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ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS
BOX 290, PENNSYLVANIA AVENUE STATION,
WASHINGTON, D. C.

ERRATA.

Page 158, paragraph 3, line 3.—After "(dry basis)" add "and Canadian lead number, respectively".

Page 158, heading for Table 2.—Change "total ash (dry basis)" to "Canadian lead number".

Page 314, paragraph 4:

Lines 3 and 8.—Change "1924 pounds" to "2924 pounds".

Line 9.—Change "710 pounds" to "1079 pounds".

Line 11.—Change "112 pounds" to "170 pounds".

Page 316, line 2.—Change "40 acres" to "30 acres".

Page 317, paragraph 4, lines 3 and 4.—Change "sixtieth" to "both".

Page 337, line 5.—Change "(3)" to "(4)" and transfer with text following to appropriate position for "(4)".

Page 337, line 6.—Change "(4)" to "(3)" and transfer with text following to appropriate position for "(3)".

Page 340, footnote 1.—Change **"3"** to **"4"**.

Page 366, footnote 2.—Change **"16"** to **"56"**.

Page 452, footnote 2.—Change **"8"** to **"51"**.

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PROCEEDINGS OF THE THIRTY-FIFTH[1] ANNUAL CONVENTION, NOVEMBER, 1919.

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[1] No meeting was held in 1918 other than a meeting of the Executive Committee.

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TUESDAY—AFTERNOON SESSION.

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PROCEEDINGS OF THE THIRTY-FOURTH ANNUAL CONVENTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, 1917.

OFFICERS, REFEREES, ASSOCIATE REFEREES AND COMMITTEES OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, FOR THE TWO YEARS ENDING NOVEMBER, 1919.

Honorary President.

H. W. **Wiley**, Woodward Building, Washington, D. C.

President.

P. F. **Trowbridge**, Agricultural Experiment Station, Agricultural College, N. Dak.

Vice-President.

H. C. **Lythgoe**, State Department of Health, Boston, Mass.

Secretary-Treasurer.

C. L. **Alsberg**, Box 744, 11th Street Station, Washington, D. C.

Additional Members of the Executive Committee.

B. B. **Ross**, Polytechnic Institute, Auburn, Ala.
G. G. **Frary**, State Food and Drug Department, Vermilion, S. Dak.

Referees.

Phosphoric acid: (Not appointed.)
Nitrogen: I. K. Phelps, Bureau of Chemistry, Washington, D. C.
Potash: T. E. Keitt, Agricultural Experiment Station, Ga.
Soils: C. B. Lipman, University of California, Berkeley, Calif.
Inorganic plant constituents: J. H. Mitchell, Clemson Agricultural College, Clemson College, S. C.
Insecticides and fungicides: O. B. Winter, Agricultural Experiment Station, E. Lansing, Mich.
Water: J. W. Sale, Bureau of Chemistry, Washington, D. C.
Foods and feeding stuffs: G. L. Bidwell, Bureau of Chemistry, Washington, D. C.
Dairy products: Julius Hortvet, State Dairy and Food Department, Old Capitol, St. Paul, Minn.
Saccharine products: W. L. Owen, Penick & Ford, Ltd., New Orleans, La.
Drugs: G. W. Hoover, U. S. Food and Drug Inspection Station, Transportation Building, Chicago, Ill.

Testing chemical reagents: L. F. Kebler, Bureau of Chemistry, Washington, D. C.
Microanalytical methods: B. J. Howard, Bureau of Chemistry, Washington, D. C.
Food preservatives: A. F. Seeker, U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, New York, N. Y. (Since deceased.)
Coloring matters in foods: W. E. Mathewson, Bureau of Chemistry, Washington, D. C.
Metals in foods: W. D. Collins, Bureau of Chemistry, Washington, D. C.
Fruits and fruit products: D. B. Bisbee, U. S. Food and Drug Inspection Station, Old Custom House, St. Louis, Mo.
Canned vegetables: W. D. Bigelow, National Canners Association, 1739 H Street, N. W., Washington, D. C.
Cereal foods: J. A. LeClerc, Miner-Hillard Milling Co., Wilkes-Barre, Pa.
Wines: J. M. Humble, U. S. Food and Drug Inspection Station, U. S. Custom House, Cincinnati, Ohio.
Soft drinks (bottlers' products): W. W. Skinner, Bureau of Chemistry, Washington, D. C.
Distilled liquors: J. I. Palmore, Bureau of Chemistry, Washington, D. C.
Beers: (Not appointed.)
Vinegars: W. A. Bender, Douglas Packing Company, Rochester, N. Y.
Flavoring extracts: A. E. Paul, U. S. Food and Drug Inspection Station, Transportation Building, Chicago, Ill.
Meat and meat products: Ralph Hoagland, Bureau of Animal Industry, Washington, D. C.
Eggs and egg products: C. E. Marsh, State Department of Health, Boston, Mass.
Gelatin: C. R. Smith, Bureau of Chemistry, Washington, D. C.
Edible fats and oils: R. H. Kerr, Bureau of Animal Industry, Washington, D. C.
Spices and other condiments: H. E. Sindall, Austin, Nichols & Co., Inc., New York, N. Y.
Cacao products: Leicester Patton, U. S. Food and Drug Inspection Station, Federal Building, Buffalo, N. Y.
Coffee: H. A. Lepper, Bureau of Chemistry, Washington, D. C.
Tea: E. M. Bailey, Agricultural Experiment Station, New Haven, Conn.
Baking powder: H. E. Patten, Provident Chemical Works, St. Louis, Mo.

Associate Referees.

Phosphoric acid:
    Basic slag, to cooperate with committee on vegetation tests on the availability of phosphoric acid in basic slag: (Not appointed.)
Nitrogen:
    Special study of the Kjeldahl method: H. W. Daudt, Jackson Laboratory, E. I. Du Pont Company, Wilmington, Del.
Potash: (Not appointed.)
Soils:
    Nitrogenous compounds: (Not appointed.)
    Lime absorption coefficient: W. H. McIntire, Agricultural Experiment Station, Knoxville, Tenn.
Inorganic plant constituents: W. L. Latshaw, State Agricultural College, Manhattan, Kans.
Insecticides and fungicides: J. J. T. Graham, Bureau of Chemistry, Washington, D. C.
Water: L. H. Enslow, Miraflores Filtration Plant, Ancon, Panama, C. Z.

Foods and feeding stuffs:
- Sugar: A. H. Bryan, Arbuckle Bros., Old Slip and Water Streets, New York, N. Y. (Since deceased.)
- Crude fiber: L. D. Haigh, University of Missouri, Columbia, Mo.
- Stock feed adulteration: Miss B. H. Silberberg, Bureau of Chemistry, Washington, D. C.
- Organic and inorganic phosphorus: J. B. Rather, Standard Oil Company, Chemical Laboratory, Brooklyn, N. Y.
- Water: J. O. Clarke, U. S. Food and Drug Inspection Station, Old Custom House, Savannah, Ga.

Dairy products:
- Separation of nitrogenous substances in milk and cheese: L. L. Van Slyke, Agricultural Experiment Station, Geneva, N. Y.

Saccharine products:
- Maple products: J. F. Snell, Macdonald College, Quebec, Canada.
- Honey: (Not appointed.)
- Sugar house products: F. W. Zerban[1], Sugar Experiment Station, New Orleans, La.;
  W. O. Whaley[2], Penick & Ford, Ltd., New Orleans, La.;
  D. D. Sullivant[2], Penick & Ford, Ltd., New Orleans, La.

Drugs:
- Medicinal plants: Arno Viehoever, Bureau of Chemistry, Washington, D. C.
- Alkaloids: A. R. Bliss, jr., School of Medicine, Emory University, Atlanta, Ga.
- Synthetic products: C. D. Wright, Bureau of Chemistry, Washington, D. C.
- Balsams and gum resins: E. H. Grant, Wm. S. Merrell Co., Cincinnati, Ohio.
- Enzyms: J. F. Brewster, Bureau of Chemistry, Washington, D. C.

Fruits and fruit products: H. J. Wichmann, U. S. Food and Drug Inspection Station, Tabor Opera House Building, Denver, Colo.

Meat and meat products:
- Separation of nitrogenous compounds in meat products: Walter Ritchie[1], University of Missouri, Columbia, Mo.
  L. C. Mitchell[3], Wilson & Co., Chemical Laboratory, Chicago, Ill.
- Meat extracts: H. H. Mitchell[1], University of Illinois, Urbana, Ill.
  C. R. Moulton[3], University of Missouri, Columbia, Mo.

PERMANENT COMMITTEES.

Cooperation with Other Committees on Food Definitions.

William Frear (State College, Pa.), *Chairman.*
Julius Hortvet, St. Paul, Minn.
C. D. Howard, Concord, N. H.

Recommendations of Referees.

(Figures in parentheses refer to year in which appointment expires.)

B. B. Ross (Auburn, Ala.), *Chairman.*

SUBCOMMITTEE A: A. J. Patten (1918), (Agricultural Experiment Station, E. Lansing, Mich.), *Chairman*, C. C. McDonnell (1922), B. B. Ross (1920). [Phosphoric acid

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[1] Associate referee for the year ending November, 1918.
[2] The work on sugar house products for the year ending November, 1919, was divided between W. O. Whaley and D. D. Sullivant.
[3] Associate Referee for the year ending November, 1919.

(basic slag, to cooperate with committee on vegetation tests on the availability of phosphoric acid in basic slag), nitrogen (special study of the Kjeldahl method), potash, soils (nitrogenous compounds, lime absorption coefficient), inorganic plant constituents, insecticides and fungicides, and water.]

Subcommittee B: H. C. Lythgoe (1920), (State Department of Health, Boston, Mass.), *Chairman*, J. M. Bartlett (1918), C. A. Browne (1922). [Foods and feeding stuffs (sugar, crude fiber, stock feed adulteration, organic and inorganic phosphorus, water), dairy products (separation of nitrogenous substances in milk and cheese), saccharine products (maple products, honey, sugar house products), drugs (medicinal plants, alkaloids, synthetic products, balsams and gum resins, enzyms), testing of chemical reagents, and microanalytical methods.]

Subcommittee C: R. E. Doolittle (1920), (Transportation Building, Chicago, Ill.), *Chairman*, W. W. Randall (1918), J. P. Street (1922). [Food preservatives, coloring matters in foods, metals in foods, fruits and fruit products, canned vegetables, cereal foods, wines, soft drinks (bottlers' products), distilled liquors, beers, vinegars, flavoring extracts, meat and meat products, (separation of nitrogenous compounds in meat products, meat extracts), eggs and egg products, gelatin, edible fats and oils, spices and other condiments, cacao products, coffee, tea, baking powder.]

Board of Editors.

C. L. Alsberg (Box 744, 11th Street Station, Washington, D. C.), *Chairman.*
C. B. Lipman (1918).
R. E. Doolittle (1919).
L. L. Van Slyke (1920).
E. F. Ladd (1921).

Special Committees.

Editing Methods of Analysis.

R. E. Doolittle (Transportation Building, Chicago, Ill.), *Chairman.*
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G. W. Hoover.
A. J. Patten.
A. F. Seeker (Since deceased).
W. A. Withers.

Vegetation Tests on the Availability of Phosphoric Acid in Basic Slag.

C. B. Williams (College of Agriculture and Mechanic Arts, W. Raleigh, N. C.), *Chairman.*
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H. D. Haskins.
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Committee on Methods of Sampling Fertilizers to Cooperate with a Similar Committee of the American Chemical Society.

C. H. Jones (Agricultural Experiment Station, Burlington, Vt.), *Chairman.*
E. G. Proulx.
B. F. Robertson.

Committee on Revision of Methods of Soil Analysis.

C. B. Lipman (University of California, Berkeley, Calif.), *Chairman*
A. W. Blair.
W. H. McIntire.
E. C. Shorey.
R. Stewart.

MEMBERS AND VISITORS PRESENT, 1917 MEETING.

Albrech, M. C., The R. T. French Company, Rochester, N. Y.
Allison, F. E., Bureau of Markets, Washington, D. C.
Almy, L. H., 1833 Chestnut Street, Philadelphia, Pa.
Alsberg, C. L., Bureau of Chemistry, Washington, D. C.
Anderson, M. S., Bureau of Soils, Washington, D. C.
Appleman, C. O., State College of Agriculture, College Park, Md.
Atkinson, F. C., American Hominy Co., Indianapolis, Ind.

Bailey, C. H., University Farm, St. Paul, Minn.
Bailey, H. S., Southern Cotton Oil Company, Savannah, Ga.
Bailey, L. H., Bureau of Chemistry, Washington, D. C.
Balch, R. T., Bureau of Chemistry, Washington, D. C.
Bartlett, G. M., Joseph Campbell Co., Camden, N. J.
Bartlett, J. M., Agricultural Experiment Station, Orono, Me.
Baston, G. H., Bureau of Markets, Washington, D. C.
Bates, Frederick, Bureau of Standards, Washington, D. C.
Baughman, W. F., Bureau of Chemistry, Washington, D. C.
Beal, W. H., States Relations Service, Washington, D. C.
Bennett, Miss B. M., States Relations Service, Washington, D. C.
Bidwell, G. L., Bureau of Chemistry, Washington, D. C.
Bigelow, W. D., National Canners Association, 1739 H Street, N. W., Washington, D.C.
Blair, A. W., Agricultural Experiment Station, New Brunswick, N. J.
Blanck, F. C., National Canners Association, Easton, Md.
Bohart, G. H., National Canners Association, 1739 H Street, N. W., Washington, D. C.
Bohn, R. M., Advance Malt Products Co., 305 South La Salle Street, Chicago, Ill.
Borden, N. H., Sherwin-Williams Co., Chicago, Ill.
Boyle, Martin, Bureau of Chemistry, Washington, D. C.
Boyles, F. M., McCormick & Co., Baltimore, Md.
Brackett, R. N., Clemson Agricultural College, Clemson College, S. C.
Bradbury, C. M., Department of Agriculture and Immigration, Richmond, Va.
Bradley, L. W., State Department of Agriculture, Atlanta, Ga.
Brattain, P. H., Corby Co., Langdon, D. C.
Breckenridge, J. E., American Agricultural Chemical Co., Carteret, N. J.
Brewster, J. F., Bureau of Chemistry, Washington, D. C.
Broughton, L. B., State College of Agriculture, College Park, Md.
Brown, B. E., Bureau of Plant Industry, Washington, D. C.
Brown, H. H., Pegepscot Paper Co., Brunswick, Me.
Browne, C. A., New York Sugar Trade Laboratory, 80 South Street, New York, N. Y.
Buckner, G. D., Agricultural Experiment Station, Lexington, Ky.

Campbell, V. H., Gibbs Preserving Co., Baltimore, Md.
Carothers, J. N., Bureau of Soils, Washington, D. C.
Cathcart, C. S., Agricultural Experiment Station, New Brunswick, N. J.
Charron, A. T., Official Provincial Laboratory, St. Hyacinthe, Canada.
Chesnut, V. K., Bureau of Chemistry, Washington, D. C.
Churchill, J. B., 80 South Street, New York, N. Y.
Clarke, W. F., Bureau of Chemistry, Washington, D C.
Clay, C. L., State Board of Health, New Orleans, La.
Coleman, D. H., Bureau of Markets, Washington, D. C.

Collins, W. D., Geological Survey, Washington, D. C.
Cook, F. C., Bureau of Chemistry, Washington, D. C.
Craig, R. S., City Health Department, Baltimore, Md.
Custis, H. H., Bureau of Animal Industry, Washington, D. C.

Daudt, H. W., Jackson Laboratory, E. I. Du Pont Co., Wilmington, Del.
Davidson, J., Bureau of Chemistry, Washington, D. C.
Davis, R. A., Post Office Department, Washington, D. C.
DeBord, G. G., Department of Medicine and Hygiene, Harvard Medical School, Boston, Mass
Deuel, H. J., Home Economic Laboratory, White Bear Lake, Minn.
Doolittle, R. E., Transportation Building, Chicago, Ill.
Doyle, Miss A. M., 1365 Oak Street, N. W., Washington, D. C.
Dubois, W. L., Berlin Arcade Building, Milwaukee, Wis.
Du Mez, A. G., Hygienic Laboratory, Washington, D. C.
Dunbar, P. B., Bureau of Chemistry, Washington, D. C.
Dyer, D. A., Bureau of Animal Industry, Washington, D. C.

Edmonds, J., Davison Chemical Co., Baltimore, Md.
Eimer, W. R., 205 Third Avenue, New York, N. Y.
Ellett, W. B., Agricultural Experiment Station, Blacksburg, Va.
Emery, W. O., Bureau of Chemistry, Washington, D. C.
Emmons, F. W., Washburn-Crosby Co., Minneapolis, Minn.
Enslow, L. H., Miraflores Filtration Plant, Ancon, Panama, C. Z.
Ewing, C. O., United Drug Company, Boston, Mass.

Fellers, C. R., Sanitary Inspector, New Brunswick, N. J.
Ferris, L. W., Bureau of Chemistry, Washington, D. C.
Fitzgerald, F. F., National Canners Association, 1739 H Street, N. W., Washington, D. C.
Flint, E. R., States Relations Service, Washington, D. C.
Frary, G. G., State Food and Drug Commission, Vermilion, S. Dak.
Frear, William, Agricultural Experiment Station, State College, Pa.
French, D. M., Alexandria Fertilizer & Chemical Co., Alexandria, Va.
Frisbie, W. S., State Department of Agriculture, Lincoln, Nebr.
Fry, W. H., Bureau of Soils, Washington, D. C.
Fuller, A. V., Bureau of Animal Industry, Washington, D. C.
Fuller, H. C., Institute of Industrial Research, Washington, D. C.
Furber, F. B., Lederle Laboratories, New York, N. Y.

Garby, C. D., Bureau of Chemistry, Washington, D. C.
Gardiner, R. F., Bureau of Soils, Washington, D. C.
Gascoyne, W. J., Gascoyne & Co., Inc., Baltimore, Md.
Geagley, W. C., State Food and Drug Department, Lansing, Mich.
Geidel, C. D., State Dairy and Food Department, Old Capitol, St. Paul, Minn.
Gillespie, L. J., Bureau of Plant Industry, Washington, D. C.
Goodrich, C. E., Bureau of Chemistry, Washington, D. C.
Gordon, W. O., Industrial Appliance Co., Chicago, Ill.
Gowen, P. L., National Canners Association, Easton, Md.
Grab, E. G., National Fruit Product Co., Washington, D. C.
Graham, J. J. T., Bureau of Chemistry, Washington, D. C.
Grant, D. H., Bureau of Chemistry, Washington, D. C.

