It was the final consensus of the Committee that few chemists were actually misled by the present system, which had advantages sufficient to make it undesirable to change it.

Because the increasing number of methods was threatening to make the size of the new edition unmanageable, it was decided that not only should most of the "a"s and "the"s be omitted as they were in the 6th edition, but that a number of other common words should be abbreviated, using the abbreviations adopted by "Chemical Abstracts."

The Committee discussed the advisability of reproducing in the book, as a convenience to readers, certain methods that were parts of the Federal Definitions and Standards of Food but that had never been studied by the Association. It was decided that, since those chemists who were concerned with the standards must necessarily have copies of the standards themselves, no good purpose would be served by adding these methods to the book.

Because the Executive Committee had voted to delete the chapters on Naval Stores, and Leathers and Tanning Materials, it became necessary to renumber most of the chapters. This brought up the question of whether the sequence of chapters should be completely revised. General Committee opinion did not favor rearrangement of chapters on a strictly alphabetical basis; it was finally decided that the chapters should be divided into 6 groups as follows, with chapters in alphabetical sequence within each group, except that the beverage chapters would be grouped together:

| *Group* | *Material covered* |
|---|---|
| 1 | Soils and materials related thereto |
| 2 | Miscellaneous materials not foods or drugs |
| 3 | Foods |
| 4 | Drugs and cosmetics |
| 5 | General methods |
| 6 | Reference tables |

The adoption by the Association last year of the recommendations of the Committee on Classification of Methods meant that there would be no more tentative methods but that all methods that were named as "methods" would have to be classified as either "first action" or "official."

It also meant that in the future no method would be adopted as even "first action" until it had been studied collaboratively. A type of method to be known as a "procedure" was recognized, for which collaborative study was not to be required, but the only methods that were supposed to fall in this class were the few that were not readily susceptible to collaborative study. This change in the classification system required careful individual consideration of what to do with a large number of tentative methods now in the 6th edition. In sending the manuscript of the chapters to the referees the chairman of the Committee on Revision of Methods attempted to call the attention of each referee to each tentative method in his chapter, in order that no referee might overlook any method concerning which he would have to make a recommendation to the Association for a change in status. It will probably be necessary for the Association, at this meeting, to approve the adoption as "first action" under suspension of the rules of a number of methods that have not been studied collaboratively and that cannot be studied collaboratively in time for their inclusion in the 7th edition. There are some present tentative methods of long standing that appear to be too valuable to omit from the book because of a technicality.

In the 7th edition the Enzyme chapter will be expanded in the same manner as the Vitamin chapter was made a reality in the 6th edition, namely, by the transfer to it of enzyme methods now in other chapters.

The Committee and the Association were fortunate to be able to obtain the services of Mrs. Otis. Her experience in the compilation of the last four editions has made her preëminently qualified to assist the Committee in the task of preparing the 7th edition, a task that has been particularly difficult because of the many changes in the status of methods due to the new classification system and in the cross references due to the changing of the chapter numbers.

The present status of the revision is as follows: All methods adopted since the publication of the 6th edition have been inserted in the chapters, the enlarged chapters have been read and corrected independently by Mrs. Otis and the chairman of the Revision Committee, and the corrected manuscript of each chapter has been forwarded to the appropriate referee. All except five of the chapters have been returned to the chairman by the referees and their suggested corrections entered in the manuscript. These chapters will now only require the insertion of changes made by this meeting before they can be sent to the printer.

The Committee cannot predict with certainty, publication difficulties being what they are, just when the new edition will be in your hands. If everything goes well the book may be off the press before the 1950 meeting of the Association; at the very worst it should appear very early in 1951.

Finally, the Committee wishes to express its indebtedness to the referees for their careful work in reviewing the chapters, and the other members

of the Committee also wish to express their deep appreciation of the invaluable aid of Mrs. Otis.

Approved.

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## REPORT OF COMMITTEE TO CONFER WITH AMERICAN PUBLIC HEALTH ASSOCIATION ON STANDARD METHODS FOR THE EXAMINATION OF DAIRY PRODUCTS

In behalf of the Committee on the Examination of Standard Methods for the Examination of Dairy Products of the American Public Health Association, I am pleased to commend your Referee on Dairy Products for the practical considerations he has given to the several methods for the determination of residual phosphatase in heat-treated dairy products. A report on his collaborative work on phosphatase methods will appear at other places in this volume. The announcement last fall of a new modification, known as the Cornell Method for Phosphatase, makes it desirable to include it among phosphatase methods to be studied collaboratively in the near future.

Attention has been directed to bacteriological transfer pipettes, probably of wartime origin, which do not conform to APHA specifications. Measurements disclose that some pipettes by one jobber varied appreciably from the bore specifications at the dispensing end. Manufacturers in general state that they have no difficulty in conforming with specifications. In order that responsible officials may be guided uniformly when determining conformance to specifications, a set of simple directions is being developed.

It is gratifying to know that collaborative work has been completed to the point where methods for sediment in milk have been adopted, first action. These procedures are essentially identical with those recorded in the Ninth Edition of Standard Methods for the Examination of Dairy Products. An opinion exists that the standard sediment material, used for preparing sediment discs, may have been of such a coarse mesh that it serves best for comparison with sediment in producers' milk as delivered at receiving plants, and that possibly a different standard sediment mixture, prepared with finer mesh material, should be recognized for comparison with sediment tests on retail samples of milk. This opinion is based largely on an attempt to extend the application, by the Production and Marketing Administration of the Department of Agriculture, of the present sediment test mixture to standards for retail milk supplies, and on the declaration by one State health department that they had found this sediment mixture unsatisfactory for their work on retail milks.

A problem which needs careful consideration results from the use, by

veterinarians and by many lay milk-producers, of certain antibiotics for controlling and/or reducing mastitis infections, with little or no thought after treatment as to how soon the milk from the cow so treated will be included with the herd milk. When such milk is included, some producers have shown its presence to seriously retard starter growth during cheese making. Others have shown its effect on retarding the usual rate of decolorization when certain dye reduction tests are applied to milk samples. Data are not available to show conclusively that the presence of traces of antibiotics will retard colony growth on agar plates. Of related interest is the comment by an expert on cucumber pickle processing, who observed that when spore forming bacteria were prevalent in the brines, the normal growth of the lactic acid bacteria was often retarded.

The aim should be not to restrict the use of the magic drugs, but to devise some means which may be applied, if the need arises, to make their fraudulent use readily detectable, either if deliberately added to milk at the farm or in the milk plant, or if present as a result of therapeutic use. Unless the use of some readily detectable, harmless, and unmistakable marker, to be added by the manufacturer, is authorized, the need is first for a qualitative, and then for a quantitative, objective test for added antibiotic substances. Because substances with distinct antiseptic tendencies other than true antibiotics may be present, or may be added to defeat the purpose of certain recognized microbiological methods, it is obvious that objective tests will be far from simple, and that they may not be successfully applied routinely to milk supplies.

A. H. ROBERTSON, *Chairman*
WM. HORWITZ
J. O. CLARKE

Approved.

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## REPORT OF THE COMMITTEE ON RECOMMENDATIONS OF REFEREES

WM. F. REINDOLLAR, *Chairman*

The plan for expediting the handling and distribution of referee reports prior to the annual meeting, outlined in the 1947 report and put into effect in 1948, was continued during the past year and has proved fairly satisfactory. Not only has the burden on referees of mailing copies to a half dozen individuals been eliminated but, in addition, the several subcommittees have received some of their material sufficiently in advance of the meeting to give it adequate consideration. Approximately one hundred referees and associate referees submitted reports prior to the first of October.

Inasmuch as the publication of the seventh edition of the Official Methods of Analysis is planned for 1950, the important task of reviewing the particular chapter or chapters dealing with his field was assigned to each general referee. This time it became necessary in addition to the routine work of checking references, incorporating new methods and changes made since the last revision, etc., to reclassify many of the procedures as required in the Report of the Committee on Classification of Methods which was adopted at the 1948 Conference. To this end reprints of the report, together with a letter explaining its provisions, were sent to all referees. The cooperation of referees and collaborators has been generally satisfactory and to them the chairman extends his sincere appreciation. It is largely through their labors that the new Book of Methods has been made possible.

The reclassification of the methods, with the deletion of the designation "tentative," will in some instances require the retention or revision of some methods through special action. The vote of the Association will be called for in these instances.

Approved.

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## REPORT OF SUBCOMMITTEE A ON RECOMMENDATIONS OF REFEREES*

By H. A. HALVORSON, Department of Agriculture, Dairy and Food, St. Paul, Minnesota, *Chairman;* E. L. GRIFFIN, and J. B. SMITH

### FEEDING STUFFS

It is recommended—

(1) That the method for galactan, **27.40,** be made first action.

(2) That the acid titration method, **27.49,** and alkaline titration method, **27.50,** for hydrocyanic acid formed by hydrolysis of glucosides in beans, be made first action.

(3) That the Knapheide-Lamb method for iodine in mineral mixed feeds, **27.54–27.56,** and the Elmslie-Caldwell method, **27.57,** be made first action.

(4) That work on the following be continued:

(a) Calcium and iodine in mineral mixed feeds.
(b) Lactose in mixed feeds.
(c) Adulteration of condensed milk products.
(d) Crude fat or ether extract
(e) Fluorine.
(f) Protein evaluation in fish and animal products.
(g) Hydrocyanic acid glucosides.

* These recommendations submitted by Subcommittee A were approved by the Association. Unless otherwise given all references are to *Methods of Analysis,* A.O.A.C., 1945.

(h) Sampling and analysis of condensed buttermilk.
(i) Microscopic examination of feeds.
(j) Tankage (hide, hoof, horn, and hair content).
(k) Fat in fish meal.

(5) That further study be made of the method for crude fibre as suggested by the Associate Referee.

(6) That the method for sulfaquinoxaline in feeds as recommended by the Associate Referee be adopted, first action, and that the work be continued and studies of methods for sulfaguanidine and other drugs be undertaken.

(7) That the method for determination of acid-soluble manganese be revised by changing the wording beginning "Heat nearly to boiling point" in **27.59** to read "Heat nearly to boiling point, and with stirring or swirling add 0.3 g of $KIO_4$ for each 15 mg. of Mn present. Maintain at 90–100°C. for 30–60 minutes or until color development is complete. Cool, make to measured volume of 50 or 100 ml, and mix. Compare with standard $KMnO_4$ in a colorimeter or in a spectrophotometer at 530 m$\mu$. Calculate p.p.m of Mn in the sample."

(8) That the following statement " . . . In the case of wheat grains multiply the results by 5.7" be inserted as a note following **27.10.**

(9) That the methods for calcium and phosphorus in feeds, as recommended by the Associate Referee for minerals in feeds, be adopted, first action; that the method adopted as official, first action, *This Journal*, **32,** 95, be dropped.

(10) That the procedure for sampling, **27.1,** be changed to agree with the new procedure for sampling fertilizers.

### FERTILIZERS

It is recommended—

(1) That the method for acid-forming or nonacid-forming quality, **2.64–2.65,** be adopted, first action, and study be continued.

(2) That the method for water-soluble boron, **2.44,** be deleted, and that the "Identical *p*H" method, as reported by the Associate Referee, be adopted, first action.

(3) That the short volumetric method for copper, **2.60,** be adopted, first action.

(4) That the tentative methods for zinc. **2.61** and **2.62,** be adopted, first action.

(5) That the official gravimetric method for acid-soluble magnesium, **2.51,** be revised as suggested by the Associate Referee.

(6) That the volumetric method for acid-soluble magnesium, **2.52,** be revised as suggested by the Associate Referee.

(7) That the methods for water-soluble magnesium, **2.53** and **2.54,** be

rewritten as suggested by the Associate Referee and the combined methods be adopted, first action.

(8) That the colorimetric method for acid-soluble manganese, **2.55,** be revised as suggested by the Associate Referee and studies on magnesium and manganese be continued.

(9) That the air-flow method be made official for determining free water in fertilizers.

(10) That the vacuum desiccation method, with a drying period of 16–18 hours, be made official for determining free water in fertilizers,

(11) That study of the oven-drying method be continued.

(12) That the procedure for oven drying be studied to establish a specific drying temperature.

(13) That the formaldehyde titration method for nitrogen, as stated in the Associate Referee's report, be adopted as official.

(14) That the comparison of the Devarda procedure with the Kjeldahl procedure for determination of nitrogen in sodium nitrate be discontinued.

(15) That the method for total nitrogen, **2.27,** be changed as suggested by the Associate Referee.

(16) That further study be made of high percentage nitrogen in high nitrate-chloride mixtures.

(17) That the official method for determination of citrate-insoluble phosphoric acid be revised by changing "Acidulated samples" in line 1 of **2.16(a)** to "Acidulated samples and mixed fertilizers" and by omitting the words "other than basic slag" from the first line of **2.16(b).**

(18) That section **2.17** (official) be changed by deleting the words "in acidulated samples, dicalcium phosphate, precipitated bone phosphate, and precipitated bone."

(19) That the methods for citric acid-soluble phosphoric acid in basic slag, **2.18–2.20,** be deleted.

(20) That editorial changes as suggested by the Associate Referee be made in **2.16(a)** and **(b).**

(21) That alpha phosphate and phosphate rock-magnesium silicate glass be evaluated by the neutral ammonium citrate method, preferably with continuous agitation during the citrate digestion, and that collaborative work on determination of available $P_2O_5$ in these materials be discontinued.

(22) That work on methods for phosphoric acid in fertilizers be continued as suggested by the Associate Referee.

(23) That additional collaborative work on determination of potash in fertilizers be carried out as recommended by the Associate Referee.

(24) That in section **2.41(b)** the factor weight "2.425 g" be changed to "2.422 g."

(25) That studies on the effect of fineness of grinding on the insoluble phosphoric content of fertilizers be discontinued.

(26) That in section **2.1** on directions for sampling the words "but preferably 2 pounds" be inserted after "material" in line 3.

(27) That in the procedure for sampling, **2.1,** the directions beginning with "Remove a core" be changed to read: "Remove a core diagonally from end to end thru the bag lying horizontally. Take cores as follows: From a lot of 1 to 10 bags, sample all bags; from a lot of 11 to 20 bags, sample 10 bags; from a lot of 21 to 40 bags, sample 15 bags; from a lot of 41 or more bags, sample 20 bags. Take 1 core from each bag sampled, except that for lots of 1 to 4 bags take enough cores from each bag—following the same path—to total 5 or more cores."

(28) That the directions for sampling fertilizer in bulk lots and in small containers, recommended in *This Journal,* **31,** 71 (1948) be retained.

(29) That the final sentence in section **2.1** be changed as follows: "Preferably send all cores to the laboratory for quartering. Thoroly mix portions taken on clean oilcloth or paper, reduce by quartering to quantity of sample required, and place in air-tight container."

(30) That in section **2.2,** line 2, the sentence "Alternate procedure—grind not less than ½ pound of the reduced sample without previous sieving" be inserted after the second sentence.

(31) That the investigation of sampling and preparation of sample be continued.

(32) That the method for free sulfur, **2.63,** be adopted, first action.

(33) That the methods for acid-soluble calcium, **2.49–2.50,** be adopted, first action.

(34) That studies be continued on inert materials.

### ECONOMIC POISONS

It is recommended—

(1) That the alcoholic caustic method for 2,2-bis(*p*-chlorophenyl)-1,1,1-trichloroethane (DDT), **6.151–6.152,** be deleted.

(2) That Method III for determining total arsenic, **6.9–6.10,** be adopted, first action.

(3) That the distillation method for determining total fluorine, **6.22–6.23,** be adopted, first action.

(4) That the method for determining fluorine present as sodium fluosilicate, **6.24–6.25,** be adopted, first action.

(5) That the method for determining total arsenic oxide, **6.38–6.39,** be adopted, first action.

(6) That the official methods for the analysis of magnesium arsenate, **6.50, 6.51, 6.52,** and **6.53,** be deleted.

(7) That the method for determining pyrethrin II in pyrethrum powder, **6.114,** be adopted, first action.

(8) That the method for determining pyrethrin I in pyrethrum extracts in mineral oil, **6.115–6.116,** be adopted, first action.

(9) That the iodine titration methods for the analysis of lime-sulfur solutions, **6.123–6.124, 6.127, 6.129,** and **6.132,** be deleted.

(10) That the method for determining sulfide sulfur in lime-sulfur solutions, **6.130,** be adopted, first action.

(11) That Methods I through VI for determining DDT, *This Journal,* **30,** 319 (1947), and amended, *ibid.,* **31,** 368 (1948), be adopted as official.

(12) That the method for determining DDT in emulsions as published, *ibid.,* **31,** 371 (1948), be adopted as official.

(13) That Methods No. 20 and No. 21 for determining 2,4-D in herbicides, as given in the report of the Associate Referee, be adopted, first action.

(14) That Methods No. 23A and No. 23B for determining 2,4-D in herbicides be revised as suggested by the Associate Referee and subjected to further study.

(15) That the study of methods for determining parathion be continued.

(16) That the study of methods for determining oil in oil emulsions be continued.

(17) That the study of methods for the analysis of dimethyl dithio carbamates be continued.

(18) That the study of methods for determining organic thiocyanates in economic poisons be continued.

(19) That the method for determining alphanaphthyl thiourea based on a determination of its nitrogen content, as described in the report of the Associate Referee on rodenticides, be adopted, first action, and that the method be given further study.

(20) That further work be done on methods for the detection and determination of sodium fluoroacetate (1080).

(21) The the method for determining tetraethyl pyrophosphate, as described in the report of the Associate Referee, be adopted, first action.

(22) That the modified partition chromatographic method [(Harris, T. H.) "The Determination of Gamma Benzene Hexachloride in Insecticide Products." *This Journal,* **32,** 684 (1949); and the infrared spectrometer method. (Tufts, L. E. and Kimball, R. H.) "The analysis of Hexachlorocyclohexane by Infrared Spectroscopy," Hooker Electrochemical Company Report No. 4706. May 15, 1949)] for the determination of the gamma isomer in technical benzene hexachloride be adopted as alternate methods, first action.

(23) That the modified partition chromatographic method for the determination of the gamma isomer of benzene hexachloride in wettable powder and insecticidal dust formulations be adopted, first action.

(24) That the investigation of methods for the determination of the gamma isomer of benzene hexachloride in emulsion concentrates, solutions, and formulations containing other organic insecticides be continued.

DISINFECTANTS

It is recommended—

(1) That the "Phenol Coefficient—official test," **6.153–6.157,** be revised as follows (first action):

(a) Change the names and abbreviations for the test organisms *Eberthella typhosa* and *Staphylococcus aureus* to *Salmonella typhosa* and *Micrococcus pyogenes var. Aureus,* to conform with the nomenclature used for these bacteria in the sixth edition (1948) of Bergey's Manual.

(b) Provide a more complete identification of the test organisms according to the strain numbers recorded in the American Type Culture Collection.

(c) Make agitation of medicant tubes and subculture tubes mandatory during the test, as suggested by the Referee.

(d) Stipulate a sterilization time for media at 15 lbs. steam pressure for 20 min. instead of the present 40 min. period.

(e) List nutrient broth, fluid thioglycollate medium U.S.P. XIII, and letheen broth as alternate subculture media for use dependent upon whichever one gives the lowest result, as suggested by the Referee.

(f) Allow for use of platinum alloy transfer loops in addition to platinum loops for making culture transfers.

(2) That an Associate Referee be appointed to initiate studies directed toward the development of a more precise chemically-defined medium for maintaining and propagating test cultures used in the phenol coefficient method.

(3) That the method for "Fungicidal Test," **6.158–6.162,** be made first action.

PLANTS

It is recommended—

(1) That the Associate Referees continue the studies of their assignments.

(2) That the methods for carotene, **12.75** and **12.76,** be adopted, first action.

(3) That in section **12.77** "Supplementary Information," the paragraph numbered "4" be changed as suggested by the Referee.

(4) That study of methods for carotene be continued.

(5) That the method for zinc, **12.24–12.30,** be adopted, first action.

(6) That studies on methods for zinc in plants be continued.

(7) That studies of the methods for copper and cobalt be continued as suggested by the Referee.

(8) That the magnesium uranyl acetate method for sodium, **12.20–12.21,** be adopted, first action.

(9) That the study of methods for sodium in plant material be continued.

(10) That studies be continued on methods for sugars and starch as suggested by the Referee.

(11) That the following methods be adopted, first action:

**12.3** Moisture.
**12.4** Ash.
**12.11** Micro method for aluminum only.
**12.18** Potassium, perchloric acid method.
**12.22–12.23** Copper.
**12.31–12.32** Arsenic.
**12.48** Selenium.
**12.50** Reducing sugars—Munson and Walker general method.
**12.51–12.52** Reducing sugars—Quisumbing and Thomas method.
**12.53** Sucrose.
**12.54** Ether extract.
**12.55** Crude fiber
**12.56** Total nitrogen.
**12.57** Organic and ammoniacal nitrogen.
**12.64–12.67** Lignin.
**12.78** Boron.

(12) That the following methods be deleted:

**12.47** Iodine.
**12.58–12.59** Ammonia in tobacco.
**12.60–12.61** Free nicotine in tobacco.
**12.62–12.63** Nitrate nitrogen in tobacco.

(13) That the 72 per cent sulfuric acid method for lignin, *This Journal*, **32**, 287–291 (1949), be adopted, first action.

### SPECTROGRAPHIC METHODS

It is recommended that study of spectrographic methods be continued as outlined by the Referee.

### SOILS AND LIMING MATERIALS

It is recommended—

(1) That the methods for sampling, **1.1–1.2,** and **3.1–3.2,** be made procedures, and that the following methods be made first action.

Methods for soils, **1.3–1.53.**
Neutralization value, **3.3–3.4.**
Carbon dioxide, **3.7.**
Total calcium oxide, **3.8.**
Total magnesium oxide, **3.9.**
Mechanical analysis of ground limestone, **3.10.**
Neutralization value of calcium silicate slags, **3.11.**

(2) That studies on the "combination dithizone-spectrographic method" and on the polarographic procedure for the determination of zinc in soils be continued.

(3) That the study of the determination of copper in soils be continued.

(4) That the utilization of carmin as an indicator in the determination of boron content of soils be studied further and that p-nitrobenzenazo-1, 8-dihydroxy naphthalene-3, 6-disulphonic acid, or "chromotrope-B" be

studied as a suitable reagent for the determination of boron in soils.

(5) That further studies of *p*H in soils of arid and semi-arid regions be based upon soil systems of moisture content representative of an air-dry condition.

(6) That a study be made as to the adequacy of calcium hydroxide as a fixative for fluorine in soil charges of 1 to 1 proportion, with calcination of 500°C. in 5–60 minute periods.

(7) That the direct distillation of unignited soil with sulfuric acid at 165°C., followed by distillation of an aliquot with perchloric acid at 135°C., be studied collaboratively.

(8) That the "2-point" barium hydroxide-barium acetate titration procedure for the determination of exchangeable hydrogen in soils be studied further in relation to calcite equilibria in a variety of soils.

(9) That the survey and comparison of methods for the determination of phosphorus (a) that fraction in "available" state and (b) the proportions of organic-inorganic forms therein, be continued (*This Journal*, **30**, 43, 1947).

(10) That the survey and comparisons of methods for the determination of exchangeable potassium in soils be continued (*This Journal*, **30**, 44, 1947).

(11) That the Associate Refereeship on exchangeable calcium and magnesium be maintained.

### STANDARD SOLUTIONS

It is recommended—

(1) That the procedure for preparing standard solutions of potassium dichromate by using the theoretical quantity of the National Bureau of Standards standard sample, or the equivalent amount of suitable commercial lots that have been compared for oxidimetric strength with the standard sample, be adopted, first action.

(2) That the method for the standardization of sulfuric acid by standard borax, **43.14–43.15**, be made official.

(3) That the procedure for standardization of bromide-bromate solutions against arsenous oxide, *This Journal*, **31**, 119 (1948), be adopted as official.

(4) That the methods for preparation of buffer solutions for calibration of *p*H equipment, as suggested by the Referee, be adopted, first action.

(5) That the descriptions of the method for hydrochloric acid, **43.10(d)**, and for sodium thiosulfate solution, **43.29**, be revised as suggested by the Referee.

### VITAMINS

It is recommended—

(1) That the spectrophotometric method for Vitamin A in fish liver oils as adopted last year, be modified to include the use of the U.S.P. Reference Standard for Vitamin A and the Morton-Stubbs correction, first action.

(2) That work on Vitamin A in mixed feeds be continued.

(3) That the method for carotene in feedstuffs, after modification as suggested by the Referee, be adopted as official.

(4) That further study of the method be continued as recommended by the Referee.

(5) That the method for Vitamin D in poultry feed supplements, **36.61–36.63,** be adopted, first action.

(6) That when the U.S.P. Vitamin $D_3$ Reference Standard becomes official, the method for Vitamin D in poultry feed supplements be changed as recommended by the Referee.

(7) That the toe-ash and X-ray procedures be studied to determine their suitability as alternate procedures as recommended by the Referee.

(8) That the growth method for the determination of thiamine and the fluorometric method for its determination be adopted as official.

(9) That the fermentation method for thiamine, modified as suggested by the Referee, be adopted as official.

(10) That the microbiological method for riboflavin, modified as suggested by the Referee, be adopted as official.

(11) That the fluorometric method for the assay of riboflavin, with editorial changes as suggested by the Referee, be adopted as official.

(12) That the microbiological method for the assay of nicotinic acid or nicotinamide, with editorial changes as suggested by the Referee, be adopted as official.

(13) That the sublimation method, **36.46,** be deleted and that an Associate Referee to study other methods be appointed.

(14) That the microbiological assay method for folic acid using *S. faecalis* as the test organism, be adopted, first action.

(15) That an Associate Referee on methods for vitamin $B_{12}$ and the animal protein factor be appointed.

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## REPORT OF SUBCOMMITTEE B ON RECOMMENDATIONS OF REFEREES*

By G. Robert Clark (Division of Cosmetics, Food and Drug Administration, Federal Security Agency, Washington, D. C.), *Chairman;* H. J. Fisher, and F. H. Wiley

### NAVAL STORES

It is recommended—

(1) That study of the topic be discontinued.

* These recommendations submitted by Subcommittee B were approved by the Association. Unless otherwise given, all references are to *Methods of Analysis, A.O.A.C.,* 1945.

### RADIOACTIVITY

It is recommended—

(1) That methods **38.6–38.12** be modified to eliminate the use of the electroscope constant.

(2) That the "standard deviation" be used instead of the "probable error" where specified in method **38.15.**

(3) That the subject be continued.

### VEGETABLE DRUGS AND THEIR DERIVATIVES

It is recommended—

(1) That the following methods be adopted, first action:

**39.20** and **39.21** Phenacetin and salol.
**39.45** Aminopyrine, acetophenetidin, phenobarbital, and caffeine.
**39.73** Arecoline hydrobromide.
**39.78** Cocaine (Method II).
**39.99** Physostigmine salicylate in tablets.
**39.121** and **39.122** Alkaloids in ipecac.
**39.123** and **39.124** Ipomea and jalap.
**39.125** Podophyllum.
**39 126** and **39.127** Belladonna and stramonium ointments.
**39.191** Santonin in santonica.
**39.184** Quinine (Spectrophotometric method).
**39.152–39.154** Guaiacol (from alkoxyl determination).
The proposed method (*This Journal*, **30**, 464, 1947) for quinine ethyl carbonate.

(2) That method **39.72,** a qualitative test for aconitine in aconite root, be adopted as a procedure.

The committee recommends the following in accordance with the Referee's recommendation:

(3) Adoption as official of method for ethyl morphine in sirups, in absence of other alkaloids.

(4) Deletion of the methods **39.107,** quinine and strychnine, and **39.113–39.114,** cascara sagrada, and continuance of the topic to study spectrophotometric procedures.

(5) Continuance of the topics: Theobromine and phenobarbital; Aminopyrine, ephedrine, and phenobarbital; rutin in tablets.

### SYNTHETIC DRUGS

It is recommended in concurrence with the recommendations of the Referee—

(1) *Butacaine:* That the proposed methods be adopted, first action, for determination of the drug in the following dosage forms: crystalline, solutions, and ointments containing 2 per cent or more.

(2) *Carbromal:* That collaborative study of the proposed method be continued to include analysis of a mixture of known composition.

(3) *Synthetic Estrogens:* That study of the topic be continued.

(4) *Methylene Blue:* That study of the topic be continued.

(5) *Phenolphthalein in Chocolate Preparations:* That the official method **39.168–39.170** be amended, first action, to incorporate the modification recommended by the Associate Referee.

(6) *Propadrine Hydrochloride:* That the study of the topic be continued.

(7) The committee recommends that study of the topic *Propyl-thiouracil* be continued in order to develop a more specific and more generally applicable method.

(8) That study of the following topics be continued: Benadryl and Pyribenzamine, Spectrophotometric Methods, Sulfanilamide Derivatives.

### MISCELLANEOUS DRUGS

The committee recommends continuance of the following topics:

Microscopic tests for alkaloids and synthetics.
Mercury compounds.
Organic iodides and separation of halogens.
Alkali metals.
Glycols and related compounds.
Preservative and bacteriostatic agents in ampul solutions.
Estrone and estradiol.
Methyl Alcohol.

### COSMETICS

It is recommended—

(1) That the following methods be adopted, first action:

Deodorants and anti-perspirants, methods **4.1, 4.2, 4.3,** and the proposed method for zinc in the presence of aluminum.

Cosmetic creams, the proposed methods for type of emulsion—water, ash, and chloroform-soluble material.

Face powders, the proposed methods for boric acid and zinc.

(2) That the following topics be continued:

Deodorants and anti-perspirants.
Cosmetic creams.
Cosmetic powders.
Cosmetic skin lotions.
Mascaras, eyebrow pencils, and eye shadows.
Sun tan preparations.
Hair dyes and rinses.

(3) That the topic, moisture in cosmetics, be discontinued.

### COAL-TAR COLORS

The committee concurs in the recommendation for deletion of methods **21.17–21.63,** inclusive, and **21.68** and the substitution of the rewritten material submitted by the Referee.

# REPORT OF SUBCOMMITTEE C ON RECOMMENDATIONS OF REFEREES*

By J. O. CLARKE, Food and Drug Administration, Federal Security Agency, Washington, D. C., *Chairman;* PAUL A. CLIFFORD; and A. H. ROBERTSON

### PROCESSED VEGETABLE PRODUCTS

It is recommended—

(1) That work on methods for determining quality factors in canned and frozen fruits and vegetables, on determination of moisture in dried vegetables, and enzymatic activity in frozen vegetables, be continued.

It is also recommended—

(1) That section **35.1** be made a procedure.

(2) That sections **35.3, 35.4, 35.5, 35.9,** and **35.18** be made official.

(3) That sections **35.20, 35.21,** and **35.22** be deleted.

(4) That the method for catalase, *This Journal,* **30,** 76 (1947), be adopted as first action.

### FILL OF CONTAINER METHODS

It is recommended—

(1) That studies of methods for fill of container for foods, drugs, and cosmetics be continued.

### COFFEE AND TEA

It is recommended—

(1) That in section **18.2,** coloring matters in coffee, the designation "tentative" be dropped and the matter referred to the Committee on Revision.

(2) That the Bailey-Andrew method for caffeine in tea be adopted as an official method for caffeine in coffee, and that the following directions be given under the topic "Caffeine" (in coffee): "Proceed as under **18.41** using 10 grams of sample prepared as in **18.4.**"

(3) That methods for chlorogenic acid in coffee be developed.

(4) That more modern methods for the determination of moisture in coffee and tea be developed.

(5) That section **18.24,** "Chicory Infusion" be designated a procedure and placed elsewhere than under the topic "Coating and Glazing Substances."

It is also recommended—

(1) That sections **18.1, 18.3, 18.22, 18.23, 18.25, 18.26, 18.44, 18.45,** and **18.46** be designated as procedures.

(2) That sections **18.6, 18.17, 18.18, 18.20, 18.38, 18.42,** and **18.43** be designated as official.

(3) That sections **18.5, 18.15,** and **18.21** be deleted.

* These recommendations submitted by Subcommittee C were approved by the Association. Unless otherwise given all references are to *Methods of Analysis, A.O.A.C.,* 1945.

### COLORING MATTERS IN FOODS

(1) The Referee, with commendable conservatism, recommends that the method given in his report for the codetermination of Tartrazine (FD&C Yellow No. 5) and Sunset Yellow (FD&C Yellow No. 6) be subjected to continued study. The committee believes that the work already done by the Referee is sufficiently comprehensive to justify adoption of the method as first action.

(2) That investigational work be undertaken to separate and determine quantitatively Sunset Yellow FCF (FD&C Yellow No. 6) in presence of Amaranth (FD&C Red No. 2).

(3) That investigational work be undertaken to separate and determine quantitatively Light Green S.F. Yellowish (FD&C Green No. 2), Fast Green FCF (FD&C Green No. 3), and Brilliant Blue FCF (FD&C Blue No. 1).

(4) That investigational work be undertaken to separate quantitatively Yellow AB (FD&C Yellow No. 3), Yellow OB (FD&C Yellow No. 4), Orange SS (FD&C Orange No. 2), and Oil Red XO (FD&C Red No. 32).

(5) That section **21.3** be dropped and the method suggested to the revision committee by the Referee, for the detection of oil-soluble coal-tar dyes, be adopted as first action.

(6) That the word "procedure" in the titles of **21.2** and **21.4** be deleted.

(7) That section **21.8** be dropped and the method for preparation of solution, as suggested by the Referee, be adopted, first action.

(8) That work on coloring matters in foods be transferred to Subcommittee B, which already handles the bulk of the material of Chapter 21.

It is also recommended—

(1) That section **21.1** be made a procedure.

(2) That sections **21.14, 21.15,** and **21.16,** be designated first action.

(3) That sections **21.2, 21.4, 21.9, 21.10, 21.11,** and **21.12** be made official.

### DAIRY PRODUCTS

It is recommended—

(1) That the Sanders-Sager method for the detection of residual phosphatase, with minor clarifying changes, be adopted as official for milk and cream, cheddar type cheese, and soft uncured cheeses, and as first action for all other dairy products.

(2) That the Long test, **22.43,** be adopted as first action and be made applicable only to fluid milk and cream, together with an alternative photometric procedure suggested by the Referee.

(3) That the Rapid test for phosphatase **22.49–22.57,** be deleted.

(4) That methods for the determination of ash in milk and in evaporated milk be further studied.

(5) That the clarifying changes suggested by the Referee in section **22.25** be made.

(6) That the change in the method for fat in cheese suggested last year

be made official and that methods of sampling of hard cheeses be further studied.

(7) That studies of methods for the determination of the acidity of milk be continued.

(8) That methods **22.108** and **22.109,** preparation of butter samples, and the tentative method, *This Journal,* **31,** 91 (1948) be made procedures and that the Referee and Associate Referee make diligent effort to devise one or more methods, whether or not involving the use of mechanical shakers, stirrers, etc., that will be satisfactory to analysts.

(9) That studies of methods for the detection of reconstituted milk be continued.

(10) That the Associate Referee conduct fundamental studies of the acetic serum method **(22.28)** and the copper serum method **(22.30);** and that the sour serum method **(22.29)** be deleted (final action).

(11) That studies be continued to ascertain whether the present Babcock method for fat in milk should be modified for use in determination of fat in homogenized milks and chocolate drinks, and that the Associate Referee submit collaborative data comparing the modified Babcock method with the standard Babcock method and evaluate the comments of collaborators.

(12) That studies be undertaken on sampling and preparation of samples of soft cheeses.

(13) That studies be continued on methods for frozen desserts.

(14) That the Referee review the methods for moisture and ash in the various dairy products with a view towards their unification.

It is also recommended—

(1) That methods **22.22, 22.23–22.24, 22.97, 22.137, 22.143–22.145** be made official.

(2) That methods **22.4, 22.5–22.7, 22.16, 22.19–22.20, 22.96, 22.98, 22.99, 22.101, 22.104,** and **22.138–22.141** be adopted, first action.

(3) That methods **22.1, 22.2, 22.3, 22.15, 22.36–22.38, 22.59, 22.60, 22.73, 22.85, 22.94, 22.95, 22.106, 22.107, 22.120, 22.123, 22.132, 22.135,** and **22.146** be designated procedures.

(4) That methods **22.121** and **22.122** be deleted.

### EGGS AND EGG PRODUCTS

It is recommended—

(1) That **23.25–23.26,** the glycerol qualitative test, with the slight modification proposed by the Associate Referee, be designated a procedure; and that the present quantitative method, **23.27–23.28,** be dropped and the proposed periodate method be made first action. That further work be done on egg mixtures containing added glycerol with sugars.

(2) That the modified method given by the Associate Referee for the acidity of ether extract in dried eggs be made official.

(3) That the present methods for acidity of ether extract, **23.30, 23.31** and **23.32(b)** be dropped and that **23.32(a),** as modified by the Associate Referee, together with his proposed method for liquid eggs, be made official and that the subject be closed.

(4) That the acid hydrolysis method for fat, **23.8** and **23.9,** be made official.

(5) That the method given, **23.33** and **23.34,** for ammonia nitrogen be dropped and that the method proposed by the Associate Referee be studied collaboratively.

(6) That the method for the determination of volatile fatty acids be modified according to the new chromatographic technic recommended by the Referee on Decomposition in Foods.

It is also recommended—

(1) That section **23.1** be designated as a procedure.

(2) That section **23.6(b)** (dried eggs only) be dropped.

(3) That sections **23.10–23.12** be designated first action.

### EXTRANEOUS MATERIALS IN FOODS AND DRUGS

It is recommended—

(1) That a change be made in the title of Chapter 42 from "Extraneous Materials in Foods and Drugs" to "Extraneous Materials: Isolation."

It is further recommended—

(2) That work be continued on drugs, spices, and miscellaneous materials; cereal products; dairy and egg products; nut products; baked products; beverage materials; fruit products; vegetable products; sediment tests for milk and cream; mincemeat; peas and beans, with collaborative work where possible.

(3) That the method for stone fragments and decomposed peanuts in coarse peanut butter, *This Journal,* **30,** 102 (1947), be adopted as first action.

(4) That methods **42.26, 42.27, 42.28, 42.29, 42.30, 42.41, 42.42, 42.43, 42.54,** and **42.77** be deleted, and that the methods suggested by the Associate Referee be substituted therefor as first action.

(5) That method **42.44,** as modified by the Associate Referee, be adopted as first action.

(6) That methods **42.60, 42.78,** and **42.84** be adopted as first action, with the changes suggested by the Associate Referee.

(7) That the method for sediment in fluid milk, amended by the Referee, be adopted, first action.

(8) That the tentative methods for the following products, which have been adopted since the sixth edition of the *Book of Methods* was published, be changed to procedures:

Popcorn.
Mold in cranberries.

Brewer's grits.
Canned fish.
Chicken giblet paste.
Meat scraps.
Chewing gum.
Shredded coconut.
Jams and jellies.
Frozen and canned blueberries.
Dried mushrooms.
Soybean flour.

It is also recommended—

(1) That sections **42.1** to **42.4**, inclusive, directions for apparatus, reagents, etc., remain as in the present *Book of Methods* without specific method classification.

(2) That methods **42.12, 42.13, 42.15, 42.16, 42.17, 42.18, 42.19, 42.39, 42.40, 42.48, 42.49, 42.52, 42.74,** and **42.75** be made procedures.

(3) That methods **42.5, 42.6, 42.7, 42.10, 42.21(a), 42.22, 42.23, 42.24, 42.25, 42.31, 42.33, 42.34, 42.35, 42.36, 42.38, 42.45, 42.46, 42.50, 42.55, 42.56, 42.61, 42.62, 42.64, 42.66(a), 42.67, 42.68, 42.69, 42.70, 42.71, 42.79, 42.80, 42.81, 42.82, 42.83, 42.85, 42.86, 42.87, 42.88, 42.89, 42.90, 42.91, 42.92, 42.93, 42.94, 42.95, 42.96, 42.97, 42.98, 42.99,** and **42.100** be adopted, first action.

(4) That methods **42.8, 42.9, 42.11, 42.14, 42.21(b), 42.37, 42.47, 42.53, 42.59, 42.63, 42.65, 42.66(b), 42.72, 42.73,** and **42.76** be deleted.

### DECOMPOSITION AND FILTH IN FOODS (CHEMICAL INDICES)

It is recommended—

(1) That the official method for the determination of volatile acids in fish and other marine products, and eggs be modified as suggested by the Referee and adopted, first action.

(2) That the method for water-insoluble acids in fish and other marine products be studied collaboratively.

(3) That the method for succinic acid in fish and other marine products be studied collaboratively.

(4) That the method for indole in shrimp, oysters, clams, and crabmeat be adopted, first action, and further studied.

(5) That the method for water-insoluble acids in butter and cream be adopted as official.

(6) That the method for the determination of volatile acids in butter and cream involving the chromatographic technique be adopted, first action.

(7) That the proposed method for catecholase activity in pineapple be studied further as a means for the identification of that type of decomposition which is characterized by the darkening of the fruit tissue.

(8) That the study of other methods for detection of decomposition in fruits be further pursued.

(9) That the method for water-insoluble acids in eggs and egg products be made first action.

(10) That the method for succinic acid in eggs and egg products be made first action.

### GELATIN, DESSERT PREPARATIONS, AND MIXES

It is recommended—

(1) That the method proposed by the Associate Referee for the determination of jelly strength of gelatin, including the use of the modified one-half inch plummet, be substituted for method **9.6,** and adopted, first action.

(2) That the method proposed by the Associate Referee for the determination of jelly strength of gelatin dessert powders, including the use of the modified one inch plummet, be substituted for method **9.12,** and adopted, first action.

(3) That methods **9.13–9.15,** sucrose and dextrose, and **9.20–9.21,** starch, be made first action, and that work be continued.

It is also recommended—

(1) That sections **9.1, 9.7,** and **9.16** be designated as procedures.

(2) That sections **9.2, 9.3, 9.4, 9.5, 9.8, 9.9, 9.10, 9.11** (as revised), **9.17, 9.18,** and **9.19** be made official.

### FISH AND OTHER MARINE PRODUCTS

It is recommended—

(1) That the method devised by the Associate Referee for determination of crude fat by acid hydrolysis be adopted as official.

(2) That work be continued on methods for the determination of total solids in fish.

(3) That the parenthetical statement limiting method **24.3** to oysters and scallops be changed to read "shucked oysters, scallops, and soft shell clams," and that the method be made first action.

(4) That sections **24.9, 24.10,** and **24.11** be dropped and the method for volatile fatty acids recommended by the Referee on Decomposition in Foods be substituted therefor and made first action.

(5) That the determination of indole in shrimp, oysters, and crabmeat be made first action.

(6) That the method for drained liquor in shucked oysters referred to in the Referee's report be adopted as official.

(7) That sections **24.1** and **24.2** be combined and adopted as a procedure, with the omission of **24.1(c)** and of **24.2(f)** (the skimmer and method for drained liquor are provided in recommendation 6). That **24.1(g)** be expanded to permit use of the Waring blendor. That minor changes be made to include clams where oysters and scallops are now mentioned.

(8) That paragraph **24.7** be deleted.

### GUMS IN FOODS

It is recommended—

(1) That the studies be continued on the detection of gums in cheese and cheese foods, frozen desserts, cacao products, and catsup and related tomato products.

(2) That method **33.57**, gums in mayonnaise and French dressing, be adopted as official.

(3) That method **22.138–22.141** be made first action.

### MEAT AND MEAT PRODUCTS

It is recommended—

(1) That collaborative work be continued on methods for soybean flour in sausage, for creatin and creatinin in meat products, and for horse meat in ground meat.

(2) That an Associate Referee be again appointed to study starch in meat products.

(3) That section **28.3**, added water in sausage, be adopted as a procedure. The committee believes that paragraph **28.3** represents an interpretation of chemical results and questions the propriety of adopting an interpretation as a method. Since further study may indicate otherwise, the committee is recommending that paragraph **28.3** remain in the Book of Methods as a procedure and further studied.*

(4) That the methods for nitrates and nitrites, **28.11–28.13**, and sections **28.14–28.17**, as modified by the Referee, be adopted as first action.

(5) That the method for starch, **28.18**, modified as suggested by the Referee, be adopted as a procedure.

(6) That section **28.33**, creatin, be rewritten in accordance with the suggestions of the Referee and remain as an official method.

(7) That the method for salt, **28.5**, as revised by the Referee, be adopted as first action.

(8) That the qualitative test for skim-milk solids, as given by the Referee, be adopted as a procedure.

(9) That the method for the determination of lactose, as suggested by the Referee, be adopted as first action.

It is also recommended—

(1) That sections **28.1, 28.20,** and **28.42** be designated as procedures.

(2) That sections **28.11, 28.12, 28.13, 28.34, 28.35,** and **28.36** be adopted as first action methods.

(3) That the following paragraphs be deleted: **28.9, 28.10, 28.19, 28.21, 28.22, 28.23, 28.24, 28.28, 28.29, 28.30, 28.31, 28.32, 28.37, 28.38, 28.47, 28.49, 28.50, 28.51, 28.52, 28.53, 28.54, 28.55, 28.58, 28.59,** and **28.60.**

* NOTE.—The above recommendation was not approved by the Association, which, upon motion, voted to authorize the Chairman of the Committee on Revisions to exercise his editorial judgment upon the proper disposition of paragraph 28.3.

METALS, OTHER ELEMENTS, AND RESIDUES IN FOODS

It is recommended—

(1) That the proposed method for the determination of cadmium in foods be designated first action, and that further work be discontinued to await the call of the Referee.

(2) That line 13 in section **29.13** reading "add 1 ml in excess" be changed to read "add 10 ml in excess," and study of methods for the determination of copper in foods be continued.

(3) That the study of methods for the determination of zinc in foods be continued.

(4) That the present mercury method be deleted and replaced by a reference to mercury methods in the literature as supplied by the Referee and that work on mercury be continued.

(5) That the colorimetric Schechter-Haller method for DDT in plant and animal products be adopted as first action and that the total chlorine method for chlorinated hydrocarbons without specific designation be adopted as first action. Further work on these methods is also recommended.

(6) That the effect of the canning process on the decomposition of the newer insecticides be further studied, with respect to the nature of possible decomposition products and their effects on methods of analysis.

(7) That studies on the determination of parathion be continued.

(8) That the method for sodium fluoroacetate (1080) reported at this meeting be adopted as first action and that work be continued.

(9) That studies on the determination of methoxychlor and especially its differentiation from DDT, particularly in dairy products, be begun.

It is also recommended—

(1) That sections **29.6–29.10, 29.11–29.21, 29.64–29.68, 29.69–29.77** be designated first action.

(2) That methods **29.22–29.33** and **29.34–29.52** be made official.

MICROBIOLOGICAL METHODS

It is recommended—

(1) That work be continued on eggs and egg products, sugar, canned vegetables, canned meat, canned fish, acid canned foods, nuts and nut products, and frozen fruits and vegetables.

It is also recommended—

(1) That all official methods retain their first action status and that the tentative methods, **40.22–40.25** and **40.26–40.27** be deleted.

MICROCHEMICAL METHODS

It is recommended—

(1) That the method recommended by the Referee for the microdetermination of nitrogen be adopted as first action, and further studied, and that method **41.3–41.6** be deleted.

(2) That the micro method for the determination of methoxy and ethoxy groups, **41.1–41.2,** be made official.

### NUTS AND NUT PRODUCTS

It is recommended—

(1) That methods for moisture, crude fat, crude protein, crude fiber, ash, reducing sugars, and salt be further studied.

(2) That the methods for preparation and preservation of samples, *This Journal,* **32,** 96 (1949), be adopted as procedures and be further studied, and that all other methods listed in this reference be adopted, first action.

(3) That sorting methods for moisture and fat and methods for added starch in nut butters and pastes be studied.

(4) That method **30.11** be designated as a procedure, and that methods **30.12** and **30.18** be adopted, first action.

### OILS, FATS, AND WAXES

It is recommended—

(1) That the changes in the S.P.A. method for unsaponifiable matter proposed by the Associate Referee be accepted, and that the method be made official and the subject closed.

(2) That the modified Bellier test described by the Associate Referee be adopted as official and work be discontinued.

(3) That work on methods for the quantitative estimation of peanut oil be initiated.

(4) That work on methods for the detection and estimation of antioxidants in oils be continued.

(5) That studies on spectroscopic methods for the analysis of oils be initiated.

(6) That the minor editorial changes proposed by the Referee be accepted.

It is also recommended—

(1) That sections **31.1, 31.44, 31.49, 31.53,** and **31.54** be designated as procedures.

(2) That sections **31.22, 31.23, 31.35, 31.36, 31.52, 31.56,** and **31.57** be designated first action.

(3) That sections **31.6, 31.7,** and **31.58–31.70,** incl., be deleted.

### PRESERVATIVES AND ARTIFICIAL SWEETENERS

It is recommended—

(1) That the method for the determination of quaternary ammonium compounds in eggs, given in the Associate Referee's report, be adopted, first action.

(2) That collaborative study be continued on quantitative methods for the determination of quaternary ammonium compounds in fruit juices,

bottled soda, milk, mayonnaise and salad dressing, sandwich spreads, and pickles and relishes.

(3) That methods for the determination of quaternary ammonium compounds in shrimp be further studied.

(4) That the method for monochloracetic acid referred to under "Preservatives and Artificial Sweeteners," *This Journal*, **32**, 97 (item 3) be adopted as official for carbonated beverages, fruit juices, and for beer and wine. The method should include the modifications given in recommendations 2 and 6 of the Associate Referee's 1949 report.

(5) That the method for the determination of monochloracetic acid in commercial preservatives, *This Journal*, **32**, 97 (item 4) be made official.

(6) That the qualitative tests for monochloracetic acid in carbonated beverages, orange juice, beer and wine, *This Journal*, **32**, 99, be made official.

(7) That the qualitative tests for monochloracetic acid in commercial preservatives, *This Journal*, **32**, 97–99 of the *Journal* be adopted as official.

(8) That further work be done on the determination of monochloracetic acid in fruit juices other than orange juice.

(9) That the method for thiourea in oranges and orange juice, identified as the "rapid oxidation method," *This Journal*, **32**, 100, with the slight modifications suggested by the Associate Referee, be adopted as first action.

(10) That the qualitative test for thiourea in orange juice, *This Journal*, **31**, 104 be designated as a procedure.

(11) That the method for thiourea in frozen peaches, *This Journal*, **31**, 102, be adopted, first action.

(12) That the chromotropic acid test and the Hehner-Fulton test for formaldehyde, as given in the report of the Associate Referee, be adopted, first action.

(13) That the Denigès-Tourron method and the modified LaParola-Mariani method for the detection of dulcin, as described by the Associate Referee, be adopted, first action, and further studied.

(14) That the method for saccharin described by the Associate Referee, *This Journal*, **30**, 494 be adopted, first action.

(15) That work be initiated on methods for the detection and estimation of propoxy, 2-amino, 4-nitrobenzene.

(16) That the method for formic acid be re-studied.

(17) That an associate referee be appointed to develop more sensitive qualitative methods for fluorides.

(18) That the method for volatile fatty acids in baked products, involving a rapid distillation of the volatile acids as directed by the Associate Referee, and chromatographic identification of propionic acid, be adopted, first action and replace tentative method, *This Journal*, **31**, 99 (1948), and study continued.

It is also recommended—

(1) That methods **32.13** and **32.16** be adopted as official.

(2) That sections **32.19–32.26**, incl., **32.34, 32.35, 32.36, 32.37**, and **32.38** be deleted.

SPICES AND OTHER CONDIMENTS

It is recommended—

(1) That studies of methods for the detection of caramel in vinegar be continued.

(2) That studies on the determination of tartrates in vinegar be continued.

(3) That the method for "Permanganate Oxidation Number" as described by the Referee, be adopted as official and that paragraph **33.91–33.92** be dropped.

(4) That methods for the determination of free mineral acids in vinegar be further studied.

(5) That methods for the determination of sorbitol, and the value of these methods for the detection of cider vinegar in wine vinegar, be studied.

(6) That the method for the determiation of volatile oil in spices, **33.17,** as modified by the Associate Referee, be made first action.

(7) That the tentative method for volatile oil and resin in ginger, **33.25,** be made first action after deletion of references to optical rotation and acid and ester numbers.

(8) That methods for the determination of ash in prepared mustard be studied.

(9) That studies of methods for the determination of sugars and ether extract in prepared mustard be continued.

(10) That studies of methods for the determination of pungent principles in prepared mustard and mustard flour be continued.

(11) That the tentative method for total fat in mayonnaise and salad dressing, **33.54,** with the slight modification described by the Associate Referee, be made first action.

(12) That the efficiency of the method for the preliminary removal of fat in the official method for total nitrogen in mayonnaise, **33.52,** be checked and, if necessary, further studied.

(13) That studies be made of methods for determining seed and stem content of chili peppers.

It is also recommended—

(1) That sections **33.18, 33.48, 33.49,** and **33.50,** be designated as official.

(2) That sections **33.3 33.23, 33.37, 33.64, 33.65,** and **33.81** be designated first action.

(3) That sections **33.1, 33.29–33.33, 33.34, 33.46, 33.58, 33.59,** and **33.89** be designated as procedures.

(4) That sections **33.2, 33.12, 33.19, 33.21, 33.22, 33.24, 33.55, 33.83,** and **33.84** be deleted.

ENZYMES

It is recommended—

(1) That method **5.1–5.4,** proteolytic activity of papain, be designated as first action.

(2) That this chapter be expanded. The committee notes an increasing use of the various enzymes. The Referee on Extraneous Materials in Foods and Drugs has expressed a need for more active vegetable protein and carbohydrate ferments and, in particular, an enzyme which will effectively digest the protein of cheese and other dairy products. Further need for an active lipolytic enzyme is expressed in *This Journal* **30,** 343 (1947). Methods for the preparation and/or testing of such enzymes would be valuable.

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## REPORT OF SUBCOMMITTEE D ON RECOMMENDATIONS OF REFEREES*

By Kenneth L. Milstead (1950) (Food and Drug Administration, Cincinnati, Ohio), *Chairman*; J. Walter Sale (1952);† and C. S. Ferguson (1954)‡

ALCOHOLIC BEVERAGES

*Malt Beverages, Brewing Materials, and Allied Products*:

It is recommended—

(1) That study of methods for determination of essential oil and resins in hops be continued.

(2) That the dye color method for the estimation of color in wort and beer, described in *This Journal,* **32,** 81 (1949), be adopted as official.

(3) That work on photometric beer color evaluations be continued.

(4) That study of beer turbidity methods be continued.

(5) That the Mathers test for caramel, *This Journal,* **31,** 76 (1948), be adopted for beer as first action.

(6) That the method for carbon dioxide in beer, described in the report of the Associate Referee this year, be adopted as first action to replace **14.24–14.25.**

(7) That work be continued on polarographic and spectrographic methods for tin in beer.

(8) That the method for total solids in yeast, **14.112–14.115,** as revised in *This Journal,* **31,** 174 (1948), and *Book of Methods, A.S.B.C.,* 5th edition, 1949, be adopted as official.

(9) That the following tentative methods for malt beverages, sirups and extracts, and brewing materials be adopted as first action:

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* These recommendations submitted by Subcommittee D were approved by the Association. Unless otherwise given all references are to *Methods of Analysis, A.O.A.C.,* 1945.

† John B. Wilson served for Sale.

‡ Frank A. Vorhes served for Ferguson.

**14.16** Dextrin.
**14.21–14.23,** incl. Carbon Dioxide.
**14.28–14.30** Iron.
**14.33** End Fermentation.
**14.42** Alternate Bushel Weight.
**14.57** Extract in Caramel Malt.
**14.58** and **14.59** Color in Caramel and Black Malts.
**14.94** Diastatic Power of Malt Sirups.
**14.116–14.124** Spent Grains.

and the following new methods:

Dextrose, under Brewing Sugars and Sirups, *Book of Methods, American Society Brewing Chemists,* 5th Ed. (1949) p. 57, **13.**
Protein, under Malt, *Book of Methods A.S.B.C.,* p. 109, **9.**
Wort Nitrogen, under Malt, *Book of Methods, A.S.B.C.,* p. 110, **10.**

(10) That the methods for mold, **14.47;** length of acrospire, **14.43;** mealiness, **14.44;** assortment, **14.46;** and foreign seeds and broken kernels, **14.48,** be classed as procedures.

(11) That the official (*This Journal,* **31,** 56 (1948)) method for hops, **14.80(b)** and **14.81** be revised as described in the report of the Associate Referee for malt beverages, and by the Associate Referee on hops, and adopted as a procedure, except **14.81(b)** color and luster, and **(e)** aroma, which should be dropped.

(12) That studies be continued on the determination of iron in beer, giving attention to both the wet-ashing orthophenanthraline procedure and the direct non-ashing procedure as suggested by the Associate Referee.

*Wines*:

It is recommended—

(1) That chromatographic studies of wine be continued.

(2) That the tentative method for volatile acidity—exclusive of $SO_2$, **15.25**—be dropped and that the tentative method II, **15.26,** for the same constituent be made first action.

(3) That the tentative method for citric and malic acids, **15.30,** be adopted as first action.

(4) That the methods for nitrates, **15.36(a)** and **(b),** be dropped.

(5) That the general reference to Chapter 21, "Coloring Matters—Tentative" **(15.37),** be dropped.

(6) That **15.15** be made a procedure.

*Distilled Liquors:*

It is recommended—

(1) That the study of methods of analysis with reference to the aging or maturing of whisky in laminated (plywood) barrels be continued.

(2) That the study of colorimetric methods for fusel oil be continued.

(3) That the official method, **16.29,** for methanol by the immersion refractometer method be studied in the light of the findings of Beyer and Reeves, *This Journal,* **28,** 800 (1945).

(4) That the rapid method for proof of distilled spirits as described in the report of the Associate Referee for 1948, *This Journal,* **32,** 154 (1949), be adopted as official.

(5) That study be continued of the official Denigè's method for methanol, **16.25,** and the tentative method for methanol in **39.161** and **39.162** to bring about uniformity in these procedures as far as possible.

(6) That the tentative method for "Detection of Acetone, Ketones, Isopropanol, and Tertiary Butyl Alcohol" **(16.20** and **16.21)** be made first action after changing "one or more of the above-mentioned compounds" to "acetone, other ketones or tertiary butyl alcohol."

(7) That the tentative tetramethylammonium iodide method for methanol, **16.31** to **16.34,** incl., be dropped.

(8) That the method for water-insoluble color, **16.36,** be made first action, after revision as recommended by the Associate Referee this year.

(9) That the methods listed below be adopted as first action, after revision as described in the Associate Referee's report this year.

**16.13** Esters—Official.
**16.16** Aldehydes Volumetric Method—Official, first action.
**16.22** Fusel Oil—Official.

(10) That the tentative method for color insoluble in amyl alcohol, **16.37,** be dropped.

(11) That the following methods be made first action:

**16.38** Marsh test, caramel.
**16.40** Cyclohexanol test, caramel.
**16.42–16.43** Tannin.

(12) That the last sentence in **XVI,** 5, 5th Ed., "The alcohol content of distillate may be checked by determining immersion refractometer reading and obtaining percentage of alcohol from Table 20, **XLIII**" be included in the 7th Edition. This was apparently omitted from the 6th Edition by oversight as its deletion was never recommended.

(13) That the Fulton test for caramel, *This Journal,* **31,** 77 (1948), be adopted, first action, for distilled liquors and for cordials and liqueurs.

*Cordials and Liqueurs:*

It is recommended—

(1) That **16.44**—"Physical Examination" be designated a procedure.

(2) That the caption of **16.63**—"Preliminary Procedure*** Acids" be changed to **16.63**—"Characteristic Acids—Preparation of Sample—Procedure."

(3) That the following methods be adopted as official:

**16.45** Specific Gravity.
**16.46** Alcohol.
**16.47** Methanol.
**16.48** Aldehydes.
**16.49** Furfural.
**16.50** Fusel Oil.

(4) That the method for "total solids—from the specific gravity of the dealcoholized sample," as given in this year's report, be adopted as first action to replace **16.51(a).**

(5) That the method for "total solids by evaporation," as given in this year's report, be adopted as first action to replace **16.51(b).**

(6) That the "Mather's Test for Caramel," as given in the report of the Associate Referee on caramel, be adopted as first action.

(7) That the method for "Total Acidity," **16.62,** be adopted as first action.

(8) That the qualitative method for Thujone, **16.73,** be adopted as first action.

(9) That the following methods be adopted as official:

**16.52** Glycerol.
**16.53** Sucrose by Polarization.
**16.54** Sucrose by Reducing Sugars.
**16.55** Ash.
**16.56** Soluble and Insoluble Ash.
**16.57** Alkalinity of Soluble Ash.
**16.58** Alkalinity of Insoluble Ash.
**16.59** Phosphoric Acid.

(10) That the following methods be adopted as first action:

**16.64** Tartaric Acid, deleting reference to **26.35.**
**16.65** Citric Acid.
**16.66** Active Malic Acid.
**16.67** Inactive Malic Acid.

### CACAO PRODUCTS

It is recommended—

(1) That the methods proposed by the Associate Referee for the determination of pectic acid in cacao products be adopted as first action and the method be subjected to further study, and that the tentative method, **19.16,** for the determination of pectic acid, be dropped.

(2) That the following parenthetical statement be inserted in **19.23** immediately following the caption "Quantitative Determination": "Not applicable to cacao products containing milk ingredients or those prepared with sugar and water by cooking and drying."

(3) That method II under fat **(19.24)** be deleted and the modified acid hydrolysis method, *This Journal,* **28,** 482 (1945), for fat determination studied by the Associate Referee be substituted as first action, with the

option of using a 150 mm Büchner fritted glass funnel (fine or medium porosity), and with the caption "Applicable to cacao products containing milk ingredients or those prepared with sugar and water, by cooking and drying" and also providing for the use of a sample of 10–20 g. in the case of milk chocolate.

(4) That **19.25** be made a procedure, retaining the present directions and adding at the beginning: "**(a)** Not applicable to cacao products containing milk ingredients or those prepared with sugar and water by cooking and drying" and adding a new section, "**(b)** Applicable to cacao products containing milk ingredients or those prepared with sugar and water by cooking and drying," reading "Proceed as under Method II using 20 g. sample and combining fat obtained in duplicate determinations for examination"; and that studies on separation and preparation of fat for identification be continued.

(5) That the study of the method for the determination of lactose in cacao products in the presence of other reducing sugars be continued.

(6) That the study of methods for the determination of maltose in cacao products be continued.

(7) That the method, **19.1(a)**, and the method **19.1(b)**, for the preparation of sample of chocolate products, be designated as procedures.

(8) That the following methods be adopted as first action:

**19.2** Moisture.
**19.10–19.14**, incl.—Shell in cacao nibs.
**19.32** Detection of coconut and palm kernel oil in cacao butter and fat extracted from milk chocolate.
**19.33–19.34** Silver number for detection of coconut and palm kernel oils.
**19.35–19.36** Critical temperature of dissolution of fat in acetic acid.
**19.38** Milk fat in milk chocolate.

(9) That the following methods be dropped:

**19.9** Coloring Matters.
**19.37** Acetone-carbon tetrachloride test for fat.
**19.41** Milk ash (from calcium).
**19.44** Corn syrup.
Tannin and pigments, *This Journal*, **31**, 78 (1948).

(10) That the following methods be adopted as first action:

**19.22** Chocolate liquor.
**19.30** Milk fat in milk chocolate.
**19.43** Dextrose.
**19.45** Starch. 1. Direct acid hydrolysis method.
**19.46** Starch. 2. diastase method.
**19.47** Theobromine.

(11) That the change recommended by the Referee be made in the method, **19.42**, for sucrose.

(12) That work on cacao constituents be continued.

(13) That the method for the determination of lecithin in cacao products (*This Journal*, **32,** 168 (1949), be adopted as first action and that it be studied further.

CEREAL FOODS

It is recommended—

(1) That the studies on the determination of starch in raw and cooked cereals be continued.

(2) That the method for the determination of fat acidity in grain and flour, **20.18–20.21,** incl., **20.76,** be made official.

(3) That the study of the modification of the method, **20.28, 20.29** and **20.30,** for the determination of reducing and non-reducing sugars in bakery products as recommended in the Associate Referee report be continued.

(4) That the method for benzoic acid in flour in the Associate Referee's report be adopted as first action,

(5) That the methods for the determination of lactose in bread be further studied.

(6) That the method for the determination of fat and fat number in bread, *This Journal*, **32,** 85 (1949), be adopted as official.

(7) That the method for the determination of proteolytic activity of flour and malted wheat flour, *This Journal*, **32,** 86 (1949), with the changes recommended in the Associate Referee's report, be adopted as official and that the work be continued as suggested in the report.

(8) That the methods for soybean flour, *This Journal*, **32,** 87 (1949), for moisture, ash, nitrogen and oil or petroleum benzine extract be further studied.

(9) That the method for the determination of added inorganic material in phosphated flour (qualitative), *This Journal*, **32,** 88 (1949), be adopted as official.

(10) That the method referred to in *This Journal*, **25,** 83, (1942), for the determination of unsaponifiable matter and sterols in noodles be studied to determine their applicability to bakery products containing eggs.

(11) That the study of methods for the determination of albumen in noodles and macaroni products be conducted.

(12) That the study on the determination of moisture, fat, crude fiber, ash, and protein in bakery products be continued.

(13) That the study on the determination of moisture in flour products containing sodium bicarbonate as one of its constituents be continued.

(14) That the study on the determination of bromates in flour be continued.