Grant, E. H., Wm. S. Merrell Co., Cincinnati, Ohio.
Gray, M. A., Chemist, Minneapolis, Minn.
Griffin, E. L., Bureau of Chemistry, Washington, D. C.

Hand, W. F., Agricultural and Mechanical College, Agricultural College, Miss.
Harris, H. L., Pacific Coast Borax Co., 100 William Street, New York, N. Y.
Hart, B. R., 530 St. Paul Street, Baltimore, Md.
Hartwell, B. L., Agricultural Experiment Station, Kingston, R. I.
Haskins, H. D., Agricultural Experiment Station, Amherst, Mass.
Haywood, J. K., Bureau of Chemistry, Washington, D. C.
Hazard, I. W., Red Wing Preserving Co., Fredonia, N. Y.
Hazen, William, Bureau of Soils, Washington, D. C.
Heath, W. H., Bureau of Chemistry, Washington, D. C.
Hellmuth, E. A., National Canners Association, 1739 H Street, N. W., Washington, D. C.
Hoagland, D. R., Agricultural Experiment Station, Berkeley, Calif.
Holmes, A. D., Jackson Laboratory, E. I. Du Pont Co., Wilmington, Del.
Hoover, G. W., U. S. Food and Drug Inspection Station, Transportation Building, Chicago, Ill.
Hortvet, Julius, State Dairy and Food Department, St. Paul, Minn.
Houghton, H. W., Hygienic Laboratory, Washington, D. C.
Howes, C. C., Davison Chemical Co., Baltimore, Md.
Hoyt, C. F., 318 Federal Building, Salt Lake City, Utah.
Hubbard, W. S., U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, New York, N. Y.
Hurst, L. A. Bureau of Plant Industry, Washington, D. C.
Huston, H. A., 42 Broadway, New York, N. Y.

Ingle, M. J., Albion, N. Y.
Irwin, W. H., Swift & Co., Chicago, Ill.
Irwin, Mrs. W. H., Swift & Co., Chicago, Ill.

Jackson, R. F., Bureau of Standards, Washington, D. C.
Jacobs, B. R., Bureau of Chemistry, Washington, D. C.
Jamieson, G. S., Bureau of Chemistry, Washington, D. C.
Jarrell, T. D., Bureau of Chemistry, Washington, D. C.
Johns, C. O., Bureau of Chemistry, Washington, D. C.
Johnson, J. M., Hygienic Laboratory, Washington, D. C.
Jones, C. H., Agricultural Experiment Station, Burlington, Vt.
Jones, W. P., Union Trust Building, Washington, D. C.

Kebler, L. F., Bureau of Chemistry, Washington, D. C.
Keenan, G. L., Bureau of Chemistry, Washington, D. C.
Keister, J. T., Bureau of Chemistry, Washington, D. C.
Kellogg, J. W., State Department of Agriculture, Harrisburg, Pa.
Klein, David, Hollister-Wilson Laboratories, Chicago, Ill.
Knight, H. L., States Relations Service, Washington, D. C.
Knight, O. D., Insecticide and Fungicide Board, Washington, D. C.
Krazbill, H. R., Bureau of Plant Industry, Washington, D. C.
Kunke, W. F., Bureau of Chemistry, Washington, D. C.

Langenbeck, Karl, Agricultural Lime Bureau of the National Lime Manufacturers, Washington, D. C.
Lathrop, E. C., 8096 Du Pont Building, Wilmington, Del.
LeClerc, J. A., Miner-Hillard Milling Co., Wilkes-Barre, Pa.
LeCompte, T. R., Bureau of Soils, Washington, D. C.
Lepper, H. A., Bureau of Chemistry, Washington, D. C.
Lewis, H. F., University of Maine, Orono, Me.
Linder, W. V., Bureau of Internal Revenue, Washington, D. C.
Lipman, C. B., University of California, Berkeley, Calif.
Lodge, F. S., Armour Fertilizer Works, Chicago, Ill.
Lynch, W. D., Bureau of Chemistry, Washington, D. C.
Lyons, Mrs. M. A., Bureau of Chemistry, Washington, D. C. (Since deceased.)
Lythgoe, H. C., State Department of Health, Boston, Mass.

McCall, A. G., Agricultural Experiment Station, College Park, Md.
McDonnell, C. C., Bureau of Chemistry, Washington, D. C.
McDonnell, H. B., Agricultural Experiment Station, College Park, Md.
McGeorge, W. T., U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, San Francisco, Calif.
McIntire, W. H., Agricultural Experiment Station, Knoxville, Tenn.
Magnuson, H. P., Bureau of Soils, State Farm, Lincoln, Nebr.
Magruder, E. W., F. S. Royster Guano Co., Norfolk, Va.
Makemson, W. K., Bureau of Markets, New York, N. Y.
Mallory, G. M., Bureau of Internal Revenue, Washington, D. C.
Mason, G. F., H. J. Heinz Co., Pittsburgh, Pa.
Mathewson, W. E., Bureau of Chemistry, Washington, D. C.
Menge, G. A., Hires Condensed Milk Co., 913 Arch Street, Philadelphia, Pa.
Middleton, E. S., National Canners Association, 1739 H Street, N. W., Washington, D. C.
Miller, C. F., Bureau of Soils, Washington, D. C.
Miller, H. M., National Canners Association, Los Angeles, Calif.
Mitchell, A. S., Bureau of Chemistry, Washington, D. C.
Monarch, J. L., Bureau of Chemistry, Washington, D. C.
Morgan, W. J., Bureau of Chemistry, Washington, D. C.
Mory, A. V. H., N. K. Fairbanks Co., Cincinnati, Ohio.
Munch, J. C., Bureau of Chemistry, Washington, D. C.

Nealon, E. J., Bureau of Chemistry, Washington, D. C.
Nelson, E. K., Bureau of Chemistry, Washington, D. C.
Nollau, E. H., E. I. Du Pont Co., Wilmington, Del.

Oberhelman, G. O., Bureau of Chemistry, Washington, D. C.
O'Neill, A. T., State College of Agriculture, College Park, Md.

Palkin, Samuel, Bureau of Chemistry, Washington, D. C.
Palmer, H. E., Bureau of Chemistry, Washington, D. C. (Since deceased.)
Palmore, J. I., Bureau of Chemistry, Washington, D. C.
Parkins, J. H., F. S. Royster Guano Co., Norfolk, Va.
Parkinson, Miss N. A., Bureau of Chemistry, Washington, D. C.
Patten, A. J., Agricultural Experiment Station, E. Lansing, Mich.
Patten, H. E., Bureau of Chemistry, Washington, D. C.

Patterson, H. J., Agricultural Experiment Station, College Park, Md.
Phelps, F. P., Bureau of Standards, Washington, D. C.
Phelps, I. K., Bureau of Chemistry, Washington, D. C.
Pingree, M. H., American Agricultural Chemical Co., Baltimore, Md.
Pope, W. B., 1416 Newton Street, N. W., Washington, D. C.
Powdermaker, Miss Florence, States Relations Service, Washington, D. C.
Pozen, M. A., Health Department, Washington, D. C.
Price, T. M., 1811 Irving Street, Washington, D. C.
Proulx, E. G., Agricultural Experiment Station, La Fayette, Ind.
Pulizzi, T. O., Bureau of Chemistry, Washington, D. C.

Quaintance, C. F., Coors Porcelain Co., Golden, Colo.

Rabak, Frank, Bureau of Plant Industry, Washington, D. C.
Randall, W. W., State Department of Health, 16 W. Saratoga Street, Baltimore, Md.
Rask, O. S., Bureau of Chemistry, Washington, D. C.
Rather, J. B., Agricultural Experiment Station, Fayetteville, Ark.
Read, Miss E. A., Bureau of Chemistry, Washington, D. C.
Redfield, H. W., U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, New York, N. Y.
Reed, J. B., Bureau of Chemistry, Washington, D. C.
Remington, R. E., Agricultural Experiment Station, Fargo, N. Dak.
Remsburg, C. G., State College of Agriculture, College Park, Md.
Roark, R. C., General Chemical Company, Baltimore Works, Baltimore, Md.
Robb, J. B., State Department of Agriculture, Richmond, Va.
Roberts, O. S., Agricultural Experiment Station, La Fayette, Ind.
Robinson, C. H., Dominion Experimental Farms, Ottawa, Canada.
Rodes, William, Agricultural Experiment Station, Lexington, Ky.
Ross, B. B., Polytechnic Institute, Auburn, Ala.
Ross, S. H., Cudahy Packing Co., E. Chicago, Ind.
Ross, W. H., Bureau of Soils, Washington, D. C.
Round, L. A., State House, Providence, R. I.
Rudnick, Paul, Armour & Company, Chicago, Ill.
Runkel, Homer, Bureau of Chemistry, Washington, D. C.
Runyan, E. G., Hutchins Building, Washington, D. C.
Ruprecht, R. W., F. W. Lunnell & Co., Philadelphia, Pa.

Sale, J. W., Bureau of Chemistry, Washington, D. C.
Schreiner, Oswald, Bureau of Plant Industry, Washington, D. C.
Seeker, A. F., U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, New York, N. Y. (Since deceased.)
Seidell, Atherton, Hygienic Laboratory, Washington, D. C.
Sellers, W. S., American Can Company, New York, N. Y.
Shorey, E. C., 2706 North Harrison Street, Wilmington, Del.
Shrader, J. H., Bureau of Plant Industry, Washington, D. C.
Shulenberger, F. W., Eimer & Amend, New York, N. Y.
Sievers, A. F., Bureau of Plant Industry, Washington, D. C.
Silberberg, Miss B. H., Bureau of Chemistry, Washington, D. C.
Sindall, H. E., Austin, Nichols & Co., Inc., New York, N. Y.
Sive, B. E., Bureau of Standards, Washington, D. C.
Smalley, F. M., Southern Cotton Oil Co., Savannah, Ga.
Smith, C. R., Bureau of Chemistry, Washington, D. C.

Smith, E. E., Bureau of Chemistry, Washington, D. C.
Smith, H. R., U. S. Food and Drug Inspection Station, Park Avenue Building, Baltimore, Md.
Smith, J. G., Bureau of Soils, Washington, D. C.
Smither, F. W., Bureau of Standards, Washington, D. C.
Spears, H. D., Agricultural Experiment Station, Lexington, Ky.
Spencer, G. C., Bureau of Chemistry, Washington, D. C.
Stillwell, A. G., Stillwell Laboratories, New York, N. Y.
Street, J. P., 405 Indiana Street, Indianapolis, Ind.
Sullivan, A. L., State Food and Drug Commission, 16 W. Saratoga Street, Baltimore, Md.
Sutton, C. G., B. B. Culture Laboratory, 176 Palisade Avenue, Yonkers, N. Y.

Taber, W. C., U. S. Food and Drug Inspection Station, Park Avenue Building, Baltimore, Md.
Taylor, G. B., Bureau of Animal Industry, Washington, D. C.
Taylor, J. N., Bureau of Animal Industry, Washington, D. C.
Thatcher, A. S., Loose-Wiles Biscuit Co., Long Island City, New York, N. Y.
Thomas, E. O., Norfolk, Va.
Thompson, E. C., Director of Laboratories, New York, N. Y.
Thornton, E. W., State Department of Agriculture, Raleigh, N. C.
Todd, A. R., State Food and Drug Department, Lansing, Mich.
Toll, J. D., The American Fertilizer, Philadelphia, Pa.
Tolman, L. M., Wilson & Company, Chicago, Ill.
Treuthardt, E. L. P., Munitions Building, Washington, D. C.
Trowbridge, P. F., Agricultural Experiment Station, Agricultural College, N. Dak.

Valaer, Peter, jr., Bureau of Internal Revenue, Washington, D. C.
Van Slyke, L. L., Agricultural Experiment Station, Geneva, N. Y.
Veitch, F. P., Bureau of Chemistry, Washington, D. C.
Viehoever, Arno, Bureau of Chemistry, Washington, D. C.
Vollertsen, J. J., Morris & Company, Chicago, Ill.

Walton, G. P., Bureau of Chemistry, Washington, D. C.
Weber, F. C., Bureau of Chemistry, Washington, D. C.
Weems, J. B., State Department of Agriculture, Richmond, Va.
Wessling, Miss H. L., States Relations Service, Washington, D. C.
Wherry, E. T., Bureau of Chemistry, Washington, D. C.
White, W. S., City Food and Drug Inspection, Cleveland, Ohio.
Wihlfahrt, J. E., Fleischmann Co., New York, N. Y.
Wiley, H. W., Woodward Building, Washington, D. C.
Wiley, S. W., Wiley & Co., Inc., Baltimore, Md.
Wilson, J. B., Bureau of Chemistry, Washington, D. C.
Withers, W. A., College of Agriculture and Mechanic Arts, Raleigh, N. C.
Wright, C. D., Bureau of Chemistry, Washington, D. C.

Yanovsky, Elias, Norwalk Tire and Rubber Co., Norwalk, Conn.

PRESIDENT'S ADDRESS[1] [2].

INSECTICIDE AND FUNGICIDE LEGISLATION IN THE UNITED STATES, WITH ESPECIAL REFERENCE TO THE FEDERAL INSECTICIDE ACT OF 1910.

By J. K. HAYWOOD (Bureau of Chemistry, Washington, D. C.), *President.*

MEMBERS OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS:

I have read carefully the addresses of past presidents of this association and find they have so fully covered the work of the association that I feel I can not add anything that will be of interest to you. I have, therefore, taken the liberty of departing, to some extent, from the time-honored custom of addressing you relative to the direct work of this association—either past, present or future—and have chosen for my subject "Insecticide and Fungicide Legislation in the United States, with Especial Reference to the Federal Insecticide Act of 1910". While this subject is one which does not specifically deal with the work of the association, I believe that it will interest many, if not all of you, since it is closely allied to our work and is of direct interest to all who are engaged in the enforcement of insecticide and fungicide laws.

I will not attempt to follow in detail the numerous insecticide and fungicide laws in the various States which have been enacted and repealed, but will attempt to explain the State laws as they existed seven years before the passage of the Insecticide Act of 1910; in a general way, the history of the Insecticide Act of 1910, the provisions of the act, and the method of its enforcement; and the State laws as they exist at the present time, seven years after the enactment of the Insecticide Act of 1910.

EARLY STATE INSECTICIDE AND FUNGICIDE LAWS.

In 1903, seven years before the passage of the Insecticide Act of 1910, only six States had passed insecticide laws[3], namely, California, Louisiana, New York, Oregon, Texas and Washington.

In so far as I can learn, the first insecticide law passed in the United States was Act No. 131 of the General Assembly of the State of Louisiana[4],

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[1] Presented Tuesday morning, November 20, 1917, as special order of business for 11.30 o'clock.

[2] Since this address was prepared, the writer has discovered that certain States had passed insecticide and fungicide laws which should properly have been included in the address. Reference is therefore made to the following State laws: Colorado, Connecticut, Kentucky, Ohio, and Wisconsin [U. S. Dept. Agr., S. R. A., Insecticide, 21: (1918)]. Subsequent to November, 1917, a considerable number of new State insecticide and fungicide laws have been passed which will shortly be reported in the Service and Regulatory Announcements of the Insecticide and Fungicide Board of the United States Department of Agriculture.

[3] U. S. Bur. Chem. Bull. 76: (1903), 57.

[4] Acts of Louisiana, 1890, No. 131, 171; U. S. Bur. Chem. Bull. 76: (1903), 58.

relative only to Paris green, approved by the governor July 10, 1890, and becoming effective September 1, 1890. This law, among other provisions, required the board of agriculture or the commissioner of agriculture to distribute circulars each season, setting forth the brands of Paris green and their percentage of arsenic as claimed by the dealer, and directed that brands containing 50 per cent or more of arsenic be classed as "strictly pure" and brands containing less than 50 per cent of arsenic be classed as "impure". It further directed that the commissioner of agriculture prepare labels for Paris green marked "Guaranteed Strictly Pure" or "Guaranteed Impure", and containing certain other information. These labels were to be furnished to the manufacturer on payment of a certain sum, if said manufacturers had complied with the various provisions of the act. It was made the duty of every person offering Paris green for sale as an insecticide to attach one of these labels to each of their packages of Paris green, a violation of this provision being punishable by a fine. It will thus be noted that the law, to all intents and purposes, established a standard for Paris green, which standard required that pure green contain 50 per cent of arsenic. It is probable that the law really meant 50 per cent of white arsenic (As_2O_3) and should have so stated, since even the purest Paris green does not contain so much as 50 per cent of metallic arsenic.

The next State insecticide law passed in the United States was an act to amend the agricultural law of the State of New York[1] to prevent fraud in the sale of Paris green. This act became a law March 23, 1898 by the approval of the governor. It required that State manufacturers and dealers in original packages of Paris green file a certificate with the commissioner of agriculture, setting forth the brand of Paris green sold, the number of pounds contained in each package offered for sale, the name of the manufacturer and the place of manufacture, and the amount of arsenic that the Paris green contained. This statement was considered a guarantee to the purchaser that every package of Paris green contained not less than the amount of arsenic set forth in the statement. It further directed that the commissioner of agriculture furnish dealers and State manufacturers of Paris green, who had complied with the above-mentioned provision of the act, with a certificate which would authorize them to deal in Paris green in New York State. The law required that all Paris green, or any product analogous to it, sold or offered for sale in the State as such, contain at least 50 per cent of arsenious oxid, thus establishing a legal standard for this article. Fines for violations of the act were included and a method of enforcing the law outlined.

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[1] Laws of New York, 1898, **1**: ch. 113, 215; U. S. Bur. Chem. Bull. **76**: (1903), 59.