(15) That the following methods be adopted as first action:

**20.39** Water-soluble protein-nitrogen precipitable by 40% alcohol.
**20.46–20.47** Starch, polarimetric method.

**20.80** Crude fiber (soy bean flour).
**20.87** Citric acid.
**20.98** Solids.
**20.99** Ash.
**20.100** Protein.
**20.101** Fat.
**20.102** Crude fiber.
**20.103** Sugars.
**20.117** Water-soluble protein-nitrogen precipitable by 40% alcohol.

(16) That the following tentative methods be dropped:

**20.44–20.45** Starch.
**20.53** Benzoic acid (rye flour).
**20.54** Gasoline color value.
**20.81** Crude fat or ether extract.

(17) That the following tentative methods be adopted as first action:

**20.48** Chlorine in fat of flour.
**20.51** Nitrite nitrogen.
**20.55–20.59** Carotene.
**20.69** Soybean flour in uncooked cereal products.
**20.112** Original ash in macaroni products containing added salt but not containing added eggs.

(18) That the following change be made in the method for chlorides in ash as sodium chloride, **20.113**: change "does not give" in line 4 to "gives only approximate."

(19) That the method prepared by the Associate Referee for the determination of pigment in flour expressed as carotene be adopted as first action.

### BAKING POWDER

It is recommended—

(1) That the qualitative test for phosphoric acid, **17.31,** be adopted as official.

(2) That the residual carbon dioxide methods, *This Journal,* **31,** 78 (1948), and ibid., **32,** 83 (1949), be adopted as official.

(3) That the methods **17.2** and **17.3,** gravimetric for total carbon dioxide; **17.7,** residual carbon dioxide; and the phrase "Subtract residual $CO_2$ **17.7,** from total $CO_2$, **17.3**" in **17.9** be dropped.

(4) That the quantitative determination of aluminum by precipitation with phenyl hydrazine, **17.24,** be dropped.

(5) That the following tentative methods be made first action:

**17.11** Neutralizing value of monocalcium phosphate.
**17.12** Neutralizing value of sodium acid pyrophosphate.
**17.13** Tartaric acid, free or combined (qualitative test).
**17.22–17.23** Aluminum (qualitative test).
**17.34** Arsenic.
**17.35** Fluorine.
**17.36** Lead.

FLAVORS AND NON-ALCOHOLIC BEVERAGES

It is recommended—

(1) That the following methods be adopted as first action:

**13.19** Tartaric Acid.
**13.20** Citric Acid.
**13.21** Malic Acid.
**25.3** Glycerol in vanilla extract.
**25.29** Glycerol in lemon, orange, and lime extracts and flavors.
**25.54** Alcohol in almond extract and designated as Method I.
**25.56** Benzoic acid.
**25.60** Alcohol in cassia, cinnamon, dried clove extracts, and designated as Method I.
**25.61** Isopropyl alcohol.
**25.64** Alcohol in ginger extract.
**25.65** Solids in ginger extract.
**25.66** Ginger (qualitative test).
**25.67** Capsicum (qualitative test).
**25.68** Alcohol in peppermint, spearmint and wintergreen extracts and designated as Method I.
**25.69** Isopropyl alcohol.
**25.73** Oil in anise and nutmeg extracts, Method I.

(2) That the following methods be adopted as official:

**13.29** Benzaldehyde.
**13.30** Gamma undecalactone.
**25.58** Hydrocyanic acid.

(3) That the following methods be adopted as first action:

**25.15** Vanilla resins.
**25.26, 25.27, 25.28** Isopropyl alcohol in lemon extract in the absence of acetone after making the following change in **25.27**, first sentence, "To 2 ml of distillate add 0.5 ml of 5% . . . "
**25.35** Oil of lemon, orange, or lime in oil base flavor, Method II by polarization.
**25.55** Benzaldehyde.
**25.63** Oil in cassia, cinnamon, and clove.
**25.71** Oil in peppermint, spearmint, and wintergreen extracts, Method II.
**25.74** Oil, Method II.
**25.75** Essential oil.

(4) That the following methods be adopted as procedures:

**13.8** Commercial Glucose.
**25.16** Qualitative test (vanilla resins).
**25.18** Color value.
**25.19** Residual color.

(5) That the following methods be dropped:

**25.23** Alcohol in lemon, orange and lime extracts, Method I.
**25.44** Lemon and orange peel color.
**25.62** Oil in cassia, cinnamon and clove extracts, Method I.
**25.70** Oil in peppermint, spearmint and wintergreen extracts, Method I.
**25.72** Methyl salicylate in wintergreen extract.

(6) That the method for alcohol in almond extract, as given by the Referee, be adopted as method II, first action.

(7) That the method for alcohol in cassia, cinnamon, and clove extracts, as given by the Referee, be designated as method II and adopted as first action.

(8) That the method for alcohol in peppermint, spearmint, and wintergreen extracts, as given by the Referee, be designated as method II and adopted as first action.

(9) That the collaborative study of the reflux method for determination of peel oil in citrus fruit juices and the use of the modified oil separation trap be continued.

(10) That collaborative work be continued on the method for determination of beta-ionone where small amounts are present.

(11) That collaborative studies on the Ripper method for determination of aldehydes in spirits as applied to lemon oils and extracts be continued.

(12) That collaborative studies of the methods proposed by the Referee for determination of esters in lemon extract be continued.

(13) That collaborative studies on the Seeker-Kirby method for determination of esters in lemon and orange oils (Dept. of Agri. Tech. Bull. 241 (1932)) be continued.

(14) That collaborative studies of extract methods containing both isopropyl alcohol and acetone be continued.

(15) That collaborative study of the photometric method for determination of vanillin and coumarin be continued.

(16) That work be continued on the determination of glycerol, vanillin, and coumarin in vanilla and imitation vanilla extracts, with special reference to the automatic extraction of vanillin and coumarin.

(17) That the study of emulsion flavors be continued.

(18) That studies on maple concentrates and imitations be continued.

(19) That study of the method for determination of diacetyl, published in *This Journal*, **25**, 255 (1942), be continued.

(20) That the methods for vanilla resins in vanilla extract, **25.15** and **25.16**, be studied collaboratively.

(21) That the method for color insoluble in amyl alcohol, **16.37**, which is being dropped from Chapter 16 be transferred to Chapter 25 and adopted as first action as applied to vanilla extract.

### FRUITS AND FRUIT PRODUCTS

It is recommended—

(1) That study of methods for determining fruit and sugar content of frozen fruits be continued.

(2) That the glass electrode method for the determination of titratable

acidity, **26.30,** be adopted as first action and that further work on this subject be discontinued.

(3) That the bitartrate method for the determination of total tartaric acid, **26.32** and **26.33,** be adopted as first action.

(4) That the method for the determination of citric acid, **26.36** and **26.37,** be adopted as first action.

(5) That the method for the determination of Laevo-malic acid, **26.38, 26.39,** and **26.40,** be adopted as first action.

(6) That the method for the determination of inactive malic acid, **26.41, 26.42,** and **26.43,** be adopted as first action.

(7) That the study of methods for the separation and determination of fruit acids be continued.

(8) That method **26.7** and the rapid procedure, *This Journal,* **32,** 94 (1949) for the determination of water-insoluble solids in fruits, and the method for the determination of seeds in berry fruits, *This Journal,* **32,** 95 (1949), be adopted as first action and that further work on these subjects be discontinued.

(9) That the changes adopted last year in method **26.18(a),** *This Journal,* **32,** 94 (1949), be adopted as official.

(10) That the sentence beginning "If recoveries are low. . . . " in **26.19,** note (3) be deleted.

(11) That the title for method **26.8,** "Soluble solids in fresh and canned fruits, jams, marmalades, and preserves," be revised by inserting "by refractometer" between "solids" and "in," and that the method be adopted as first action.

(12) That the following methods be adopted as first action:

**26.12** Total sulfur.
**26.13** Chlorine in ash.
**26.20–26.21** Manganese.
**26.22–26.23** Calcium.
**26.24** Magnesium.
**26.25** Alcohol precipitate.
**26.26** Pectic Acid.
**26.28(b)** Titratable acidity, highly colored solutions.

(13) That for purposes of clarification only, the phrase "using 0.1*N* $HNO_3$" be inserted after "if necessary" in line 5 of the gravimetric cobaltinitrite method for potassium, **26.19.**

(14) That the following tentative methods be dropped:

**26.34–26.35** Tartaric acid (Racemate method).
**26.45** Volatile acids.
**26.51** Free mineral acids.
**26.56** Dextrin.
**26.58** Gelatin.
**26.59–26.60** Agar-agar.
**26.61** Added water in grape juice.

(15) That **26.29** buffer standards, be deleted from this chapter and reference be made to the appropriate section of the chapter on standard solutions where the buffer standards will appear.

(16) That method **26.55,** for commercial glucose, be made a procedure.

### SUGAR AND SUGAR PRODUCTS

It is recommended.—

(1) That the method for the determination of lac on confectionery, *This Journal,* **32,** 102 (1949), official, first action, as amended by the Associate Referee, be adopted as official, and the subject closed.

(2) That the study of methods for the determination of moisture be continued.

(3) That the study be continued on tables of density of solutions of sugar at various temperatures.

(4) That the official method for the determination of free acid in honey, **34.99,** be modified as suggested in this year's report.

(5) That the study of methods for the detection of adulteration of honey be continued.

(6) That micro methods for reducing sugars be studied.

(7) That the method for unfermented reducing substances in molasses adopted as official, first action, in 1948, *This Journal,* **32,** 103 (1949), be adopted as official.

(8) That the Zerban and Martin values for refractive indices of dextrose and invert sugar solution, *This Journal,* **27,** 295 (1944), be adopted as first action.

(9) That the tentative methods, **34.133–34.155**, inclusive, be adopted as first action and that they be further studied.

(10) That the procedures for the measurement of transmittancy of solution of commercial sugar products be studied.

(11) That the micro method for reducing sugars, **34.63, 34.64,** and **34.65** be modified as suggested by the Associate Referee and adopted as first action after changing the words "Reducing Sugars" in the title to "Dextrose."

(12) That Ofner's method for the determination of invert sugar in the presence of sucrose be made official; (first action, *This Journal,* **32,** 103).

(13) That the method, **34.8,** for solids by refractometer be amended by adding: "In liquid products containing invert sugar, correct the per cent solids obtained from **44.7** by adding 0.022 for each per cent invert sugar present in the sample." (Final recommendation.)

(14) That the following methods be made first action:

**34.2** Moisture direct drying.
**34.16** and **34.73** Mineral adulterants in ash.
**34.78** Starch.
**34.79** Ether extract, continuous extraction.
**34.80** Ether extract, Roese-Gottlieb.

**34.81** Paraffin.
**34.92** Direct polarization.
**34.93** Invert polarization.
**34.96** Levulose.
**34.97** Dextrose.
**34.102–34.103** Commercial invert sugar, resorcinal test.
**34.104–34.105** Commercial invert sugar, aniline chloride test.
**34.130** Malic acid value.
**34.131–34.132** Sucrose, hot water digestion, Methods I and II.
**34.133–34.155** Starch conversion products methods.

(15) That the following methods be dropped:

**34.12** Mineral constituents.
**34.36–34.37** Invert sugar II. Scales method.
**34.46** Invert sugar IV. Herzfeld gravimetric method.
**34.69** Mineral constituents.
**34.83** Coloring matters.
**34.84, 34.115** Metals.

(16) That the following methods be adopted as official:

**34.38** Invert sugar, Munson-Walker general method.
**34.45** Determination of reduced copper by titration with potassium permanganate.
**34.50** Dextrose, Lane-Eynon general volumetric method.

(17) That the following methods for the preparation of sample, maple sirup, be designated as procedures:

**34.107 a(1) and a(2).**

(18) That the following methods for the preparation of sample for maple sugar and other solid or semi-solid products be designated as procedures:

**34.107, b(1) and b(2).**

(19) That the following methods be made procedures:

**34.31–34.32** Commercial glucose (approximate), polarimetric Methods I and II.
**34.77** Commercial glucose.
**34.98** Dextrin.
**34.100** Commercial glucose, qualitative.
**34.101** Commercial glucose, quantitative.
**34.106** Diastase.
**34.121** Commercial glucose.

### WATERS, BRINE, AND SALT

It is recommended—

(1) That title of Chapter 37 be changed from "Waters, Brine, and Salt" to "Mineral Waters and Salt."

(2) That section captions "Potable Water" "Industrial Water," "Irrigating Water" and "Brine" be deleted.

(3) That the following methods be deleted:

Turbidity, **37.1, 37.2.**
Color, **37.3–37.4.**

Odor, **37.5.**
Suspended matter, **37.9.**
Nitrogen in form of free and albuminoid ammonia, **37.10** (except that reagents (a) and (c) be placed under **37.18)–37.11, 37.12, 37.13,** and **37.48.**
Nitrogen in form of nitrite, **37.14, 37.15,** and **37.49.**
Oxygen required, **37.27–37.30,** incl.
Dissolved oxygen, **37.31–37.34,** incl.
Lead, **37.35–37.40,** incl.
Copper, **37.41–37.42.**
Zinc, **37.43–37.44.**
Free carbon dioxide, **37.55.**
Industrial water, **37.84–37.97.**
Irrigating water, **37.98–37.100,** incl.

(4) That method for total solids **37.6;** solids in solution **37.7,** and ignited residue, **37.8,** be transferred to the section on "Mineral Water" after "Specific gravity—Official," in place of **37.46** and **37.47;** and that present methods **37.46** and **37.47** be dropped.

(5) That methods for nitrogen in form of nitrate, **37.16–37.19,** incl., and chloride, **37.20** and **37.21,** be transferred to the section on "Mineral Water" in place of **37.50** and **37.51,** and that present methods **37.50** and **37.51** be dropped.

(6) That colorimetric methods using dithizone for the determination of lead, copper, and zinc in mineral waters be studied.

(7) That methods for fluorine, **37.22–37.26,** incl., be transferred to the section on "Mineral Water" in place of **37.52,** and that present method **37.52** be dropped.

(8) That the following be deleted from method **37.19** "Nesslerize, and compare with standards as in determinations of free $NH_3$, **37.11.** Subtract quantity of *N* found in blank from that found in sample. Calculate to mg/liter of N.," and substitute therefor the following from method **37.11:** "at rate of ca 1 tubeful in 10 min. into 50 ml. Nessler tubes until $NH_3$ ceases to be given off (4–5 tubes are usually sufficient). Add 2 ml. of Nessler reagent to each tube and let stand 10 min. From a small buret measure definite quantity of the $NH_4Cl$ soln. into Nessler tubes. Dilute to 50 ml. with $NH_3$ free $H_2O$, add 2 ml. of the Nessler reagent, and compare depth of color with Nesslerized distillate. Report as mg/liter of N."

(9) That method **37.59,** line 3, be changed from "add 5% $(NH_4)$ SCN soln." to "add 3 ml. 5% $(NH_4)$ SCN soln."

(10) That method **37.60,** line 1, be changed from "with fused $KHSO_4$" to "with about 1 gram fused $KHSO_4$."

(11) That the following methods be adopted as official:

Strontium, **37.63.**
Bromide in presence of chloride and iodide, **37.105–37.107,** incl.

(12) That method **37.71,** "Phosphoric Acid" line 5, "freshly prepared" be inserted between words "add" and "molybdate soln."

(13) That method **37.81 (a)** Boric Acid, *qualitative procedure* be retained deleting only at the end, "**32.17.**"

(14) That the present reference to the quantitative method for boric acid under method **37.81** be deleted and the method recommended by the Referee in this year's report be adopted as first action.

(15) That in title of method **37.83** "Equivalent combining weights and their Reciprocals based on International Atomic Weights, 1943" date be changed to "1948."

(16) That the following methods be designated as procedures:

Boric acid, qualitative test, **37.81(a)**, as revised
Reporting of results for water, **37.82.**
Preparation of sample (salt), **37.108.**
Preparation of solution for sulfate, calcium, and magnesium **37.112.**
Reporting results, **37.119.**

(17) That the following methods be adopted as first action:

Iodide and bromide, **37.77–37.78.**
Bromide in presence of chloride but not iodide, **37.101–37.104**, incl.
Salt, **37.109–37.111**, incl.
Sulfate, **37.113.**
Calcium, **37.114.**
Magnesium, **37.115.**

## CHANGES IN OFFICIAL AND TENTATIVE METHODS OF ANALYSIS MADE AT THE SIXTY-THIRD ANNUAL MEETING, OCTOBER 10, 11, AND 12, 1949*

This report gives the newly adopted methods by title and lists those on which revisions were made. It also includes the changes in designations of methods made to conform with the classifications provided in by-laws 5 and 6 as adopted at the 1948 meeting (*This Journal*, **32**, 38 (1949)). The details of the new methods and the revisions will be published in full in the seventh edition of *Methods of Analysis*, which is expected to be ready for distribution by October 1950. In some cases the details of revision are given in the reports of the appropriate subcommittees, A. B. C. D., pages 36 to 71.

### 1. SOILS

(1) Methods for sampling, **1.1**, and preparation of sample, **1.2**, were made procedures.

(2) Methods **1.3** to **1.53**, incl., were made first action.

### 2. FERTILIZERS

(1) The following methods were adopted as official:

(a) Free water, air-flow method, *This Journal*, **32**, 72 (1949).
(b) Free water, vacuum-desiccation method, *This Journal*, **32**, 73 (1949).

* Unless otherwise given all references in this Report are to *Methods of Analysis*, *A.O.A.C.*, 1945.

(2) The formaldehyde titration method for nitrogen in ammonium nitrate, *This Journal*, **32**, 71 (1949), was revised and adopted as official.

(3) The following official methods were revised:

(a) Sampling, **2.1.**
(b) Preparation of sample, **2.2.**
(c) Citrate-insoluble phosphoric acid, **2.16.**
(d) Citrate-soluble and available phosphoric acid, **2.17.**
(e) Total nitrogen, Kjeldahl modified to include nitrate N, **2.27.**
(f) Potash Lindo-Gladding method, **2.41(b).**
(g) Acid-soluble magnesium, gravimetric, **2.51.**
(h) Acid-soluble magnesium, volumetric, **2.52.**
(i) Acid-soluble manganese, colorimetric, **2.55.**

(4) The official methods for citric acid-soluble phosphoric acid, **2.18–2.20,** incl., and for water-soluble boron, **2.44** were deleted.

(5) The following methods were made first action:

(a) Acid-soluble calcium, **2.49–2.50.**
(b) Copper, short volumetric, **2.60.**
(c) Zinc, gravimetric **2.61,** and colorimetric **2.62.**
(d) Free sulfur, **2.63.**
(e) Acid-forming or nonacid-forming quality, **2.64** and **2.65.**

(6) The methods for water-soluble magnesium, **2.53** and **2.54,** were rewritten and adopted, first action.

(7) The "identical *p*H" method for boron was adopted, first action.

### 3. AGRICULTURAL LIMING MATERIALS

(1) Methods for sampling, **3.1,** and preparation of sample, **3.2,** were made procedures.

(2) Methods **3.3, 3.4,** and **3.7–3.11,** incl., were made first action.

(3) The methods for neutralizing values for blast furnace slags, *This Journal*, **31**, 71 (1948), and for sulfide sulfur in calcium silicate slags, *This Journal*, **32**, 73, 1949, were made first action.

### 4. COSMETICS

(1) The methods for aluminum and zinc in deodorants, and antiperspirants, **4.1–4.3,** incl., were made first action.

(2) A new method for zinc and boric acid in face powders was adopted, first action.

(3) New methods for type of emulsion, water, ash, and chloroform-soluble matter in vanishing cream were adopted, first action.

### 5. ENZYMES

(1) The method for proteolytic activity of papain, **5.1–5.4,** incl., was made first action.

## 6. ECONOMIC POISONS

(1) The following methods were adopted as official:

(a) DDT (I–VI, incl.), *This Journal*, **30**, 319 (1947), and **31**, 368 (1948).
(b) DDT in emulsions, *This Journal*, **31**, 371 (1948).

(2) The method for phenol coefficient, **6.153–6.157**, incl., was revised and retained as official.

(3) The following methods were made first action:

(a) Total arsenic, Method III, **6.9–6.10.**
(b) Total fluorine, distillation method, **6.22–6.23.**
(c) Fluorine present as sodium fluosilicate, **6.24–6.25.**
(d) Total arsenic oxide, **6.38–6.39.**
(e) Pyrethrin III in pyrethrum powder, **6.114.**
(f) Pyrethrin I in pyrethrum extracts in mineral oil, **6.115–6.116.**
(g) Sulfide-sulfur in lime-sulfur solutions, **6.130.**
(h) Fungicidal test, **6.158–6.162**, incl.

(4) The following new methods were adopted, first action:

(a) 2,4-D in herbicides.
(b) Alphanaphthyl thiourea in rodenticides.
(c) Tetraethyl pyrophosphate.
(d) Gamma benzene hexachloride, chromatographic.
(e) Gamma benzene hexachloride, infrared spectrometric.
(f) Gamma benzene hexachloride in wettable powder and insecticidal dust, chromatographic.

(5) The following methods were deleted:

(a) Monosulfide equivalent, iodine titration, **6.123–6.124, 6.127, 6.129**, and **6.132.**
(b) Alcoholic caustic method for 2,2-bis (*p*-chlorophenyl)-1,1,1-trichloroethane (DDT), **6.151–6.152.**
(c) Magnesium arsenate, **6.50–6.53**, incl.

## 7. CAUSTIC POISONS

No additions, deletions, or other changes.

## 8. NAVAL STORES

This chapter was deleted.

## 9. GELATIN, DESSERT PREPARATIONS, AND MIXES

(1) The following methods were made official:

(a) Moisture, **9.2, 9.8**, and **9.17.**
(b) Ash, **9.3, 9.9**, and **9.18.**
(c) Total phosphorus, **9.4.**
(d) Nitrogen, **9.5, 9.10**, and **9.19.**
(e) Total acidity, **9.11**, as revised.

(2) The following new methods were adopted, first action:

(a) Jelly strength of gelatin, to replace **9.6.**
(b) Jelly strength of gelatin dessert powders, to replace **9.12.**

(3) The methods for sucrose and dextrose, **9.13–9.14, 9.15,** and **9.20,** and for starch, **9.21,** were made first action.

(4) Methods for preparation of sample, **9.1, 9.7,** and **9.16,** were made procedures.

### 10. LEATHERS, AND 11. TANNING MATERIALS

These chapters were deleted, *This Journal,* **32,** 81 (1949).

### 12. PLANTS

(1) The following methods were made first action:

(a) Moisture, **12.3.**
(b) Ash, **12.4.**
(c) Micro method for aluminum only, **12.11.**
(d) Potassium, perchloric acid method, **12.18.**
(e) Sodium, magnesium uranyl acetate, **12.20–12.21.**
(f) Copper, **12.22–12.23.**
(g) Zinc, **12.24–12.30,** incl.
(h) Arsenic, **12.31–12.32.**
(i) Selenium, **12.48.**
(j) Reducing sugars, Munson-Walker, **12.50.**
(k) Reducing sugars, Quisumbing and Thomas, **12.51–12.52.**
(l) Sucrose, **12.53.**
(m) Ether extract, **12.54.**
(n) Crude fiber, **12.55.**
(o) Total nitrogen, **12.56.**
(p) Organic and ammoniacal nitrogen, **12.57.**
(q) Lignin, **12.64–12.67,** incl.
(r) Boron, **12.78.**

(2) The method for carotene **12.75–12.77,** incl., with minor revision, was made first action.

(3) The 72% sulfuric acid method for lignin, *This Journal,* **32,** 287 (1949), was adopted, first action.

(4) The following methods were deleted;

(a) Iodine, **12.47.**
(b) Ammonia in tobacco, **12.58–12.59.**
(c) Free nicotine in tobacco, **12.60–12.61.**
(d) Nitrate nitrogen in tobacco, **12.62–12.63.**

### 13. BEVERAGES (NONALCOHOLIC) AND CONCENTRATES

(1) The following methods were made official:

(a) Benzaldehyde, **13.29.**
(b) Gamma undecalactone, **13.30.**

(2) The following methods were made first action:

(a) Tartaric acid, **13.19.**
(b) Citric acid, **13.20.**
(c) Malic acid, **13.21.**

(3) The method for commercial glucose, **13.8,** was made a procedure.

## 14. MALT BEVERAGES, SIRUPS AND EXTRACTS, AND BREWING MATERIALS

(1) The dye color method for estimation of color in wort and beer, *This Journal*, **32**, 81 (1949), was adopted as official.

(2) The method for total solids in yeast, **14.112–14.115**, incl., was revised and adopted as official.

(3) The following new methods were adopted, first action:

(a) Dextrose, under brewing sugars and sirups.
(b) Protein, under malt.
(c) Wort nitrogen, under malt.

(4) The following methods were made first action:

(a) Dextrin, **14.16.**
(b) Carbon dioxide, chemical method, **14.21–14.23**, incl.
(c) Iron, **14.28–14.30**, incl.
(d) End fermentation as increase in degree of fermentation, **14.33.**
(e) Bushel weight, alternate method, **14.42.**
(f) Extract in caramel malt, **14.57.**
(g) Color in caramel and black malts, **14.58** and **14.59.**
(h) Diastatic power of malt sirups, **14.94.**
(i) Spent grains, **14.116–14.124**, incl.

(5) The Mather's test for caramel, *This Journal*, **31**, 76 (1948), was adopted, first action, for beer.

(6) The method given by the Associate Referee for carbon dioxide in beer was adopted, first action, to replace **14.24–14.25.**

(7) The following methods were made procedures:

(a) Mold, **14.47.**
(b) Length of acrospire, **14.43.**
(c) Mealiness, **14.44.**
(d) Assortment, **14.46.**
(e) Foreign seeds and broken kernels, **14.48.**

(8) The method for hops, **14.80(b)** and **14.81** as revised, were made first action, with deletion of **14.81(b)**, color and luster, and **(e)** aroma.

## 15. WINES

(1) The following methods were made first action:

(a) Volatile acidity, exclusive of $SO_2$, Method II, **15.26.**
(b) Citric and malic acids, **15.30.**

(2) The method for commercial glucose, **15.15**, was made a procedure.

(3) The following methods were deleted:

(a) Volatile acidity, exclusive of $SO_2$, Method I, **15.25.**
(b) Nitrates, **15.36.**

## 16. DISTILLED LIQUORS

(1) The rapid method for proof of distilled liquors, *This Journal*, **32**, 154 (1949), was adopted as official.

(2) The last sentences of XVI, 5, of the 5th edition, was restored in the method for alcohol.

(3) The following methods were made official:

(a) Specific gravity, **16.45**
(b) Alcohol, **16.46.**
(c) Methanol, **16.47.**
(d) Aldehydes, **16.48.**
(e) Furfural, **16.49.**
(f) Fusel oil, **16.50.**
(g) Glycerol, **16.52.**
(h) Sucrose by polarization, **16.53.**
(i) Sucrose by reducing sugars, **16.54.**
(j) Ash, **16.55.**
(k) Soluble and insoluble ash, **16.56.**
(l) Alkalinity of soluble ash, **16.57.**
(m) Alkalinity of insoluble ash, **16.58.**
(n) Phosphoric acid, **16.59.**

(4) The following new methods were adopted, first action:

(a) Total solids, from specific gravity of dealcoholized sample, to replace **16.51(a).**
(b) Total solids by evaporation, to replace **16.51(b).**
(c) Mather's test for caramel.
(d) Fulton test for caramel in distilled liquors and in cordials and liqueurs, *This Journal*, **31**, 77 (1948).

(5) The following methods were revised and made first action:

(a) Esters, **16.13.**
(b) Aldehydes, volumetric method, **16.16.**
(c) Detection of acetone, ketones, isopropanol, and tertiary butyl alcohol, **16.20–16.21.**
(d) Fusel oil, **16.22.**
(e) Water-insoluble color, **16.36.**

(6) The following methods were made first action:

(a) Marsh test, caramel, **16.38.**
(b) Cyclohexanol test, caramel, **16.40.**
(c) Tannin, **16.42–16.43.**
(d) Total acidity, **16.62.**
(e) Tartaric acid, **16.64,** deleting reference to **26.35.**
(f) Citric acid, **16.65.**
(g) Active malic acid, **16.66.**
(h) Inactive malic acid, **16.67.**
(i) Thujone, **16.73.**

(7) The following methods were made procedures:

(a) Physical examination, **16.44.**
(b) Characteristic acids, after revision of title, **16.63.**

(8) The following methods were deleted:

(a) Methanol, tetramethylammonium iodide method, **16.31–16.34,** incl.
(b) Color insoluble in amyl alcohol, **16.37** (Transferred to Ch. 25.)

## 17. BAKING POWDERS AND BAKING CHEMICALS

(1) The following methods were adopted as official:

(a) Phosphoric acid, qualitative test, **17.31.**
(b) Residual carbon dioxide, gasometric method, *This Journal*, **31**, 78 (1948).
(c) Residual carbon dioxide, drying oven method, *This Journal*, **32**, 83 (1949).

(2) The following methods were made first action:

(a) Neutralizing value of monocalcium phosphate, **17.11.**
(b) Neutralizing value of sodium acid pyrophosphate, **17.12.**
(c) Tartaric acid, free or combined (qualitative test), **17.13.**
(d) Aluminum (qualitative test), **17.22–17.23.**
(e) Arsenic, **17.34.**
(f) Fluorine, **17.35.**
(g) Lead, **17.36.**

(3) The following methods were deleted:

(a) Total carbon dioxide, gravimetric method, **17.2–17.3.**
(b) Residual carbon dioxide, **17.7.**
(c) The phrase "subtract residual $CO_2$, **17.7**, from total $CO_2$, **17.3**," in **17.9.**
(d) Aluminum, quantitative determination by precipitation with phenylhydrazine, **17.24.**

## 18. COFFEE AND TEA

(1) The Bailey-Andrew method, **18.41**, was adopted as official for caffeine in coffee.

(2) The following methods were made official:

(a) Soluble solids, **18.6.**
(b) Starch, **18.17.**
(c) Sugars, **18.18.**
(d) Total acidity, **18.20.**
(e) Protein, **18.38.**
(f) Tannin, **18.42** and **18.43.**

(3) The following were made procedures:

(a) Macroscopic examination, green coffee, **18.1.**
(b) Macroscopic examination, roasted coffee, **18.3.**
(c) Coating and glazing substances on coffee, **18.22, 18.23,** and **18.25.**
(d) Chicory infusion, **18.24.**
(e) Dust, stems, and foreign leaves in tea, **18.26.**
(f) Facing in tea, **18.44, 18.45,** and **18.46.**

(4) The following methods were deleted:

(a) Moisture in coffee, **18.5.**
(b) Fendler-Stüber method for caffeine in coffee, **18.15.**
(c) Volatile acidity in coffee, **18.21.**

## 19. CACOA BEAN AND ITS PRODUCTS

(1) The following official methods were revised:

(a) Fat method I, **19.23** (applicability restriction).
(b) Sucrose, **19.42.**

(2) The following new methods were adopted, first action:

(a) Pectic acid.
(b) Acid hydrolysis method for fat, *This Journal*, **28**, 482 (1945), as revised.
(c) Lecithin, *This Journal*, **32**, 168 (1949).

(3) The following were made first action:

(a) Moisture, **19.2.**
(b) Shell in cacao nibs, **19.10–19.14,** incl.
(c) Chocolate liquor, **19.22.**
(d) Milk fat in milk chocolate, **19.30.**
(e) Detection of coconut and palm kernel oil in cacao butter and fat extracted from milk chocolate, **19.32.**
(f) Silver number for detection of coconut and palm kernel oils, **19.33–19.34.**
(g) Critical temperature of dissolution of fat in acetic acid, **19.35–19.36.**
(h) Milk fat in milk chocolate, **19.38.**
(i) Dextrose, **19.43.**
(j) Starch, direct acid hydrolysis, **19.45.**
(k) Starch, diastase method, **19.46.**
(l) Theobromine, **19.47.**

(4) The following were adopted as procedures:

(a) New directions for separation and preparation of fat designated as "applicable to cacao products containing milk ingredients or those prepared by cooking with sugar and water and drying," and **19.25** was restricted as being not applicable to such products.
(b) Preparation of sample, **19.1(a)** and **19.1(b).**

(5) The following methods were deleted:

(a) Pectic acid, **19.16.**
(b) Fat, Method II, **19.24.**
(c) Acetone-carbon tetrachloride test for fat, **19.37.**
(d) Milk ash (from calcium), **19.41.**
(e) Corn sirup, **19.44.**
(f) Tannins and pigments, *This Journal*, **31**, 78 (1948).

## 20. CEREAL FOODS

(1) The following methods were adopted as official:

(a) Fat acidity in grain and flour, **20.18–20.21,** incl., and **20.76.**
(b) Fat and fat number in bread, *This Journal*, **32**, 85 (1949).
(c) Proteolytic activity, *This Journal*, **32**, 86 (1949), as revised.
(d) Added inorganic material in phosphated flour, *This Journal*, **32**, 88 (1949).