In Bulletin 204 of the Geneva (New York) Agricultural Experiment Station, published December 1910, it is stated:

> AMENDMENT TO THE PARIS GREEN LAW.
>
> In accordance with the suggestions made by us last year, that portion of the Paris green law which related to the definition of Paris green was changed. The essential portion of the amended law embodying this change is as follows:
>
> Paragraph 112. Composition of Paris green or analogous products. Paris green, or any product analogous to it, when sold, offered or exposed for sale, as such, in this state, shall comply with the following requirements:
> First. It shall contain arsenic in combination with copper, equivalent to not less than fifty per centum of arsenious oxid.
> Second. It shall not contain arsenic in water-soluble forms equivalent to more than three and one-half per centum of arsenious oxid.

Upon looking up this supposed amendment in the New York State statutes, I failed to find it, and have been informed by the Chief of the Bureau of Plant Industry of the New York Department of Agriculture that, while the amendment was prepared, it did not become a law.

In 1899 two States, Oregon and Texas, passed insecticide laws or combined insecticide and fungicide laws. The Oregon[1] law was approved by the governor February 17, 1899, and became effective at once. This law made it unlawful for any person or corporation doing business in the State to sell Paris green, arsenic, London purple, sulphur or any spray material or compound for spraying purposes, in quantities exceeding one pound, without providing with each package sold a certificate signed by the seller, guaranteeing the quality and per cent of purity of the materials. It required that all of the materials mentioned conform to the certificate furnished, and provided a fine for a violation of the act. A method of carrying out the provisions of the act was also outlined.

The Texas[2] law was approved March 25, 1899, and became effective at once. This law was entitled "AN ACT for the better protection of the farmer in the purchase of commercial fertilizers and commercial poisons used for destroying bollworms and other pests". Relative to insecticides, it required that before any commercial poison, or any chemical or mixture used as a commercial poison, such as London purple, arsenic, Paris green or any poison used for the purpose of destroying the bollworm or other pests, be sold or offered for sale in the State, a fair sample be taken by the manufacturer, agent, importer or party selling the product, be sent to the professor of chemistry of the Agricultural and Mechanical College under seal, who was in turn to have the sample analyzed after he had been paid an analysis fee of fifteen dollars by the consignor. It was directed that the analysis be printed in the form of a label, which

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[1] General Laws of Oregon, 1899, H. B. 238, 98; U. S. Bur. Chem. Bull. 76: (1903), 61.
[2] General Laws of Texas, 1899, ch. 46, 64; U. S. Bur. Chem. Bull. 76: (1903), 61.

was to bear the name of the manufacturer, the brand of the commercial poison, the essential ingredients contained in the commercial poison and the money value of said poison, and was to be furnished to the manufacturer or dealer at a certain cost. It was made mandatory for each package or quantity of commercial poison to bear the above-mentioned label, and a fine was provided for a violation of any of the provisions of the act. The law also authorized the professor of chemistry to take samples of commercial poisons for analysis to compare them with samples furnished by the manufacturer, and authorized agriculturists or farmers or purchasers of commercial poisons in the State to take samples under official rules and regulations and forward them for free analysis to the professor of chemistry above-mentioned.

In 1901 two more States, Washington and California, passed insecticide laws or combined insecticide and fungicide laws. The Washington[1] law was approved by the governor February 26, 1901, and apparently became effective at once. This law declared it unlawful for any person, firm or corporation doing business in the State of Washington to sell or offer for sale adulterated or low-grade Paris green, arsenic, London purple, sulphur or any spray material or compound for spraying purposes. The law set the following standard for Paris green, that it contain not less than 50 per cent of arsenic trioxid in combination and not more than 4 per cent of water-soluble arsenic trioxid; and also the following standard for commercial arsenic, that it contain not less than 96 per cent of arsenic trioxid. A fine for a violation of the act was provided, as well as a method for its enforcement.

The California[2] act became a law by constitutional limitation February 28, 1901, and only applied to Paris green to be used as an insecticide. It provided that all manufacturers in the State and all dealers in original packages of Paris green, which was used as an insecticide and was manufactured outside the State, submit a sample of the Paris green to the State Agricultural Experiment Station before it was offered or exposed for sale. The sample was to be accompanied by a statement giving the brand of Paris green, the number of pounds in each package to be placed on the market, the name of the manufacturer, place of manufacture, and the amount of combined arsenic contained in the Paris green. This statement was to be considered as constituting a guarantee to the purchaser that the Paris green contained not less combined arsenic than the amount stated. It further specified that the Director of the State Agricultural Experiment Station give all parties who had complied with the above-mentioned provision of the act a

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[1] Session Laws of Washington, 1901, ch. 22, 19; **U. S. Bur. Chem. Bull. 76:** (1903), 63.

[2] Statutes and Amendments to the Codes of California, 1901, ch. 53, 69; **U. S. Bur. Chem. Bull. 76:** (1903), 57.

certificate authorizing them to deal in Paris green in California. It further provided that no person should be entitled to deal in Paris green unless he held such a certificate. The law prescribed the following standard for Paris green: That it contain at least 50 per cent of arsenious oxid and not contain more than 4 per cent of the same in the uncombined state. Other provisions of the law provided penalties and a method for its enforcement.

THE FEDERAL INSECTICIDE AND FUNGICIDE ACT, KNOWN AS "THE INSECTICIDE ACT OF 1910".

HISTORY.

It may reasonably be claimed that the Insecticide Act of 1910 was the incentive for the more comprehensive and more intelligent State legislation relative to insecticides and fungicides such as exists in the United States at the present time. The credit of suggesting Federal legislation relative to this subject is to be given to the Association of Economic Entomologists of the United States, and more especially to Professor E. D. Sanderson of that association. On January 7, 1908 Professor Sanderson, Director of the New Hampshire Agricultural Experiment Station, wrote to Dr. H. W. Wiley, then Chief of the Bureau of Chemistry, Department of Agriculture, stating that at the December 1907 meeting of the Association of Economic Entomologists, the committee on proprietary insecticides, of which he was chairman, had been instructed to ascertain if there were any possibility of securing an interpretation of the Federal Food and Drugs Act which would bring proprietary insecticides and fungicides within its scope and, if not, to determine whether it was feasible to secure an amendment to the law so that it would cover proprietary insecticides and fungicides. On January 14, 1908 Professor Sanderson was informed by Dr. Wiley that the Federal Food and Drugs Act did not cover insecticides and that in his opinion the matter could best be handled by a special insecticide law.

On February 3, 1908 Professor Sanderson requested that Dr. Wiley formulate a Federal insecticide law. This request was favorably considered by Dr. Wiley, and the preparation of the proposed law was entrusted to the writer, the proposed law being submitted to Professor Sanderson March 4, 1908. Since this was the first draft of the present Federal Insecticide Act, which has been followed to a large extent in subsequent State legislation, it is believed that it will be of some historic interest, and it is therefore printed at the end of this address. It will be noted that the law, as originally drawn up, differed principally from the present law in the following particulars: (1) It applied only to insecticides; (2) it provided in certain cases for larger fines than are provided for in the present law; (3) it directed that the act be enforced by the

Bureau of Chemistry; (4) it defined original unbroken package; (5) it stated that the amount of arsenious oxid in Paris green must be 55 per cent; (6) it stated that arsenic in water-soluble forms in lead arsenate must not be equivalent to more than 1 per cent of arsenic oxid; (7) it did not allow the addition of water to lead arsenate under certain restrictions; (8) it did not declare a product adulterated if it contained substances injurious to vegetation. After this law was submitted to Professor Sanderson, he requested that it be rewritten to cover fungicides. This was done, and the amended law sent to him March 14, 1908. Certain further correspondence was conducted between Professor Sanderson and the Bureau of Chemistry, relative to the desirability of incorporating a section in the law to prevent the sale of insecticides or fungicides which would injure vegetation. It was finally decided to incorporate the paragraph which now constitutes the last paragraph in Section 7 of the act.

The proposed law as finally corrected, with the exception of the clause relative to injuring vegetation, was introduced in the Senate April 6, 1908 (60th Congress, 1st Session), as Senate Bill 6515. Containing the clause relative to injuring vegetation, it was introduced in the House April 20 (calendar day April 25), 1908, by Mr. E. A. Hayes as House Bill 21262, and April 20 (calendar day April 27), 1908, by Mr. F. O. Lowden (60th Congress, 1st Session) as House Bill 21318.

On June 6, 1908 Professor Sanderson wrote to various manufacturers, entomologists and agricultural chemists, inviting them to attend a meeting in New York City on June 18, 1908 to discuss the proposed law introduced as Senate Bill 6515 and House Bill 21318, for the purpose of securing an agreement, so that all could unite upon desirable legislation. The writer was present at this conference. At the meeting certain changes in the bills were agreed to and the presentation of these amendments before the proper authorities was entrusted to a committee composed of Professor E. D. Sanderson, Director of the New Hampshire Agricultural Experiment Station, Durham, N. H.; Professor H. E. Summers, Iowa State Entomologist, Ames, Iowa; J. P. Street, Chemist, Connecticut Agricultural Experiment Station, New Haven, Conn.; R. G. Harris, Grasselli Chemical Company, Cleveland, Ohio; and H. F. Baker, President of the Thomsen Chemical Company, Baltimore, Md. This committee met and organized as an executive committee.

A change in the standard for lead arsenate, outlined in the original proposed bill and incorporated in the House and Senate bills above mentioned, was suggested by certain interested parties. Accordingly, a meeting of manufacturers with the executive committee was called in New York on December 8, 1908. The executive committee was instructed to request that the standard for lead arsenate, appearing in the original bill, be dropped and that the standard for this article, appearing in the present Insecticide Act of 1910, be substituted.

House Bill 21318 was referrred to the Committee on Interstate and Foreign Commerce in the House, but was not considered by the committee during that session of Congress. Senate Bill 6515 was referred to the Committee on Agriculture and Forestry and was reported by Mr. H. E. Burnham with amendments on February 1, 1909 (Report No. 895). The amended bill differed principally from the bill as originally introduced in the Senate in that it dropped the definition of "original unbroken package", changed the total amount of arsenious oxid required in Paris green from 55 to 50 per cent, replaced the original standard for lead arsenate by the standard as it appears in the present Insecticide Act of 1910, permitted the addition of water to lead arsenate under certain restrictions, and contained a clause relative to certain products subject to the act, declaring them to be adulterated if they contained substances injurious to vegetation.

The bill was next introduced as House Bill 2218 in the 61st Congress, 1st Session, on March 18, 1909, by Mr. E. A. Hayes, but still contained some of the provisions objected to by all those interested in the passage of the bill. It was again introduced in the 61st Congress, 2nd Session, February 15, 1910, by Mr. F. O. Lowden as House Bill 20989, in practically the same form as the present Insecticide Act of 1910, except that House Bill 20989 provided that the law be enforced by the Bureau of Chemistry, whereas the present law provides that the law be enforced by such existing bureau or bureaus as the Secretary of Agriculture may designate.

In the Senate the bill was again introduced as Senate Bill 6131 by Mr. T. E. Burton in the 61st Congress, 2nd Session, on February 4, 1910, and referred to the Committee on Agriculture and Forestry. The bill as introduced was practically the same as the amended Senate Bill 6515, reported by Mr. Burnham on February 1, 1909 (Report No. 895). It was reported to the Senate by Mr. Simon Guggenheim March 23, 1910 with amendments (Report No. 436), bringing the Senate Bill in accord with House Bill 20989.

Senate Bill 6131 was passed by the Senate April 4, 1910 (with slight amendments), and referred to the Committee on Interstate and Foreign Commerce in the House on April 5, 1910. It was reported to the House with amendments on April 12, 1910 (Report No. 990). The only amendment worthy of note made in the bill by the House Committee was an amendment to Section 4, evidently intended to direct that the enforcement of the law be carried on by such existing bureau or bureaus of the Department of Agriculture as the Secretary of Agriculture might direct, instead of by the Bureau of Chemistry. The change made in Section 4 was ambiguous. Therefore this section was changed on the floor of the House to read as it appears in the present Insecticide Act of 1910. The bill finally passed the House on April 18, 1910. It was referred back to the Senate and passed that body as amended by the House on April 19, 1910. It was approved by the President, April 26, 1910, and became effective on and after the first day of January, 1911.

DISCUSSION OF THE ACT AND METHOD OF ITS ENFORCEMENT.

The Insecticide Act of 1910 is a Federal[1] enactment designed to prevent the manufacture, sale or transportation in interstate commerce of adulterated or misbranded insecticides, fungicides, lead arsenates, or Paris greens; to prevent the importation of such misbranded and adulterated articles into the United States, and the exportation of such articles from the United States. Under the provisions of the act the Government is empowered to proceed criminally against parties who ship misbranded or adulterated insecticides, fungicides, lead arsenates or Paris greens in interstate commerce; against parties who manufacture, sell or offer for sale any of such misbranded or adulterated articles in the District of Columbia or any of the Territories; against parties who export such articles; and against parties who import such articles, and having imported, deliver or offer to deliver in original unbroken packages.

The Government is further empowered to make seizures of any of such misbranded or adulterated articles which are being transported from one State, Territory or district to another for sale, or which, having been so transported, remain unloaded, unsold, or in original unbroken packages, and furthermore may seize any such misbranded or adulterated articles which are manufactured, sold or offered for sale in the District of Columbia or any Territories of the United States.

The act further authorizes the Government to exclude from the country any such adulterated or misbranded articles, or any such articles as are forbidden entry into, or forbidden to be sold, or restricted in sale in the country in which they are made or from which they are exported, or any such article which is otherwise dangerous to the health of the people of the United States.

Under the criminal section of the act any person who shall be convicted of a violation of the law may be fined, for the first offense not to exceed two hundred dollars; and for each subsequent offense not to exceed three hundred dollars, or may be sentenced to imprisonment not to exceed one year, or by both such fine and imprisonment, in the discretion of the court.

Some of the principal features of this act are as follows:

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[1] U. S. Statutes at Large, 1909–11, 36: (I), 331; U. S. Dept. Agr., Office of the Secretary, Circ. 34, rev.: (1917), 12.

(a) Definite standards for lead arsenates and Paris greens are stated, and it is required that all lead arsenates and Paris greens subject to the act shall conform to these rigid specifications.

(b) All insecticides and fungicides (other than lead arsenates and Paris greens) which contain inert ingredients shall bear a statement upon the face of the principal label of each and every package giving the name and percentage amount of each and every inert ingredient contained therein and the fact that it is inert, or, in lieu of this, a statement of the name and percentage amount of each and every active ingredient which has insecticidal or fungicidal properties, together with the total percentage of inert ingredients.

(c) For insecticides (other than lead arsenates and Paris greens) and for fungicides which contain arsenic or compounds of this metal, a statement must be made on the face of the principal label of the total arsenic, expressed as per cent of metallic arsenic, and total arsenic in water-soluble forms, similarly expressed.

(d) No statement, design or device appearing on the label of an insecticide, fungicide, Paris green or lead arsenate shall be false or misleading in any particular. It will at once be seen that all false or exaggerated claims relative to the efficacy of the article constitute misbranding, and the Government is empowered to institute criminal or seizure proceedings as outlined above.

(e) All insecticides and fungicides (other than lead arsenates and Paris greens) must be up to the standard under which they are sold

(f) No substance or substances shall be contained in any insecticide or fungicide (other than lead arsenates and Paris greens) which shall be injurious to the vegetation on which such articles are intended to be used.

The above provisions are the most important ones in the act. While there are various other requirements not so important, it is by a strict enforcement of these provisions specifically mentioned that the consumer is largely protected against those products which bear misleading claims, which are absolute fakes, and which, while killing insects and fungi, may be injurious to the vegetation on which they are intended to be used.

The law is enforced by a board of four members: A representative from the Bureau of Chemistry; from the Bureau of Plant Industry; from the Bureau of Entomology; and from the Bureau of Animal Industry.

Working under the direction of the representative from the Bureau of Chemistry are chemists, bacteriologists and microscopists, who make examinations of the insecticides, fungicides and disinfectants (other than those used primarily on horses, cattle, sheep, swine, or goats) to determine their composition, and whether or not the claims for the disinfectants are misleading.

The representative from the Bureau of Plant Industry has a corps of plant pathologists, who test the efficacy claims made on the various fungicide labels and determine further whether or not such fungicides are injurious to the vegetation upon which they are to be used.

Under the direction of the representative from the Bureau of Entomology there is a staff of entomologists, who test all entomological claims appearing upon the labels and in the literature and, in certain cases, determine whether the insecticides submitted to them are injurious to vegetation when used as directed.

Working under the direction of the representative from the Bureau of Animal Industry are chemists and bacteriologists, who examine and determine the composition of the various insecticides, fungicides and disinfectants which are to be used primarily on horses, cattle, sheep, swine or goats, and also determine whether the claims made upon the label for such articles are misleading.

In addition to the various scientists actually employed to enforce the Insecticide Act of 1910 in the four bureaus involved, various experts in their respective bureaus are freely consulted and aid the board in determining whether or not the claims for the various products which come under the act are misleading. Among the scientists consulted are pharmacologists and medico-chemical experts in the Bureau of Chemistry, entomologists who are experts along special lines in the Bureau of Entomology, plant pathologists who are experts along special lines in the Bureau of Plant Industry, and veterinarians, animal pathologists and zoologists in the Bureau of Animal Industry.

The board has an executive officer, whose duty it is to direct the activities of all inspectors of the board and see that the various insecticides and fungicides which appear upon the market are collected for examination; to attend to all fiscal and business affairs of the board; and to take all necessary action for carrying out the recommendations of the board, including arrangements for hearings, collection of evidence and preparation of cases for reference to the Solicitor of the Department.

The exact method of collecting and examining samples may be of interest. The inspectors travel throughout the United States on carefully prepared itineraries, and collect samples of insecticides and fungicides for examination and test to determine whether or not they are in violation of the act. These samples are transmitted to the Insecticide and Fungicide Board at Washington, under the seal of the inspectors, with complete records identifying the sample with a specific interstate shipment. These samples are assigned by the board to one or more of the four groups mentioned above that are engaged in the enforcement of the act. If these samples are not used primarily on horses, cattle, sheep, swine or goats, they are submitted to the scientists working in the Bureau of Chemistry, for chemical, bacteriological and microscopical examination; if the samples bear insecticidal claims they are also submitted to the entomologists of the board for tests; if they bear plant pathological claims they are further submitted to the plant pathologists of the board for test; and, if they are samples which are for use primarily on horses, cattle, sheep, swine or goats, they are submitted to the chemists, bacteriologists and consulting scientists of the Bureau of Animal Industry.

If, upon examination, any samples are found to violate the provisions of the law, appropriate charges are prepared covering such violations. These charges are submitted to the board, and, if it is considered that there has been a substantial violation of the law, the manufacturer is cited to a hearing and given an opportunity to show any fault or error in the findings of the Department of Agriculture. If, upon examination, no violation of the law is shown, the case is placed in permanent abeyance. If a non-flagrant violation of the law is shown, the matter is brought to the attention of the manufacturer by correspondence, and he is given an opportunity to correct his labels without resort to the courts. After a manufacturer has answered a citation, a full report of the hearing, together with all the papers in the case, is again submitted to the board for decision as to whether the case shall be placed in permanent abeyance, taken up by correspondence, or prosecuted. If prosecution is decided upon, the executive officer of the board assembles the various reports upon the sample, completes the evidence if necessary, and transmits the case to the Solicitor of the Department of Agriculture with the recommendation of the board. On the basis of the facts and charges presented to him, the Solicitor decides from the legal point of view whether or not in his opinion the law has been violated. If he concludes that the law has been violated, the case is then transmitted to the Secretary of Agriculture for his action. If he concurs in the findings of the board and the Solicitor, the case is transmitted to the Department of Justice and is forwarded by this Department to the proper United States Attorney for prosecution. Upon the conclusion of cases referred to the courts for prosecution, notices of judgment are prepared and published for the information and benefit of the public.

Not only is the law enforced in this manner, but the Insecticide and Fungicide Board issues from time to time Service and Regulatory Announcements and Decisions, interpreting the various provisions of the act for the benefit of shippers and giving them information to aid them in bringing their labels and products into conformity with the provisions of the act. Various basic scientific investigations necessary for the enforcement of the law are carried on, and in some instances the results of the work are published. Thousands of investigations have been made and are being made by the entomologists and plant pathologists of the board to determine whether certain particular ingredients entering into the composition of insecticides and fungicides are active or inert against various classes of insects and fungi. Investigations are also made to determine whether or not specific substances are injurious to the vegetation for which the manufacturer recommends them. The chemists of the board study the methods of manufacturing insecticides and fungicides and the basic reactions that occur in their preparation, develop new insecticides and fungicides, and investigate and evolve new analytical methods for their examination.

PRESENT STATE INSECTICIDE AND FUNGICIDE LAWS.

(Including laws relative to disinfectants.)

At the present time, seven years after the passage of the Insecticide Act of 1910, twenty-one States have passed laws, now in force, relative either to insecticides or fungicides (including disinfectants), or both. These States are: California, Colorado, Idaho, Kentucky, Louisiana, Maine, Maryland, Michigan, Minnesota, Montana, New Hampshire, New Jersey, New York, North Dakota, Ohio, Oregon, Pennsylvania, Washington, Wisconsin, Connecticut and Iowa. Some of these laws were passed before the passage of the Federal Insecticide Act of 1910, but most of them were passed after the Federal law became effective, and are in the main more or less patterned after this law.

No attempt will be made to discuss these various State laws in detail, since it was not contemplated that this address would attempt to give all the facts relative to the various State laws. However, reference will be made to all of these laws and the more important features contained therein.

In 1904 the State of Louisiana[1] passed a Paris green law, which was approved by the governor July 6, 1904, became effective August 9, 1904, and is still in force. The law provides for its enforcement by the Louisiana State Board of Agriculture and Immigration, and for the annual distribution by the board of circulars setting forth the brands of Paris green and their percentage of arsenic as claimed by the dealer, and classes them as "strictly pure" if they contain 50 per cent or more of arsenic, and "impure" if they contain less than 50 per cent of arsenic. It is further required that the Commissioner of Agriculture and Immigration prepare labels for Paris green marked "Guaranteed Strictly Pure" or "Guaranteed Impure" and sell these to dealers for a certain sum, and that dealers in Paris green for insecticidal purposes use these

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[1] **Acts of Louisiana, 1904, No. 174, 355; U. S. Dept. Agr., S. R. A., Insecticide, 13: (1916), 114.**

labels to designate their quality of Paris green. It is provided that any person manufacturing, dealing in, selling or soliciting orders in the State of Louisiana for the sale of Paris green shall, when he has agreed to sell any lot of Paris green, notify in writing the Chief State Inspector of Fertilizers of the sale and all the necessary facts relative to the transaction. The inspector of fertilizers, in turn, is directed to sample the Paris green and forward the sample to the State chemist for analysis. According to the law, no fraudulent Paris green shall be sold in the State, and the price of fraudulent green shall not be collected by any process of law. It is further enacted that Paris green sold in the State or for use in the State, which has not been inspected or stamped, shall be presumed to be fraudulent.

The law also makes it the duty of the manufacturer or dealer in original packages of Paris green in the State to give certain facts to the commissioner of agriculture relative to his product, among which shall be a guarantee of the amount of arsenic in the green, which shall be considered a guarantee to the purchaser, and the law further requires that, upon the filing of such a statement, the commissioner issue a certificate to the manufacturer or dealer, licensing him to sell Paris green in the State. Penalties are provided for a violation of the act.

It will be noted that the law, to all intents and purposes, established a standard for Paris green of 50 per cent of arsenic, which probably means 50 per cent of white arsenic, since even the purest Paris green does not contain so much as 50 per cent of metallic arsenic.

In 1905 the State of North Dakota[1] passed a formaldehyde law, which became effective February 17, 1905, and is still in force. Among other provisions, it requires that formaldehyde when sold, offered or exposed for sale as a fungicide, shall contain at least 40 per cent by weight of formaldehyde and be considered adulterated if it contains less than 38 per cent. Penalties are provided for a violation of the law and a method for its enforcement is outlined.

In 1907 Colorado passed a horticultural inspection law, which was approved April 9, 1907, went into effect in 1909, was amended in 1917[2], and is still in force. Among other provisions, this law states:

> Sec. 10. It shall be deemed a violation of this Act for any one to sell in Colorado, insecticide poisons such as Paris green, London purple, white arsenic, arsenate of lime, arsenate of lead, acetate of lead, arsenate of zinc, cyanid of potassium, hellebore, pyrethrum powder, or any other materials or preparations sold or offered for sale, for the control of insect pests or plant diseases, that are diluted or mixed with other substances, unless the kind and amount of the adulterations or mixtures are conspicuously printed in the English language upon each and every package sold. Upon all packages

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[1] Compiled Laws of North Dakota, 1913, 2: ch. 97; 2346. U. S. Dept. Agr., S. R. A., Insecticide, **13**: (1916), 135.

[2] Laws of Colorado, 1917, ch. 131, 473; U. S. Dept. Agr., S. R. A., Insecticide, **21**: (1918), 435.

> of arsenate of lead or arsenite of zinc sold in paste form, the percentage of water, by weight, must be guaranteed.
>
> The State Entomologist may inspect, examine and make analyses of insecticide, fungicide or other materials held or offered for sale within the State for the purpose of determining their purity, their strength and their value for the destruction of insects or plant diseases in any stage of their development. He shall have free access during all reasonable business hours upon or into any premises or structures to make examinations of insecticides or fungicide materials, and upon tendering payment therefor at the current value, may take any sample or samples for examination, analyses, or tests, the results of which may be published for the information of the public.

Penalties are provided for violations of the act and a method for its enforcement is also outlined.

In 1907 a law was passed by the State of Pennsylvania[1] regulating the sale of Paris green in said State. The law was approved May 29, 1907, and became effective September 1, 1907. Since the law has been repealed and a more comprehensive one passed to take its place, it will not be discussed.

In 1908 the State of Kentucky[2] passed a law known as "An Act for Preventing the Manufacture and Sale of Adulterated or Misbranded Food, Drugs, Medicines and Liquors and Providing Penalties for Violations Thereof", which was approved March 13, 1908, became effective for drugs on and after January 1, 1909, and is still in force, as amended March 23, 1916. This law is largely patterned after the Federal Food and Drugs Act[3], but is, of course, adapted for State use. Under the definition of "drug", it includes Paris green and all other insecticides and fungicides, so that the various provisions of the act which apply to drugs also apply to Paris green and all other insecticides and fungicides. Besides defining adulteration and misbranding, providing for penalties and a method of enforcing the law, etc., it provides for the publication of the analyses and the results of the inspection of samples taken or submitted for examination, under certain restrictions.

On March 23, 1916, an amendment[4] to this law was passed which went into immediate effect, and was designed to provide funds for the enforcement of the law.

In 1908 the State of New York[5] passed a law for the prevention of fraud in sale of Paris green and other substances, which became effective May 18, 1908, and is still in force. The law is applicable to Paris green, arsenate of lead, sulphur, lime sulphids, miscible combinations of mineral or vegetable oils, sulphate of copper, Bordeaux mixture, or any insecticide or fungicide, or essential ingredient thereof, used for

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[1] Laws of Pennsylvania, 1907, No. 235, 309; U. S. Dept. Agr., S. R. A., Insecticide, **13**: (1916), 145.

[2] Acts of Kentucky, 1908, ch. 4, 10; U. S. Dept Agr., S. R. A., Insecticide, **13**: (1916), 110.

[3] U. S. Statutes at Large, 1905–7, **34** (I): ch. 3915, 768; U. S. Dept. Agr., Office of the Secretary, Circ. 21, rev.: (1913), 18.

[4] Acts of the General Assembly of Kentucky, 1916, ch. 44, 486; U. S. Dept. Agr., S. R. A., Insecticide, **21**: (1918), 411.

[5] Laws of New York, 1908, **1**: ch. 279, 766; U. S. Dept. Agr., S. R. A., Insecticide, **13**: (1916), 134.

the control of insects, or fungous diseases or any other purpose, within the State. It requires that State manufacturers and dealers in original packages of the above-mentioned commodities file a certificate with the Commissioner of Agriculture setting forth the brands of the commodities, the number of pounds contained in each package offered for sale, the name of the manufacturer and place of manufacture, the percentages and chemical compositions of all essential substances or ingredients. It further requires that all packages of preparations containing arsenic, free or in combination, bear a statement of the percentage of arsenious oxid or its equivalent, soluble or insoluble in distilled water, and that all packages of all the commodities covered by the act bear a label giving all the facts, which are directed to be filed with the Commissioner of Agriculture.

The law directs that the Commissioner of Agriculture furnish purchasers of original packages of the commodities covered by the law which are manufactured outside the State and are intended to be sold or offered for sale, and manufacturers of the commodities within the State who have complied with the above-mentioned provision of the act, with a certificate which will authorize them to deal in the commodities covered by the law in New York State. The law provides that any person who fails to file the statement shall not be entitled to deal, within the State, in the commodities covered by the law.

The law requires that all Paris green, or products analogous to it, sold, offered or exposed for sale in the State, contain at least 50 per cent of arsenious oxid, and defines insecticides and fungicides in much the same way as they are defined in the Insecticide Act of 1910. The procedure to be followed in taking samples and having them examined is outlined.

In 1909 the State of Minnesota[1] passed an act to prevent deception in the sale of Paris green and other insecticides, which became effective from and after August 1, 1909, and is still in force. The law applies to Paris green and other insecticides, and "insecticide" is defined by the act as Paris green and any other substance or mixture of substances intended to be used for preventing, destroying, repelling or mitigating any and all insects which may infest vegetation. The law requires that Paris green contain at least 50 per cent of arsenious oxid in combination with copper, not more water-soluble arsenic than the equivalent of 3.5 per cent of arsenious oxid, and no substance that injuriously affects its quality or strength, and requires that lead arsenate contain at least 50 per cent of actual lead arsenate, 12.5 per cent of arsenious oxid[2], no more water-soluble arsenic than the equivalent of 1 per cent of arsenic oxid, and no

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[1] Laws of Minnesota, 1909, ch. 62, 60; ch. 100, 91; U. S. Dept. Agr., S. R. A., Insecticide, **13:** (1916), 126.

[2] Probably intended for 12.5 per cent of arsenic oxid.

added substance that will injuriously affect the quality or strength of the lead arsenate.

It is required that the label of the insecticide state in English the name and residence of the manufacturer or distributor or party for whom the insecticide is manufactured, and state the name and, with substantial accuracy, the percentage of each ingredient contained therein. A penalty for the violation of the act and a method for its enforcement are provided.

In 1910 the State of Maryland[1] passed an act relative to foods, drugs, disinfectants, etc., which was approved April 5, 1910, and became effective from and after July 1, 1910. The law is modeled after the Federal Food and Drugs Act, but is adapted for State use and contains the following provision relative to disinfectants:

> That for the purposes of this Act an article shall be deemed to be misbranded:
>
> * * * * * * *
>
> As to disinfectants:
>
> If in the case of disinfectants manufactured or sold in this State, the manufacturers, sales agents or dealers fail to show on the labels the carbolic acid coefficient or relative germicidal strength of such disinfectants as compared with pure carbolic acid; provided, however, that deodorants and antiseptics having no germicidal strength must be plainly labeled and sold as such and such preparations as have no such germicidal strength, shall not be labeled "disinfectants".

Penalties for violations of the act and a method for its enforcement are provided.

During 1911 six States, Montana, Oregon, Idaho, Maine, Wisconsin and California, passed laws relative to the control of insecticides or fungicides, or both.

The Montana[2] law was approved February 15, 1911, became effective at once, and is still in force. It follows closely the provisions of the Insecticide Act of 1910, but is adapted for State use.

A law relative to lime-sulphur solutions and compounds was filed in the office of the Oregon[3] Secretary of State on February 21, 1911, became immediately effective, and is still in force. It requires that no lime and sulphur solution or compound for spraying purposes shall be sold or offered or exposed for sale unless it has a specific gravity of 30° Baumé or more, and contains only products which arise from boiling lime and sulphur in water without the addition of salt or other soluble substances. It further requires that each package of the above-mentioned products be labeled to show the contents of the compound or solution and the gravity thereof. A penalty is provided for a violation of the act.

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[1] Laws of Maryland, 1910, ch. 156, 146; U. S. Dept. Agr., S. R. A., Insecticide, **13**: (1916), 120.
[2] Laws of Montana, 1911, ch. 26, 38; U. S. Dept. Agr., S. R. A., Insecticide, **13**: (1916), 127.
[3] General Laws of Oregon, 1911, ch. 146, 198; U. S. Dept. Agr., S. R. A., Insecticide, **13**: (1916), 144.

The Idaho law relative to insecticides is an amendment to the horticultural law of Idaho[1]. The law was amended in 1911[2] (approved and effective March 8, 1911) to add Sections 1326-C and 1326-D relative to lime-sulphur solution and further amended in 1913[3] (approved and effective February 24, 1913) to add Sections 1326-G and 1326-H relative to lead arsenate, and make changes in Sections 1326-C and 1326-D relative to lime-sulphur solution. Following are the only provisions of the act which are of interest in connection with this paper:

> Sec. 1326-C. All spray solution known as a lime and sulphur liquid shall be conspicuously labeled as to the strength of the solution showing a guaranteed strength of lime and sulphur combined in solution as sulphates and sulphids, of which solution not less than seventy per cent (70 per cent) by weight shall be sulphur, and such label or labels shall also contain a direction as to the proportions of water to be used in any mixture containing a four per cent (4 per cent) solution by weight of lime and sulphur combined as sulphates and sulphids, of which solution not less than seventy per cent (70 per cent) by weight shall be sulphur.
>
> Any violation of the provisions of this section shall constitute a misdemeanor and shall be punished as provided in Section 1324.
>
> Sec. 1326-G. * * * * * *
>
> All arsenate of lead sold for the purpose of being manufactured into arsenate of lead solution shall contain not more than fifty per cent water, not less than twelve and one-half per cent arsenic oxid, not more than three-fourths of one per cent water-soluble arsenic oxid. Any violation of the provisions of this section shall constitute a misdemeanor and shall be punished as provided in Section 1324.

It will be noted that the standard for lime-sulphur solution stated in this act is an impossible standard for lime-sulphur solution, since lime and sulphur do not combine merely to form sulphates and sulphids, but combine to form sulphids, thiosulphates, sulphates and possibly sulphites. A solution of lime-sulphur can not possibly contain 70 per cent by weight of sulphur.

The Maine[4] law, known as "An Act to amend and unify the laws regulating the sale of agricultural seeds, commercial feeding stuffs, commercial fertilizers, drugs, foods, fungicides and insecticides", was approved March 28, 1911, and became effective January 1, 1912. Action was taken to amend and unify the law[5] in 1913, and the law which is now in force became effective January 1, 1914. This law, in so far as it applies to insecticides and fungicides, is modeled closely after the Federal Insecticide Act of 1910, but is adapted for State use. It differs principally from the Federal act in that it requires a statement on the label of the number of net pounds in the package and in that the manufacturer is not given the choice of stating on his label the names and percentage amounts of each and every active ingredient and the total percentage

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[1] Idaho Revised Codes, 1908, **1**: 605; U. S. Dept. Agr., S. R. A., Insecticide, **13**: (1916), 108.

[2] Idaho Session Laws, 1911, ch. 58, 159; U. S. Dept. Agr., S. R. A., Insecticide, **13**: (1916), 108.

[3] *Ibid.*, 1913, ch. 18, 87-8; U. S. Dept. Agr., S. R. A., Insecticide **13**: (1916), 109.

[4] Laws of Maine, 1911, ch. 119, 114; U. S. Dept. Agr., S. R. A., Insecticide, **13**: (1916), 117.

[5] *Ibid.*, 1913, ch. 140, 180; ch. 164, 214; U. S. Dept. Agr., S. R. A., Insecticide, **13**: (1916), 117.

of inert ingredients, in lieu of stating the names and percentage amounts of each and every inert ingredient. The section of the Maine law covering this matter reads as follows:

> For the purpose of this act an article shall also be deemed to be misbranded.
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> * * * * * * *
>
> In case of fungicide and insecticide:
>
> * * * * * * *
>
> Sixth. If it consists partially or completely of an inert substance or substances which do not prevent, destroy, repel, or mitigate insects or fungi and does not have the percentage amount of such inert ingredient plainly and correctly stated on the label.