(2) The following new methods were adopted, first action:

(a) Benzoic acid in flour.
(b) Pigment in flour.
(c) Succinic and propyonic acids in bread and cake.

(3) The following methods were made first action:

(a) Water-soluble protein-nitrogen precipitable by 40% alcohol, **20.39.**
(b) Starch, polarimetric method, **20.46–20.47.**

(c) Chlorine in fat of flour, **20.48.**
(d) Nitrite nitrogen, **20.51.**
(e) Carotenoids, **20.55–20.59,** incl.
(f) Soybean flour in uncooked cereal products, **20.69.**
(g) Crude fiber (soybean flour), **20.80.**
(h) Citric acid, **20.87.**
(i) Solids, **20.98.**
(j) Ash, **20.99.**
(k) Protein, **20.100.**
(l) Fat, **20.101.**
(m) Crude fiber, **20.102.**
(n) Sugars, **20.103.**
(o) Original ash in macaroni products containing added salt but not containing added eggs, **20.112.**
(p) Water-soluble protein-nitrogen precipitable by 40% alcohol, **20.117.**

(4) The following methods were deleted:

(a) Starch, **20.44–20.45.**
(b) Benzoic acid, **20.53.**
(c) Gasoline color value, **20.54.**
(d) Crude fat or ether extract, **20.81.**
(e) Volatile fatty acids in bakery products, *This Journal*, **31,** 99 (1948).

## 21. COLORING MATTERS

(1) The following methods were made official:

(a) Separation by wool dyeing, **21.2,** after deletion of word "procedure" in title.
(b) Separation by immiscible solvents, **21.4–21.7,** incl., and **21.9,** after deletion of word "procedure" in title.
(c) Identification, **21.10–21.12,** incl.

(2) The following new methods were adopted, first action:

(a) Determination of tartrazine (FD&C Yellow No. 5) and Sunset Yellow (FD&C Yellow No. 6).
(b) Oil-soluble coal-tar dyes, to replace **21.3.**
(c) Preparation of solution, to replace **21.8.**

(3) The following methods were made first action on natural coloring matters:

(a) Separation, **21.14.**
(b) Identification, **21.15** and **21.16.**

(4) Method for pigments and lakes, **21.1,** was made a procedure.

(5) The methods on Commercial Coal-Tar Colors beginning on page 283, including **21.17–21.63,** and **21.68** were rewritten.

## 22. DAIRY PRODUCTS

(1) The Sanders-Sager method for phosphatase test for pasteurization, *This Journal*, **31,** 82 (1948), was adopted as official for milk and cream, cheddar type cheeses, and soft uncured cheeses, and as first action for all other dairy products.

(2) The method for phosphatase test for pasteurization, Long test,

and an alternate photometric method, were adopted, first action, as applicable to fluid milk and cream.

(3) The rapid test for phosphatase, **22.49–22.57,** incl., was deleted.

(4) The official method for fat, Roese-Gottleib, **22.25,** was editorially revised.

(5) The changes in the method for fat in cheese, **22.130,** recommended in *This Journal,* **32,** 91 (1949), were made official.

(6) The following methods were made first action:

(a) Lactose, **22.22–22.24.**
(b) Protein, **22.97.**
(c) Lactose in process cheese, **22.137.**
(d) Weight per unit volume of packaged ice cream, **22.143–22.145,** incl.
(e) Water-insoluble acids in butter and cream, *This Journal,* **32,** 91 (1949).

(7) The following methods were made first action:

(a) Acidity, **22.4.**
(b) Citric acid, **22.5–22.7,** incl.
(c) Ash, **22.16.**
(d) Casein, Method II, **22.19–22.20.**
(e) Moisture, **22.96.**
(f) Casein in malted milk and chocolate malted milk, **22.98.**
(g) Ash, **22.99.**
(h) Fat in malted milk, **22.101.**
(i) Citric acid in dried milk, **22.104.**
(j) Gums in soft curd cheese, **22.138–22.141.**
(k) A method for volatile acids in butter and cream involving the chromatographic technic.

(8) The methods for collection of sample, **22.1, 22.59, 22.94, 22.107,** and **22.123,** and for preparation of sample of various dairy products, **22.2, 22.60, 22.73, 22.85, 22.95, 22.123,** and **22.146,** were made procedures.

(9) The following methods were made procedures:

(a) Specific gravity, **22.3.**
(b) Total solids, approximate, **22.15.**
(c) Hypochlorites and chloramines, **22.36–22.38,** incl.
(d) Microscopic identification of malted milk and its flavored products, **22.106.**
(e) Preparation of butter samples, **22.108** and **22.109,** and method in *This Journal,* **31,** 91 (1948).
(f) Microscopic examination, **22.120.**
(g) Tartaric acid, qualitative test, **22.132.**
(h) Citric acid, qualitative test, **22.135.**

(10) The following methods were deleted:

(a) Sour serum, **22.29.**
(b) Renovated butter and oleomargarine, foam test, **22.121,** and melted fat test, **22.122.**

## 23. EGGS AND EGG PRODUCTS

(1) The method for volatile fatty acids, **23.36,** was modified to include a chromatographic technique and made first action.

(2) The following methods were adopted as official:

(a) Fat by acid hydrolysis, **23.8–23.9.**
(b) Acidity of ether extract, Rapid Method II, **23.32(a)** as revised.
(c) New method, acidity of ether extract, liquid eggs.

(3) The following methods were adopted, first action:

(a) A new method for glycerol in eggs, quantitative.
(b) Lipoids and lipoid phosphoric acid ($P_2O_5$), **23.10–23.12,** incl.
(c) A new method for water-insoluble acids.
(d) A new method for succinic acid.

(4) The following methods were made procedures:

(a) Collection and preparation of sample, **23.1.**
(b) Glycerol, qualitative test, **23.25–23.26.**

(5) The following methods were deleted:

(a) Water-soluble nitrogen and crude albumin nitrogen, **23.6(b),** dried eggs.
(b) Glycerol quantitative method, **23.27–23.28.**
(c) Acidity of ether extract, **23.30, 23.31, 23.32(b).**
(d) Ammonia nitrogen, **23.33–23.34.**

## 24. FISH AND OTHER MARINE PRODUCTS

(1) New methods for the following determinations were adopted as official:

(a) Crude fat by acid hydrolysis, *This Journal,* **32,** 93 (1949).
(b) Drained liquor in shucked oysters.

(2) The following methods were adopted, first action:

(a) Indole in shrimp, oysters, and crabmeat, *This Journal,* **31,** 96 (1948), as revised.
(b) Total solids, **24.3,** revised to include soft shell clams.
(c) The method for volatile fatty acids, **24.9–24.11,** incl., was modified to include a chromatographic technic.

(3) The following methods were made procedures:

(a) Apparatus, **24.1,** and preliminary treatment and preparation of sample, **24.2,** with revisions, combined as one section, and including clams where oysters and scallops are mentioned.

(4) The method for salt, II, with calcium acetate as fixative, **24.7,** was deleted.

## 25. FLAVORING EXTRACTS

(1) The method for hydrocyanic acid, quantitative, **25.58,** was made official.

(2) The following new methods were adopted, first action:

(a) Alcohol in almond extract, designated as Method II.
(b) Alcohol in cassia, cinnamon, and clove extracts and designated as Method II.

(c) Alcohol in peppermint, spearmint, and wintergreen extracts and designated as Method II.

(d) Color insoluble in amyl alcohol, transfer of **16.37.**

(3) The following methods were made first action:

(a) Glycerol in vanilla extract, **25.3.**
(b) Vanilla resins, **25.15.**
(c) Isopropyl alcohol in lemon extract, **25.26–25.28,** incl., as revised.
(d) Glycerol in lemon, orange, and lime extracts and flavors, **25.29.**
(e) Oil of lemon, orange, or lime in oil base flavors, polarization, **25.35.**
(f) Alcohol in almond extract, **25.54,** designated as Method I.
(g) Benzaldehyde, **25.55.**
(h) Benzoic acid, **25.56.**
(i) Alcohol in cassia, cinnamon, and dried clove extracts, **25.60,** designated as Method I.
(j) Isopropyl alcohol, **25.71,** in cassia, cinnamon, and clove extracts.
(k) Oil in cassia, cinnamon, and clove, **25.63.**
(l) Alcohol in ginger extract, **25.64.**
(m) Solids in ginger extract, **25.65.**
(n) Ginger (qualitative test), **25.66.**
(o) Capsicum (qualitative test), **25.67.**
(p) Alcohol in peppermint, spearmint, and wintergreen extracts, **25.68,** designated as Method I.
(q) Isopropyl alcohol in peppermint, spearmint, and wintergreen extracts, **25.69.**
(r) Oil in peppermint, spearmint, and wintergreen extracts, Method II, **25.71.**
(s) Oil in anise and nutmeg extracts, **25.73,** Method I.
(t) Oil in anise and nutmeg extracts, **25.74,** Method II.
(u) Essential oil, **25.75.**

(4) The following methods were made procedures:

(a) Vanilla resins (qualitative test), **25.16.**
(b) Color value, **25.18.**
(c) Residual color after precipitation with lead acetate, **25.19.**

(5) The following methods were deleted:

(a) Alcohol in lemon, orange, and lime extracts, **25.23,** Method I.
(b) Lemon and orange peel color, **25.44.**
(c) Oil in cassia, cinnamon, and clove extracts, **25.62,** Method I.
(d) Oil in peppermint, spearmint, and wintergreen extracts, **25.70,** Method I.
(e) Methyl salicylate in wintergreen extract, **25.72.**

## 26. FRUITS AND FRUIT PRODUCTS

(1) Method **26.18(a)** as revised, *This Journal,* **32,** 94 (1949), was adopted as official.

(2) The following methods were made first action:

(a) Water-insoluble solids, **26.7.**
(b) Water-insoluble solids, rapid method, *This Journal,* **32,** 94 (1949).
(c) Seeds in berry fruits, *This Journal,* **32,** 95 (1949).
(d) Soluble solids in fresh and canned fruits, jams, marmalades, and preserves, **26.8,** as revised.

(e) Total sulfur, **26.12.**
(f) Chlorine in ash, **26.13.**
(g) Manganese, **26.20–26.21.**
(h) Calcium, **26.22–26.23.**
(i) Magnesium, **26.24.**
(j) Alcohol precipitate, **26.25.**
(k) Pectic acid, **26.26.**
(l) Titrable acidity, with indicator, highly colored solutions, **26.28(b).**
(m) Titrable acidity, glass electrode method, **26.30,** with reference to appropriate section in Chapter 43 on buffer solutions, in place of **26.29.**
(n) Total tartaric acid, bitartrate method, **26.32–26.33.**
(o) Citric acid, **26.36–26.37.**
(p) Laevo-malic acid, **26.38–26.40,** incl.
(q) Inactive malic acid, **26.41–26.43,** incl.

(3) The following methods were made procedures:

(a) Sampling, **26.1.**
(b) Sampling frozen fruit in barrels, *This Journal,* **30,** 72 (1947)
(c) Preparation of sample, **26.2**
(d) Commercial glucose, **26.55.**

(4) The following methods were deleted:

(a) Titratable acidity, II, with glass electrode, reagents, **26.29.**
(b) Tartaric acid, Racemate method, **26.34–26.35.**
(c) Volatile acids, **26.45.**
(d) Free mineral acids, **26.51.**
(e) Dextrin, **26.56.**
(f) Gelatin, **26.58.**
(g) Agar agar, **26.59–26.60.**
(h) Added water in grape juice, **26.61.**

## 27. GRAIN AND STOCK FEEDS

(1) The following official methods were revised:

(a) Crude protein, **27.10.**
(b) Acid soluble manganese, **27.59.**

(2) The following new methods were adopted, first action:

(a) Sulfaquinoxaline.
(b) Calcium and phosphorus.

(3) The following methods were made first action:

(a) Galactan, **27.40.**
(b) Hydrocyanic acid formed by hydrolysis of glucosides in beans, acid titration, **27.49,** and alkaline titration, **27.50.**
(c) Iodine in mineral mixed feeds, Knapheide-Lamb, **27.54–27.56,** incl. and Elmslie-Caldwell, **27.57.**

(4) The directions for sampling, **27.1,** were changed to conform to those adopted for fertilizers, and adopted as procedures.

(5) The official, first action, method for calcium and phosphorus, *This Journal,* **32,** 95 (1949), was deleted.

## 28. MEATS AND MEAT PRODUCTS

(1) The method for creatin, **28.33,** was revised and adopted as official.

(2) The following methods were made first action:

(a) A new method for lactose.
(b) Salt, **28.5,** as revised.
(c) Nitrates, ferrous chloride method, **28.11–28.13,** incl.
(d) Nitrates, xylenol method, **28.14–28.17,** incl., as revised.
(e) Amino nitrogen, **28.34–28.36,** incl.

(3) The following methods were made procedures:

(a) Skim milk solids, qualitative test.
(b) Preparation of sample, **28.1** and **28.42.**
(c) Starch (qualitative test) **28.18,** as revised.
(d) Soybean flour (qualitative test), **28.20.**

(4) The method for added water in sausage, **28.3,** was rewritten and **28.3(c)** was made a procedure.

(5) The following methods were deleted under meat:

(a) Ammonia, **28.9–28.10.**
(b) Starch (quantitative method), **28.19.**
(c) Glycogen, qualitative, **28.21,** and quantitative, **28.22–28.23,**
(d) Dextrose, **28.24.**
(e) Soluble and insoluble nitrogen, **28.28–28.29.**
(f) Coagulable nitrogen, **28.30.**
(g) Proteose, peptone, and gelatin nitrogen, **28.31.**
(h) Meat bases, **28.32.**
(i) Total soluble phosphorus, **28.37.**
(j) Separation of soluble inorganic and organic phosphorus, **28.38.**

(6) The following methods under meat products and similar products were deleted:

(a) Fat, **28.47.**
(b) Ammonia, **28.49.**
(c) Insoluble nitrogen, **28.50.**
(d) Coagulable nitrogen, **28.51.**
(e) Proteoses and gelatin, **28.52.**
(f) Gelatin, **28.53.**
(g) Amino nitrogen, **28.54.**
(h) Acid alcohol-soluble nitrogen, **28.55.**
(i) Nitrates, **28.58.**
(j) Glycerol, **28.59.**
(k) Sugar, **28.60.**

## 29. METALS, OTHER ELEMENTS, AND RESIDUES IN FOODS

(1) The following methods were made official:

(a) Fluorine, **29.22–29.33,** incl.
(b) Lead, **29.34–29.52,** incl.

(2) The following methods were adopted, first action:

(a) DDT.

(b) Cadmium.
(c) Sodium fluoroacetate (1080)

(3) The following methods were made first action:

(a) Arsenic bromate method, **29.6–29.10,** incl.
(b) Copper, **29.11–29.21,** incl.
(c) Tin, **29.64–29.68,** incl.
(d) Zinc, **29.69–29.77,** incl.

(4) The method for mercury, **29.54–29.56,** incl., was deleted.

## 30. NUTS AND NUT PRODUCTS

(1) The following methods, *This Journal,* **32,** 96 (1949) were made first action:

(a) Moisture.
(b) Crude fat.
(c) Crude protein.
(d) Crude fiber.
(e) Ash.
(f) Reducing sugars.
(g) Sucrose.
(h) Sodium chloride.
(i) Water-insoluble organic residue.

(2) The following methods were made first action:

(a) Starch, **30.12.**
(b) Shredded coconut glycerol, **30.18.**

(3) The following were made procedures:

(a) Peanut butter, **30.11.**
(b) Methods for preparation of sample, *This Journal,* **32,** 96 (1949).

## 31. OILS, FATS, AND WAXES

(1) The following methods were adopted as official, as revised:

(a) Unsaponifiable matter, S.P.A. method, **31.40.**
(b) Peanut oil, modified Bellier test, **31.47–31.48.**

(2) The following methods were made first action:

(a) Thiocyanogen number, **31.22–31.23.**
(b) Cholesterol and phytosterol in mixtures of animal and vegetable oils, **31.35 and 31.36.**
(c) Foreign fats containing tristearin in lard, **31.52.**
(d) Coal-tar colors, **31.56–31.57.**

(3) The following methods were made procedures:

(a) Preparation of sample, **31.1.**
(b) Rosin oil, qualitative, **31.44.**
(c) Cold test, **31.49.**
(d) Fish oil and marine animal oils in presence of vegetable oils and in absence of metallic salts, **31.53.**
(e) Mineral oil (qualitative), **31.54.**

(4) The following methods were deleted:

(a) Specific gravity at temperature of boiling water, **31.6–31.7.**
(b) Cottonseed, **31.58–31.70,** incl.

### 32. PRESERVATIVES AND ARTIFICIAL SWEETENERS

(1) The methods for monochloroacetic acid determination and qualitative tests in carbonated beverages, fruit juices, beer, and wine, and in commercial preservatives, *This Journal,* **32,** 97–99 (1949), were adopted as official.

(2) The following methods were made official:

(a) Saccharin qualitative phenol-sulfuric acid test, **32.13.**
(b) Saccharin quantitative phenol rapid method for non-alcoholic beverages, **32.16.**

(3) The following methods were adopted, first action:

(a) Thiourea in oranges and orange juice, Rapid oxidation method, *This Journal,* **32,** 100 (1949).
(b) Thiourea in frozen peaches, *This Journal,* **31,** 102 (1948).
(c) Formaldehyde, chromotropic acid and the Hehner-Fulton tests.
(d) Dulcin, Denigès-Tourron and modified LaParola-Mariani methods.
(e) Saccharin, *This Journal,* **30,** 494 (1947).
(f) Volatile fatty acids, modified to include a chromatographic technic, to replace tentative method, *This Journal,* **31,** 99 (1948).
(g) Quarternary ammonium compounds in eggs.

(4) The qualitative test for thiourea in orange juice, *This Journal,* **31,** 104 (1948), was made a procedure.

(5) The following methods were deleted:

(a) Formaldehyde, **32.19–32.26,** incl.
(b) Beta naphthol, **32.34.**
(c) Abrastol, **32.35–32.36.**
(d) Sucrol or dulcin, **32.37–32.38.**

### 33. SPICES AND OTHER CONDIMENTS

(1) The following methods were adopted as official:

(a) "Permanganate oxidation number" for vinegar, *This Journal,* **32,** 102 (1949), with addition of 2d and 3d full paragraphs, *This Journal,* **32,** 338 (1949).
(b) Gums in mayonnaise and French dressing, **33.57.**

(2) The following methods were made official:

(a) Specific gravity of volatile oil, **33.18.**
(b) Reducing sugars before and after inversion, **33.48–33.49.**
(c) Sucrose, **33.50.**

(3) The following methods were adopted, first action:

(a) Volatile oil in spices, **33.17,** as revised.
(b) Volatile oil and resin in ginger, **33.25,** after deletion of references to optical rotation and acid and ester numbers.
(c) Total fat in mayonnaise and salad dressing, **33.54,** as revised.

(4) The following methods were made first action:

(a) Moisture distillation with toluene, **33.3.**
(b) Eugenol in volatile oil, **33.23.**
(c) Ether extract, **33.37.**
(d) Soluble and insoluble phosphoric acid, **33.64–33.65.**
(e) Polarization, **33.81.**

(5) The following methods were made procedures:

(a) Preparation of sample, **33.1, 33.34, 33.46, 33.59,** and **33.89.**
(b) Microscopic examination, **33.29–33.33,** incl.
(c) Organoleptic examination, **33.58.**

(6) The following methods were deleted:

(a) Moisture drying with heat, **33.2.**
(b) Cold water extract, **33.12.**
(c) Optical rotation of volatile oil, **33.19.**
(d) Acid number volatile oil, **33.21.**
(e) Ester number of volatile oil, **33.22.**
(f) Ketone and aldehyde in volatile oil, **33.24.**
(g) Calculation of composition, **33.55.**
(h) Tartaric acid and tartrates, qualitative, **33.83,**and quantitative, **33.84.**

## 34. SUGAR AND SUGAR PRODUCTS

(1) The following methods were adopted as official:

(a) Lac on confectionery, *This Journal*, **32,** 102 (1949), as revised.
(b) Reducing substances in molasses, *This Journal*, **32,** 103 (1949).
(c) Solids by refractometer, **34.8,** as revised.
(d) Ofner's method for invert sugar in presence of sucrose, **34.47–34.48.**
(e) Free acid in honey, **34.99,** as revised.

(2) The following methods were made official:

(a) Invert sugar, Munson-Walker general method, **34.38.**
(b) Determination of reduced copper by titration with potassium permanganate, **34.43.**
(c) Dextrose, Lane-Eynon general volumetric method, **34.50.**

(3) The following methods were adopted, first action:

(a) Zerban and Martin values for refractive indices of dextrose and invert sugar solution, *This Journal*, **27,** 295 (1944).
(b) Micro method for reducing sugars, **34.63–34.65,** incl., as revised.

(4) The following methods were made first action:

(a) Moisture, direct drying, **34.2.**
(b) Mineral adulterants in ash, **34.16** and **34.73.**
(c) Starch, **34.78.**
(d) Ether extract, continuous extractor method, **34.79,** and Roese-Gottlieb method, **34.80.**
(e) Paraffin, **34.81.**
(f) Polarization, direct, **34.92,** and invert, **34.93.**
(g) Levulose, **34.96.**

(h) Dextrose, **34.97.**

(i) Commercial invert sugar, resorcinol test, **34.102–34.103,** and aniline chloride test, **34.104–34.105.**

(j) Malic acid value, **34.130.**

(k) Sucrose in sugar beets, **34.131–34.132.**

(l) Starch conversion products, **34.133–34.155,** incl.

(5) The following methods were made procedures:

(a) Commercial glucose, **34.31–34.32, 34.77, 34.100, 34.101, 34.121.**

(b) Dextrin (approximate), **34.98.**

(c) Diastase, **34.106.**

(d) Preparation of sample, **34.107-(a) 1 and 2;** and **(b) 1 and 2.**

(6) The following methods were deleted:

(a) Mineral constituents, **34.12** and **34.69.**

(b) Invert sugar II, Scales method, **34.36–34.37;** and IV, Herzfeld gravimetric method, **34.46.**

(c) Metals, **34.84** and **34.115.**

## 35. PROCESSED VEGETABLE PRODUCTS

(1) The following methods were made official:

(a) Insoluble solids, **35.3.**

(b) Soluble solids, **35.4.**

(c) Specific gravity, **35.5.**

(d) Sodium chloride, Rapid method, **35.9.**

(e) Field corn in canned mixtures of field and sweet corn, **35.18.**

(2) Method for catalase, *This Journal,* **30,** 76 (1947), was adopted, first action.

(3) The method for preparation of sample, **35.1,** was made a procedure.

(4) The following methods were deleted:

(a) Volatile fatty acids in vegetable juices, **35.20–35.21.**

(b) Moisture, dried products, **35.22.**

## 36. VITAMINS

(1) The following methods were adopted as official:

(a) Carotene in feedstuffs, *This Journal,* **31,** 111 (1948), as revised.

(b) Thiamine, growth method **36.16–36.23,** incl.; fluorometric method, **36.24–36.26,** incl., as revised; fermentation method, **36.27–36.31,** incl., as revised.

(c) Riboflavin, microbiological method, *This Journal,* **32,** 105 (1949), as revised.

(d) Riboflavin, fluorometric method, *This Journal,* **32,** 108 (1949), as revised.

(e) Nicotinic acid, microbiological method, **32,** 110 (1949), as revised.

(2) The vitamin A, spectrophotometric method, *This Journal,* **32,** 103 (1949), as revised this year, was adopted, first action.

(3) The microbiological method for folic acid, using *S. faecalis* as the test organism, was adopted as first action.

(4) The method for vitamin D in poultry feed supplements, **36.61–36.63,** incl., was adopted, first action.

(5) The sublimation method for nicotinic acid, **36.46,** was deleted.

### 37. WATERS, BRINE, AND SALT

(1) The title of this chapter was changed to "Mineral waters and salt" and captions "Potable water" "Industrial water," "Irrigating water," and "Brine," were deleted.

(2) The following methods were revised and retained as official:

(a) Nitrogen in form of nitrite, II Reduction method, **37.19.**
(b) Iron, colorimetric method **37.59,** and volumetric method, **37.60.**
(c) Phosphoric acid, **37.71.**

(3) The following methods were made official:

(a) Strontium, **37.63.**
(b) Bromide in presence of chloride and iodide, **37.105–37.107,** incl.

(4) A new method was adopted, first action, to replace boric acid, quantitative method, **37.81.**

(5) The following methods were made first action:

(a) Iodide and bromide, **37.77–37.78.**
(b) Bromide in presence of chloride but not iodide, **37.101–37.104,** incl.
(c) Salt, moisture, **37.109;** matters insoluble in water, **37.110;** and matters insoluble in acid, **37.111.**
(d) Sulfate, **37.113.**
(e) Calcium, **37.114.**
(f) Magnesium, **37.115.**

(6) The following were made procedures:

(a) Boric acid qualitative test, **37.81,** as revised.
(b) Reporting results for waters, **37.82.**
(c) Preparation of sample (salt), **37.108.**
(d) Preparation of solution for sulfate, calcium, and magnesium, **37.112.**
(e) Reporting results, **37.119.**

(7) The following methods were deleted:

(a) Turbidity, **37.1–37.2.**
(b) Color, **37.3–37.4.**
(c) Odor, **37.5.**
(d) Suspended matter, **37.9.**
(e) Nitrogen in form of free and albuminoid ammonia, **37.10** (except reagents (a) and (c) placed under **37.18**), and **37.11–37.13,** incl., and **37.48.**
(f) Nitrogen in form of nitrite, **37.14, 37.15, 37.49.**
(g) Oxygen required, **37.27–37.30,** incl.
(h) Dissolved oxygen, **37.31–37.34,** incl.
(i) Lead, **37.35–37.40,** incl.
(j) Copper, **37.41–37.42.**
(k) Zinc, **37.43–37.44.**
(l) Solids in solution, **37.46.**

(m) Ignited residue, **37.47.**
(n) Nitrogen in form of nitrite, **37.50.**
(o) Chloride, **37.51.**
(p) Fluorides, **37.52.**
(q) Free carbon dioxide, **37.55.**
(r) Industrial waters, **37.84–37.97,** incl.
(s) Irrigating waters, **37.98–37.100,** incl.

### 38. RADIOACTIVITY

(1) The method **38.6–38.12,** incl., was revised to eliminate the use of the electroscope constant and the method retained as official.

(2) "Standard deviation" was substituted for "probable error" in **38.15.**

### 39. DRUGS

(1) A new method for ethylmorphine in sirups, in absence of other alkaloids, was adopted as official.

(2) The following methods were made first action:

(a) Acetophenetidin (phenacetin) and salol, acid hydrolysis method, **39.20;** alkaline hydrolysis method, **39.21.**
(b) Aminopyrine, acetophenetidin, phenobarbital, and caffeine, **39.45.**
(c) Arecoline hydrobromide, **39.73.**
(d) Cocaine, method II, **39.78.**
(e) Physostigmine salicylate, tablets, **39.99.**
(f) Alkaloids in ipecac, fluid extract, automatic extractor, **39.121;** hand extraction, **39.122.**
(g) Ipomea and jalap, **39.123–39.124.**
(h) Podophyllum, **39.125.**
(i) Belladonna and stramonium ointments, Methods I and II, **39.126** and **39.127.**
(j) Santonin in santonica, **39.191.**
(k) Quinine (spectrophotometric method), **39.184.**
(l) Guaiacol (from alkoxyl), **39.152–39.154**
(m) The method for quinine ethyl carbonate, *This Journal,* **30,** 464 (1947).
(n) Butacaine sulfate in crystals, tablets, solutions, and ointments containing 2% or more.
(o) Phenolpthalein in chocolate preparations, **39.168–39.170,** as amended.

(3) The qualitative test for aconitine in aconite root (**39.72**) was made a procedure.

(4) The following methods were deleted:

(a) Quinine and strychnine, **39.107.**
(b) Cascara sagrada, **39.113–39.114.**

### 40. MICROBIOLOGICAL METHODS

(1) The following methods were deleted:

(a) Examination of canned fruits and other canned foods, **40.22–40.25,** incl.
(b) Examination of nuts and nut products, **40.26–40.27.**

### 41. MICROCHEMICAL METHODS

(1) A new method for microdetermination of nitrogen was adopted, first action.

(2) Method for methoxyl and ethoxyl groups, **41.1–41.2,** was made official, as revised.

(3) The method for nitrogen, **41.3–41.6,** incl., was deleted.

## 42. EXTRANEOUS MATERIALS IN FOODS AND DRUGS

(1) The title of this chapter was changed to Extraneous Materials: Isolation.

(2) The following methods were adopted, first action:

(a) Stone fragments and decomposed peanuts in coarse peanut butter, *This Journal,* **30,** 102 (1947).

(b) Methods given *This Journal,* **32,** 322–324 (1949) be substituted for **42.26–42.30,** incl., **42.41–42.43,** incl,., **42.54,** and **42.77.**

(c) Insect and rodent filth, apple butter, **42.44,** as revised.

(d) Rot in canned tomatoes, **42.60,** as revised.

(e) Light and heavy filth in ground spices, **42.78** and **42.84,** as revised.

(f) Sediment in fluid milk.

(3) The following were made first action:

(a) Canned citrus juices, mold, **42.5,** fly eggs and maggots, **42.6,** and rodent fragments, **42.7.**

(b) Filth in cocoa, **42.10.**

(c) Filth in butter, **42.21(a).**

(d) Filth in shelled nuts, **42.22.**

(e) Peanut butter filth, **42.23,** water-insoluble residue, **42.24,** light filth, **42.25.**

(f) Insect excreta in flour, **42.31.**

(g) Insects, insect fragments, and rodent hairs in whole corn meal, **42.33.**

(h) Rodent excreta, degerminated corn meal, **42.34.**

(i) Cream corn meal, rodent excreta, **42.35,** insects, insect fragments, and rodent hairs, **42.36.**

(j) Filth in starch, **42.38.**

(k) Dried apples, heavy filth, **42.45,** insects and light filth, **42.46.**

(l) Maggots in blueberries and cherries, **42.50** (*This Journal,* **30,** 96 (1947).

(m) Filth in sugar, **42.55,** and in sirups, **42.56.**

(n) Tomato products, fly eggs and maggots, **42.61,** and insect fragments, **42.62.**

(o) Mold in tomato soup and products containing tomato sauce, **42.64.**

(p) Puréed infant food, mold count, **42.66(a),** and insects and rodent filth, **42.67,** fly eggs and maggots, **42.68.**

(q) Weevils in peas and beans, **42.69.**

(r) Canned greens and broccoli, insects, **42.70,** light filth, **42.71.**

(s) Cinnamon, heavy filth and sand, **42.79,** light filth, **42.80.**

(t) Light filth in turmeric, **42.81.**

(u) Light and heavy filth in onion powder, **42.82,** and in black and white pepper, **42.83.**

(v) Rot based on mold count in spices, **42.85.**

(w) Pepper sauce, light filth, **42.86** and **42.88;** heavy filth, **42.87** and **42.89.**

(x) Filth in mayonnaise and salad dressing, **42.90.**

(y) Filth in whole pickles, **42.91,** and in chopped pickles, **42.92.**

(z) Rodent and insect excreta in condimental seeds, **42.93.**

(aa) Filth in prepared mustard, **42.94;** in whole spices, **42.95.**

(bb) Light filth in tamarind pulp, **42.96.**

(cc) Cloth, urine stains, **42.97**, urease test for urea, **42.98**, xanthydrol test for urea, **42.99**, and extraction of urea and crystallization of urea nitrate, **42.100**.

(4) The following methods were made procedures:

(a) New method for shredded coconut.
(b) Cheese products, filth (quantitative), **42.12**, qualitative, **42.13**.
(c) Dried milk, filth (filtration on paper), **42.15**, filtration on sediment pad, **42.16**.
(d) Filth in cream, **42.17**.
(e) Evaporated and condensed milk, filtration on paper, **42.18**, filtration on sediment pad, **42.19**.
(f) Eggs—frozen or dried, extraneous matter, **42.39**, chicken excrement, **42.40**.
(g) Insects in berries and cherries, **42.48**, in blackberries, **42.49**.
(h) Fruit paste, **42.52** (*This Journal*, **30**, 97 (1947).
(i) Filth in canned mushrooms, **42.74**, dried mushrooms, **42.75**.
(j) Those adopted as tentative since publication of the sixth edition, *Methods of Analysis*, for—
Popcorn.
Mold in Cranberries.
Brewer's Grits.
Canned Fish.
Chicken Giblet Paste.
Meat Scraps.
Chewing Gum.
Shredded Coconut.
Jams and Jellies.
Frozen and Canned Blueberries.
Dried Mushrooms.
Soybean Flour.

(5) The following methods were deleted:

(a) Tea, rodent and insect excreta, **42.8**, insects and insect fragments and hairs, **42.9**.
(b) Dairy products, manure fragments, **42.11**.
(c) Mites, in cheese products (**42.14**).
(d) Filth, evaporated and condensed milk, filtration on sediment pad, **42.21(b)**.
(e) Filth, alimentary pastes, **42.37**.
(f) Rot in canned blackberries and raspberries, **42.47**.
(g) Filth in mince meat, **42.53**.
(h) Tomato products, rot fragments, **42.59**, sand, **42.63**.
(i) Mold in dehydrated tomato products, **42.65**.
(j) Rot in puréed infant foods, **42.66(b)**.
(k) Insects in canned asparagus, **42.72**.
(l) Decomposed material in canned mushrooms, **42.73**.
(m) Insects in Brussels sprouts, **42.76**.

## 43. STANDARD SOLUTIONS

(1) The following methods were adopted as official:

(a) Sulfuric acid, standard borax, **43.14–43.15**.
(b) Bromide-bromate, *This Journal*, **31**, 119 (1948).
(c) The reagent **43.10(d)** was revised in the method for hydrochloric acid.
(d) The method for sodium thiosulfate **43.29**, was revised.

(2) The following methods were adopted, first action:

(a) A new method for potassium dichromate.
(b) Methods for preparation of buffer solutions.

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## REPORT OF REPRESENTATIVE ON THE BOARD OF GOVERNORS OF THE CROP PROTECTION INSTITUTE

The Institute has continued to serve as a liaison agency between the research staffs of the Agricultural Experiment Stations and the manufacturers of chemicals in the testing and screening of experimental materials. Two of those on the Board are Directors of Experiment Stations. The office of the Board and the laboratory and greenhouse facilities are provided in the New Hampshire Experiment Station at Durham. The investigations are dispersed through allocations to the various appropriate experiment stations, and the appointees to fellowships upon problems become residents there and subject to the direction and collaboration of personnel of the appropriate research departments. The operations of the Institute are helpful to the personnel of the State institutions, to the ethical manufacturer, and to the users of chemical materials in agricultural practices.

Your representative participated in a recent meeting of the Board and in a subsequent testimonial dinner at which the Board's Chairman, Doctor W. C. O'Kane, was lauded by the administration of the University of New Hampshire and presented with a multi-autographed plaque in recognition of his distinguished service through the operation of the Institute.

It is hoped that this useful and effective agency will continue to function as a channel through which the findings obtained by research workers can be implemented into beneficial practice.

Approved.

W. H. MacIntire

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## REPORT OF THE SECRETARY-TREASURER

By Henry A. Lepper

The Executive Committee met in the Board Room of the Cosmos Club Sunday, October 9, 1949. All members were present. The audit by John Bisselle and Company was presented and accepted.

# ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, INC.

## BALANCE SHEET—SEPTEMBER 30, 1949

### ASSETS

| | | |
|---|---|---|
| *Current Assets:* | | |
| Cash, Lincoln National Bank | $7,842.83 | |
| Office cash fund | 20.66 | $ 7,863.49 |
| Accounts receivable | $2,280.60 | |
| Less reserve for doubtful accounts | 51.30 | 2,229.30 |
| Accrued interest receivable, Government bonds | | 250.00 |
| Inventories | | 7,047.20 |
| *Total Current Assets* | | $17,389.99 |
| *Fixed Assets:* | | |
| Furniture and fixtures | | 756.01 |
| *Investments* | | 53,292.00 |
| *Total Assets* | | $71,438.00 |

### SURPLUS

| | |
|---|---|
| *Balance, October 1, 1948* | $74,777.37 |
| Less: Net *loss* for the fiscal year ended September 30, 1949 | *3,339.37* |
| *Balance, September 3, 1949* | $71,438.00 |

The balance sheet for the last fiscal year showed a loss. The expenses of the Association were increased over the previous year because of higher printing costs, increases in salaries, and additional clerical and editorial help. These, together with the decline in the sale of *Methods of Analysis*, resulted in the loss indicated. It was to be expected that the demand for the sixth edition would lessen with the contemplated publication of the seventh edition in 1950. The deficit is chargeable to the publication of our *Journal*. While the Executive Committee considers it desirable to continue to support the *Journal*, in part, from the general funds of the Association, for the reasons set forth at last year's meeting, *This Journal*, **32**, 66 (1949), it was felt that the advance in costs should be met to some extent by an increase in the subscription price. Accordingly, it is recommended that this be set at $7.50 domestic and $8.00 foreign per year, postage prepaid, with a 20 per cent discount to members.

The Secretary was authorized to hire additional office help when necessary and additional editorial help with the preparation of the manuscript for the seventh edition of *Methods of Analysis*. The secretary was further authorized to contract for the printing of the seventh edition and its sale price set at not less than $7.50, with the same discounts as allowed

on previous editions. The furnishing of reprints of the changes in methods, adopted in the interim between editions of the book of methods, was approved, with the cost of printing and mailing to be borne by such purchasers of the book as desire the service. The expenditure of $170 for a new typewriter was authorized.

Dr. W. F. Reindollar was reappointed Chairman of the Committee on Recommendations of Referees. The secretary was instructed to arrange the time of the next meeting in cooperation with the other associations holding meetings in consecutive sessions with the A.O.A.C., so as to confine the series of meetings within one week. By shortening the meeting of the A.O.A.C. to two and one-half days instead of three days, and that of the Association of American Feed Control Officials to one and one-half days instead of two, it would be possible to have the meetings of these two associations and those of the Associations of American Fertilizer Control Officials and the Economic Poisons Control Officials meet beginning Monday and ending on Saturday. Accordingly, the meeting of the A.O.A.C. has been set for October 2, 3, and 4, 1950, at the Shoreham Hotel in Washington, D. C.

The re-establishment of the custom of having a guest speaker on a subject of general interest was approved with authorization to pay travelling and other expense, where necessary.

Approved.

## REPORT OF THE COMMITTEE ON NECROLOGY

### Martin Boyle

Mr. Boyle was born in Galway, Ireland, January 20, 1876; he died on February 25, 1949. Mr. Boyle received his B.S. degree in Chemistry from Massachusetts Institute of Technology, 1898, and L.L.B. from Georgetown University in 1913. He worked 6 years as a chemist for the Towle Manufacturing Co., Newburyport, Mass., and on July 12, 1904, joined the old Bureau of Chemistry, U.S.D.A., where he served as a chemist on various investigations on foods until he was made an administrative officer of the Food and Drug Administration. On June 30, 1942, he went on optional retirement.

### Sam Byall

Sam Byall, who served for nearly 20 years as a chemist in the Bureau of Agricultural and Industrial Chemistry of the U. S. Department of Agriculture, died on December 27, 1948. Following graduation from the A. and M. College of Mississippi in 1911, he served as a chemist in feed and fertilizer control work at the Louisiana State Agricultural Experiment Station until 1920, except for a year spent in the Armed Forces during

World War I. During the interval between 1920 and his entry into the Federal service, Mr. Byall was engaged in industrial work largely in sugar technology. His entire career in the Department was spent in sugar chemistry research. He was engaged in investigations on the cultural, technological, and chemical factors which influence the composition and quality of beet and cane sugars and other products. In 1943 he was reassigned from the Washington laboratories to the Southern Regional Research Laboratory at New Orleans. For a considerable period both before and after the reassignment Mr. Byall was associated with the development work on sugars and syrups at the Meridian Mississippi Field Station. In the interval between July 1945 and his death, he was in charge of the Station. He was an author of over 40 articles and reports dealing with sugar chemistry and technology.

Mr. Byall was born in Baltimore, Maryland, on October 6, 1890, and moved to Mississippi at an early age. His career was cut short by his untimely death.

### Jehiel Davidson

Jehiel Davidson, chemist in various Bureaus of the U. S. Department of Agriculture for 31 years, died on January 11, 1948 in Washington, D. C. Much of his career was devoted to studies on wheat. His earlier contributions related to the influence of soil and climate on the protein content of wheat. These were followed by studies on types of wheat suited for the making of macaroni along with improvements in procedures. Much time in later years was devoted to quality characteristics of wheat in relation to bread making. In addition he maintained a wide interest in the biology of plant life and soil and nutrient factors. Noteworthy were his fundamental studies on the culturing of wheat seedlings in nutrient solutions to determine the influence of different nutrients on plant growth.

Dr. Davidson was born in Russia on October 13, 1875. He came to the United States at an early age and received most of his formal education in this country. He received his B.S. degree at Cornell University in 1911 and his PhD. in 1914. His entire professional career up to retirement in January 1945 was spent in the Department of Agriculture.

### Benjamin R. Hart

Mr. Hart, a native of Kentucky, received his B.S. and M.S. degrees from the State University of Kentucky. He died at his home near Lexington, Kentucky, on July 7, 1949. Mr. Hart was appointed as a Food Chemist in the Bureau of Chemistry in 1907, serving in Washington and in Cincinnati, later as Chief of Western District; and was Chief of Eastern District of the Food and Drug Administration when he resigned in 1920. He later worked in the Foodstuffs Division, U. S. Department of Commerce, and in private industry, until ill health forced his retirement.

WILLIAM VANARSDALL LINDER

Mr. Linder was born March 12, 1881, and died May 29, 1949. He graduated from Wabash College, Indiana, with B.S. in Chemistry in 1906, and from Ohio State in 1907 with M.S. in Chemistry. He was appointed in the Treasury Department on July 16, 1907, and at the time of his death was Head, Chemistry Div., Alcohol Tax Unit, Bureau of Internal Revenue. Mr. Linder was a faithful member of A.O.A.C. attending most of its meetings since 1907, and a member of A.C.S. for 42 years. He was a recognized authority on distilled liquors, denaturants, oleomargarine, and butter. He was deeply respected for his knowledge and much beloved for his fine character.

CLIFFORD HANKS ROBINSON

Clifford Hanks Robinson, B.A., F.C.I.C. Dominion Agricultural Chemist, Science Service, Department of Agriculture, died April 11, 1949, at the Civic Hospital, Ottawa. He had been in poor health for some months and suffered a stroke on February 22, from which he did not recover.

Mr. Robinson was born in Lambton County, Ontario, and matriculated from Dutton High School. Even at that time he demonstrated high scholastic ability, winning the Sir William Mulock scholarship in mathematics and science. He entered the University of Toronto in 1905 and became an ardent disciple of the late Professor Lash Miller. He graduated in 1909 with a bachelor of arts degree with first class honours in chemistry.

Mr. Robinson accepted a position as assistant chemist in the Division of Chemistry at the Central Experimental Farm, Ottawa, and in 1914 became chief assistant to Dr. F. T. Shutt, Dominion Chemist. On the retirement of Dr. Shutt and in 1933, he was appointed Acting Dominion Agricultural Chemist and was confirmed in that position in 1935.

He was a leading authority on agricultural chemistry in Canada. Under his direction the activities of the Division of Chemistry expanded rapidly until at the present time they include research in animal nutrition, plant chemistry, food chemistry, soils and fertilizers, pesticides, vitamins, and animal physiology, carried out at the central laboratories at Ottawa and in branch laboratories at Kentville, N. S., Summerland and Saanichton, B. C.

LUCIUS M. TOLMAN

Mr. Tolman, retired director of research at Wilson and Co., Inc., Chicago, died on April 29, 1949, at 74 years. After graduation from Pomona College in 1896 and the University of California in 1898, he entered the Government service and by 1907 was Chief of the Food Inspection Laboratories of the U. S. Department of Agriculture. Mr. Tolman went to Wilson and Co. as Chief Chemist in 1918 and rose through various execu-

tive positions to head of the research and technical departments of that firm. In 1901, he joined A.C.S., in 1910 was President of the Washington Sec. and later of the Chicago Sec. He also was President of the American Oil Chemists Society and Chairman of the Committee on Scientific Research of the American Meat Institute.

V. E. MUNSEY, *Chairman*
W. R. ELLIS
O. W. FORD

## REPORT OF THE COMMITTEE ON NOMINATIONS

Your committee proposes the following nominees and moves their election to the respective offices, as designated:

*President,* W. A. Queen, Food and Drug Administration, Washington, D. C.

*Vice-President,* H. A. Halverson, St. Paul, Minnesota.

*Secretary-Treasurer,* Henry A. Lepper, Food and Drug Administration, Washington, D. C.

As additional members of the Executive Committee: W. B. White, Washington, D. C.; H. J. Fisher, New Haven, Connecticut; E. L. Griffin, U. S. Department of Agriculture, Washington, D. C.; L. S. Walker, Past President, Burlington, Vermont.

Approved.

W. H. MACINTIRE, *Chairman*
R. C. BERRY
GUY G. FRARY

## REPORT OF THE COMMITTEE ON RESOLUTIONS

*Whereas,* the officers of our Association have capably and efficiently performed their duties during the past year and have carefully planned and successfully conducted this the 63rd Annual Meeting at the close of the 65th year of our organization, therefore be it

*Resolved,* that we express our appreciation and thanks to President L. S. Walker, Vice-President W. A. Queen, and Secretary-Treasurer Henry A. Lepper and to the members of the Executive Committee for their able service.

*Whereas,* the accurate and prompt dissemination of knowledge concerning progress in methodology is one of the principal duties of this organization, therefore be it

*Resolved,* that we express our commendation of the able work done by W. B. White, and the Editorial Board of the *Journal* and to H. J. Fisher and his co-workers in the revision of the Book of Methods.

*Whereas,* the progress of the work of this Association on research in

methodology depends very largely upon the effective services of the members of the Committee on Recommendations of Referees, our Referees, Associate Referees, and collaborators, therefore be it

*Resolved*, that this Association express its appreciation to these workers who have given so generously of their time and efforts toward the accomplishment of the aims of this organization.

*Whereas*, the registration at this meeting indicates the presence of representatives of numerous and widely varied State and Federal agencies and institutions in all parts of the nation and of Canada, therefore be it

*Resolved*, that we hereby express our thanks to those agencies who have made possible the attendance of so many individuals who have contributed much to the success of our meeting.

*Whereas*, the success of any meeting such as this is in considerable degree dependent upon the availability of ample and satisfactory meeting places, therefore be it.

*Resolved*, that the Secretary express to the management of the Shoreham Hotel the thanks of our Association for making available rooms and other facilities suitable for the successful conduct of this meeting.

Approved.

J. F. FUDGE, *Chairman*
R. C. BERRY
G. H. MARSH

## CONTRIBUTED PAPERS

# BOILING TEMPERATURES OF KJELDAHL DIGESTION MIXTURES*

By C. L. Ogg and C. O. Willits
(Eastern Regional Research Laboratory[1])

The widely divergent results obtained in the 1948 collaborative studies of the micro Kjeldahl procedure emphasized that inherent errors existed in the methods generally used. A report of work at this Laboratory (1)

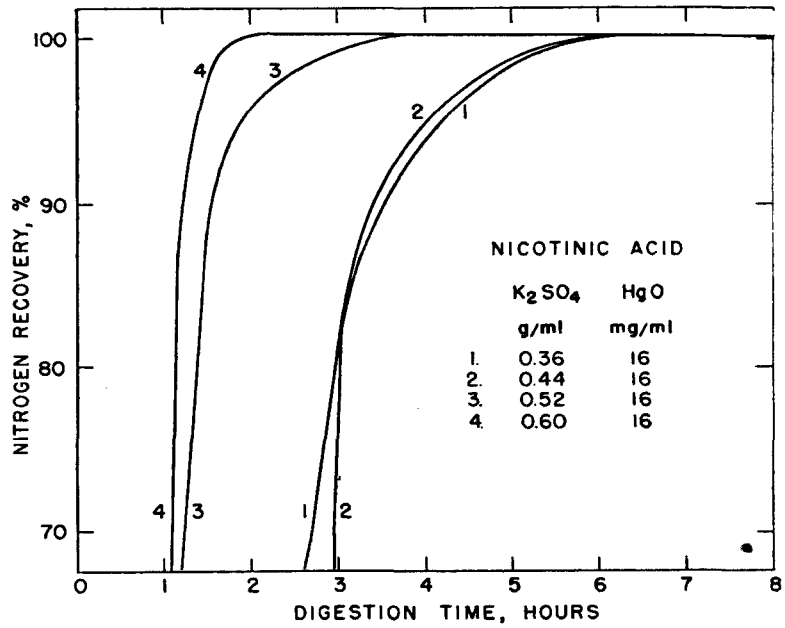


FIGURE 1

FIG. 1.—Effect of Time of Digestion and Potassium Sulfate Concentration on Recovery of Nitrogen.

indicated that not only was the salt concentration critical but also that the required time of digestion and the amount of salt were closely related (Figure 1). As a result, the 1949 collaborators were instructed to use a high

* Presented at the Annual Meeting of the Association of Official Agricultural Chemists, held at Washington, D. C., October 10, 11, and 12, 1949.

[1] One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, United States Department of Agriculture.

concentration of potassium sulfate (0.625 g. per ml.). Some, however, still did not obtain the desired results. On investigation it was found that some of these collaborators had misinterpreted the instructions for boiling the digestion mixture. In most instances, those who obtained the low nitrogen values used a lower digestion temperature than that specified. This was evident because (a) the digestion mixture did not *boil vigorously* and (b) the acid did not distill up into the neck of the flask. When the heat was

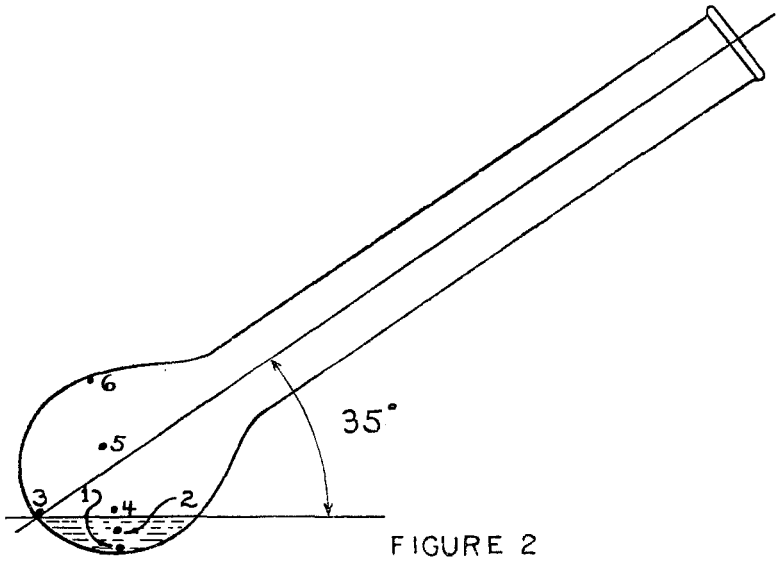


FIG. 2.—Locations at which Temperatures were Determined.

increased enough to produce these conditions, good results were obtained indicating that the temperature of the digestion mixture was important and that its variation, resulting from differences in salt concentration as well as in the amount of heat applied, was the cause of many erratic nitrogen results. Consequently, a study was made of the boiling temperatures of several digestion mixtures having the potassium sulfate concentration range normally used.

*Procedure.*—Temperature measurements were made in eight solutions; six were heated on a micro gas digestion rack, one on a micro, and one on a macro, electrically heated rack. These measurements were made only when the digestion mixtures were boiling vigorously enough to cause the sulfuric acid to distill at least one-third of the way up the neck of the flask.

A platinum-platinum 10 per cent rhodium thermocouple with a bead slightly larger than a pinhead was used to make spot temperature measurements at the six locations shown in Figure 2. The thermocouple wires

were separated by a porcelain rod insulator, which also served as a handle to place the thermocouple bead in the desired location. The thermocouple was checked against a calibrated 360°C. thermometer.

Table 1 shows the temperatures at the locations indicated in Figure 2. Although the concentration of potassium sulfate ranged from 0.25 to 0.875 g. per ml. of sulfuric acid, the three concentrations of greatest interest were 0.375, 0.50, and 0.625 g./ml., since they cover the range of concentrations more commonly used. These three concentrations correspond closely to 9, 12, and 15 g. of potassium sulfate per 25 ml. of sulfuric acid.

If the temperature differences of the solutions (location 2) are compared with the digestion rate curves of nicotinic acid (Figure 1), it will be noted that the rate of digestion as shown by minimum digestion times is roughly

TABLE 1.—*Boiling temperatures of Kjeldahl digestion mixtures*

Temperature, °C.

Grams of $K_2SO_4$ per ml. $H_2SO_4$

| LOCATION | 0.250 | 0.375 | 0.500 | 0.625 | | | 0.750 | 0.875 |
|---|---|---|---|---|---|---|---|---|
| 1 | 334* | 342* | 345* | 353* | 356† | 356‡ | 360* | 365* |
| 2 | 332 | 336 | 342 | 349 | 349 | 349 | 354 | 358 |
| 3 | 332 | 336 | 342 | 349 | 349 | 348 | 353 | 354 |
| 4 | 327 | 333 | 333 | 342 | 344 | 345 | 345 | 348 |
| 5 | 315 | 325 | 318 | 334 | 341 | 338 | 323 | 339 |
| 6 | 265 | 270 | 272 | 282 | 288 | 285 | 278 | 283 |

* Readings made in 30-ml. Kjeldahl flasks on micro gas digestion rack.
† Readings made in 500-ml. Kjeldahl flask on macro electric digestion rack.
‡ Readings made in 30-ml. Kjeldahl flask on micro electric digestion rack.

doubled for each 10°C. increase in temperature. For example, the minimum time for complete digestion for potassium sulfate concentrations of 0.60 and 0.36 g. per ml. was 2 and 6 hours, respectively, and the difference between the boiling temperatures of the two corresponding solutions, 0.625 and 0.375 g. per ml. was 13°C.

Since the reaction rate appears to be so dependent on temperature, the amount of potassium sulfate and the digestion time are critical, especially when analyzing refractory nitrogenous materials. The amount of potassium sulfate and time of digestion usually used probably exceed the critical limits for easily decomposed materials such as amides and amines. However, when the collaborators studying the micro Kjeldahl method in 1948 were asked to analyze nicotinic acid, their results (2) ranged from less than 1 to 11.5 per cent nitrogen (theoretical value, 11.38 per cent).

In the light of the data presented here, this great discrepancy of results may be attributed principally to the boiling temperatures of the different digestion mixtures or to the use of insufficient heat. In all cases, the excess water must be distilled from the flask and the remaining solution boiled

briskly. In the temperature range of the boiling points of these different mixtures, the minimum time of digestion varies inversely with the temperature, that is, the higher the boiling temperature the shorter the digestion time. Apparently the time can be further shortened by superheating the layer of digestion mixture in contact with the walls of the flask. The minimum digestion time for nicotinic acid by the macro procedure was 2 hours, whereas 3 hours were required when using the micro gas heated rack. The temperatures of the boiling mixtures were the same (Table 1, location 2, columns 4 and 5), but the temperature at the bottom of the electrically heated macro flask (location 1,) was 7°C. higher than that of the liquid (location 2); the temperature difference between these locations in the gas-heated micro flask was only 4°.

LITERATURE CITED

(1) WILLITS, C. O., COE, M. R., and OGG, C. L., *This Journal*, **32**, 118 (1949).
(2) WILLITS, C. O., and OGG, C. L., *Ibid.*, **31**, 565 (1948).

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# THE DETERMINATION OF PROPYLENE GLYCOL IN VANILLA EXTRACTS*

By CHARLES F. BRUENING (Food and Drug Administration, Federal Security Agency, Baltimore, Maryland)†

Propylene glycol is used in many preparations in the food, drug, and cosmetic fields, including imitation vanilla extracts. At present, satisfactory methods for the determination of propylene glycol in vanilla extract are not found in the literature. The usual separation or isolation of propylene glycol by direct distillation is unsatisfactory because of poor recoveries and charring of the sugar in these preparations. Extraction procedures have been unsuccessful because of poor separation from other interfering ingredients and difficulties in recovering propylene glycol due to its volatility.

The method developed depends on the isolation of propylene glycol from the extract by co-distillation with an organic solvent such as heptane. The sample is distilled with the organic solvent, the distillate collected in a modified Dean and Stark water receiver, and the propylene glycol in the distillate determined by the periodate method (1). The method used is based on observations made by Newburger (2) who, in determining water in a sunburn preventive cream by a xylene distillation, reported that propylene glycol also distilled over with the water and xy-

* Presented at the Annual Meeting of the Association of of Official Agricultural Chemists, held at Washington, D.C., October 10–12, 1949.
† Present address, Food and Drug Association, Chicago, Ill.

lene; and he also noted that the substitution of a lower boiling liquid, such as heptane, did not prevent the distillation of propylene glycol with the water. Metayer and co-workers (3), in a study of co-distillation applicable to the analysis of glycols, showed that propylene glycol could be distilled over only partially with benzene, xylene, and toluene, but that cyclohexane, methycyclohexane, and dimethylcyclohexane distilled propylene glycol quantitatively. Tetralin and decalin gave quantitative recoveries for both propylene glycol and glycerol.

In this work the pure glycol or a mixture of glycols was placed in the distilling flask with 40 to 50 times the volume of the suitable solvent, heated, and the condensed vapors collected in a Dean and Stark receiver. The amount of glycol recovered was ascertained by determining the volume, specific gravity, and refractive index of the layer containing the glycols. In the present work heptane was selected because it boils at a comparatively low temperature, 96°–100°C., and thus decomposition of sugar and other ingredients in extracts does not occur; it is very efficient in the codistillation of propylene glycol; it holds many volatile substances in solution in the upper layer in the receiver, thus lessening the contamination of the glycol-water layer; and although small amounts of glycerol codistill with the heptane, the method provides a correction for this contaminant. Alcohol does not interfere. Glycols, other than propylene glycol and glycerol, are poisonous and are not considered here since this paper deals only with unadulterated extracts. The method, although designed for vanilla extracts, is generally applicable, with suitable modification, to the determination of propylene glycol in many food, drug, and cosmetic preparations.

## METHOD

### APPARATUS

Modified Dean and Stark type distilling tube receiver of 10 ml capacity. The apparatus used was similar to that described in the Book of Methods (4) except it had two ground glass joints, one fitting into a straight tube condenser and the other into a 500-ml Erlenmeyer flask with interchangeable joint. It also had a glass stopcock at the bottom of the receiver tube which permitted the distilled liquid to be readily transferred to a volumetric flask. A suitable 10 ml receiver can be purchased.[1]

### REAGENTS

*Heptane.*—Boiling Point 96°–100°C.[2]

*Sand.*—Washed 50 mesh.

*Bromcresol purple indicator.*—Dissolve 0.1 gram of indicator in 100 ml of alcohol.

*Potassium Periodate .02 M.*—Dissolve 4.6 grams $KIO_4$ in ca 500 ml hot water. Dilute to ca 900 ml with water, cool to room temperature, and make to 1000 ml.

*Potassium Arsenite .02 N.*—Dilute 1 volume of USP 0.1 *N* potassium arsenite to 5 volumes.

*Sodium hydroxide.*—0.02 *N.*

[1] Ace Glass Co., catalog 40, #7745 is satisfactory.
[2] Obtainable from Eastman or Phillips Petroleum Co.

### PROCEDURE

Place 10 ml of sample (or smaller volume if necessary to insure that not more than 2.0 g of propylene glycol is taken, adding water if necessary to make the total volume 10 ml) in a 500 ml Erlenmeyer flask and add a few glass beads. If the sample contains much sugar or other solid material add 20 g of washed sand. Add 50 ml of heptane and connect flask to the distilling tube of the receiver which has previously been connected to the condenser. Heat, using a variable heat hot plate, and distil at such a rate that a continuous stream of distillate flows from the condenser tip, and 6–8 ml of aqueous distillate comes over in 10 min. after boiling begins. Distil 3 hours and permit distilled liquid to cool.

Open stopcock and transfer aqueous and heptane layers to a 250-ml volumetric flask. Wash distilling tube receiver by pouring successive 10-ml portions of water down condenser, transferring each portion to the volumetric flask. Make aqueous soln in flask to volume with water, shake contents, allow layers to separate, pipette off upper heptane layer and discard.

To an aliquot of the aqueous soln containing not more than 45 mg of propylene glycol (or its equivalent if glycerol is present) add 35 ml of .02 *M* $KIO_4$ soln, make up to ca 100 ml and permit soln to stand 1 hour. Add ca 1.0 g of $NaHCO_3$, 0.5 g of KI, and 5 ml of starch soln. Titrate immediately with the .02 *N* $KAsO_2$ soln to the disappearance of the blue color.

Standardize 35 ml of the .02 *M* $KIO_4$ by the same titration procedure. The difference between the two titrations represents the amount of periodate reduced.

1 ml of .02 *N* $KAsO_2$ = 0.76 mg propylene glycol
1 ml of .02 *N* $KAsO_2$ = 0.46 mg glycerol

If the original sample contains glycerol, proceed as follows:

Distil 8 hours and determine the periodate consumption as directed above. To another aliquot of the same volume add a drop of bromcresol purple indicator and add .02 *N* sodium hydroxide until a light purple-colored soln is obtained. Add 35 ml of .02 *M* $KIO_4$ soln, make up to ca 100 ml, and allow soln to stand 1 hour. Then add 10 drops of propylene glycol (ca 0.5 ml), mix well, wash down sides of flask with water, and allow to stand for 10 min. Add 3 drops of indicator and titrate with .02 *N* sodium hydroxide to a light purple end point.

1 ml of .02 *N* NaOH = 1.84 mg glycerol
Propylene glycol = [ml of .02 *N* $KAsO_2$ − (4 × ml .02 *N* NaOH)] × 0.76 mg

## EXPERIMENTAL

In the attempt to determine the accuracy of the above method an aqueous solution containing approximately 1 g of propylene glycol per

TABLE 1.—*Recovery of propylene glycol occurring alone in aqueous solutions*

| TIME OF DISTILLATION | PROPYLENE GLYCOL | | |
|---|---|---|---|
| | TAKEN | FOUND | RECOVERY |
| (*Hrs.*) | (*g*) | (*g*) | *per cent* |
| 1 | 0.990 | 0.99 | 100 |
| 1 | 0.990 | 0.99 | 100 |
| 2 | 0.990 | 0.98 | 99 |
| 3 | 0.990 | 0.98 | 99 |
| 4 | 0.990 | 0.98 | 99 |

10 ml was distilled for varying periods of time. The results are given in Table 1. At the same time a 10-ml portion of water was also distilled for 1 hour and treated with periodate; no periodate was consumed, indicating that the practical heptane used introduced no ingredients oxidizable by periodate.

The results show that almost theoretical recovery of propylene glycol was obtained in the one hour heating period on propylene glycol in aqueous solutions. As the time of distillation was increased recoveries remained constant.

It was anticipated that glycerol, which may occur in vanilla extracts with propylene glycol, might cause interference with the method. Accordingly, 10 ml aliquots of a solution containing approximately 1 g of glycerol were distilled for varying periods of time. The amount of glycerol distilled over was determined by measuring the amount of periodate consumed in oxidizing the glycerol. The results are shown in Table 2.

TABLE 2.—*The distillation of glycerol from aqueous solution*

| TIME OF DISTILLATION | GLYCEROL | | |
|---|---|---|---|
| | TAKEN | FOUND | RECOVERY |
| (*Hrs.*) | (*g*) | (*g*) | *per cent* |
| 1 | 1.028 | .035 | 3.4 |
| 2 | 1.028 | .059 | 5.7 |
| 3 | 1.028 | .082 | 8.0 |

The recoveries obtained indicate that glycerol distills over slowly and recoveries are proportional to the time of distillation. Experimentation with a second distilling apparatus showed that the recovery for a given period varied with the two sets of apparatus. Thus it is difficult to predict the amount of glycerol that will distill over from a given sample and apparatus.

Though glycerol comes over in variable amounts, propylene glycol distils quantitatively, and the amount of propylene glycol in any distillate can be determined after correcting for the amount of glycerol in the same distillate. Application was made of the method of Newburger and Bruening (5) for the estimation of glycerol in the presence of propylene and ethylene glycols with periodate. With this reagent glycerol yields formic acid, formaldehyde and water, while propylene glycol yields formaldehyde, acetaldehyde and water. Thus in the oxidation mixture formic acid can be titrated with standard alkali to determine the amount of glycerol. The periodate required to oxidize this amount of glycerol can then be calculated. If this amount of periodate is subtracted from the periodate required to oxidize both glycols, the difference represents the amount of periodate required to oxidize only the propylene glycol. Presumably,

propylene glycol could also be determined directly by ascertaining the amount of acetaldehyde formed in the oxidation since glycerol does not give acetaldehyde on oxidation.

To determine the accuracy of the method for propylene glycol in vanilla extracts when no other glycol is present, four typical imitation vanilla extracts were prepared. All contained many of the ingredients common to these extracts. The formula given below lists the ingredients in all the extracts; the amount of propylene glycol was varied so that the four extracts contained approximately 5, 10, 15, and 20 g per 100 ml, respectively. The exact amount in any extract can be obtained from Table 3 since 10 ml samples were taken in all determinations.