The Maine law also specifically authorizes the Commissioner of Agriculture to fix and publish standards of purity, quality or strength, when such standards are not fixed by law.

The Oregon[1] law, which is still in force, known as "AN ACT Prohibiting the sale of misbranded insecticides, Paris green, lead arsenate, or fungicide, etc.", was filed in the office of the Secretary of State February 23, 1911, and by reason of its not containing an emergency clause, went into effect May 20, 1911, ninety days from the end of the session of the Legislature Assembly at which it was enacted. The law contains a provision repealing the former State insecticide act of Oregon, mentioned at the beginning of this paper. This law is closely modeled after the Federal Insecticide Act of 1910, but is adapted for State use. The section of the law requiring a statement relative to inert ingredients on the label is different from a similar section in the Federal act and reads as follows:

> That for the purpose of this act an article shall be deemed to be misbranded—
>
> * * * * * * *
>
> In the case of insecticides (other than Paris greens and lead arsenates) and fungicides: * * * *third*, if it consists partially or completely of an inert substance or substances, which do not present [prevent], destroy, repel, or mitigate insects or fungi, and does not have the names and percentage amounts of each and every one of such inert ingredients plainly and correctly stated on the label.

Also, this law contains the following important provision which does not appear in the Federal Insecticide Act:

> That for the purpose of this act an article shall be deemed to be misbranded—
>
> In the case of insecticides, Paris greens, lead arsenates, and fungicides: * * * *fourth*, if the label does not state the chemical formula of the compound or compounds which shall constitute the insecticide, Paris green, lead arsenate or fungicide, contained within the package.

The Wisconsin[2] law was approved and became effective in June 1911, and is still in force as amended in 1915. It is modeled closely after the Federal Insecticide Act, but is adapted for State use. A slight mistake

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[1] General Laws of Oregon, 1911, ch. 205, 328; U. S. Dept. Agr., S. R. A., Insecticide, 13: (1916), 142.

[2] Wisconsin Statutes, 1911, ch. 61, 991; U. S. Dept. Agr., S. R. A., Insecticide, 13: (1916), 150.

was made in quoting one section of this law in Service and Regulatory Announcement, Insecticide, No. 13, U. S. Department of Agriculture, which was due to an error in the Wisconsin State leaflet issued by the entomologist of Wisconsin, giving the text of the law.

Under the section "Misbranding defined.—Section 1494—10v. 2. (b)", the provision relative to active and inert ingredients should read:

> In the case of insecticides (other than Paris green and lead arsenates) and fungicides: * * * third, if it consists partially or completely of an inert substance or substances which do not prevent, destroy, repel or mitigate insects or fungi, and does not have the names and percentage amounts of each and every one of such inert ingredients plainly and correctly stated on the label; that in lieu of naming and stating the percentage amount of each and every *inert* ingredient the producer may at his discretion state plainly upon the label the correct names and percentage amounts of each and every ingredient of the insecticide or fungicide having insecticidal or fungicidal properties, and make no mention of the inert ingredients, except in so far as to state the total percentage of inert ingredients present.

In 1915 the following amendments were made in this law[1]:

A section known as Section 1494–2 was passed, which reads in part as follows:

> Section 1494–2. It shall be the duty of the state entomologist to enforce the laws relating to * * * the inspection of insecticides and fungicides.

Section 1494–10q was repealed.

Section 1494–10w was amended to read as follows:

> A fee not to exceed five dollars may be collected for the examination or analysis of each sample of insecticide or fungicide submitted by any manufacturer, wholesaler, jobber or dealer. Such fees shall be paid into the * * * State treasury.

The California[2] law, as first passed, became effective July 1, 1911, but was amended[3] in 1913, so that at the present time it is in force in the form reported in Service and Regulatory Announcement, Insecticide, No. 13[4]. It specifically repeals the 1901 California Paris green law, previously mentioned in this paper. The law, as at present in force, is largely modeled after the Federal Insecticide Act of 1910; however, it contains certain additional important provisions.

It requires that commercial insecticides and fungicides and materials to be used for insecticidal or fungicidal purposes bear a label stating the name, brand and trade-mark, the name and address of the manufacturer, importer or dealer, the place of manufacture, a correct general statement of the nature and composition, and the total percentage of the substance or substances alleged to have insecticidal or fungicidal properties. It further directs that the provisions of Section 7 of the act, relative to

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[1] Laws of Wisconsin, 1915, ch. 413, 518; U. S. Dept. Agr., S. R. A., Insecticide, **21**: (1918), 446.
[2] Statutes and Amendments to the Codes of California, 1911, ch. 653, 1248.
[3] *Ibid.*, 1913, ch. 211, 363; ch. 612, 1141.
[4] U. S. Dept. Agr., S. R. A., Insecticide, **13**: (1916), 102.

adulteration, shall not apply to an article if the standard of strength, quality or purity be plainly stated on the container, even though the standard differs from that determined by Section 7 of the act. The law also exempts certain insecticidal and fungicidal substances from the application of the act, outlines a method of taking samples and directs that results of analyses and other important information be published at least yearly.

On September 1, 1911, a Texas[1] fertilizer law became effective, which is still in force. This law, in Section 19, repeals Chapter 46 of the General Laws of 1899, previously mentioned. The State of Texas, therefore, has no insecticide law at the present time.

In 1912 "AN ACT to regulate the sale of insecticides" was passed by the State of New Jersey[2], was approved March 19, 1912, became immediately effective, and is now in force. The law, with provisions adapting it to State use, is largely modeled after the Federal Insecticide Act of 1910, except that it is not nearly so comprehensive. The law applies only to insecticides and not to fungicides, and does not include most of the misbranding provisions contained in Section 8 of the Federal Insecticide Act. It does require, however, that every manufacturer of insecticides in the State, and every dealer in original packages of insecticides manufactured outside the State, shall each year submit to the State chemist a statement giving the brand or brands of insecticides to be sold, the name of the manufacturer and place of manufacture and the minimum amount of total arsenic and maximum amount of water-soluble arsenic. If the insecticide does not contain arsenic, the professed standard must be stated. The law further requires that the statement furnished shall be printed and attached to each package sold by the retail dealer and that it shall be considered a guarantee of the composition of the material. It is further directed that if the material is sold in bulk, this guarantee must be attached to the container and a copy of the guarantee given upon a request from the purchaser. The State chemist is directed to issue a certificate to the party filing the above-mentioned statement, which authorizes the party to deal in the brand of insecticide named.

A method of enforcing the act and penalties for its violation are provided, and the State chemist is directed to publish the analyses and other pertinent facts at least annually.

In 1913 the State of North Dakota[3] passed an insecticide and fungicide act, which was approved February 21, 1913, became effective July 1, 1913, and is still in force. This law is patterned very closely after the

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[1] General Laws of Texas, 1911, ch. 109, 218.

[2] Laws of New Jersey, 1912, ch. 89, 122; U. S. Dept. Agr., S. R. A., Insecticide, **13**: (1916), 132.

[3] Compiled Laws of North Dakota, 1913, **2**: ch. 97, 2346; U. S. Dept. Agr., S. R. A., Insecticide, **13**: (1916), 135.

Federal Insecticide Act of 1910. Penalties are provided, as well as a method for the enforcement of the act.

In 1913 the State of Ohio[1] passed "AN ACT to regulate the manufacture and sale of insecticides and fungicides in Ohio", which was approved April 16, 1913, and is still in force in a very slightly modified form. The changes made in the 1913 law consist in using the term "Secretary of Agriculture" wherever the words "The Agricultural Commission of Ohio" are used and in dropping the following words in Section 14, "Any unexpended balance shall be credited to the agricultural fund".

This law is modeled after the Federal Insecticide Act of 1910, but contains certain important additional provisions. It requires that every package of insecticide, fungicide or essential ingredients thereof, sold or manufactured in the State, bear a statement giving the net pounds of solids, the net pounds of paste arsenate of lead on a 50 per cent water basis, the number of gallons of liquids, the name, brand, trade-mark, name of manufacturer and place of manufacture, and the percentages and chemical compositions of all essential substances or ingredients. In the case of lime-sulphur solution, it is required that the label state the degree Baumé and the per cent of sulphur. All the above information is to be considered as constituting a guarantee to the purchaser. A standard is given in the act, both for paste arsenate of lead and powdered arsenate of lead. The law provides penalties for violations of the act and contains provisions outlining the method of enforcement.

In 1913 the State of Michigan[2] passed "AN ACT For preventing the manufacture, sale or transportation of adulterated or misbranded Paris greens, lead arsenates and other insecticides, and also fungicides, and for regulating traffic therein", which was approved May 7, 1913, and is still in force. This law is modeled very closely after the Federal Insecticide Act, but differs from it in one important particular. Instead of the wording of the Federal act relative to the statement of inert ingredients or active and inert ingredients, the Michigan law simply says:

> For the purpose of this act an article shall be deemed to be misbranded,
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> * * * * * * *
>
> In the case of insecticides (other than Paris green and lead arsenates) and fungicides:
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> * * * * * * *
>
> Third, If it does not state plainly upon the label the correct names and percentage amounts of each and every ingredient of the insecticide or fungicide having insecticidal or fungicidal properties and the total percentage of inert ingredients present.

Penalties are provided and a method of enforcing the law outlined.

In 1915 the State of Washington[3] passed "AN ACT Relating to horticulture and horticultural plants and products, etc.", which was

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[1] Legislative Acts and Joint Resolutions of Ohio, 1913, **103**: H. B. No. 230, 161; U. S. Dept. Agr., S. R. A., Insecticide, **13**: (1916), 139.

[2] Public Acts of Michigan, 1913, No. 254, 476; U. S. Dept. Agr., S. R. A., Insecticide, **13**: (1916), 124.

[3] Laws of Washington, 1915, ch. 166, 494; U. S. Dept. Agr., S. R. A., Insecticide, **13**: (1916), 146.

approved by the governor March 19, 1915, became immediately effective, and is still in force. This act, in so far as it applies to insecticides and fungicides, is modeled closely after the Federal Insecticide Act. Penalties are provided and a method of enforcing the act is outlined.

In 1915 the State of New Hampshire[1] passed "AN ACT To regulate the sale and to standardize the strength and purity of fungicides and insecticides", which was approved April 14, 1915, became effective September 1, 1915, and is still in force. This law is modeled after the Federal Insecticide Act, but differs from it in the following important particulars:

Section 1 of the act reads as follows:

> Every lot or package of fungicide or insecticide which is manufactured, sold, distributed, or offered or exposed for sale in this State shall have affixed in a conspicuous place on the outside thereof a plainly printed statement clearly and truly stating the net ounces or pounds in the package or container, the name or trade-mark under which the article is sold, the name and address of the manufacturer or shipper, the place of manufacture, and also a statement of the chemical or physical composition of the material as follows: First, in case of Paris green and lead arsenate, the minimum per centum of total arsenic and the maximum per centum of water-soluble arsenic which it contains; second, in the case of fungicides and insecticides, other than Paris green and lead arsenate, the name and per centum of active ingredients, or the quality or strength under which the material is sold, and in addition the per centum of inert materials which it contains, as hereinafter provided.

The act, in Section 7, has the same requirements relative to a statement of total and water-soluble arsenic and inert ingredients or active and inert ingredients, as the Federal Insecticide Act.

The act also specifically requires a statement on the label of the weight or measure and contains a standard for powdered lead arsenate. Penalties are provided and a method of enforcing the act outlined.

In 1917 Connecticut, Iowa and Pennsylvania passed laws relative to insecticides or fungicides (including disinfectants), or both.

The Connecticut[2] law relates only to disinfectants and is as follows:

> AN ACT concerning the testing and labeling of disinfectants. *Be it enacted by the Senate and House of Representatives in General Assembly convened:*
>
> The receptacle containing any disinfectant for external use, the phenol coefficient of which can be determined by a bactericidal test, manufactured, sold, or offered for sale within the State shall bear a label showing the carbolic acid coefficient or relative germicidal value of such preparation as compared with pure carbolic acid. The relative germicidal value of a disinfectant shall be determined by the application of either the Rideal-Walker or the Hygienic Laboratory method. Any such disinfectant shall be misbranded if the statement contained on the label is false. Any person who shall misbrand any disinfectant within the meaning of this act or shall sell or offer the same for sale shall be fined not more than one hundred dollars, or imprisoned not more than sixty days, or both.

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[1] Laws of New Hampshire, 1915, ch. 118, 135; U. S. Dept. Agr., S. R. A., Insecticide, **13**: (1916), 130.

[2] Public Acts of Connecticut, 1917, ch. 314, 230; U. S. Dept. Agr., S. R. A., Insecticide, **21**: (1918), 437.

The Iowa[1] law, entitled "AN ACT to prevent the manufacture and sale of adulterated or misbranded insecticides and fungicides within the State", was approved April 25, 1917, became effective July 4, 1917, and is still in force. The law is modeled after the Federal Insecticide Act, but contains certain different important provisions, as follows:

It requires that insecticides and fungicides sold in package form be labeled to show the quantity of contents in terms of weight, measure or numerical count and provides that reasonable variations shall be permitted and tolerances established by the State Dairy and Food Commissioner.

The law also has the following requirement relative to lime-sulphur:

> All spray solution known as a lime and sulphur liquid shall be conspicuously labeled as to the strength of the solution, showing a guaranteed strength of lime and sulphur combined in solution as sulphates and sulphids, of which solution not less than seventy per cent, 70 per cent, by weight shall be sulphur, and such label or labels shall also contain a direction as to the proportions of water to be used in any mixture containing a four per cent, 4 per cent, solution by weight of lime and sulphur combined as sulphates and sulphids, of which solution not less than seventy per cent, 70 per cent, by weight shall be sulphur.
>
> Every package of such compound or solution sold, offered or exposed for sale shall be plainly labeled with black faced type, in letters of not less than one-half of an inch in height, stating the contents of the compound or solution and the gravity test thereof.

It will be noted that the standard stated for lime-sulphur solution is an impossible standard, since lime and sulphur do not combine to form only sulphates and sulphids, but to form sulphids, thiosulphates, sulphates and possibly sulphites. Also, lime-sulphur solution can not possibly contain 70 per cent by weight of sulphur. The law provides for penalties and a method for its enforcement.

In the pharmacy laws of the State of Iowa[2] a drug is defined as follows:

> Section 4999—a33. The term "drug", as used in this act, shall include all medicines and preparations recognized in the United States Pharmacopœia or National Formulary for internal or external use, and any substance or mixture of substances intended to be used for the cure, mitigation or prevention of disease of either man or other animals, or for the destruction of parasites.

Therefore, any substance used for the destruction of parasites is subject to the various drug provisions of the pharmacy laws of the State of Iowa.

The Pennsylvania[3] law, known as "AN ACT Preventing the manufacture, sale, or transportation within the Commonwealth of adulterated or misbranded Paris greens, lead arsenates, lime-sulphur compounds, and other insecticides and fungicides, and regulating traffic therein; providing for inspection of such materials, and imposing penalties", was

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[1] Laws of Iowa, 1917, ch. 385, 416; U. S. Dept. Agr., S. R. A., Insecticide, 21: (1918), 438.

[2] Supplement to the Iowa Code, 1913, ch. 10-B, 1817.

[3] Laws of Pennsylvania, 1917, No. 124, 224; U. S. Dept. Agr., S. R. A., Insecticide, 21: (1918), 443.

approved May 17, 1917, became effective June 1, 1917, and is still in force. This law repeals the Pennsylvania Paris green law of May 29, 1907, previously mentioned. The law is modeled after the Federal Insecticide Act, but contains the following important additional provisions:

> Section 2. That it shall be unlawful for any person to defraud any other person by misrepresenting the value of any treatment applied to trees, shrubs, vines, or other plant material, or to any animal, for preventing, destroying, repelling, or mitigating any insects, fungus, or bacterial disease, or for accelerating its growth or productive power.

The act requires that the quantity of contents be plainly and correctly marked on the outside of the package, in terms of weight, measure, or numerical count.

The act does not follow the wording of the Federal Insecticide Act in certain respects, but is changed so as to make the meaning plainer. Fines are provided and a method of enforcing the act outlined.

As a whole, this act is probably the most comprehensive and effective insecticide and fungicide law that is now in force.

FIRST DRAFT OF PRESENT FEDERAL INSECTICIDE ACT OF 1910.

AN ACT For preventing the manufacture, sale, or transportation of adulterated or misbranded Paris greens, lead arsenates, and other insecticides and for regulating traffic therein, and for other purposes.

Be it enacted by the Senate and House of Representatives of the United States of America in Congress assembled, That it shall be unlawful for any person to manufacture within any Territory or the District of Columbia any Paris green or lead arsenate or other insecticide which is adulterated or misbranded, within the meaning of this Act; and any person who shall violate any of the provisions of this section shall be guilty of a misdemeanor, and for each offense shall, upon conviction thereof, be fined not to exceed five hundred dollars or shall be sentenced to one year's imprisonment, or both such fine and imprisonment, in the discretion of the court, and for each subsequent offense and conviction thereof shall be fined not less than one thousand dollars or sentenced to one year's imprisonment, or both such fine and imprisonment, in the discretion of the court.

Sec. 2. That the introduction into any State or Territory or the District of Columbia from any other State or Territory or the District of Columbia, or from any foreign country, or shipment to any foreign country of any Paris green or lead arsenate or other insecticide which is adulterated or misbranded, within the meaning of this Act, is hereby prohibited; and any person who shall ship or deliver for shipment from any State or Territory or the District of Columbia to any other State or Territory or the District of Columbia, or to a foreign country, or who shall receive in any State or Territory or the District of Columbia from any other State or Territory or the District of Columbia, or foreign country, and having so received, shall deliver, in original unbroken packages, for pay or otherwise, or offer to deliver to any other person, any such article so adulterated or misbranded within the meaning of this Act, or any person who shall sell or offer for sale in the District of Columbia or the Territories of the United States any such adulterated or misbranded Paris green, or lead arsenate or other insecticide or export or offer to export the same to any foreign country, shall be guilty of a misdemeanor, and for such offense be fined not exceeding two hundred dollars for the first offense, and upon conviction for each subsequent offense not exceeding three hundred dollars or be imprisoned not exceeding one year, or both, in the discretion of the court; *Provided*, That no article shall be deemed misbranded or adulterated within the provisions of this Act when intended for export to any foreign country and prepared or packed according to the specifications or directions of the foreign purchaser when no substance is used in the preparation or packing thereof in conflict with the laws of the foreign country to which said article is intended to be shipped; but if said article shall be in fact sold or offered for sale for domestic use or consumption, then this proviso shall not exempt said article from the operation of any of the other provisions of this Act.