*Composition of Imitation Vanilla Extract with Propylene Glycol*

| | | |
|---|---|---|
| Vanillin | | .75 g |
| Coumarin | | .075 g |
| Alcohol (Anhydrous) | | 20 ml |
| Sugar | | 5 g |
| Caramel | | .5 g |
| Propylene glycol | | 5, 10, 15 or 20 g |
| Water q.s. | to | 100 ml |

In addition, four extracts containing both propylene glycol and glycerol were prepared that contained the same ingredients in the same quantities as listed above except that all these preparations contained approximately 10 g per 100 ml of glycerol, and the alcohol content was reduced to 10 per cent. The exact amount of propylene glycol and glycerol in any extract can be obtained from Table 4.

The results obtained on the four extracts containing propylene glycol without glycerol are shown in Table 3. With the extract containing approximately 10 g of propylene glycol per 100 ml results for the one and two hour distilling periods are somewhat low, but almost quantitative recoveries were obtained in three hours. With the extracts containing 15 and 20 g of propylene glycol per 100 ml satisfactory results were obtained.

The results obtained on the extract containing 5 g of propylene glycol per 100 ml are somewhat low. Apparently the presence of sugar and other solid material retards the distillation of propylene glycol. In this preparation we have water soluble solids to the extent of 5 g of sugar and 0.5 g of caramel per 100 ml. When the total, 5.5 g, is compared to the amount of propylene glycol 5 g per 100 ml it is noted that the ratio is approximately 1 to 1. Various attempts were made to increase this recovery when the solids and propylene glycol occurred in a ratio of 1 to 1, but more nearly quantitative recoveries could not be obtained. The use of sand spreads out the solid material in comparatively thin layers and permits the more rapid removal of propylene glycol. Longer distillation periods did not improve the recovery to any appreciable extent. Generally, propylene glycol distills over in large quantities in the first hour and longer boiling is only

TABLE 3.—*Recovery of propylene glycol in imitation vanilla extract*

| TIME OF DISTILLATION | PROPYLENE GLYCOL | | |
|---|---|---|---|
| | TAKEN | FOUND | RECOVERY |
| (*Hrs.*) | (*g*) | (*g*) | *per cent* |
| 1 | 1.007 | .97 | 96 |
| 1 | 1.002 | .98 | 98 |
| 2 | 1.002 | .97 | 97 |
| 2 | 1.002 | .97 | 97 |
| 3 | 1.002 | .99 | 99 |
| 3 | 1.002 | 1.00 | 100 |
| 3 | 2.006 | 1.98 | 99 |
| 3 | 1.510 | 1.48 | 98 |
| 3 | .501 | .48 | 96 |

needed to remove the propylene glycol entrapped in the solid material or in various parts of the apparatus. It is possible that the apparatus is responsible for low recovery through condenser losses or in other ways. Study is contemplated on this problem in an attempt to increase the recovery, but for most purposes the recovery obtained may be satisfactory.

These results in general indicate that none of the ingredients listed cause interference with the method. Alcohol distills over but does not interfere with the method because it is not oxidized by periodate. The vanillin and coumarin that may volatilize are held in the heptane layer and do not interfere. Generally no charring of the sugar takes place which would result in the formation of sugar decomposition products which presumably might interfere with the periodate oxidation.

The results obtained on the four extracts containing both propylene glycol and glycerol are shown in Table 4. The recoveries for a three hour distillation period are slightly lower than for the eight hour period. As indicated by the results, as the time of distillation is increased from three

TABLE 4.—*Recovery of propylene glycol in imitation vanilla extract when occurring with glycerol*

| TIME OF DISTILLATION | GLYCEROL | | | PROPYLENE GLYCOL | | |
|---|---|---|---|---|---|---|
| | TAKEN | FOUND | RECOVERY | TAKEN | FOUND | RECOVERY |
| (*Hrs.*) | (*g*) | (*g*) | *per cent* | (*g*) | (*g*) | *per cent* |
| 3 | 1.003 | .091 | 9.1 | 1.002 | .95 | 95 |
| 3 | 1.003 | .100 | 10.0 | 1.002 | .96 | 96 |
| 8 | 1.003 | .227 | 22.6 | 1.002 | 1.00 | 100 |
| 8 | 1.003 | .241 | 24.0 | 1.002 | .98 | 98 |
| 8 | 1.001 | .213 | 21.3 | 2.006 | 1.98 | 99 |
| 8 | 1.001 | .250 | 25.0 | 1.510 | 1.49 | 99 |
| 8 | 1.001 | .273 | 27.3 | .501 | .49 | 98 |

to eight hours more glycerol distils over. This is not significant in the determination of the propylene glycol content of the extract because the correction for glycerol is made on each individual distillate. However, the longer time seems desirable because the propylene glycol recoveries are higher and more closely approach the theoretical amount present. In general, all the results are considered satisfactory for most analytical purposes.

## CONCLUSION

A method is proposed for the determination of propylene glycol in imitation vanilla extract when occurring alone or with glycerol. The method involves a codistillation of propylene glycol with heptane at a relatively low boiling point. The amount of propylene glycol is determined in the aqueous distillate using the periodate method.

When glycerol is present it is determined by an alkalimetric determination of the formic acid resulting from periodate exudation of glycerol, and the total periodate consumption is corrected for this glycerol before the propylene glycol is calculated.

When typical extracts of vanilla containing measured amounts of propylene glycol alone and with glycerol were analyzed, generally satisfactory recoveries of propylene glycol were obtained.

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# STUDIES ON THE RECOVERY OF INSECTS AND INSECT PARTS FROM FIG PASTE BY CERTAIN PROCEDURES*

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During the course of some recent work on the recovery of insects and insect fragments from fruit concentrates and certain fruit pastes, using the procedure outlined in the A.O.A.C. method for "Light Filth in Fruit Pastes" (**42.52**) (1) it was found that not all of the insect parts present in the product were recovered in the castor oil layer. Results of experiments conducted with commercially ground fig paste indicate that castor

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TABLE 1.—*Insects parts recovered, using method* **42.52** *followed by one trapping with gasoline*

100 gram samples examined

| SAMPLE NO. | TOTAL WHOLE INSECTS, LARVAE & HEADS (EXCL. OF VINEGAR FLIES) | | TOTAL INSECT & LARVAE PIECES AND PARTS | | VINEGAR FLY LARVAE | |
|---|---|---|---|---|---|---|
| | *Oil* | *Gasoline* | *Oil* | *Gasoline* | *Oil* | *Gasoline* |
| 1 | 11 | 2 | 38 | 11 | | |
| 2 | 10 | 3 | 28 | 14 | | |
| 3 | 13 | 1 | 51 | 21 | | |
| 4 | 6 | 5 | 28 | 19 | | |
| 5 | 5 | 4 | 22 | 14 | | |
| 6 | 19 | 2 | 34 | 11 | | |
| 7 | 7 | 1 | 39 | 7 | | |
| 8 | 8 | 1 | 21 | 6 | | |
| 9 | 15 | 4 | 32 | 7 | | |
| 10 | 6 | 0 | 15 | 6 | | |
| 11 | 9 | 1 | 23 | 8 | | |
| 12 | 8 | 2 | 35 | 14 | | |
| 13 | 2 | 2 | 6 | 14 | | |
| 14 | 1 | 7 | 5 | 18 | | |
| 15 | 7 | 2 | 22 | 9 | | |
| 16 | 1 | 4 | 10 | 13 | | |
| 17 | 6 | 1 | 24 | 9 | | |
| 18 | 0 | 5 | 31 | 12 | | |
| 19 | 3 | 1 | 17 | 4 | | |
| 20 | 0 | 0 | 3 | 11 | | |
| 21 | 1 | 5 | 3 | 24 | | |
| 22 | 0 | 3 | 3 | 19 | | |
| 23 | 0 | 3 | 2 | 5 | | |
| 24 | 1 | 4 | 3 | 4 | | |
| 25 | 3 | 0 | 6 | 2 | | |
| 26 | 0 | 6 | 0 | 11 | 0 | 1 |
| 27 | 1 | 0 | 4 | 3 | 0 | 1 |
| 28 | 31 | 1 | 61 | 12 | 0 | 1 |
| 29 | 19 | 3 | 55 | 5 | 0 | 0 |
| 30 | 19 | 2 | 34 | 7 | 0 | 2 |
| 31 | 18 | 1 | 53 | 2 | 0 | 0 |
| 32 | 13 | 1 | 36 | 4 | 0 | 0 |
| 33 | 21 | 1 | 49 | 7 | 0 | 0 |
| 34 | 9 | 2 | 42 | 0 | 0 | 1 |
| 35 | 6 | 0 | 30 | 14 | 0 | 1 |
| Total Number: | 271 | 78 | 837 | 347 | 0 | 7 |
| Per Cent | 77.6 | 22.4 | 70.7 | 29.3 | | |
| Including vinegar fly larvae: Total No. | 271 | 85 | | | | |
| Per Cent | 76.1 | 23.9 | | | | |

oil, if carefully and thoroughly agitated with the sample, brings about flotation of a high percentage of the insect fragments from certain types of infestation; gasoline flotation following the castor oil increases the yield of insect parts, including a small fraction of the vinegar fly insect forms; and the old pan-pickout method used by B. J. Howard still seems to be necessary where a quantitative picture of vinegar fly infestation is needed.

The data in Table 1, obtained on 35 samples, show that castor oil isolates many of the insect parts, but that a second trapping using gasoline yields significantly more.

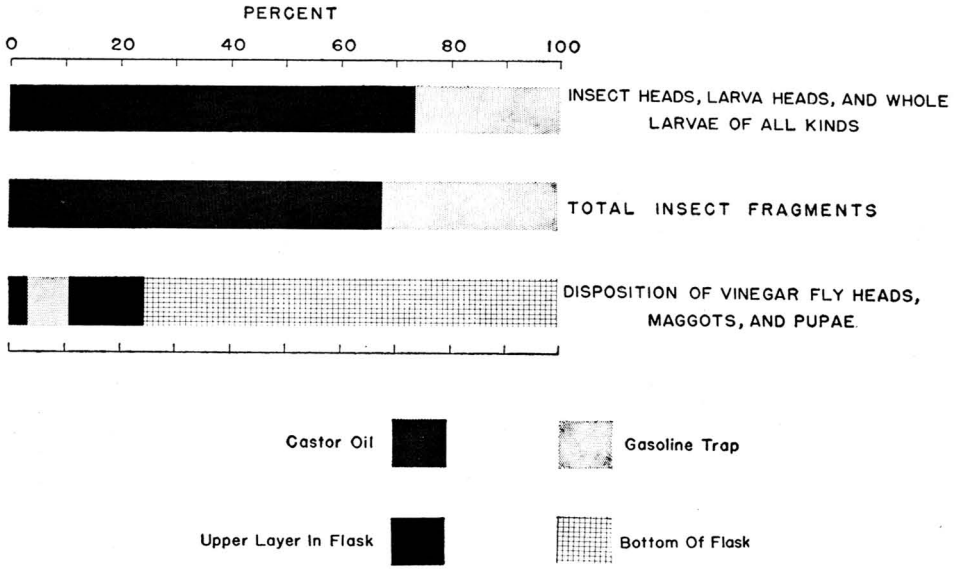


FIG. 1.—Per Cent Location of Insects and Insect Fragments in Various Extraction Media.

For unaccountable reasons this double extraction with castor oil followed by gasoline does not show an overall consistent pattern, since the oil float apparently may or may not bring up most of the parts. It is probable that time, degree, and manner of agitating the oil-and-paste mixture, as well as the physical state of the sample, are important factors influencing the number of insect parts recoverable by the oil, and the gasoline probably only acts as a sweeping agent. However, for maximum recovery both agents appear to be necessary.

Another series of experiments was run on specially prepared paste containing figs heavily infested with insects, including some sour figs infected with vinegar flies. Although a number of Drosophila parts and larvae were recovered in the oil-gas float, it was obvious from the nature of the in-

going fruit that the bulk of such material remained in the flask after the extraction operation. Further examination of the flask contents showed, on the average, about 13 per cent of the vinegar fly maggots to be amongst the fruit tissue which rose to the upper part of the flask after gasoline treatment, but most of them remained at the bottom. Changes made in the specific gravity of the liquid phase, and the use of various floating agents, failed to dislodge these vinegar fly larvae and pupae.

The results tabulated in Table 2 show recoveries of various insect forms in the oil and gas procedure and also the relative location throughout the trap flask of the vinegar fly population. Four 100-gram samples of fig paste and one infested fig were examined.

TABLE 2.—*Elements of insect contamination recovered by castor oil, gasoline and adapted cherry maggot procedure*

| SAMPLE NO. | TOTAL WHOLE INSECTS, LARVAE & HEADS (EXCLUSIVE OF VINEGAR FLIES) | | TOTAL INSECTS & LARVAE, PIECES AND PARTS | | VINEGAR FLY ADULT HEADS, LARVAE, AND/OR PUPAE | | | |
|---|---|---|---|---|---|---|---|---|
| | OIL | GASOLINE | OIL | GASOLINE | OIL | GASOLINE | UPPER LAYERS IN FLASK | BOTTOM OF FLASK |
| 1 | 16 | 1 | 25 | 22 | 2 | 2 | 0 | 37 |
| 2 | 34 | 7 | 75 | 49 | 2 | 2 | 0 | 135 |
| 3 | 12 | 0 | 79 | 68 | 10 | 12 | 41 | 41 |
| 4 | 18 | 4 | 27 | 11 | 0 | 0 | 0 | 38 |
| 5 (one fig) | 1 | 0 | 7 | 1 | 1 | 22 | 24 | 109 |
| Total number | 81 | 12 | 213 | 151 | 15 | 38 | 65 | 360 |
| Per cent | 87.1 | 12.9 | 58.5 | 41.5 | 3.1 | 7.9 | 13.6 | 75.3 |

From a breakdown of these results it is interesting to note that castor oil brought up the majority of beetle and moth type heads and larvae, and adult vinegar fly heads; gasoline brought up the majority of insect eggs and, on the average, about two and one-half times as many vinegar fly larvae as the castor oil; while about three-quarters of the vinegar fly maggots lodged at the bottom of the flask.

It seems unavoidable therefore that the old pan-pickout method, or some modification of it, must be resorted to in order to arrive at a reasonably accurate insect count, and especially to estimate the degree of vinegar fly infestation present in fruit paste. This method consists of searching, either with or without the aid of a magnifying glass, successive small portions of a water suspension of the material, spread thinly over the bottom of a large white pan, removing the insects and parts with small forceps and verifying them with the aid of a jewelers lens (about 4X). The procedure is time consuming, tedious, and subject to many inconsistencies arising from the personal factors of aptitude, experience, patience, and

visual acuity of the analyst. It has certain compensations however, in that it enables the detection of some insect forms that have a stubborn tendency to sink. In a search for an acceptable modification of this procedure, it was found that by adapting the method for recovering cherry maggots (**42.51**) a fairly satisfactory vinegar fly larva and pupa count could be obtained on fig paste residue in the trap flask. This may entail a loss of perhaps 10–15 per cent of the larvae or pupae which might float off with the light fruit tissue, but possibly about this same number might be overlooked if the entire charge remaining in the flask were examined by the pickout method.

Let us consider for a moment the case against the prolific Drosophila, in evaluating the importance of the evidence of its presence. Vinegar fly or pomace fly infestation is not at all uncommon in dried fruit, especially figs. Frequently "souring" occurs most heavily in certain orchard areas, and under adverse drying or growing conditions. Mrak *et al* (2) state that "souring as such (acetic acid formation) is not likely to be the result of independent action of yeasts, but rather the association between yeasts and acetic acid bacteria; or the independent activity of lactic acid bacteria." Nicols and Reed (3) conclude that spoilage of figs by insect infestation and microorganisms may be reduced more effectively by dehydration than by sun drying, but any marked reduction is possible only when figs are *picked* from the tree, indicating that souring may occur on the tree and that infestation is secondary, until the insects themselves become agents of inocculation. The insects multiply with proverbial rapidity—a generation may be completed in about ten days, and copious numbers of them, in various forms, may be found in a single fig. Fig flesh heavily infested with Drosophila becomes characteristically riddled and matted in appearance, the seeds loosen and in later stages the interior of the fruit becomes dry and granular. This type of material probably does not, and certainly should not escape the sorters of sliced figs, and many times may be detected in the whole fruit through physical changes in the exterior of the fig.

Fruit affected with vinegar fly infestation, then, almost always has two counts against it, as compared with that showing beetle or moth type infestation. An affected unit not only contains the insects and parts, but in most instances it is also disagreeably sour or fermented in odor and flavor.

Following is the method used in the experimental work for recovering insects and insect parts from fruit paste:

METHOD

Place 100 g of the paste in a 2-liter trap flask, and disintegrate with sodium hydroxide and hydrochloric acid, as in **42.52**. When the contents of the flask is cool, add 30 ml of castor oil and agitate gently, avoiding a beating motion, for about 3 min. Add water to bring the oil into the neck of the flask and allow to stand about 30 min. With the plunger, work down floating fruit pulp and seeds with a downward

motion that breaks the interface. Repeat this several times, and occasionally during the standing period stir the lower layer. Trap off the oil. Add gasoline to nearly fill the neck of the flask and stir contents of the flask in such a manner as to bring the gasoline in contact, as much as possible, with all the material. Allow to stand 20–30 min., trap off gasoline layer, including about one-half inch of the material at the interface. The two trappings may be combined for filtration on paper and examination. Remove the contents remaining in the flask to a large round white pan. Add warm water, allow to settle for a moment or two, then slowly decant the water and light pulp from the pan. Add more water and repeat decantation, taking care to pour off as little as possible of the lower layer of material which has a tendency to creep up along the inner inclined or pouring edge of the pan, especially if decantation is made while the suspension is in a swirling motion. The residue in the pan can be reduced to a small volume by repeated decantation, filtered on about 80-mesh bolting cloth, and examined under the wide-field microscope. If there are no indications of vinegar fly population in the oil-gas layers, however, it may not be considered necessary to examine the settlings.

Taking overall percentage figures derived from the combined results tabulated in Tables 1 and 2, the accompanying graph is presented to give a picture of recoveries of insect and larvae heads and whole larvae of all kinds, also of total insect fragments, using **42.52** followed by gasoline. The graph also depicts the relative locations in which vinegar fly population may be found following the above described recovery procedure.

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# A MODIFICATION OF THE OFFICIAL MICRO METHOD FOR THE DETERMINATION OF PHOSPHORUS CONTENT OF PLANT TISSUE

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The colorimetric method for the determination of phosphorus was evolved by Osmund (6) and modified by Denigès (4) and by Truog and Meyer (7). The procedure, referred to widely as the "Deniges Method," is based upon the stannous chloride reduction of the phosphomolybdic acid, which reacts with inorganic phosphates to induce development of a blue color. The intensity of the color is measured through comparisons with that of phosphate solutions of known concentration.

The use of hydroquinone to effect the reduction of the phosphomolybdic

acid was introduced by Bell and Doisy (2), and modified by Briggs (3). In another technique, the reduction of the phosphomolybdic acid was effected by means of 1, 2, 4-aminonaphtholsulfonic acid (5). The Briggs modification was adopted as official by the Association of Official Agricultural Chemists (8, 9). In the use of that method, however, certain disadvantages were encountered and these have been overcome through the modification described in the present paper.

## ADAPTATION OF THE METHOD TO SULFURIC ACID-SELENIUM OXYCHLORIDE DIGESTATES

It appeared expedient to determine the phosphorus content of plants through the use of the sulfuric acid-selenium oxychloride digestates (5) that are prepared for the micro determination of nitrogen content. However, when the official method for the determination of phosphorus was applied on 5-ml aliquots of the digestates intended for nitrogen determinations, the values obtained for phosphorus content were not in agreement with those found by means of the official volumetric procedure. The color in the standard solutions was developed without addition of sulfuric acid and a curve constructed from transmittance readings of those solutions. However, when the standards were modified through additions of 1 ml of $H_2SO_4$ (1+2), the phosphorus values then obtained were comparable to those found by means of the official colorimetric and volumetric procedures. Hence, to obtain accurate values through the use of standard calibration curves, it appeared that the standards should contain sulfuric acid in amounts corresponding to those in the aliquots of the unknowns.

The data in Table 1 afford comparisons of the phosphorus content of 25 vegetation samples as determined volumetrically and by means of the official micro colorimetric method applied to $H_2SO_4$-selenium oxychloride digestates (5), but with addition of 1 ml of dilute $H_2SO_4$ to the standards.

## THE TIME FACTOR IN THE DEVELOPMENT OF COLOR AND IN THE READING OF TRANSMITTANCE

The official micro method stipulates that the solutions of the unknowns and of standards be allowed to stand 30 minutes after addition of the reagents and that the readings of color then be made immediately, but the duration of the color, and thus the maximal time permissible for standing, is not stated. When the color is developed at once in a large number of solutions, the over-all time required for the readings may be as much as 3 or 4 hours. In such case, the time allowed for development of color in some of the unknowns may be as much as three hours longer than the time afforded for such development in the standards from which the curve was made. Since the disparity in time may affect the intensity of color, it seemed desirable that the number of unknowns should be no greater than can be read within an hour.

TABLE 1.—*Comparison of $P_2O_5$ content of ryegrass as determined by the official (volumetric) and the modified (colorimetric) methods*

| SAMPLE NO. | OFFICIAL VOLUMETRIC[a] | MODIFIED COLORIMETRIC[b] |
|---|---|---|
| | *per cent* | *per cent* |
| 88 | 0.36 | 0.37 |
| 90 | 0.50 | 0.50 |
| 92 | 0.49 | 0.46 |
| 94 | 0.48 | 0.47 |
| 96 | 0.50 | 0.49 |
| 98 | 0.58 | 0.57 |
| 100 | 0.54 | 0.53 |
| 102 | 0.56 | 0.56 |
| 104 | 0.56 | 0.56 |
| 106 | 0.46 | 0.47 |
| 108 | 0.43 | 0.41 |
| 110 | 0.45 | 0.45 |
| 112 | 0.40 | 0.40 |
| 114 | 0.58 | 0.58 |
| 116 | 0.51 | 0.50 |
| 118 | 0.53 | 0.53 |
| 120 | 0.54 | 0.56 |
| 122 | 0.58 | 0.60 |
| 124 | 0.53 | 0.52 |
| 126 | 0.51 | 0.52 |
| 128 | 0.52 | 0.53 |
| 130 | 0.81 | 0.81 |
| 132 | 0.67 | 0.69 |
| 134 | 0.66 | 0.69 |
| 136 | 0.63 | 0.64 |

[a] From digestions of 2-gm charges.
[b] From sulfuric acid-selenium oxychloride digestions. 1 ml (1+2) sulfuric acid was added only to standards and the blue solutions were allowed to develop overnight: analytical charges of 0.2 gm.

The data of Table 2 and the curves of Figure 1 show the changes in phosphorus values that are indicated, with and without $H_2SO_4$, during periods up to 24 hours. Since the color intensities undergo only slight change after 8 to 10 hours, it is deemed admissible and advisable that the standards and unknowns be allowed to stand overnight before the readings are made, in case many determinations are to be made.

## PREPARATION OF THE ANALYTICAL CHARGE OF PLANT MATERIAL

The preparation of the plant material for analysis by the official colorimetric procedure involves the processing of a 2-gram charge through the steps of incineration, dehydration, filtration, and washing. The over-all procedure is lengthy, tedious, and not feasible for rapid microchemical analysis. Moreover, when the blue color is developed in the 5-ml aliquot of the solution of the 2-gram charge in the stipulated 10-ml flask, the inten-

TABLE 2.—*Phosphorus equivalents of transmittance readings made after various intervals of standing to allow development of color*

| MILLIGRAMS P | | | | | | | | PER CENT OF P RECOVERY | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| STANDARD | AFTER HOUR INTERVALS | | | | | | | AFTER HOUR INTERVALS | | | | | | | |
| ½ | 1 | 2 | 4 | 6 | 8 | 10 | 24 | ½ | 1 | 2 | 4 | 6 | 8 | 10 | 24 |
| Without Addition of $H_2SO_4$ | | | | | | | | | | | | | | | |
| 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | | | |
| .01 | .01 | .01 | .01 | .01 | .01 | .01 | .013 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 130 |
| .02 | .02 | .02 | .021 | .021 | .022 | .023 | .026 | 100 | 100 | 100 | 105 | 105 | 110 | 115 | 130 |
| .03 | .03 | .031 | .032 | .033 | .033 | .034 | .039 | 100 | 100 | 103 | 107 | 110 | 110 | 113 | 130 |
| .05 | .051 | .052 | .054 | .055 | .056 | .057 | .062 | 100 | 102 | 104 | 108 | 110 | 112 | 114 | 124 |
| .07 | .071 | .073 | .076 | .078 | .079 | .079 | .085 | 100 | 101 | 104 | 109 | 111 | 113 | 113 | 121 |
| .10 | .103 | .107 | .111 | .112 | .113 | .115 | .121 | 100 | 103 | 107 | 111 | 112 | 113 | 115 | 121 |
| With Addition of $H_2SO_4$ | | | | | | | | | | | | | | | |
| 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | | | |
| .01 | .01 | .011 | .012 | .012 | .012 | .012 | .013 | 100 | 110 | 120 | 120 | 120 | 120 | 120 | 130 |
| .02 | .021 | .022 | .024 | .025 | .025 | .025 | .026 | 100 | 105 | 110 | 120 | 125 | 125 | 125 | 130 |
| .03 | .032 | .034 | .036 | .037 | .037 | .038 | .040 | 100 | 107 | 113 | 120 | 123 | 123 | 127 | 133 |
| .05 | .054 | .058 | .062 | .064 | .065 | .065 | .068 | 100 | 108 | 108 | 124 | 128 | 130 | 130 | 136 |
| .07 | .075 | .082 | .087 | .090 | .091 | .091 | .096 | 100 | 107 | 107 | 124 | 129 | 130 | 130 | 137 |
| .10 | .109 | .119 | .127 | .130 | .132 | .134 | .137 | 100 | 109 | 109 | 127 | 130 | 132 | 134 | 137 |

TABLE 3.—*Comparison of the $P_2O_5$ content of plants, as determined by the "Official" volumetric and colorimetric methods and by the modified colorimetric method*

| SAMPLE NO. | PLANT MATERIAL | $P_2O_5$ | | | | |
|---|---|---|---|---|---|---|
| | | VOLUMETRIC[a] | COLORIMETRIC | | | |
| | | | OFFICIAL[b] | MODIFIED[c] | | |
| | | | | d | e | f |
| | | *per cent* | *per cent* | *per cent* | *per cent* | *per cent* |
| 1 | Soybeans | 0.26 | 0.23 | 0.23 | 0.23 | 0.09 |
| 2 | Soybeans | 0.99 | 0.99 | 0.99 | 0.97 | 0.85 |
| 3 | Soybeans | 0.95 | 0.94 | 0.94 | 0.94 | 0.80 |
| 4 | Soybeans | 0.39 | 0.37 | 0.34 | 0.35 | 0.23 |
| 5 | Soybeans | 0.27 | 0.25 | 0.25 | 0.24 | 0.11 |
| 6 | Soybeans | 0.87 | 0.87 | 0.87 | 0.85 | 0.73 |
| 7 | Ryegrass | 0.44 | 0.41 | 0.41 | 0.40 | 0.25 |
| 8 | Ryegrass | 0.89 | 0.91 | 0.94 | 0.87 | 0.76 |
| 9 | Ryegrass | 0.81 | 0.84 | 0.84 | 0.78 | 0.69 |
| 10 | Sudan grass | 0.44 | 0.43 | 0.40 | 0.40 | 0.30 |
| 11 | Sudan grass | 0.58 | 0.56 | 0.56 | 0.52 | 0.39 |
| 12 | Clover | 0.50 | 0.48 | 0.49 | 0.47 | 0.32 |

[a] Solutions prepared according to the official micro method (1).
[b] Official micro method (1).
[c] Solutions prepared by $HNO_3$-$HClO_4$ digestion.
[d] Color developed in unknowns and standards as stipulated in the modified procedure for solutions on which nitrogen is *not* to be determined.
[e] 1 ml of $H_2SO_4$ (equal to the amount present in 5-ml aliquots of the $H_2SO_4$-selenium oxychloride digestates) added to both unknowns and standards.
[f] 1 ml of $H_2SO_4$ added to unknowns, but not to standards.

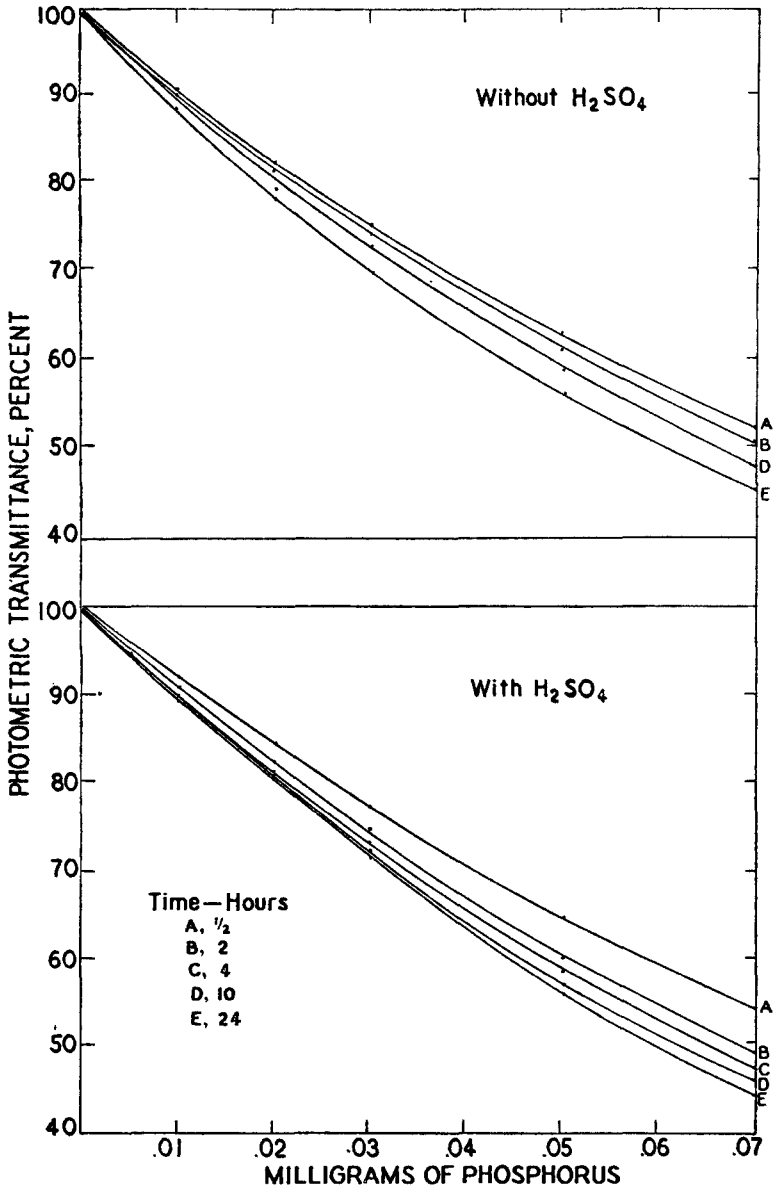


FIG. 1.—Calibration Curves of Phosphorus Equivalents from Transmittance Readings of Solutions after Various Intervals of Standing for Color Development.

sity of the color may be too great for accurate colorimetric readings. Furthermore, a volume of 10 ml is insufficient for the transmittance readings, if the analyst wishes to use some of the solution for the rinsing of the absorption tube of the electrophotometer.

In the present modification, a 0.2-gram charge of plant tissue is prepared rapidly through digestion in a mixture of $HNO_3$ and $HClO_4$. To diminish the intensity of the color and provide solution volume sufficient to allow for the rinsing of the absorption tube, the color is developed in the diluted aliquot in a 50-ml volumetric flask. The transmittance then is read by means of an electrophotometer and the $P_2O_5$ values are computed from a calibration curve constructed from readings against standard phosphate solutions, the color of which was developed simultaneously in like manner.

The data of Table 3 register determinations of $P_2O_5$ on plant materials by means of three methods, and show that the values obtained by the more rapid modified procedure are in accord with the values by the official volumetric and colorimetric methods.

## THE PROPOSED MODIFICATION

### REAGENTS

(1) *Ammonium molybdate soln.* Dissolve 50 g of finely ground ammonium molybdate in 350 ml of water in a liter flask. Add 150 ml of sulfuric acid to 300 ml of water in a beaker; mix and allow to cool. Pour the diluted sulfuric acid into the ammonium molybdate slowly, while the soln is being stirred and cooled by means of running water. Make the cooled soln to volume, filter, and store in a brown bottle.