Sec. 3. That the Secretary of the Treasury, the Secretary of Agriculture, and the Secretary of Commerce and Labor shall make uniform rules and regulations for carrying out the provisions of this Act, including the collection and examination of specimens of Paris greens, lead arsenates and other insecticides, manufactured or offered for sale in the District of Columbia, or in any Territory of the United States, or which shall be offered for sale in unbroken packages in any State other than that in which they shall have been respectively manufactured or produced, or which shall be received from any foreign country, or intended for shipment to any foreign country, or which may be submitted for examination by the Director of the Experiment Station, or agent of any State, Territory, or the District of Columbia, or at any domestic or foreign port through which such product is offered for interstate commerce, or for export or import between the United States and any foreign port or country.

Sec. 4. That the examinations of specimens of Paris greens, lead arsenates and other insecticides shall be made in the Bureau of Chemistry of the Department of Agriculture, or under the direction and supervision of such Bureau, for the purpose of determining from such examination whether such articles are adulterated or misbranded within the meaning of this Act; and if it shall appear from any such examination that any of such specimens are adulterated or misbranded within the meaning of this Act, the Secretary of Agriculture shall cause notice thereof to be given to the party from whom such sample was obtained. Any party so notified shall be given an opportunity to be heard, under such rules and regulations as may be prescribed as aforesaid, and if it appears that any of the provisions of this Act have been violated by such party, then the Secretary of Agriculture shall at once certify the facts to the proper United States district attorney, with a copy of the results of the analysis or the examination of such article duly authenticated by the analyst or officer making such examination, under the oath of such officer. After judgment of the court, notice shall be given by publication in such manner as may be prescribed by the rules and regulations aforesaid.

Sec. 5. That it shall be the duty of each district attorney to whom the Secretary of Agriculture shall report any violation of this Act, or to whom any Director of Experiment Station, or agent of any State, Territory, or the District of Columbia shall present satisfactory evidence of any such violation, to cause appropriate proceedings to be commenced and prosecuted in the proper courts of the United States, without delay, for the enforcement of the penalties as in such case herein provided.

Sec. 6. That the term "insecticide" as used in this Act shall include any substance or mixture of substances intended to be used for destroying, repelling or mitigating any and all insects which may infest vegetation, man or other animals, or households, or be present in any environment whatsoever. The term "Paris green" as used in this Act, shall include the product sold in commerce as "Paris green" and chemically known as "aceto-arsenite of copper". The term "lead arsenate" as used in this Act, shall include the product or products sold in commerce as lead arsenate and consisting chemically of products derived from arsenic acid (H_3AsO_4) by replacing one or more hydrogen atoms by lead. The term "original unbroken package" or "unbroken package", as used in this Act, shall signify the original package, carton, case, can, box, barrel, bottle, phial or other receptacle put up by the manufacturer, to which the label is attached, or which may be suitable for the attachment of a label, making one complete package of the Paris green, lead arsenate or other insecticide. The original package contemplated includes both the wholesale and the retail package.

Sec. 7. That for the purpose of this Act an article shall be deemed to be adulterated—

In the case of Paris green:

First. If it does not contain at least 55 per cent of arsenious oxid.

Second. If it contains arsenic in water-soluble forms equivalent to more than three and one-half per cent of arsenious oxid.

Third. If any substance has been mixed and packed with it so as to reduce or lower or injuriously affect its quality or strength.

In the case of lead arsenate:

First. If it contains more than 50 per cent of water.

Second. If it contains arsenic in water-soluble forms equivalent to more than one per cent of arsenic oxid.

Third. If any substance has been mixed and packed with it so as to reduce or lower or injuriously affect its quality or strength.

In the case of insecticides, other than Paris green:

First. If its strength or purity falls below the professed standard or quality under which it is sold.

Second. If any substance has been substituted wholly or in part for the article.

Third. If any valuable constituent of the article has been wholly or in part abstracted.

Sec. 8. That the term "misbranded" as used herein shall apply to all Paris greens, lead arsenates, or other insecticides, or articles which enter into the composition of insecticides, the package or label of which shall bear any statement, design, or device regarding such article, or the ingredients or substances contained therein which shall be false or misleading in any particular and to any Paris green, lead arsenate or other insecticide which is falsely branded as to the State, Territory or country in which it is manufactured or produced.

That for the purpose of this Act an article shall be deemed to be misbranded—

In the case of Paris green, lead arsenates and insecticides other than Paris greens:

First. If it be an imitation or offered for sale under the name of another article.

Second. If it be labeled, or branded so as to deceive or mislead the purchaser, or if the contents of the package as originally put up shall have been removed in whole, or in part and other contents shall have been placed in such package.

Third. If in package form, and the contents are stated in terms of weight, or measure, they are not plainly and correctly stated on the outside of the package.

In the case of insecticides other than Paris greens:

First. If it contains arsenic in any of its combinations or in the elemental form and the total amount of arsenic present (expressed as per cent of metallic arsenic) is not stated on the label.

Second. If it contains arsenic in any of its combinations or in the elemental form and the amount of arsenic in water-soluble forms (expressed as per cent of metallic arsenic) is not stated on the label.

Third. If it consists partially, or completely of an inert substance, or substances, which do not destroy, repel, or mitigate insects, and does not have the names and percentage amounts of each and every one of such inert ingredients plainly and correctly stated on the label: *Provided, however,* that in lieu of naming and stating the percentage amounts of each and every inert ingredient the producer may at his discretion plainly state upon the label the correct names and percentage amounts of each and every ingredient of the insecticide having insecticidal properties and make no mention of the inert ingredients, except in so far as to state the total percentage of inert ingredients present.

Sec. 9. That no dealer shall be prosecuted under the provisions of this Act when he can establish a guaranty signed by the wholesaler, jobber, manufacturer, or other party residing in the United States, from whom he purchases such articles, to the effect that the same is not adulterated or misbranded within the meaning of this Act, designating it. Said guaranty, to afford protection, shall contain the name and address of the party or parties making the sale of such articles to such dealer, and in such case said party or parties shall be amenable to the prosecutions, fines, and other penalties which would attach, in due course, to the dealer under the provisions of this Act.

Sec. 10. That any Paris green, lead arsenate or other insecticide that is adulterated or misbranded within the meaning of this Act, and is being transported from one State, Territory, District, or insular possession to another for sale, or, having been transported, remains unloaded, or in original unbroken packages, or if it be sold or offered for sale in the District of Columbia or the Territories, or insular possessions of the United States, or if it be imported from a foreign country for sale, or if it is intended for export to a foreign country, shall be liable to be proceeded against in any district court of the United States within the district where the same is found, and seized for confiscation by a process of libel for condemnation. And if such article is condemned as being adulterated or misbranded within the meaning of this Act, the same shall be disposed of by destruction or sale, as the said court may direct, and the proceeds thereof, if sold, less the legal costs and charges, shall be paid into the Treasury of the United States, but such goods shall not be sold in any jurisdiction contrary to the provisions of this Act or the laws of that jurisdiction: *Provided, however,* That upon the payment of the costs of such libel proceedings and the execution and delivery of a good and sufficient bond to the effect that such articles shall not be sold or otherwise disposed of contrary to the provisions of this Act, or the laws of any State, Territory, District, or insular possession, the court may by order direct that such articles be delivered to the owner thereof. The proceedings of such libel cases shall conform, as near as may be, to the proceedings in admiralty, except that either party may demand trial by jury of any issue of fact joined in any such case, and all such proceedings shall be at the suit of and in the name of the United States.

Sec. 11. The Secretary of the Treasury shall deliver to the Secretary of Agriculture upon his request from time to time samples of Paris green, lead arsenates and other insecticides which are being imported into the United States or offered for import, giving notice thereof to the owner or consignee, who may appear before the Secretary of Agriculture, and have the right to introduce testimony, and if it appear from the examination of such samples that any Paris green or lead arsenate or other insecticide offered to be imported into the United States is adulterated or misbranded within the meaning of this Act, or is otherwise dangerous to the health of the people of the United States, or is of a kind forbidden entry into, or forbidden to be sold or restricted in sale in the country in which it is made or from which it is exported, or is otherwise falsely labeled in any respect, the said article shall be refused admission, and the Secretary of the Treasury shall refuse delivery to the consignee and shall cause the destruction of any goods refused delivery which shall not be exported by the consignee within three months from the date of notice of such refusal under such regulations as the Secretary of the Treasury may prescribe: *Provided*, That the Secretary of the Treasury may deliver to the consignee such goods pending examination and decision in the matter on execution of a penal bond for the amount of the full invoice value of such goods, together with the duty thereon, and on refusal to return such goods for any cause to the custody of the Secretary of the Treasury, when demanded, for the purpose of excluding them from the country, or for any other purpose, said consignee shall forfeit the full amount of the bond: *And provided further*, That all charges for storage, cartage, and labor on goods which are refused admission or delivery shall be paid by the owner or consignee, and in default of such payment shall constitute a lien against any future importation made by such owner or consignee.

Sec. 12. That the term "Territory" as used in this Act shall include the insular possessions of the United States. The word "person" as used in this Act shall be construed to import both the plural and the singular, as the case demands, and shall include corporations, companies, societies, and associations. When construing and enforcing the provisions of this Act, the act, commission, or failure of any officer, agent, or other person acting for or employed by any corporation, company, society, or association, within the scope of his employment or office, shall in every case be also deemed to be the act, omission, or failure of such corporation, company, society, or association as well as that of the person.

Sec. 13. That this Act shall be in force and effect from and after the ——— day of ———, nineteen hundred and ———.

FIRST DAY.

MONDAY—MORNING SESSION.

The thirty-fourth annual convention of the Association of Official Agricultural Chemists was called to order by the President, J. K. Haywood of Washington, D. C., on the morning of November 19, 1917 at 10.15 at the New Willard, Washington, D. C.

No referee on the subject of foods and feeding stuffs was appointed and no report on this subject was presented.

No report on sugar in foods and feeding stuffs was made by the associate referee.

REPORT ON CRUDE FIBER.

By C. K. **Francis**[1] (Agricultural Experiment Station, Stillwater, Okla.), *Associate Referee.*

A sample of wheat shorts and one of cottonseed meal, together with a piece of muslin for use in filtering, were sent to each collaborator. Determinations of crude fiber were made by the one filtration method and by the official method. The results are shown in the following table.

Examination of the table shows that the results obtained by the one filtration method were considerably higher than those obtained by the official method. Moreover, results by the one filtration method are not so concordant as those by the official method. With the one filtration method it was particularly difficult for the collaborators to check each other.

CONCLUSIONS.

In the opinion of the collaborators, no saving in time is effected by using the one filtration method, and the failure of the results to check with those obtained by the official method raises a doubt as to whether the proposed method can be used with satisfaction.

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[1] Present address, Cosden & Company, Tulsa, Okla.

Determination of crude fiber.

ANALYST	WHEAT SHORTS		COTTONSEED MEAL	
	Official method	Proposed method	Official method	Proposed method
	per cent	*per cent*	*per cent*	*per cent*
Percy O'Meara, Agricultural Experiment Station, E. Lansing, Mich.	5.42	6.02	11.97	12.37
	5.22	5.62	12.28	12.31
	....	5.53	....	12.59
Average	5.32	5.72	12.12	12.42
T. E. Friedemann, Agricultural Experiment Station, E. Lansing, Mich.	5.18	6.17	12.50	13.97
	5.35	5.85	12.30	14.12
	5.00	6.15	....	12.60
	....	5.95	....	....
Average	5.17	6.03	12.40	13.56
Wm. Weber, State Department of Agriculture, Harrisburg, Pa.	5.24	7.61	11.92	13.73
	5.09	6.32	11.76	13.29
Average	5.16	6.96	11.84	13.51
E. R. Harrouff, Agricultural Experiment Station, College Station, Texas.	5.30	6.30	12.62	12.00
	5.22	6.70	12.65	12.95
	5.90	6.46	....	13.82
	5.60	6.25	....	14.60
	....	....	....	15.70
Average	5.50	6.42	12.63	13.81
W. L. Dubois, Hershey Chocolate Co., Hershey, Pa.	5.49	7.12	11.92	14.23
	6.01	6.07	13.97	14.73
Average	5.75	6.59	12.94	14.48
W. D. Richardson, Swift & Company, Chicago, Ill.	6.03	6.43	12.58	13.02
	5.95	6.35	12.85	13.34
	6.25	6.31	12.52	13.12
	6.20	6.30	12.51	13.36
	6.11	6.31	.....	.....
Average	6.11	6.34	12.61	13.21
E. H. Berry, U. S. Food and Drug Inspection Station, Transportation Building, Chicago, Ill.	5.15	6.40	12.45	13.58
	5.05	6.43	12.65	14.00
Average	5.10	6.41	12.55	13.79
Cornelia Kennedy, University of Minnesota, University Farm, St. Paul, Minn.	5.80	6.14	12.68	14.61
	5.84	5.75	13.21	14.17
Average	5.82	5.94	12.94	14.39

Determination of crude fiber.—Concluded.

ANALYST	WHEAT SHORTS		COTTONSEED MEAL	
	Official method	Proposed method	Official method	Proposed method
	per cent	*per cent*	*per cent*	*per cent*
R. A. Thuma, University of Minnesota, University Farm, St. Paul, Minn.	6.18 6.15	6.67 6.63	13.46 13.67	13.74 14.17
Average	6.16	6.65	13.56	13.95
L. D. Elliott, U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, San Francisco, Calif.	5.69 5.50	6.32 6.27	12.20 12.05	15.75 15.51
Average	5.60	6.30	12.13	15.63
J. H. Roop, Purdue Agricultural Experiment Station, La Fayette, Ind.	5.50 5.48 5.50	5.87 5.57 6.00	11.70 11.62 12.45	12.47 13.67 13.30
Average	5.49	5.81	11.92	13.14

REPORT ON STOCK FEED ADULTERATION.

By B. H. Silberberg (Bureau of Chemistry, Washington, D. C.), *Associate Referee.*

The work during the past year on feed adulteration consisted of two parts: the first part on scratch feed based somewhat upon the recommendations in the 1916 report on feed adulteration: the second dealing with a method for the determination of hulls in cottonseed meal.

PART I.

The work on scratch feeds will be considered first. Two samples were prepared in the following manner and sent to each collaborator:

The weed seeds and chaff were carefully cleaned out of some good quality commercial scratch feed which contained no grit. Each jar of both samples was then prepared separately. Three per cent of commercial grit and 2 per cent of a weed seed mixture were added to each jar of Sample 1. Six per cent of commercial grit and 8 per cent of a weed seed mixture were added to each jar of Sample 2. The weed seed mixture contained the following seeds, which represent large, medium-sized and small seeds:

Agrostemma githago (corn cockle);
Vaccaria vaccaria;
Polygonum convolvulus (wild buckwheat);
Chaetochloa glauca (pigeon grass);
Chenopodium album (lamb's quarters).

The collaborators were asked to make quantitative determinations of both grit and weed seeds, making two analyses if possible, using for one about 10 grams, and for the other about 20 grams. Later, through the work of A. J. Patten, one of the collaborators, and the results of experimentation, the fact was developed that the method used for mixing and drawing out the sample was of considerable importance. The samples were so clean and the ingredients for the most part of such size that the personal factor in making the separations was practically eliminated. The factors which would tend to cause a variation in results were thus narrowed down to two—the method of mixing and sampling, and the size of sample used—principally the former. The results obtained on this year's work would hardly justify drawing definite conclusions in regard to either of these points. However, for the time being, it would appear advisable to follow the recommendations of the former referee that, with a coarse scratch feed, no less than 20 grams be used for a sample, although if a thoroughly satisfactory method of sampling were devised, it might be possible to obtain accurate results on a smaller sample.

As to the method of mixing and sampling, the results indicate that some mechanical method of mixing and sampling, in which the personal factor is reduced to a minimum, is practically a necessity for concordant results. Table 1 shows that in one instance an analyst obtained very good results by mixing on sampling cloth, reducing by quartering, etc., on a sample weighing only 9.8 grams. But by the same method of mixing and on a sample weighing about twice as much, the same analyst obtained results 25 per cent off on grit and 40.5 per cent off on weed seeds. In another case an analyst obtained correct results by this method of mixing on a sample weighing 37.0 grams, but the same analyst, on a sample weighing 19.8 grams, was 6.6 per cent off on grit and 15 per cent off on weed seeds, while on a sample weighing 10.38 grams, about the same amount as that on which the other analyst mentioned obtained very good results, his are 40.3 per cent off on grit and only 1 per cent off on weed seeds. One analyst who used the sampling cloth for mixing and sampling was 74.0 per cent off on weed seeds and grit combined on a sample weighing 15.26 grams, and another analyst using this method of mixing and a sample weighing slightly more (16.63 grams) was 91.2 per cent off. While these figures are taken from results on Sample 1, those on Sample 2 would lead to the same conclusion, that mixing and quartering on a sampling cloth is not a reliable method of drawing out a sample.

Another method proposed is to separate the sample into three portions by passing it through 10 and 20 mesh sieves. This makes it easier to mix each portion more uniformly. Then, after mixing each of the three portions, weigh and take one-tenth of each for a sample. While this method has not been given so thorough a test as it would seem to merit, the results obtained so far indicate that, while it gives very satisfactory results in some instances, uniformly reliable results are not obtained.

TABLE 1.

Collaborative results on Sample 1, scratch feed, containing 3 per cent of grit and 2 per cent of weed seeds.

METHOD OF SAMPLING AND ANALYST	WEIGHT OF SAMPLE	REPORTED	
		Grit	Weed seeds
Mixed on sampling cloth, reduced by quartering until of desired size:	*grams*	*per cent*	*per cent*
A. J. Patten, Agricultural Experiment Station, E. Lansing, Mich.	9.787	3.00	1.79
R. B. Deemer, State Chemist, Department of Agriculture, La Fayette, Ind.	10.38	4.3	2.2
	10.76	4.6	3.2
Swift & Company, Chicago, Ill.			
Anaylyst "A"	15.26	8.70*	....
Analyst "B"	16.63	9.56*	....
A. J. Patten	19.60	2.25	1.19
R. B. Deemer	19.8	2.8	2.3
	20.98	2.2	2.0
	37.0	3.0	2.0
Sample separated into three portions with 10 and 20 mesh sieves, each portion weighed, mixed, and one-tenth of each taken for examination:			
A. J. Patten	14.764	2.98	2.02
B. H. Silberberg	14.77	2.0	2.4
Kny-Scheerer mixer and duster used:			
H. E. Gensler, State Department of Agriculture, Harrisburg, Pa.	7.302	2.12	2.18
	19.720	2.88	2.4
Entire sample mixed; reduced by passing through sampler, Eimer & Amend No. 6086:			
A. J. Patten	19.61	2.41	2.22
	23.907	2.86	1.77
	37.2	3.23	2.09
Mixed and sampled in Boerner sampler:			
B. H. Silberberg	18.20	2.53	2.03
	37.19	3.23	2.21
	74.39	2.86	2.14

* Combined figure on grit and weed seeds.

There are insufficient data at hand on the results obtained with the use of mechanical mixers and samplers to permit drawing conclusions as to the reliability of any of them for products of this kind.

It is respectfully recommended that the work on scratch feeds be continued another year with the idea of securing a uniformly accurate and satisfactory method of mixing and sampling. In this connection some data as to the size of the sample to be used would also be necessary.

PART II.

The second part of the work on feed adulteration consisted of testing a method devised by the referee for the quantitative determination of cottonseed hulls in cottonseed meal. Several years ago two analysts in the Microchemical Laboratory of the Bureau of Chemistry ran a set of twenty-one samples, each making two determinations on each sample, one on coarsely ground meal and the other on the same meal finely ground. The analysts checked themselves and each other so well, and

TABLE 2.

Collaborative results on Sample 2, scratch feed, containing 6 per cent of grit and 8 per cent of weed seeds.

METHOD OF SAMPLING AND ANALYST	WEIGHT OF SAMPLE	REPORTED	
		Grit	Weed seeds
	grams	*per cent*	*per cent*
Mixed on sampling cloth; reduced by quartering until of desired size:			
A. J. Patten	8.043	4.32	6.32
Swift & Company:			
Analyst "A"	14.16	18.87*	....
Analyst "B"	19.74	21.41*	....
A. J. Patten	28.56	6.32	8.58
R. B. Deemer	30.7	4.7	7.0
Sample separated into three portions with 10 and 20 mesh sieves, each portion weighed, mixed, and one-tenth of each taken for examination:			
A. J. Patten	14.465	7.13	8.06
Kny-Scheerer mixer and duster used:			
H. E. Gensler	10.301	6.20	8.20
	19.223	6.29	9.19
Entire sample mixed; reduced by passing through sampler, Eimer & Amend, No. 6086:			
A. J. Patten	18.945	6.54	8.62
	49.16	6.37	8.28
Mixed and sampled in Boerner sampler:			
B. H. Silberberg	8.97	7.24	7.47
	37.42	6.15	8.18
	73.69	5.79	8.60

* Combined figure on grit and weed seeds.

the results agreed so closely with other known data, that the method, although not scientifically accurate, seems to have a decided practical value.

Since it is impossible to simulate under laboratory conditions a commercial cottonseed meal, ordinary commercial samples had to be used for collaborative work instead of meals with known hull content, which would have been preferable. Two samples were used, Sample 1 representing meal manufactured under present milling conditions, with almost no lint left on the seed, and Sample 2 representing meal manufactured under conditions prevailing several years ago, when the seed was not so thoroughly delinted. Unfortunately, Sample 2 being several years old was very rancid, which may have had something to do with its not lending itself readily to a separation of meal and hull by the method under investigation. Samples representing the same type of meal, which were run several years ago while comparatively fresh, did not present this difficulty. For this reason it does not appear justifiable to judge of the merits of the method by the results reported on Sample 2.

In both samples, "A" represents material which was ground until it all passed through a sieve with holes 1 mm. in diameter, and "B" represents the same material ground but not sieved, the major portion thus being coarser than "A".

The directions as sent to the collaborators are as follows:

METHOD FOR DETERMINATION OF HULLS IN COTTONSEED MEAL.

After mixing the sample weigh out 50 grams. Place in a mortar (one which has become smooth seems to be even more satisfactory than a rough one). Add sufficient water to make a thin paste. Let stand about 15 minutes, or more, when it may have thickened, making it necessary to add a little more water before grinding. Grind about 10 minutes. During grinding, as the meal breaks up, it is sometimes necessary to add a little more water. After grinding add sufficient water to carry off easily the finely divided part of the meal by decantation. Stir, and decant into a beaker the yellow mixture which does not settle readily. Continue washing and decanting until the fine portion is washed out. If in any decantation too much hull (brown material) is carried off, return it to the mortar. When the washing seems no longer effective in removing meal without too much hull, repeat the grinding and continue as before until the hulls are practically free from meal. Each analyst may develop slight variations in procedure which seem to him expedient. When this operation is complete and the water is poured off the hulls, spread them out, preferably on a glass plate, and allow them to dry in the air. (Where the atmospheric humidity is high, it may be found preferable to dry the hulls in a drying oven at low temperature. They should be stirred frequently, until they feel quite dry to the touch. They should then be allowed to stand a few minutes before weighing.) When they feel dry to the touch, weigh, and multiply the weight in grams by 2 for the percentage of hulls. This figure represents the minimum percentage of hulls present.

It was recommended that each analyst making analyses of these samples first run a few other samples in order to familiarize himself somewhat with the technique of the method. The referee requested a report as to the number of such preliminary analyses made by the analyst and in only three cases was such a report made. It is therefore assumed that in other cases the analyst was entirely unfamiliar with the method.

Several variations in procedure were introduced by different analysts. H. E. Gensler of the Pennsylvania State Department of Agriculture suggests the use of hot water to obtain greater differences in the specific gravity of hull and water. The Barrow-Agee Laboratories suggest that more nearly accurate results could be obtained, and more quickly, if moisture were determined on the meal, then the hulls made moisture-free before weighing, and the moisture calculated back into them on the basis of that originally present in the meal. R. B. Deemer of the Purdue Agricultural Experiment Station introduced the sample into a liter cylinder, stirred it with a mechanical stirrer and decanted at about 15 minute intervals, thus disposing of a large part of the meal, and then made the final separation in the mortar. None of these variations in procedure appears to have a very material effect upon the results. J. H. Roop, also of the Purdue Agricultural Experiment Station, offers the following method:

GRAVITY METHOD WITH CALCIUM CHLORID.

Use a calcium chlorid solution 40 grams to 100 cc. of water, sp. gr. 1.40, sp. gr. of the cottonseed meal 1.39. Sp. gr. of the cottonseed hulls approximately 1.54.

Grind the material in the mortar with a small amount of the calcium chlorid solution, pour the material into a large beaker and add about 200 cc. more of the calcium chlorid solution, stir and allow the hulls to settle, and decant off the calcium chlorid solution. The sample will then be almost entirely freed of cottonseed meal. Grind and treat as before about three times, and transfer the hulls to a linen cloth, washing free from calcium chlorid with hot water, dry at 65°C. and let stand for several hours in air, weigh and calculate the percentage of hulls.

It was suggested by the referee on foods and feeding stuffs that from a chemical standpoint it would be of interest and value to have crude fibers run on the samples. In accordance with this the collaborators were requested to make these analyses if time permitted.

Reference to the table will show that while the collaborators checked themselves and each other satisfactorily, their results were uniformly lower than those obtained by the referee and also lower than the hull content as indicated by the crude fiber figures. In the opinion of the referee this is largely due to the use of a rough mortar, and leads to the addition of the following notes to the directions:

Use a *large, smooth* mortar. The object is not to grind but to crush the meal. If the mortar is rough, it grinds the hulls too much. Fill the mortar with water before each

TABLE 3.

Hull determination in cottonseed meal.

ANALYST	1A		1B		2A		2B	
	Hulls	Crude fiber	Hulls	Crude fiber	Hulls	Crude fiber	Hulls	Crude fiber
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
H. E. Gensler	21.4*	....	24.6*	....	9.6*	....	10.6*	....
	22.0*	....	24.8*	....	9.8*	....	11.0*	....
	22.8*	....	....	....	....	....	....	....
Swift & Company:								
Analyst "A"	22.3	....	23.8	....	9.1	....	10.7	....
Analyst "B"	19.7	15.26	22.5	13.60	9.6	8.86	8.9	10.19
Analyst "C"	20.1	....	22.2	....	8.7	....	10.1	....
C. G. Remsburg, State College of Agriculture, College Park, Md.	22.0	....	22.0	....	10.0	....	8.5	....
	22.0	....	22.0	....	10.0	....	8.4	....
Barrow - Agee Laboratories†, Memphis, Tenn.	22.60	12.07	23.16	12.35	9.14	7.00	9.80	7.78
R. B. Deemer	23.5	13.11	21.3	13.12	....	....	....	....
	22.6‡	....	22.0‡	....	....	....	....	....
J. H. Roöp§, State Department of Agriculture, La Fayette, Ind.	20.5**	....	....	....	8.9	....	....	....
H. J. Nimitz, State Department of Agriculture, La Fayette, Ind.	....	....	....	....	....	....	9.4	....
E. F. Berger, Agricultural Experiment Station, E. Lansing, Mich.	23.60	13.46	23.66	13.39	9.40	7.60	9.54	7.70
	23.76	....	24.00	....	....	....	9.76	....
W. K. Makemson, Bureau of Chemistry, Washington, D. C.	23.5*	....	26.1*	....	....	....	....	....
	....	....	24.3*	....	....	....	....	....
G. P. Walton, Bureau of Chemistry, Washington, D. C.	....	14.9	....	14.7	....	8.7	....	8.6
B. H. Silberberg	26.34	....	27.8	....	11.3	....	15.06	....
	26.2*	....	26.8*	....	14.2	....	14.06	....
L. D. Elliott, U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, San Francisco, Calif.	17.00	....	25.00	....	....	....	....	....
	20.00	....	22.00	....	8.00	....	9.00	....
	....	....	23.00	....	8.00	....	10.00	....

* Hot water used.

† Hulls dried and moisture calculated back on basis of that in original meal.

‡ Stirred in a liter cylinder and decanted, then finished in a mortar.

§ Gravity method.

** Hull determination mixture of 1A and 1B.

decantation. Scrape the meal down from the sides of the mortar before each settling. The time necessary for settling will be shorter with each successive decantation. Even the first settling seldom requires more than a minute. Examine the decanted portion for shell by looking at the bottom of the beaker after it has settled. If it is practically free from hulls, it may be discarded. The grinding and decanting may have to be repeated four or five times. Toward the end of the process, when not much meal remains with the hulls, it seems expedient to decant immediately after adding the water to carry off all the remaining meal, even though considerable hull is also carried off. Then remove the hulls which are free from meal from the mortar to the glass plate for drying. After so doing, return the mixture of meal and hulls which was poured off and grind this portion until it is as free from meal as seems practicable, and add these hulls to those previously removed. It must be remembered that the hulls cannot be made absolutely free from meal, but since some hulls are sure to be lost in the decantations, and the hulls are heavier than the meal, those which are thrown away more than compensate for the meal retained.

It is recommended that the work of developing a method for the quantitative determination of cottonseed hulls in cottonseed meal be continued another year.

One of the recommendations carried over from 1914 is that methods for the detection of peat dried at high temperatures in feed stuffs be investigated. It might possibly dispose of this recommendation to say that the referee has never found any difficulty in identifying peat in feeds with the use of the compound microscope, as it retains the sphagnum structure which is very characteristic. It is therefore recommended that this question be dropped.

As to the recommendation made in 1916 that a key or outline for the qualitative detection of adulterants in feeding stuffs be prepared and submitted at the next meeting, your referee reports that such a key has been prepared. Since, however, such enormous changes are taking place in the types of products used for feeds, due to the unsettled world conditions, it was thought better to present this later, when possibly the outline could be made more complete and up to date.

There was no associate referee on organic and inorganic phosphorus in foods and feeding stuffs and no report on this subject was presented.

REPORT ON WATER IN FOODS AND FEEDING STUFFS.

By J. O. CLARKE[1] (State Department of Agriculture, Atlanta, Ga.), *Associate Referee.*

The work on the determination of water in foods and feeding stuffs consisted in a study of the vacuum desiccator method, using sulphuric acid, lime, and calcium carbide as desiccating reagents in comparison with various heating methods.

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[1] Present address, U. S. Food and Drug Inspection Station, U. S. Custom House, Savannah, Ga.

The following samples were sent to collaborators:

INSTRUCTIONS TO COLLABORATORS.

No. 1, cottonseed meal; No. 2, wheat bran; No. 3, corn meal; No. 4, air-dried silage; No. 5, dried apples.

Samples 1, 2, 3, and 4 are prepared and ready for weighing. Sample 5 must be chopped fine with a sharp knife and weighed at once.

Determine the water in Samples 1, 2, 3, and 4 by Methods A, B, B1, D, E, and F, and in Sample 5 by Methods A, D, E, F, G, and H.

Method A.—Employ the method in use in the collaborator's laboratory for this class of substances. Report the details of the method employed.

Method B.—Dry in a water oven at the temperature of boiling water under atmospheric pressure for 5 hours.

Method B1.—Dry, as in *B*, to constant weight.

Method C.—Employ the official method[1] for direct drying. State whether vacuum or dry hydrogen was used in this method.

Method D.—Dry in vacuum over sulphuric acid without heat[1].

Method E.—Weigh 2 grams of the material into a suitable dish or crucible with a tightly fitting cover. Place in a vacuum desiccator containing about 400 grams of freshly ignited powdered lime, and exhaust with a vacuum pump. After 24 hours, open the desiccator, admitting the incoming air through concentrated sulphuric acid, and make the first weighing. After weighing, replace the dish in the desiccator and repeat the process until constant weight is obtained. The lime should be changed on the third or fourth day and, with very wet substances, again near the end of the process.

Method F.—Same method as *D* except that calcium carbide is used as the desiccating reagent. This material retains its strength until it becomes a powder. The powder should be sifted out every 3 or 4 days, returning the lumps to the desiccator.

Method G.—Dry about 5 grams of the finely chopped material for exactly 4 hours at the temperature of boiling water under atmospheric pressure.

Method H.—Dry about 5 grams of the finely chopped material to constant weight in a vacuum oven at 65°C.

The sulphuric acid should be boiled in a large Kjeldahl flask for several hours before using. If the acid from previous determinations is used, a drop of mercury or a small amount of copper sulphate boiled with it will destroy any organic matter it may contain. Avoid the use of discolored acid, as it frequently gives off some fumes of sulphur dioxid.

The lime should be powdered and ignited in a muffle for several hours, and transferred while hot to an air-tight can with friction cover.

REPORTS OF COLLABORATORS.

Three complete reports and one partial report were received. The determinations requested from the collaborators were also made by the associate referee.

DISCUSSION.

The sample of dried apples is disregarded in the following discussion, and will be taken up in a later paragraph.

Heating methods.—The different heating methods will give somewhat

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 79.

different results on the same sample. Heating in vacuum at the temperature of boiling water gives higher results than any other heating method in general use. Table 6 shows samples of wheat bran and corn meal in which the water was determined by several heating methods.

The temperature in the electric oven was accurately maintained at 98.5°C., the same temperature being held in the chamber of a well insulated water-jacketed oven. The different results observed by these two methods were probably due to the difference in ventilation of the two ovens, the water oven being completely closed, and the electric oven having four small air vents in the sides, besides being much larger.

TABLE 1.

Water in foods and feeding stuffs.

(Collaborator, G. L. Bidwell, Bureau of Chemistry, Washington, D. C.; analyst, G. P. Walton, Bureau of Chemistry, Washington, D. C.)

METHOD OF DRYING	COTTONSEED MEAL		WHEAT BRAN		CORN MEAL		AIR-DRIED SILAGE		DRIED APPLES	
	Water	Time	Water	Time	Water	Time	Water	Time	Water	Time
	per cent	*days*	*per cent*	*days*	*per cent*	*days*	*per cent*	*days*	*per cent*	*days*
(*A*) Constant weight at temperature of boiling water, 75 mm. pressure	8.59	9½*	10.55	9½*	13.53	9½*	8.06	9½*	27.41†	88½*
(*B*)	7.96	..	10.09	..	12.88	..	7.62	..	....	..
(*B1*)	8.09	24*	10.21	24*	12.97	24*	7.83	24*	....	..
(*C*)	8.73	22*	10.75	22*	13.67	22*	8.31	22*	....	..
(*D*)	7.81	5	10.20	6	13.09	6	7.50	4	15.73	6
(*E*) Initial pressure 3 mm. at time of opening desiccator, average 50 mm., last time 6 mm.	7.60	8	9.92	8	12.92	8	7.43	8	15.8	8
(*F*) Initial pressure 3 mm. at time of opening desiccator, average 52 mm., last time 10 mm.‡	7.37	8	9.72	8	12.78	8	7.32	8	15.56	8
(*G*)	....	..	....	..	....	..	....	..	20.08	..
(*H*)	....	..	....	..	....	..	....	..	19.49	49*

* Hours.

† The method used is not adapted to substances similar to Sample 5. Such substances are seldom analyzed in this laboratory. For the purposes of the experiment, the sample was dried at the temperature of boiling water in the vacuum oven under an average pressure of 88 mm. for 15 daily periods totaling 88 hours. At the end of this time the sample was considerably caramelized.

‡ Except after the first day's drying when, on opening the desiccator, the pressure was nearly atmospheric.

TABLE 2.

Water in foods and feeding stuffs.

(Collaborator, W. D. Richardson, Swift & Company, Chicago, Ill.)