(2) *Hydroquinone soln.* Dissolve 0.5 g of hydroquinone in 50 ml of water; add 1 ml of sulfuric acid (1+2); transfer to a 100 ml flask; make to volume and store in a brown bottle. Renew this reagent weekly.

(3) *Sodium sulfite soln.* Dissolve 20 g of sodium sulfite ($Na_2SO_3$) in water; transfer to 100-ml flask and make to volume; filter and store in a brown bottle.

(4) *Sulfuric acid (1+2).*—(Used only for addition to standards when charges are prepared by sulfuric acid-selenium oxychloride digestion.)

(5) *Standard potassium dihydrogen phosphate.*—

*Soln A.* Dissolve 4.3928 g of potassium dihydrogen phosphate and make to liter to provide soln to contain 1 mgm phosphorus per ml.

*Soln B.* Dilute 10 ml of soln A to liter, to provide a soln to contain .01 mgm phosphorus per ml.

*Preparation of solution of the charge.* When nitrogen content is *not* to be determined: Weigh 0.2 g of ground plant material into 50 ml beaker. Saturate the charge by adding 2 ml of nitric acid and then adding 3 ml of perchloric acid (70–72 per cent). (Observe precaution given in Addendum.) Place the watch-glass covered beaker on a cold hot-plate, on which is placed an asbestos cloth (or make similar provision to effect gradual and uniform heating). Raise the temp. gradually to evolution of white fumes and continue the digestion, with occasional rotation of beaker to assure complete contact of sample and acid, until the soln is clear, but do not allow to go to dryness. Cool and rinse the watch-glass into the beaker, transfer the soln into a 100-ml flask and make to volume. Filter or allow to settle. (Aliquots of the clear soln can be used also for the determination of Ca, K, Mg (5).)

When nitrogen content of the charge *is* to be determined: Digest a 0.2-g charge of material by means of sulfuric acid-selenium oxychloride reagents as prescribed by Kelley *et al.*, (5).

DETERMINATION

Transfer a 5-ml aliquot into a 50-ml flask and dilute with 25–30 ml of water. Add, in succession and with agitation, immediately after each addition: 1 ml of ammonium molybdate, 1 ml of sodium sulfite, 1 ml of hydroquinone. Shake the soln and allow to stand ½ hour, then make to volume and shake thoroly.

Rinse the absorption tube with a small portion of the soln, introduce 15–20 ml, and take the transmittance reading with a photoelectric colorimeter. Use filter 660 of the Evelyn photoelectric colorimeter or filter 650 of the Fisher electrophotometer.

For each set of determinations, construct a calibration curve from color readings obtained against standards made by aliquots of the potassium dihydrogen phosphate soln B that contain 0.0, 0.01, 0.02, 0.03, 0.05, and 0.07 mg of P. Develop the color in the standards and in the unknowns simultaneously, as outlined for the unknowns. In case the charges are prepared by the $H_2SO_4$-selenium oxychloride digestion, add 1 ml of $H_2SO_4$ (1+2) to the standards, prior to addition of the other reagents. Calculate per cent of phosphorus from the readings of the unknowns by means of the standard calibration curve. To convert per cent P to per cent $P_2O_5$, multiply by 2.2912.

## COMPARISON OF RESULTS OBTAINED BY THREE METHODS

The results in Table 1 show the concordance between determinations on 25 samples by means of the A.O.A.C. volumetric procedure and by the proposed modifications of the official colorimetric method. The solutions were prepared by the $H_2SO_4$-selenium oxychloride digestion, as presented by Kelley, Hunter, and Sterges for the determination of nitrogen (5). The readings were made after the solutions had stood overnight for development of color. The values reported are the means of concordant duplicate determinations. The $P_2O_5$ values obtained by the two methods were identical on 8 samples, and in the other 17 the differences were in most cases only 0.01 per cent.

Twelve samples of plant material were prepared for analysis in accordance with the A.O.A.C. colorimetric procedure (1). Aliquots of the solutions were analyzed for $P_2O_5$ content volumetrically and by the official colorimetric micro method. These samples were analyzed also by use of the $HNO_3$–$HClO_4$ digestate of a 0.2 gram charge as stipulated in the modified method. The results obtained by the more rapid modified procedure, as shown in Table 3, are in excellent concord with those obtained by the volumetric and official colorimetric methods. Also, when 1 ml of $H_2SO_4$ (1+2) was added to the standards and to the unknown solutions, the results were comparable to those obtained by the official procedures, and by the modified procedure. However, when $H_2SO_4$ was present in the unknowns (as would be the case if the solutions had been prepared for determination of nitrogen through digestion with $H_2SO_4$ and selenium oxychloride) the results were considerably lower (Table 3, (f)), unless an equivalent amount of $H_2SO_4$ was added to the standards.

The Evelyn electrophotometer was used for readings of the solutions obtained through the use of both the official method and the modified

procedure, but the Fisher photoelectric colorimeter can be used with equal facility for those determinations.

## SUMMARY AND CONCLUSIONS

A modification of the present official micro procedure for determination of $P_2O_5$ content of plant material was compared with both the official volumetric procedure and with the official colorimetric micro method.

The time limit permissible for the development of color in the solutions was determined.

The proposed modifications afford considerable saving of time in the preparation of analytical charge by elimination of incineration, evaporation, filtration, and washing, and thereby diminish chances of error. The use of a charge of only 0.2 g permits more rapid preparation of the analytical solution, decreases the amount of sample required, and the digestion procedure facilitates and expedites repetition of the determination.

A single digestate can be used for the determination of calcium, magnesium, and potassium content, as well as that of phosphorus.

When determination of the nitrogen content of plant tissue is desired, the charge can be prepared by the $H_2SO_4$-selenium oxychloride digestion (5) and the resultant solution can be used also for determination of $P_2O_5$ content. The prescribed addition of 1 ml of $H_2SO_4$ (1+2) to the standard is the only change necessary to effect uniform color development in standards and in unknowns.

## ADDENDUM

*Precaution:* The potential explosion hazard for mixtures of $HClO_4$ and organic matter is recognized. This danger has been stressed in publications and news items.* The prescribed prior saturation of the charge of plant tissue with $HNO_3$ and the low initial temperature of the digestion have led to no explosions in the making of more than 3,800 such digestions in this laboratory.

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# A STUDY OF THE EFFECT OF CLARIFICATION ON THE DETERMINATION OF REDUCING SUGARS IN PLANT MATERIALS

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Analytical methods for the determination of reducing sugars in plant materials usually include a clarification procedure to remove non-sugar-reducing substances that may be present in the plant extract. Some of the methods, including the A.O.A.C. method (1, 2), use neutral lead acetate for clarification and remove the excess lead with disodium hydrogen phosphate; this procedure has been shown to be of doubtful value when applied to some plant materials (9, 10, 11, 12). Therefore, to acquire additional information on the lead clarification procedure, various fruits and vegetables were selected at random, an alcoholic extract was made from each material, variations were made in the clarification step, and the reducing sugars obtained by the variable procedures were determined and compared.

## EXPERIMENTAL

Alcoholic extracts of the edible portion of various plant materials were prepared by a previously described method (18). Each extract was made to a definite volume with 80% redistilled ethyl alcohol and aliquots from this extract were used for all of the experiments discussed below.

The variable analytical procedures with each extract were as follows:

(a) The alcoholic extract was analyzed for reducing sugars without any previous clarification.

(b) The alcoholic extract was treated with Baker and Adamson Code 1551[2] decolorizing carbon (4, 18).

(c) The alcohol was evaporated from the extract (18) and its water solution was filtered through a mat of Celite Analytical Filter-Aid (17, 18) and analyzed for sugars without any further clarification.

(d) The same procedure outlined in (c) was followed, but in addition the water solution was treated for 15 minutes with an excess of neutral lead acetate. The lead precipitate was removed by filtration; the filtrate was de-leaded with disodium hydrogen phosphate and again filtered to remove the lead precipitate (18).

(e) The same procedure outlined in (d) was followed, but in addition the de-leaded solution was treated for 10 minutes with B & A decolorizing carbon (4, 18), and the carbon was removed by filtration.

Reducing sugars were determined by the A.O.A.C. micro copper method

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[2] Mention of manufacturers and commercial products does not imply that they are endorsed or recommended by the Department of Agriculture over others not mentioned.

TABLE 1.—*Determination of reducing sugars* in plant materials after different clarification procedures were used*

| PLANT MATERIAL | METHOD OF ANALYSIS | ALCOHOLIC EXTRACT | | | DEALCOHOLIZED EXTRACT | | |
|---|---|---|---|---|---|---|---|
| | | | TREATMENT | | TREATMENT[2] | | |
| | | PER CENT ALCOHOL CONC.[1] | NONE | CARBON[3] | CELITE | NEUTRAL LEAD ACETATE | NEUTRAL LEAD ACETATE + CARBON[3] |
| Apple, Gravenstein | A.O.A.C. | 6 | 8.43 | 8.33 | 8.40 | 8.48 | 8.44 |
| | Hassid | | 8.37 | 8.29 | 8.41 | 8.59 | 8.41 |
| Apricot† | A.O.A.C. | 24 | 2.26 | 2.28 | 2.40 | 2.25 | 2.29 |
| | Hassid | | 2.27 | 2.16 | 2.43 | 2.37 | 2.31 |
| Cabbage | Hassid | 8 | 3.77 | — | 3.77 | — | 3.67 |
| Cantaloupe | A.O.A.C. | 20 | 2.52 | 2.56 | 2.68 | 2.76 | 2.68 |
| | Hassid | | 2.45 | 2.46 | 2.45 | 2.50 | 2.47 |
| Carrot | A.O.A.C. | 40 | 2.99 | 3.08 | 3.21 | 3.20 | 3.16 |
| | Hassid | | 2.92 | 2.93 | 3.04 | 3.13 | 3.08 |
| Cherry, Bing | A.O.A.C. | 3 | 16.14 | 15.82 | 15.70 | 15.90 | 15.13 |
| | Hassid | | 16.25 | 15.30 | 15.68 | 15.88 | 15.20 |
| Crabapple | A.O.A.C. | 8 | 3.95 | 3.48 | 4.08 | 3.72 | 3.58 |
| | Hassid | | 4.76 | 3.40 | 4.82 | 3.96 | 3.49 |
| Cucumber | A.O.A.C. | 24 | 2.21 | 2.19 | 2.19 | 2.20 | 2.19 |
| | Hassid | | 2.06 | 2.07 | 2.06 | 2.11 | 2.07 |
| Egg Plant | A.O.A.C. | 16 | 2.38 | 2.51 | 2.51 | 2.55 | 2.54 |
| | Hassid | | 2.66 | 2.47 | 2.61 | 2.57 | 2.49 |
| Endive | A.O.A.C. | 32 | 1.14 | 1.26 | 1.51 | 1.44 | 1.40 |
| | Hassid | | 1.66 | 1.35 | 1.67 | 1.37 | 1.27 |
| Grape, Thompson | A.O.A.C. | 3 | 20.10 | 18.70 | 19.91 | 19.42 | 18.78 |
| | Hassid | | 18.83 | 18.75 | 18.90 | 19.46 | 18.69 |
| Grapefruit | A.O.A.C. | 8 | 5.27 | 5.11 | 5.11 | 5.17 | 4.98 |
| | Hassid | | 5.24 | 4.84 | 5.21 | 5.28 | 4.92 |
| Kohlrabi | A.O.A.C. | 20 | 2.60 | 2.72 | 2.68 | 2.71 | 2.63 |
| | Hassid | | 2.68 | 2.65 | 2.60 | 2.61 | 2.54 |
| Lettuce | Hassid | 14 | 2.48 | — | 2.65 | — | 2.53 |
| Loganberry | A.O.A.C. | 11 | 4.86 | 4.63 | 4.88 | 4.72 | 4.51 |
| | Hassid | | 5.18 | 4.50 | 5.12 | 4.70 | 4.48 |

TABLE 1.—*Continued*

| PLANT MATERIAL | METHOD OF ANALYSIS | ALCOHOLIC EXTRACT | | | DEALCOHOLIZED EXTRACT | | |
|---|---|---|---|---|---|---|---|
| | | PER CENT ALCOHOL CONCN.[1] | TREATMENT | | TREATMENT[2] | | |
| | | | NONE | CARBON[3] | CELITE | NEUTRAL LEAD ACETATE | NEUTRAL LEAD ACETATE + CARBON[3] |
| Mustard Greens | A.O.A.C. | 32 | 1.59 | 1.48 | 1.61 | 1.60 | 1.47 |
| | Hassid | | 1.81 | 1.51 | 1.84 | 1.87 | 1.49 |
| Nectarine | A.O.A.C. | 24 | 2.31 | 2.28 | 2.41 | 2.31 | 2.31 |
| | Hassid | | 2.39 | 2.05 | 2.52 | 2.29 | 2.18 |
| Onion bulb | A.O.A.C. | 24 | 3.02 | 3.10 | 3.04 | 3.08 | 3.04 |
| | Hassid | | 2.94 | 2.91 | 2.98 | 3.02 | 2.97 |
| Orange | A.O.A.C. | 10 | 3.72 | 3.71 | 3.76 | 3.88 | 3.66 |
| | Hassid | | 3.82 | 3.62 | 3.86 | 3.90 | 3.61 |
| Peach | A.O.A.C. | 24 | 2.25 | 2.22 | 2.48 | 2.46 | 2.43 |
| | Hassid | | 2.28 | 2.09 | 2.40 | 2.29 | 2.22 |
| Pear, Immature | A.O.A.C. | 9 | 4.89 | 4.86 | 4.92 | 4.92 | 4.77 |
| | Hassid | | 4.84 | 4.70 | 4.75 | 4.80 | 4.68 |
| Pepper, Green, Bell | A.O.A.C. | 20 | 2.79 | 2.79 | 2.87 | 2.91 | 2.81 |
| | Hassid | | 2.72 | 2.66 | 2.70 | 2.74 | 2.68 |
| Plum,† Santa Rosa | A.O.A.C. | 16 | 3.21 | 2.81 | 3.15 | 2.95 | 2.88 |
| | Hassid | | 3.40 | 2.89 | 3.34 | 3.00 | 2.76 |
| Squash,† Crookneck | A.O.A.C. | 20 | 2.24 | 2.41 | 2.35 | 2.42 | 2.35 |
| | Hassid | | 2.29 | 2.27 | 2.30 | 2.37 | 2.31 |
| Squash, Zucchini | A.O.A.C. | 20 | 2.18 | 2.23 | 2.13 | 2.17 | 2.10 |
| | Hassid | | 2.06 | 2.06 | 2.09 | 2.12 | 2.07 |
| Strawberry | A.O.A.C. | 10 | 5.70 | 5.26 | 5.66 | 5.40 | 5.22 |
| | Hassid | | 5.66 | 5.15 | 5.66 | 5.37 | 5.18 |
| Stringbean, Scarlet Runner | A.O.A.C. | 12 | 2.10 | 2.34 | 2.23 | 2.24 | 2.20 |
| | Hassid | | 2.33 | 2.19 | 2.33 | 2.25 | 2.16 |
| Tomato† | A.O.A.C. | 24 | 2.61 | 2.65 | 2.73 | 2.62 | 2.60 |
| | Hassid | | 2.59 | 2.56 | 2.58 | 2.60 | 2.52 |
| Youngberry | A.O.A.C. | 12 | 4.19 | 3.88 | 4.15 | 3.93 | 3.76 |
| | Hassid | | 4.71 | 3.91 | 4.63 | 4.16 | 3.87 |

*See opposite page for footnotes to table.*

(3) and the Hassid micro ferricyanide method (6, 7). The sugar concentration of the alcoholic plant extracts exceeded the amount required by the two micro methods of analysis (0.2 to 0.6 mg. reducing sugars per ml. of sample); thus it was necessary to dilute the aliquots with redistilled water when the alcoholic extracts were used for analysis. Therefore, at the time of analysis, the alcohol content of the samples varied from 3% to 40% when either of the above procedures (a) or (b) was used. In Table 1, each ferricyanide sugar value represents two, and each copper sugar value represents three, closely agreeing replicates.

## DISCUSSION

In almost 50 per cent of the plant materials studied, the reducing sugar content of the plant extract could be determined without the removal of the alcohol and without any form of clarification. The remainder of the plants required treatment of their alcoholic extracts with a selected decolorizing carbon only (Table 1).

While this study was in progress, research on fruit-cannery waste was being conducted by others in this Laboratory. Occasionally, we were called on to determine sugars in the juices pressed from these waste materials. Reducing sugars were determined by the Hassid method (6, 7) using the juice (a) as received, (b) after treatment with decolorizing carbon, (c) after lead-phosphate treatment, and (d) after lead-phosphate-carbon clarification. The sugar values obtained on a representative set of such samples are given in Table 2. The data show that clarification did not affect the analytical values.

There will be instances when the investigator will require values for the total sugar content of the plant. At such times the removal of the alcohol from the extract will be necessary to avoid possible interference with invertase hydrolysis (16). It seems plausible that the additional procedure of lead clarification would not be necessary if it proved to be of no value when determining reducing sugars on the same plant.

The fact that neutral lead acetate clarification may not improve the analytical results does not preclude the possibility that non-sugar-reducing substances may be included in the sugar value. This has been evi-

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*Footnotes to Table 1*

* Results calculated as percentage of dextrose.
† The fruit was not peeled.
‡ The concentration of ethyl alcohol in the sample at time of analysis.
§ Celite = Dealcoholized plant-concentrate filtered thru a Büchner funnel containing a thin mat of Celite Analytical Filter-Aid (17, 18).
Neutral lead acetate = Water-solution of plant extract treated with excess neutral lead acetate, allowed to stand 15 minutes, then filtered thru a filter of analytical grade. Excess lead removed from filtrate with disodium hydrogen phosphate and the precipitate removed by filtration.
Neutral lead acetate + carbon = Same as neutral lead acetate treatment plus additional 10-minute treatment of the de-leaded solution with 5 mg B&A carbon per ml of solution.
¶ Carbon = 10-minute treatment of plant extract with 5 mg of Baker & Adamson Code 1551 decolorizing carbon per ml of solution. The concentration of the reducing sugars in the extracts were adjusted to prevent possible adsorption of dextrose and levulose by the carbon (see Ref. No. 4).

denced in fermentation studies (5, 8, 13, 14) and also by Williams, *et al.* (18), who showed that non-sugar-reducing substances which were not removed from dehydrated potato extracts by neutral lead acetate clarification were removed by treatment of the extract with synthetic ion-exchange resins. Furthermore, the authors concur with other investigators (15, 17, 18) that no specific set of published data can be applied universally to a particular type of plant. Therefore, the data presented in Tables 1 and 2 are not intended to establish the fact that the plant materials listed therein can always be analyzed for sugars without including a clarification procedure. Rather, the purpose is to show that if it is necessary to deter-

TABLE 2.—*Determination of reducing sugars* in pear-waste juice after different clarification procedures were used*

| SAMPLE NO. | METHOD OF CLARIFICATION | | | |
|---|---|---|---|---|
| | NONE[1] | CARBON[2] | NEUTRAL LEAD ACETATE[2] | NEUTRAL LEAD ACETATE+CARBON[2] |
| 1 | 66.6, 65.9 | 63.6, 63.6 | 66.3, 65.8 | 67.2, 66.5 |
| 2 | 70.6, 70.9 | 66.6, 67.3 | 70.0, 70.3 | 69.0, 69.2 |
| 3 | 62.3, 62.3 | 62.6, 62.6 | 63.0, 63.0 | 62.5, 63.2 |
| 4 | 61.3, 61.6 | 60.9, 60.9 | 61.5, 61.5 | 61.2, 60.8 |
| 5 | 61.9, 62.3 | 61.6, 60.6 | 61.8, 62.0 | 61.5, 61.3 |
| 6 | 53.9, 53.9 | 53.3, 53.6 | 53.5, 53.5 | 52.5, 53.0 |

* Reducing sugars determined by the Hassid micro-ferricyanide method and the results calculated as mg of dextrose per ml of juice. Each value represents the results of a single analysis; two values for each type of analysis are given to indicate the degree of precision of the method.
[1] Used the natural juice as received.
[2] See Footnotes 2 and 3, Table 1.

mine sugars on many samples of a particular plant, it might save time and expense to make a preliminary study to determine the value of the clarification procedure. The time factor is especially important where the analytical results may be needed during the harvesting period of the plant. Also, the omission of the clarification routine would eliminate several precipitation and filtration steps, which are conducive to the accumulation of operational errors.

## CONCLUSIONS

Alcoholic extracts of some plant materials can be analyzed for reducing sugars without clarification by the neutral lead acetate procedure. Other plant materials may require treatment of the alcoholic extract with a selected decolorizing carbon only. Juices expressed from pear-cannery wastes can be analyzed for sugars without previous clarification.

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## RELATIONSHIP BETWEEN ACIDS, ESTERS, AND SOLIDS DURING THE AGING OF WHISKY

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Comprehensive studies involving the changes occurring during the aging of whisky have been published by four groups of investigators, Crampton and Tolman (1), Liebmann and Rosenblatt (2), Liebmann and Scherl (3), and Valaer and Frazier (4). While these studies contain a wealth of data on the concentrations of various congeners throughout the course of a four or eight year aging period, little is known of the interrelationships between various congeners except for the non volatile groups, such as solids, non volatile acids, tannin, and color. Conspicuous among the observed changes associated with the aging of whisky are increases in the concentrations of acids, esters, and solids. Liebmann and coworkers have stated that a relationship exists between the concentration of acids and solids. In addition, Liebmann and Scherl have suggested a relationship between the concentration of esters and solids. The nature of these two relationships, however, has not been disclosed.

Figure 1 reveals that there exists throughout the period of aging a linear relationship between the increase in acids and esters and the increase in dissolved solids. The basic data for Figure 1 were obtained from average values as reported in the summary tables of the four groups of investigators. It has been conventional to record ester concentration in terms of

ethyl acetate per 100 liters at 100 proof. In the calculation of data for Figure 1, average values for esters as ethyl acetate were recalculated to an equivalent concentration of acetic acid by multiplying by 60/88. Average values for acids, esters (as acetic acid) and solids at zero time were subtracted from corresponding values at various ages to give values representing the increases in the named congeners at comparable periods. Figure 1 represents the increase in acids plus esters (as acetic acid) plotted against the increase in solids for corresponding periods.

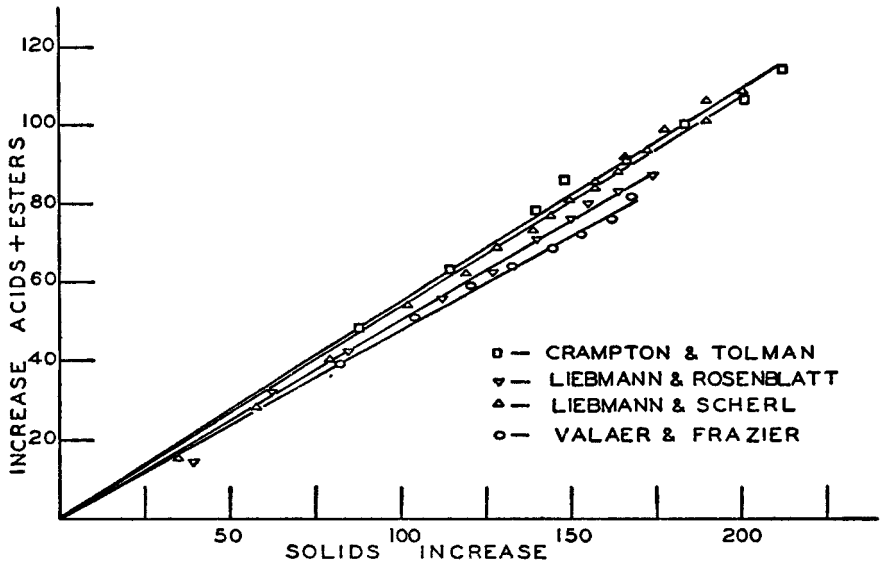


FIGURE 1.—Relationship between the increase in acids and esters (as acetic acid, g. per 100 liters at 100 proof) and the increase in dissolved solids (g. per 100 liters at 100 proof).

Stated in mathematical terms, observations summarized in Figure 1 show that acids and esters increase in direct proportion to the increase in solids, $A+E=c\,S$. In which A, E and S represent, respectively, the increase in acids, esters (as acetic acid), and dissolved solids and c is a constant. The value for c has been calculated as 0.49 for the data of Valaer and Frazier, 0.52 for the data of Liebmann and Rosenblatt, and 0.54 for the data of Crampton and Tolman and of Liebmann and Scherl. It is interesting that the relationship between the increase in acids and esters and the increase in solids is virtually the same for all four groups of observations, which cover a period of at least 40 years.

The observations summarized in Figure 1 strongly support the concept that acids and esters are formed during the aging of whisky by a process which is directly linked with the extraction of solids from the barrel. It is,

as stated by Liebmann and Rosenblatt, difficult to believe that volatile acidic material which comprises more than 80 per cent of the acid-ester fraction is extracted directly from the wood of the barrel. More attractive is the possibility that the acid-ester fraction is developed by a chemical process which depends on some precursor which in turn is extracted from the barrel at the same rate as the solids fraction. This concept is compatible with certain observations made by Crampton and Tolman on whisky aged under different conditions. These authors report average data for rye and bourbon whisky. At the time of their investigation it was customary to store rye whisky in heated warehouses while bourbon whisky was usually held in unheated warehouses. Average data reveal that the increases in acids, esters and solids were definitely greater for rye whisky than for bourbon of the same age. Yet calculations for the increase in acids and esters per unit increase in dissolved solids fail to show a significant difference; values for both rye and bourbon fluctuated around an average of 0.54 which is the same as calculated for all samples summarized by Crampton and Tolman. This suggests that greater formation of acids and esters at higher temperatures is simply a reflection of an increased rate of extraction. Also, Crampton and Tolman reported on several samples of whisky stored in uncharred barrels. With uncharred barrels the increase in acids and esters was directly proportional to the increase in solids; however, the ratio of the increase in acids and esters to the increase in solids amounted to 1.1. This is about two times the value observed for charred barrels. With uncharred barrels it is presumed that more acid-ester precursor is extracted per unit of solids than in the case of charred barrels. The preceding examples suggest that the relationship between the increase in acids and esters and the increase in solids may have application in research dealing with factors influencing the congener content of whisky.

## CONCLUSION

A survey of the published literature pertaining to the aging of whisky reveals that the increase in acids and esters (calculated as acetic acid) is directly proportional to the increase in dissolved solids. This observation suggests that the formation of acids and esters during the aging of whisky is directly dependent upon some precursor which is extracted from the barrel at the same rate as the bulk of the solids fraction.

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# ISOLATION OF DDT FROM FATS*

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DDT in dairy products and biological tissues is closely associated with the fat component, and solvent methods for its extraction from these materials always include the fat. The conventional Schechter-Haller colorimetric method for the determination of DDT is not applicable in the presence of more than traces of fat (or other organic material), and the isolation of DDT from comparatively large quantities of fat has remained a serious analytical problem. Schechter, *et al.*[1] remove fat from DDT by repeated extraction of their chloroform solution with sulfuric acid and fuming sulfuric-sulfuric acid mixtures. Clifford[2] hydrolyzes fats with a lipase preparation, neutralizes the fatty acids to form their soaps and extracts the DDT from the mixture with petroleum ether. Both methods have obvious disadvantages.

A more convenient and much faster method is presented here. It was found that celite (a commercial diatomaceous earth), impregnated with sulfuric acid-fuming sulfuric acid, and slurried with carbon tetrachloride, would hold fats within a chromatographic column while DDT passed through with the carbon tetrachloride.[3] (Other fat-soluble chlorinated insecticides may likewise be separated from interfering fat.) Recoveries of microgram quantities of DDT added to 5 grams of butter oil free from DDT ranged between about 90–100 per cent.

## PROPOSED METHOD

### APPARATUS

(1) *Chromatographic tubes.*—40 mm O.D. × 200 mm long.

(2) *Tamping rod,* of glass, diam. of flat portion somewhat less than inner diam. of chromatographic tube.

### REAGENTS

(1) *Celite 545.* (Johns Manville).

(2) *Fuming sulfuric acid.*—15 per cent $SO_3$.

(3) *Carbon tetrachloride.*—A.C.S. grade.

### PREPARATION OF SAMPLE

*Milk.*—Incorporate risen cream by warming to 35–40° and shaking vigorously. Immediately after mixing weigh 100 g into a centrifuge bottle, add an equal volume of 95% ethanol, and separate the fat with petroleum ether as directed in reference

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* Excerpt from a thesis to be submitted by Bernard Davidow in partial fulfillment of the requirements for the degree of Doctor of Philosophy at Georgetown University.

† Arnold J. Lehman, Chief.

[1] Schechter, *et al, Ind. Eng. Chem. Anal. Ed.*, 19, 51 (1947).

[2] Clifford, Paul A., *This Journal*, 30, 337 (1947).

[3] In a private communication, R. T. Hall (Hercules Experiment Station) indicated that a celite concentrated sulfuric acid column had been used in an attempt to separate toxophene from fat.

(2). (Poor separation of the alcohol-serum layer is sometimes encountered especially with homogenized milks. In such cases treat a further sample, before dilution with alcohol, by mixing with 2–3 g of commercial pancreatin or the lipase preparation described in reference (2), and heat for about 1 hour at 35–40°). Do not include appreciable quantities of the alcohol-serum phase when "blowing off" the petroleum ether fractions. Extract 3–4 times with the petroleum ether, evaporate the solvent, and dissolve the clear fatty residue in 15–20 ml of carbon tetrachloride.

*Butter.*—Warm gently on the steam bath to break the emulsion, decant the fat layer, and filter a portion through a dry folded filter in a warming oven. If a clear, water-free oil is not obtained, repeat the filtration. Weigh not over 5 g into a beaker or flask, and dissolve in 15–20 ml of carbon tetrachloride.

*Other Oils, Fats and Extracts of Biological Samples.*—Filter the oil or fat directly, as above, or filter its solvent soln before evaporation of the solvent, in order to obtain a clear, water-free sample. Weigh the sample and dissolve in 15–20 ml of carbon tetrachloride.

## CHROMATOGRAPHY

Place 30 g of Celite 545 in a mortar, and add 9 ml of conc. $H_2SO_4$ and 9 ml of the fuming $H_2SO_4$. Triturate until a homogeneous, slightly damp powder is obtained. Add 100 ml of carbon tetrachloride in divided portions and triturate the mass to a smooth slurry. Plug the constricted end of a chromatographic tube with glass wool and attach a short piece of rubber tubing fitted with a closed screw-clamp. Pour about 25 ml of carbon tetrachloride into the tube and, with a procelain or glasss spoon, add the slurry in several portions, packing it down with the tamping rod after each addition. (The finished column must appear homogeneous and of uniform firmness across its diameter. A little experience will show how firmly the column must be packed in order to obtain an optimum flow rate: ca 120 drops per minute.) After preparation of the column, a little free carbon tetrachloride should stand above it; if more than a few mm remain, drain off to about this level. Now add the carbon tetrachloride soln of the sample to the column with a serological pipet, disturbing the surface as little as possible; also add the carbon tetrachloride rinsings of the flask or beaker. Remove the tubing and allow the carbon tetrachloride to flow out freely. When the fat sinks into the column, rate of flow is slowed, and eluate collection (in a 400-ml beaker) is started at this point. After the fat soln has just passed into the column, rinse down the sides of the tube with about 25 ml of carbon tetrachloride added in 3–4 portions, and after the final rinse has sunk in, fill the tube with carbon tetrachloride and collect about 250 ml of eluate. (The column is designed to retain about 5 g of fat, and the fat will slowly move down as a horizontal yellow band. However, this band should not approach the exit end of the tube until after collection of the requisite 250 ml of eluate.) Evaporate the eluate to dryness on a warm steam-bath with the aid of a moderate jet of air. Do not allow the beaker to remain on the bath longer than is necessary for evaporation of the carbon tetrachloride.

## DETERMINATION OF DDT

Determine DDT as outlined in reference (2). If, in routine work, resolution of the isomers is unnecessary, estimate DDT from a standard curve prepared with a 3+1 proportion, respectively, of the p, p′ and o, p′ isomers.

If approximate DDT content of the fat sample is unknown, make the residue, remaining after evaporation of the carbon tetrachloride eluate, to definite volume with an appropriate solvent (ca 50 ml with benzene) and carry an exploratory 10-ml aliquot thru the procedure. Depending upon the range of the standard curve (as determined by volume of colorimetric solution and cell length) repeat, if necessary, with a more appropriate aliquot. Because the quantity of residual ma-

terial remaining after the chromatographic separation of fat is usually negligible, always include 10 mg of oleic acid (DDT-free) with the sample or sample aliquot before nitration. (See reference (2), p. 339.) Only three alkali washes (p. 347) are necessary to wash interfering yellow color from the petroleum ether extract of the DDT-nitration products.

Convert results obtained upon the clear oil from commercial butters to the butter itself, by multiplying by 0.8.

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## THE IDENTICAL *p*H METHOD FOR DETERMINATION OF BORAX IN MIXED FERTILIZERS*

By Donald S. Taylor (Research Department, Pacific Coast Borax Co., Division of Borax Consolidated, Limited, Pasadena, California)

Previous work in this laboratory on methods for determination of borax in mixed fertilizers has been reported (7). The general dissatisfaction with current A.O.A.C. methods was pointed out, and an alternative procedure was described. This alternative procedure will now be identified as the sodium carbonate-barium carbonate method to distinguish it from other methods.

The sodium carbonate-barium carbonate method has now been tried by a number of laboratories (8), with conflicting results. Some workers obtained good results in line with those obtained by four different analysts at this laboratory. Other workers reported erratic results. Therefore it seemed evident that the sodium carbonate-barium carbonate method did not represent the best possible solution to the problem. The difficulties may be due to unrecognized variations in technique. Efforts to identify these difficulties have not yet been successful, and it was felt that if possible, a shorter method should be developed.

Some preliminary efforts were spent on techniques involving distillation with methanol. This work will not be reported in detail, but the results were not encouraging. It was concluded that accurate results could be obtained by distillation procedures, but only by tedious and cumbersome procedures. The official A.O.A.C. distillation procedure (Method 2:45, Acid-Soluble Boron (1)) was found to give low results on several typical fertilizers, and it was felt that eliminating these errors would require even more tedious methods.

Accordingly, attention was turned to a method which has been employed for boron analyses on other types of materials, particularly on water samples. This method, unfortunately, requires the use of a sensitive *p*H meter, but has other characteristics which are attractive. This procedure is here identified as the "Identical *p*H" method because titration is carried from a definite *p*H without mannite to the same *p*H with man-

* Presented at the Annual Meeting of the Association of Official Agricultural Chemists, held at Washington, D. C., October 10–12, 1949.

nite. The principle involved was first employed colorimetrically by Foote (3) for determining boron in irrigation water. Wilcox (9) adapted the method to the use of potentiometric technique. Wilcox also described (10) a variation involving ignition and phosphate removal with lead nitrate for use on plant material. Others who have described applications of variations of this technique are Hollander and Rieman (4) for glass, Ruehle and Shock (5) for magnesium chloride liquors, and Fajans (2) for glass.

The principle involved in this method is that a dilute solution containing boric acid is adjusted to a selected *p*H near neutrality, mannite is added to increase the acidity of boric acid, and the solution is titrated with alkali until the same *p*H is again reached. The exact *p*H used has varied with different workers from 6.3 to 7.6. Since boric acid begins to react with alkali at about *p*H 5.5 and since boric acid with mannite is not completely neutralized until about *p*H 8.2, such a titration is not intrinsically stoichiometric. Any *p*H selected will result in either some boric acid neutralized before mannite addition, or some boric acid remaining unneutralized after titration, or both. However, if approximately constant volumes, constant amounts of mannite, and suitably low boric acid concentrations are employed, the method can be empirically standardized by merely titrating known amounts of boric acid in the same fashion. Fajans (2) reports that under certain conditions the alkali equivalence is substantially stoichiometric.

The merit of the "Identical *p*H" procedure resides in its comparative freedom from interferences, since presumably only materials reacting with mannitol to yield acid will be titrated. Unfortunately, the only common material besides boric acid which does this is phosphoric acid, though it reacts to a much smaller degree. Since phosphate is a common fertilizer component it must be removed before titration. Wilcox (10) suggested using lead nitrate and sodium bicarbonate for removal of phosphate when using plant ash, and this procedure seemed attractive for trial with fertilizers.

The main faults of the "Identical *p*H" method are its limitation to relatively low amounts of boric acid, and the errors caused by variation of salt concentration. At high boric acid concentrations, low mannite concentrations, or widely varying salt concentrations, the fraction of boric acid neutralized at the final end point varies appreciably. It is therefore best (a) To operate in such fashion that the salt content is relatively constant, (b) Use a large amount of mannite, and (c) Keep the amount of boric acid low.

## PRELIMINARY WORK

The method of sample dissolution adopted for fertilizer samples was treatment with hot hydrochloric acid. The previous report from this

laboratory (7) pointed out the necessity for using acid because of possible formation of water-insoluble borates in fertilizers on storage.

Preliminary trials of the method were made with the inclusion of an alkaline boiling to remove ammonia. It was found that this alkaline boiling in pyrex vessels resulted in appreciable dissolving of boron from the glass. The amount was low, but significant in this method because of the small amount of boron which is desired for the titration. Therefore, trials were made in presence of much ammonium sulfate. At *p*H values near neutral some of the ammonium ion originally present is probably converted to ammonium hydroxide, and the equilibria between these two forms may buffer the solution enough to decrease the sensitivity of the *p*H to alkali addition, and thereby decrease the precision. Tests in presence of ammonium sulfate were carried out by the method described later, using *p*H 6.3 as the identical *p*H, since presumably the interference would be less than at *p*H 7. Table 1 shows results found for borax on 0.5 gm. samples of commercial ammonium sulfate, with and without added boron. The boron was added as standard boric acid solution.

TABLE 1.—*Analyses for borax by "Identical pH" method in presence of 0.5 gm. commercial ammonium sulfate*

| PER CENT BORAX ADDED | PER CENT BORAX FOUND |
|---|---|
| 0.00 | 0.01 |
| 6.16 | 6.20, 6.24 |

These results are reasonably accurate, so it was decided best to set up this procedure without an ammonia removal step. By so doing the procedure is simplified, and errors due to solution from Pyrex are eliminated.

The procedure as described below was also tested on one gram samples of commercial superphosphate, to check on complete removal of phosphate by the use of lead nitrate and sodium bicarbonate. Values of 0.00, and 0.02% borax were obtained, showing satisfactory removal of phosphate.

In first trials of this method, the solution *p*H sometimes showed a slow drifting when adjusted to the value of 6.3. As alkali was added to the acid solution the *p*H would at first reach 6.3, then slowly drift back toward lower values. In extreme cases this was very annoying and resulted in high results. The trouble was finally traced to slow reaction between some lead and carbonic acid in the solution. Though lead cannot be completely removed, the carbonic acid can be, and rigorous exclusion of carbonic acid leads to normal behavior. Final neturalization with several ml. of supposedly carbonate free 0.5 *N* sodium hydroxide was sometimes enough to cause the trouble, so the procedure outlined in the method of over-acidifying, boiling out carbonic acid, then reneutralizing and slightly

acidifying before final boiling was adopted. When such a procedure was followed no drifting of *p*H occurred and reagent blanks were uniformly about 0.30 ml. of 0.025 *N* sodium hydroxide, most of which was apparently due to the sodium bicarbonate used.

In order to more or less reproduce salt content the method is set up to include dissolution using a definite volume of concentrated hydrochloric acid (3 ml). Acid which is consumed by the sample will lead to an equivalent amount of salt in solution, and any excess is reacted with sodium bicarbonate to bring the salt content up to a fairly reproducible level about equivalent to 3 grams sodium chloride. Additional amounts of neutral water soluble salts (potassium chloride, potassium sulphate, etc.) from the sample will not change the amount of salt present by any large proportion. Three grams of c.p. sodium chloride is used in the solution when standardizing to approximately reproduce this level of salt concentration.

It was decided appropriate to limit the borax content of the sample to about 40 mgms., so that one gram samples are used up to 4.0% borax, smaller samples are used for higher amounts. Usual precautions about sampling and grinding materials made up of coarse particles must, of course, be followed.

The amount of lead nitrate solution to be used was set at 10 ml of 10% solution, or one ml for each 1.2% contained phosphoric acid if the phosphoric content exceeds 12%. This appears to be a reasonable approach since the phosphoric acid content is normally known.

It was also found that crystalline D-sorbitol (Atlas) can replace mannite. It dissolves more rapidly and is less expensive, but of course blanks must be checked with the material used.

For fertilizers containing organic matter the usual titration methods require an ashing step due to weak organic acids which interfere. In this "Identical *p*H" method the organic matter could only interfere with accuracy if it either reacts with mannitol to release acid or reacts with boric acid to release acid. Neither of these possibilities seem very probable, but they cannot be rigorously excluded because of the variety of organic materials which may be present in fertilizers. Trials with one organic base containing a variety of materials were satisfactory, so the method is tentatively set up without ashing. This seems to greatly increase the desirability of the method for general use, as ashing is time-consuming and can lead to loss of boron if poorly carried out.

## METHOD

### REAGENTS

*Lead nitrate*, c.p., 10% *soln.*
*Sodium bicarbonate powder*, c.p.
*Sodium chloride*, c.p.
*Methyl red indicator* (0.1%)
*Mannitol (neutral), or crystalline D-sorbitol.*

*Hydrochloric acid*, concd, 0.5 *N*. and 0.02 *N*.

*Sodium hydroxide soln*, carbonate free, 0.5 *N*.

*Sodium hydroxide soln*, carbonate free, about 0.025 *N*. (The NaOH solns must be protected from atmospheric $CO_2$ by soda lime tubes or other suitable means.)

*Boric acid soln* (for standardizing the 0.025 *N* NaOH).—Dissolve 1.0000 g reagent grade dry $H_3BO_3$ in distd water and dilute to 1 liter.

### APPARATUS

A high sensitivity glass electrode *p*H meter is used for the titration. According to Wilcox (10) the quinhydrone electrode system and other similar assemblies can also be used and may be more convenient. An assembly with the electrodes, motor driven stirrer, and burettes arranged for convenient use with a 250-ml beaker is required. The burettes are used for the 0.025 *N* NaOH and 0.02 *N* HCl, and may be ordinary 50 ml burettes.

### PROCEDURE

Weigh sample within 0.001 g (1.0 g for up to 4.0% borax, smaller samples above that content) and place in 250 ml beaker. Add ca 50 ml water and 3 ml conc. HCl. Heat to boiling and keep hot until carbonates are decomposed. Keep soln hot but do not boil during treatment for phosphate removal which follows. Add 10% $Pb(NO_3)_2$ soln, 10 ml in usual cases, or 1 ml for each 1.2% contained $P_2O_5$ if $P_2O_5$ content is known to be above 12%. Add solid $NaHCO_3$ a little at a time until soln approaches neutrality. (This can often be observed by the formation of white precipitate in addition to insoluble matter already present.) Add few drops of methyl red indicator and continue gradual addition of bicarbonate until *just* alkaline to methyl red (yellow or very slightly orange). Keep resulting mixture hot but not boiling (water bath or steam bath is best) for 30 min., adding additional small quantities of $NaHCO_3$ if required to keep same indicator color. (In some cases the indicator will bleach because of the nitrate, and more must be added. If indicator color is obscured by organic matter external spot tests may be used to follow neutralization.)

The solution volume after neutralization and heating should be ca 40–50 ml. Filter hot soln into 250-ml beaker and wash the solids thoroughly with hot water. Acidify the filtrate with few drops of concd HCl and boil briefly to expel most of $CO_2$. Neutralize hot soln with 0.5 *N* NaOH, and reacidify with 0.5 *N* HCl, using about 0.3–0.5 ml in excess. Dilute to about 150 ml and boil quietly a few minutes to expel remaining $CO_2$. Cool to room temp. in flowing water. Roughly neutralize mixture using carbonate free 0.5 *N* NaOH and place beaker in the titration assembly with electrodes and stirrer immersed. Start stirrer and adjust *p*H to exactly 6.30 by additions of 0.025 *N* NaOH or 0.02 *N* HCl as required. (When properly adjusted a steady *p*H should be obtained, drifting usually is due to incomplete $CO_2$ removal.) With steady reading of 6.30 *p*H, read the 0.025 *N* NaOH burette, add 20 grams mannite or crystalline sorbitol, and titrate with 0.025 *N* NaOH until *p*H meter again reads exactly 6.3. (This can conveniently be done, with a slide-wire type instrument, by opening the *p*H meter circuit when mannite is added, leaving the scale setting at 6.3, and closing the circuit again when indicator color shows that end point is being approached, carefully adding the standard NaOH until galvanometer needle reaches zero again. With practice the somewhat slow approach to equilibrium which is characteristic of the glass electrode can be anticipated so that there is little danger of overrunning end point.) When end point is reached, read burette again. The amount of standard NaOH used is corrected by a reagent blank determined by carrying out same procedure without sample but with all reagents. Calculate borax content of the sample:

$$\%Na_2B_4O_7 \cdot 10H_2O = \frac{\text{mg. borax/ml (ml NaOH for Sample} - \text{ml NaOH for Reagents)}}{10 \times \text{gms Sample}}$$

NOTE. The equivalence of the 0.025 *N* NaOH is determined in terms of mg borax/ml, as follows: Pipet 25 ml of standard boric acid soln into 250-ml beaker, add 3.0 g c.p. NaCl, acidify to methyl red, make to 150 ml, boil to expel $CO_2$, cool, and titrate potentiometrically in same fashion as used for sample. Also carry out same procedure with the NaCl alone to obtain a titration blank. The soln strength is then calculated:

$$\text{Mg. borax/ml} = \frac{38.55}{\text{ml NaOH for } H_3BO_3 - \text{ml blank}}$$

## SAMPLES USED

The samples used in checking the method were the same series used in previous work at this laboratory (7). Briefly, they were:

(1) F. S. Royster Guano Company, 0-12-12 fertilizer with borax.
(2) Magruder Check Sample No. 9, 2-12-2 fertilizer with 4.4% borax.
(4) Laboratory mixture, 19% ammonium sulfate, 16% KCl, 35% superphosphate, 30% dolomite.
(5) Laboratory mixture, 19% ammonium sulfate, 16% KCl, 40% superphosphate, 25% limestone.
(6) Mixture, Downey Fertilizer Company, "Red Star Gro-Master V" (4-10-2), plus 10% superphosphate. Contains much organic matter.

These materials were analyzed, and the last three were analyzed with added borax present. The borax was added as a solid and had been mixed with the base materials nine months before analysis. Satisfactory results are also obtained when the boron was added to the weighed sample as a standard solution of boric acid just before the analysis.

## RESULTS

Results obtained in this laboratory on the several samples are shown in Table 2. It is noted that in general duplicate runs check well, and agree satisfactorily with the borax contents when these are known. The average deviation from the mean for this table is about $\pm 0.02$ per cent borax, but it is evident that the absolute deviation may run somewhat higher than this on samples containing the higher amounts (see No. 6, with 3.00%, for example).

In Table 3 the average results on some of the samples are compared with the average results by the sodium bicarbonate-barium carbonate method (7). It is apparent that the results in this laboratory obtained by both methods are substantially in agreement, and close to the known borax contents. In general the results by the "Identical *p*H" method appear equal or better than those by the sodium bicarbonate-barium carbonate method. It is concluded that the "Identical *p*H" method should be satisfactory over the range of concentrations usually encountered in commercial mixed fertilizers. However, materials such as fertilizer borates

TABLE 2.—*Results of fertilizer analyses by the "Identical pH" method*

| SAMPLE USED | PER CENT BORAX PRESENT | PER CENT BORAX FOUND | AVERAGE PER CENT BORAX | DEVIATIONS FROM AVERAGE, % BORAX |
|---|---|---|---|---|
| 1 | (?) | 3.52 | 3.55 | −0.03 |
| | | 3.56 | | +0.01 |
| | | 3.56 | | +0.01 |
| | | 3.56 | | +0.01 |
| 2 | 4.40 | 4.29 | 4.29 | 0.00 |
| (Magruder | | 4.29 | | 0.00 |
| Check Sample) | | 4.34 | | +0.05 |
| | | 4.23 | | −0.06 |
| 4 | 0.0 | 0.02 | 0.02 | 0.00 |
| | | 0.03 | | +0.01 |
| | | 0.01 | | −0.01 |
| | | 0.02 | | 0.00 |
| 4 | 1.00 | 1.03 | 1.01 | +0.02 |
| | | 1.02 | | +0.01 |
| | | 1.00 | | −0.01 |
| | | 1.01 | | 0.00 |
| | | 1.02 | | +0.01 |
| | | 0.99 | | −0.02 |
| 5 | 0.0 | 0.02 | 0.01 | +0.01 |
| | | 0.01 | | 0.00 |
| 5 | 5.00 | 5.06 | 5.09 | −0.03 |
| | | 5.12 | | +0.03 |
| 6 | 0.0 | 0.03 | 0.02 | +0.01 |
| (Organic) | | 0.02 | | −0.01 |
| 6 | 3.00 | 3.06 | 3.01 | +0.05 |
| (Organic) | | 3.01 | | 0.00 |
| | | 3.04 | | +0.03 |
| | | 3.06 | | +0.05 |
| | | 2.94 | | +0.07 |
| | | 2.96 | | −0.05 |

containing very high amounts of borax and free of phosphates can be better analyzed by other known methods (6).

In practice it was found possible to analyze a group of six samples in 2–3 hours, by the "Identical *p*H" method. It is believed that once the analyst becomes practiced the "Identical *p*H" procedure will be much more convenient than other known methods.

TABLE 3.—*Comparison of average results by $Na_2CO_3$-$BaCO_3$ and "Identical pH" methods*

| FERTILIZER SAMPLE NUMBER | PER CENT BORAX CONTENT | AVERAGE PER CENT BORAX BY "IDENTICAL pH" METHOD | AVERAGE PER CENT BORAX BY $Na_2CO_3$-$BaCO_3$ METHOD |
|---|---|---|---|
| 1 | — | 3.55 | 3.45 |
| 2 | 4.40 | 4.29 | 4.31 |
| 4 | 1.00 | 1.01 | 1.05 |
| 5 | 0.0 | 0.01 | 0.12 |
| 5 | 5.00 | 5.09 | 5.06 |
| 6 (Organic) | 3.00 | 3.01 | 2.93 |


## SUMMARY

The "Identical pH" titration principle employed by Wilcox (9) and others for special boron determinations has been adapted to analysis for borax in mixed fertilizers. The procedure developed has been tested on a number of samples and found to be reasonably accurate, and very convenient. It is believed to offer advantages over any other present method for such determinations.



## ACKNOWLEDGMENT

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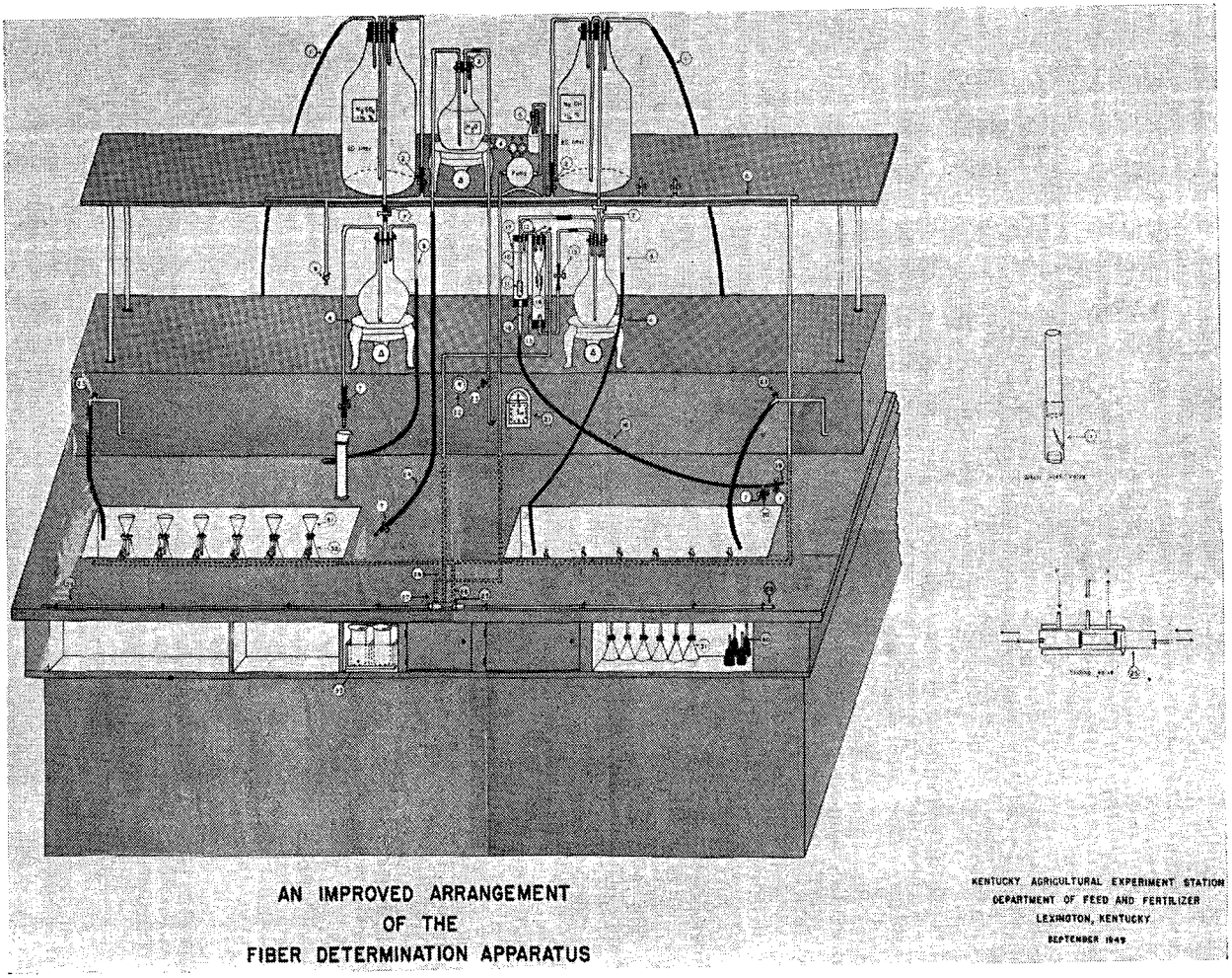

AN IMPROVED ARRANGEMENT
OF THE
FIBER DETERMINATION APPARATUS

KENTUCKY AGRICULTURAL EXPERIMENT STATION
DEPARTMENT OF FEED AND FERTILIZER
LEXINGTON, KENTUCKY
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## NOTES

# AN IMPROVED ARRANGEMENT OF THE FIBER DETERMINATION APPARATUS

By V. C. MIDKIFF AND J. A. SHRADER (Kentucky Agricultural Experiment Station,* Lexington, Kentucky)

For 35 years prior to 1946 analysts in the Kentucky Feed Control Laboratory had been using the same "home-made" fiber analysis apparatus. This was arranged very inconveniently, and in recent years it was found to be entirely inadequate to handle the number of samples collected by the inspectors because of the increase in the amount of commercial feeds sold in Kentucky.

Modern fiber equipment was ordered and plans were made for rearranging the laboratory and modeling a different fiber analysis "set-up." (See opposite page.)

The digestion and filtration apparatus, the heating units, trays, timer, and glassware now are conveniently arranged on a single laboratory table so that a minimum of steps are necessary to carry on the work. Two alberene sinks are in the table where the work is done, so that all the funnels, filter cloths, suction flasks, and beakers can be washed there. The sinks really serve three purposes: namely, to wash utensils, carry off waste material, and lower the suction flasks to a more convenient level for filtering.

The rugged little electric vacuum-pressure pump is the nerve center of the "set-up." There are many uses for it. Placed on the top shelf above the fiber table where the gauges can be easily seen, this pump is used to fill the large storage carboys above the laboratory table, to partially evacuate the suction flasks in the filtering procedures, and to operate a measuring device which measures the 200 ml. of boiling sodium hydroxide solution required in the A.O.A.C. method of analysis.

LEGEND FOR DIAGRAM*

1. Filling and vent tube (2).
2. Pinch cock on vacuum filler line (2).
3. Vent tube.
4. Hot plates (3).
5. Safety bottle.
6. Main vacuum line.
7. Pinch cocks (5).
8. Vacuum regulating valve.
9. Overflow, vent, and siphon starting tube (2).
10. 200 ml capacity measuring tube.
11. Alkali inlet valve.
12. Zero overflow tube.
13. Overflow reservoir tube.
14. Electrical connections to warning buzzer.
15. Pinch cock on overflow return to preheating reservoir.
16. Alkali outlet tube.
17. Zero level.
18. 200 ml level.
19. Fine stream nozzle.
20. Coarse stream nozzle.
21. Interval timer and clock.
22. Switch to vacuum-pressure pump.
23. Tap water valves (3).
24. Hot water line.
25. Sliding valve.
26. Vacuum line.
27. Pressure line.
28. Vacuum and pressure line to measuring device.
29. Extension rod to sliding valve.
30. Walter type gooch crucible holders (12).
31. Coors procelain Hirsch funnels (12).
32. Suction flasks (1 liter size) (12).
33. Storage tray for digestion dipots.

* This report, made in connection with a project of the Kentucky Agricultural Experiment Station, was presented at the Annual Meeting of the Association of Official Agricultural Chemists, held at Washington, D. C., Oct. 10–12, 1949, and is published by permission of the Director.

* Condensers in rear of sinks not shown.

EXPLANATION OF DIAGRAM

The 1¼% sulfuric acid and 1½% sodium hydroxide carboys are filled by vacuum from reserve mixing carboys on the floor through tubes (1). The solutions then are siphoned into 6-liter flat-bottom flasks resting on hot plates (4) which heat the solutions to boiling. The 200 ml. of boiling acid solution is measured by siphon into a graduate.

As all know, the 200 ml. of boiling alkali solution used to wash the residue from the acid digestion filtration is rather difficult to dispense. With this in mind a device was attempted which would measure exactly 200 ml. of the boiling solution and at the same time could be used to wash the residue from the filtering cloth. After constructing a number of automatic measuring devices, it was found that the corrosive action of the hot alkali solution was a factor that had to be considered. From these experiments the very simple and durable measuring apparatus shown (10 to 20) in the diagram was evolved.

This measuring device consists mainly of two 1½" diameter pyrex tubes and a rubber inlet valve. It is operated by vacuum and pressure from the previously mentioned pump. A conveniently placed sliding valve controls the vacuum which fills the measuring device, and the pressure which empties the tube through the washing nozzle.

When the sliding valve (25) is in the position shown, a partial vacuum is maintained throughout the copper tube system (6) to the sliding valve by tube (26) and to overflow reservoir tube (13). This is connected to the 200 ml. measuring tube (10) by tube (12). The vacuum will cause the slit in alkali inlet valve (11) to open and the alkali solution to flow until it reaches the level of the overflow tube (12). The amount of vacuum can be regulated by the bleeding valve (8). By moving the position of the sliding valve (25) the pressure tube (27) is in line with tube (28) and the slight pressure applied to the measuring device will cause a steady stream of the hot alkali solution to flow through the fine stream nozzle (19) when clamp (7) is released. The solution flows to a predetermined 200 ml. capacity mark (18). Merely sliding the valve one way and then the opposite by the extension rod (29) fills and empties the 200 ml. measuring tube (10). Eventually the overflow tube will have filled to make contact with electrical connections (14) to a warning buzzer. Pinch clamp (15) is then opened and the pressure forces the overflown alkali solution back into preheating flat botton flask.

This new "set-up" saves by actual count seven steps out of eight steps made before. With this arrangement twenty-four fiber samples can be run each half day by one person. Not only is this 100 per cent faster than with the old "set-up," it is more accurate and enables us to follow very closely the method as outlined by the A.O.A.C.

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## NOTE ON THE DETERMINATION OF SULFUR IN EGGS

By DONALD B. RODERICK, The Clemson Agricultural College, School of Agriculture, South Carolina Experiment Station, Clemson, South Carolina*

On account of the difficulty found in completely oxidizing the organic matter present in the whole egg, and obtaining an ash, some experimental work on this line has been conducted in order to find a suitable oxidizing agent, or a mixture of agents.

All analyses were made on a single lot of liquid whole eggs, with special precautions to insure homogeneity of samples. Four different oxidizing mixtures were employed. These were all mixed intimately with the sample, which was then evaporated

* Technical contribution number 168, from the South Carolina Experiment Station.

to dryness and ashed over a Bunsen burner. The ash was then dissolved in 1:4 hydrochloric acid and the sulphur determined by the conventional method (A.O.A. C. Methods, 6th Ed. Sec. 12.34). Reagent blanks were run in every case and the results were corrected for the small amount of sulfur present in the reagents.

The following oxidants were tried:

(A) Fifteen ml of 50% magnesium nitrate soln.

(B) Ten ml of normal potassium permanganate soln and 15 ml of concentrated nitric acid.

(C) Ten ml of normal potassium permanganate soln.

(D) Two g of magnesium oxide and 10 ml of normal potassium permanganate soln.

No experiments were performed with perchloric-nitric acid mixtures because of their dangerous character, especially since some of the above oxidants proved satisfactory.

The results obtained were as follows:

| METHOD | WEIGHT OF SAMPLE | PER CENT OF SULPHUR |
|---|---|---|
| | *gram* | |
| A | 3.8495 | .150 |
| | 6.1200 | .175 |
| | 6.1550 | .160 |
| | 6.8869 | .155 |
| | Average .160% Sulfur | |
| B | 3.5698 | .161 |
| | 4.2130 | .168 |
| | 12.9528 | .162 |
| | 13.5723 | .167 |
| | Average .164% Sulfur | |
| C | 11.4039 | .084 |
| | 12.5000 | .071 |
| | 13.4090 | .071 |
| | 15.5587 | .075 |
| | Average .075% Sulfur | |
| D | 5.4038 | .168 |
| | 5.6100 | .171 |
| | 5.7000 | .170 |
| | 7.6884 | .142 |
| | Average .162% Sulfur | |

The conclusions arrived at were: Method A was the most satisfactory from the standpoint of ease of manipulation and character of ash and ash solution. Method B gave rather more consistent results, but presented manipulation difficulties. Method C was clearly unsatisfactory. Method D gave a somewhat wider spread than method A.

**Organic Reactions.** Editor-in-Chief, Roger Adams, 446 pp. text, refs. and index. John Wiley and Sons, New York. Price $6.00.

The fifth volume of "Organic Reactions" is a valuable addition to the growing compendium of organic chemistry set forth in the first four volumes. This useful series is rapidly becoming the leading source of reference material for preparative organic chemistry because of its cleancut style, multitude of factual material, and voluminous and readily accessible bibliography. The real value of this series does not lie alone with the profuse amount of factual material compiled for each reaction, but with the critical view with which each expert author has treated the subject matter.

A particularly useful section of each chapter is devoted to the treatment of scope and limitation of each reaction. This type of compilation allows the chemist to readily evaluate the reaction in question with reference to his particular problem. The tables of yields are also valuable in predicting successful application of the reactions.

In the present volume, ten chapters are devoted, respectively, to "The Synthesis of Acetylenes," "Cyanoethylation," "The Diels-Alder Reaction: Quinones and Other Cyclenones," "Preparation of Aromatic Fluorine Compounds from Diazonium Fluoborates: The Schiemann Reaction," "The Friedel and Crafts Reaction with Aliphatic Dibasic Acid Anhydrides," "The Gattermann-Koch Reaction," "The Leuckart Reaction," "Selenium Dioxide Oxidation," "The Hoesch Synthesis," and "The Darzens Glycidic Ester Condensation." The average number of references for each chapter is about one hundred and eighty.

The fifth volume brings the total number of subjects covered in the series to forty-eight. It can be seen by examination of the cumulative index of chapters in the fifth volume that the subject matter now covers a great portion of the preparative organic chemistry field.

LEE S. HARROW

**Colloid Chemistry.** By Harry Boyer Weiser. John Wiley & Sons, Inc., New York, N. Y., 444 pages. Price $5.50.

The author's aim in this book is to present colloid chemistry as an established branch of chemistry and not a chapter lifted from physical chemistry.

The book was written primarily as a text book but it should be especially useful to the analytical chemist who wishes to more completely understand the physical nature of many applications of colloid chemistry to chemical analysis. The subject is presented in an interesting and easily read manner that is unusual in such a technical and specialized field. The text is well illustrated with a large number of graphs, tables and diagrams. Very extensive references to the literature are given.

The book is divided into five major parts; Absorption, Sols, Gels, Emulsions and Foams, and Aerosols.

Of special interest to the analytical chemist are the seven chapters (2–8) devoted to a study of the different types of adsorption. Many processes having important technical application are described. Among these are: (1) removal of coloring matter from solution by charcoal, a fact which has been known since the fifteenth century; (2) the adsorption of gases on charcoal—widely used in gas masks during World War I; (3) the absorption of solids from solution and their subsequent removal by more polar solvents—the basis of absorption chromatography; (4) exchange adsorption: well-known examples are the use of zeolites as water softeners and the more recent use of resinous ion exchangers to produce water in degree of purity equal to distilled water; (5) adsorption indicators.

Chapters 9–10 are devoted to a study of sols and descriptions of the mechanism of dialysis, electrodialysis, electrophoresis and the Tyndall Phenomenon and its utilization in the ultra-microscope.

The remainder of the book is devoted to a number of topics and includes an interesting chapter on Dyeing.

JONAS CAROL