METHOD OF DRYING	COTTONSEED MEAL		WHEAT BRAN		CORN MEAL		AIR-DRIED SILAGE		DRIED APPLES	
	Water	Time	Water	Time	Water	Time	Water	Time	Water	Time
	per cent	*days*	*per cent*	*days*	*per cent*	*days*	*per cent*	*days*	*per cent*	*days*
(*A*) 5 hrs. at 102–103°C. in air oven	7.58	..	9.57	..	11.63	..	7.61	..	22.25	..
(*B*)	7.50	..	9.34	..	11.94	..	7.80	..	....	..
(*B1*)	7.13 7.55	4* 6*	9.08 9.45	4* 6*	11.85 12.04	4* 6*	7.28 7.78	4* 6*	....	.. ..
(*C*)	6.72	..	8.61	..	10.76	..	7.43	..	....	..
(*D*) Average vacuum 20 inches.	6.39 7.53	4 13	8.61 8.92	4 13	10.40 11.93	4 9½	7.51 7.68	4 10	20.88	5 ..
(*E*) Average vacuum 20 inches.	7.15	11½ ..	8.92	12½ ..	11.82	13½ ..	6.99 7.66	14 13½	19.80	15½ ..
(*F*) Average vacuum 20 inches.	7.27	11¼	9.21	10½	11.98	10½	7.62	11½	20.01	11½
(*G*)	....	..	....	..	....	..	....	..	21.56	..
(*H*)	....	..	....	..	....	..	....	..	21.40	12*

* Hours.

TABLE 3.

Water in foods and feeding stuffs.

(Collaborator, H. R. Clarke, State Department of Agriculture, Atlanta, Ga.)

METHOD OF DRYING	COTTONSEED MEAL		WHEAT BRAN		CORN MEAL		AIR-DRIED SILAGE		DRIED APPLES	
	Water	Time	Water	Time	Water	Time	Water	Time	Water	Time
	per cent	*days*	*per cent*	*days*	*per cent*	*days*	*per cent*	*days*	*per cent*	*days*
(*B*) 5 hrs. at temperature of boiling water, atmospheric pressure.	7.30	..	8.79	..	10.54	..	6.56	..	....	..
(*B1*)	7.14	..	8.95	7*	10.92	7*	6.49	7*	....	..
(*C*)	8.43	..	10.15	..	12.97	..	7.78	..	....	..
(*D*)	8.48	4	10.51	4	13.47	4	7.36	4	18.48	4
(*E*)	8.57	7	10.31	7	13.35	7	7.63	7	18.58	7
(*F*)	8.06	6	10.34	6	12.95	6	7.70	6	18.37	6
(*G*)	....	..	....	..	....	..	....	..	21.45	..
(*H*)	....	..	....	..	....	..	....	..	20.90	..

* Hours.

Drying at 65°C. and 70 millimeter pressure seems to give about the same result as at 98.5°C. and atmospheric pressure. An examination of Tables 1 to 5, inclusive, will show a similar discrepancy in the different heating methods as reported by the several collaborators.

Sulphuric acid-vacuum method.—Comparison of this method with the

TABLE 4.

Water in foods and feeding stuffs.

(Collaborator, R. F. Monsalvatge, Southern Cotton Oil Co., New Orleans, La.; analyst, Andrew Schwartz.)

METHOD OF DRYING	COTTONSEED MEAL		WHEAT BRAN		CORN MEAL		AIR-DRIED SILAGE		DRIED APPLES	
	Water	Time	Water	Time	Water	Time	Water	Time	Water	Time
	per cent	*days*	*per cent*	*days*	*per cent*	*days*	*per cent*	*days*	*per cent*	*days*
(*A*) 5 hrs. at 105°C. in electric oven, atmospheric pressure.......	7.77	..	9.42	..	12.42	..	7.19	..	21.05	..
(*E*) Average vacuum 21 inches...............	7.45	8	9.94	8	12.68	8	6.86	8	19.50	8

TABLE 5.

Water in foods and feeding stuffs.

(Collaborator, J. O. Clarke, State Department of Agriculture, Atlanta, Ga.)

METHOD OF DRYING	COTTONSEED MEAL		WHEAT BRAN		CORN MEAL		AIR-DRIED SILAGE		DRIED APPLES	
	Water	Time	Water	Time	Water	Time	Water	Time	Water	Time
	per cent	*days*	*per cent*	*days*	*per cent*	*days*	*per cent*	*days*	*per cent*	*days*
(*B*).................	7.23	..	9.19	..	11.74	..	7.13	..	....	..
(*B1*).................	7.20	4*	9.16	4*	11.95	4*	7.30	4*	....	..
(*C*).................	8.45	4*	10.36	4*	13.50	4*	8.06	5*	....	..
(*D*) Pressure less than 1 mm.	8.32	4	10.37	3	13.39	3	8.03	5	18.63	12
	....	..	10.48	4	13.56	5	7.63	1	....	..
(*E*) Pressure less than 1 mm.	8.20	8	10.26	4	13.36	5	7.71	4	18.75	11
	8.27	11	10.36	6	13.44	6	7.80	5	....	..
	....	..	10.45	11	13.52	8	....	..	....	..
	....	..	....	..	13.62	11	....	..	....	..
(*F*) Pressure less than 1 mm.	8.04	6	10.26	3	13.36	5	7.72	4	18.80	11
	8.15	8	10.36	6	13.43	6	....	..	....	..
	....	..	....	..	13.52	11	....	..	....	..
(*G*).................	....	..	....	..	....	..	....	..	21.54	..
(*H*).................	....	..	....	..	....	..	7.40	..	21.37	..

* Hours.

TABLE 6.

Water determined by different heating methods.

METHOD	WATER IN WHEAT BRAN	WATER IN CORN MEAL
	per cent	*per cent*
Dried to constant weight at the temperature of boiling water (98.5°C.) and 70 mm. pressure	10.44	13.60
Dried to constant weight in water-jacketed oven at the temperature of boiling water (about 98.5°C., atmospheric pressure)	9.16	11.95
Dried to constant weight in electric oven at 98.5°C., atmospheric pressure	9.88	12.82
Dried to constant weight at 65°C., pressure 70 mm	9.75	12.78

official method, heating in vacuum at the boiling point of water, shows a very good agreement, considering the nature of these determinations.

The agreement among the different collaborators is as close as obtained by the official heating method at the temperature of boiling water and 70 to 75 millimeter pressure, and much closer than obtained by other heating methods.

In order to obtain the best results with this method, as complete a vacuum as possible is desirable. A comparison of Richardson's results obtained at 20 inches vacuum with those of the other collaborators obtained at less than 3 millimeters is striking. In the former case 7½ to 13 days' drying was necessary to obtain constant weight; while in the latter, constant weight was reached in 3 to 6 days. It will also be noted that Richardson's results are lower than the others, due doubtless to the same cause. On a sample of cottonseed meal containing 7.81 per cent of water, by the vacuum method at the temperature of boiling water, constant weight after loss of 7.59 per cent of water was obtained by the sulphuric acid-vacuum method in 7 days with 26 inches vacuum, while, with a pressure of less than 1 millimeter, constant weight with a loss of 7.86 per cent of water was obtained in only 5 days. A high vacuum pump is not necessary with this method, but as complete a vacuum as needed can be obtained by the use of a good aspirator pump and a tight desiccator with a little ether. For the best results a pressure of less than 2 millimeters should be maintained, the completeness of drying depending largely on the last few millimeters exhausted.

Lime-vacuum method.—This method gives slightly lower results than the sulphuric acid-vacuum method, and requires a somewhat longer time. An examination of Tables 1 to 5, inclusive, shows that the several collaborators obtained results by this method which compare favorably with those obtained by the same collaborator when the sulphuric acid-vacuum method was used, thus accounting to a certain extent for the difference in pressure. As with the sulphuric acid-vacuum method, as complete a vacuum as possible is desirable, but can only be obtained by means of a high vacuum pump. Sulphuric acid is slightly volatile in a high vacuum, while lime is not. The preparation of lime presents some difficulty, unless a high temperature muffle is employed, for it is essential that this reagent be carefully prepared by heating for several hours at a high temperature.

Carbide-vacuum method.—This method compares favorably with the sulphuric acid-vacuum method, and with the lime-vacuum method. The drying efficiency of carbide is somewhat less than that of sulphuric acid and about equal to that of lime. Carbide is easily obtainable, very cheap, and requires no further preparation for use. When its usefulness is at an end, it breaks into a fine powder, which can be shaken to the bottom of the desiccator, leaving the clean lumps exposed. The only possible objection to carbide is the acetylene gas which is set free, but apparently this does not act on any of the substances which would ordinarily be dried by this method. Carbide is used to some extent by the writer as a general desiccating reagent; practically the only substances ordinarily encountered in a food laboratory which can not be placed in the carbide desiccator are perforated porcelain crucibles containing cuprous oxid.

Dried apples.—Products of this character can not be dried at a temperature above 70°C. without decomposition. It appears that none of the vacuum desiccator methods removes all of the water, as can be seen by comparing these results with those obtained at 65°C. and 70 millimeter pressure. As determined at 65°C. in partial vacuum, the greatest difference among different collaborators in this product was 1.91 per cent, while slightly better results were obtained by the empirical method of drying for 4 hours at the temperature of boiling water. The greatest difference in this case was 1.46 per cent. The vacuum desiccator methods gave results much lower than either of the heating methods.

It was thought that the method of drying the sample might have some effect on the results of subsequent ether extractions. The writer determined the ether extract on the samples of cottonseed meal, wheat bran, corn meal, and air-dried silage as dried by all of the above methods, and no difference in this result was observed as between samples dried either by heating or desiccator methods. Although these samples did not show a difference in the ether extract with different methods of moisture determinations, some substances, which dry by heating methods to a horn-like residue, give much more satisfactory ether extraction when dried by the desiccator method. This is especially true with meat products and similar substances.

RECOMMENDATIONS.

It is recommended—

(1) That the method for the determination of water in foods and feeding stuffs by drying in vacuum over sulphuric acid[1] be adopted as official.

(2) That Method E, page 49, for the determination of water by drying over lime in vacuum be adopted as a tentative method, and receive further study in the ensuing year.

(3) That Method F, page 49, for the determination of water by drying over calcium carbide in vacuum be adopted as a tentative method, and receive further study in the ensuing year.

A NEW METHOD FOR MOISTURE DETERMINATION[2].

By G. F. Lipscomb and W. D. Hutchins (Clemson Agricultural College, Clemson College, S. C.).

The official method for the determination of moisture in fertilizers is known to be inaccurate generally. For this reason it seemed advisable to develop a method capable of giving more accurate results and requiring a shorter time for each determination. The method worked out in this laboratory is based on combined high and low temperature and vacuum. The sample is heated by means of live steam, and the moisture driven off into a vacuum cooled to $-100°C.$ by a mixture of solid carbon dioxid and ether. The apparatus employed is shown in Fig. 1.

METHOD.

Weigh 1 gram of the material into the bucket, "B". Pass steam from a suitable generator through the jacket, "J", for several minutes, and lower the bucket into place. Adjust the receptacle, "D", containing the freezing mixture, making the joint tight with a little vaseline. After a vacuum has been created, allow the sample to remain in the apparatus 5 minutes; then remove it, cool in a desiccator and weigh. Repeat the process until constant weight is obtained.

EXPERIMENTAL.

Moisture determinations were made according to this method on a number of fertilizer materials, and the results compared with those obtained by the official method, as shown in Table 1.

DISCUSSION.

When cottonseed meal is heated according to the official method it loses its bright yellow color, indicating that some decomposition takes

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 79.

[2] Presented by R. N. Brackett.

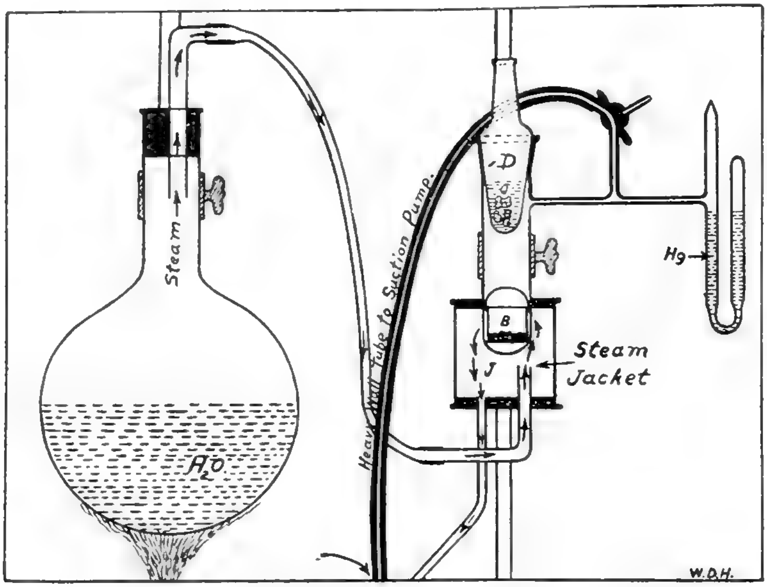

FIG. 1. APPARATUS FOR THE DETERMINATION OF MOISTURE.

place. This does not occur with samples of cottonseed meal treated according to the proposed method. The results obtained by the new method are slightly higher. The average time required for a complete determination is only 25 minutes. In estimations made by both methods the vapor from sodium nitrate gave an acid reaction; that from fish scrap was acid in the official method and neutral in the new method.

CONCLUSION.

In general, the results obtained by the new method are comparable with those of the official method. The new method has the distinct advantage of being extremely rapid and of producing very little decomposition of organic material.

TABLE 1.

Moisture in fertilizer materials, as determined by the new and the official methods.

SAMPLE	NUMBER OF SAMPLE	OFFICIAL METHOD	PER CENT MOISTURE FOUND BY NEW METHOD						
			Time in Minutes						
			5	10	15	20	25	30	35
Cottonseed meal...	1	7.35	6.06	8.24	8.36	....	....	....	....
	2	8.12	5.92	7.40	8.76	8.96	9.06	9.06	....
	3	7.44	5.98	6.76	6.83	7.49	7.50	....	....
	4	7.53	5.74	6.35	7.75	7.81	8.18	8.41	8.43
	5	7.77	5.68	6.34	7.83	7.83	....	....	....
	6	7.81	6.41	6.80	7.99	7.99	....	....	....
Sodium nitrate.....	1	1.21	1.07	1.08	1.08	....	....	....	....
	2	0.93	0.67	0.675	....	....	....	....	....
	3	0.605	0.41	0.42	....	....	....	....	....
	4	0.995	0.71	0.72	....	....	....	....	....
Acid phosphate....	1	9.64	7.87	8.02	8.34	8.34	....	....	....
	2	11.45	9.87	11.06	11.21	11.21	....	....	....
	3	12.11	10.08	11.54	11.56	....	....	....	....
	4	3.01	1.52	2.41	2.82	2.82	....	....	....
Fish blubber.......	1	13.22	11.95	12.77	13.06	13.08	....	....	....
Fish scrap.........	2	8.75	6.01	7.30	8.30	8.36	....	....	....
Starch............	1	9.59	7.98	10.02	11.07	11.10	....	....	....
	2	9.63	10.56	11.86	12.30	12.31	....	....	....
	3	8.95	8.32	9.81	10.62	10.62	....	....	....
Leather..........	1	7.30	7.12	7.33	7.39	7.39	....	....	....
	2	11.29	10.83	11.99	12.01	....	....	....	....
	3	6.72	4.25	5.57	6.22	6.57	6.59	....	....
	4	11.46	6.74	9.15	10.73	11.35	11.36	....	....
	5	14.13	11.90	12.94	13.17	13.20	13.20	....	....
Soils.............	1	1.94	2.24	2.29	2.29	....	....	....	....
	2	1.72	1.89	1.97	1.99	....	....	....	....
	3	1.27	1.46	1.58	1.58	....	....	....	....

DOUBLE MOISTURE DETERMINATIONS IN FERTILIZER MATERIALS.

By J. O. CLARKE (U. S. Food and Drug Inspection Station, U. S. Custom House, Savannah, Ga.).

It is a well known fact that the moisture content of many fertilizer materials undergoes considerable change during the grinding and preparation of the sample. This is especially true in materials containing more than 10 per cent of moisture, and is the source of considerable error when the moisture is not determined both before and after preparing the sample for analysis. It has come to the writer's attention that neglect of this point sometimes causes considerable variation in results reported on the same sample by different laboratories. The apparent error, due to failure to calculate the results back to the original moisture basis, is larger the greater the percentage of the active constituent, and in cases where the active constituent is high, as in muriate of potash, pyrites or some phosphate materials, which contain 40 to 45 per cent of phosphoric acid, a relatively small loss or gain of moisture in the preparation of the sample would considerably affect the reported result.

The writer has secured from a number of fertilizer laboratories the results on several samples of fertilizer materials, such as were found in routine fertilizer analyses when double moisture determinations were made. In most cases results were taken from the records of the laboratory concerned, and illustrate very well the possible error that would have been introduced had double moisture determinations not been made.

Table 1 contains the analytical results on a number of materials on which double moisture determinations were made. In every case except one there was a loss of moisture in the preparation of the sample. One result, however, the last, shows a gain in moisture from 5.85 to 6.85

TABLE 1*.

Variation on active constituent shown by double moisture determinations.

MATERIAL	MOISTURE		ACTIVE CONSTITUENT BASIS OF ORIGINAL SAMPLE		ACTIVE CONSTITUENT BASIS OF PREPARED SAMPLE		DIFFERENCE	
	Original sample	Ground and prepared sample	Ammonia	Phosphoric acid	Ammonia	Phosphoric acid	Ammonia	Phosphoric acid
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
Tankage	18.85	12.25	6.16	10.18	6.66	11.01	0.50	0.83
Tankage	20.70	15.24	6.90	10.07	7.38	10.76	0.48	0.69
Tankage	16.60	11.81	10.87	....	11.50	....	0.63	....
Tankage	8.85	6.24	6.61	11.49	6.80	11.82	0.19	0.33
Dried blood	14.50	12.10	15.70	....	16.14	....	0.44	....
Dried blood	13.00	11.10	16.01	....	16.36	....	0.35	....
Dried blood	9.55	8.18	15.02	....	15.25	....	0.23	....
Bone	20.25	16.70	2.87	23.52	3.00	24.57	0.13	1.05
Bone	14.85	11.10	4.16	20.65	4.35	21.56	0.19	0.91
Bone	22.47	13.63	2.06	24.24	2.30	27.00	0.24	2.76
Nitrate of soda	3.10	1.74	18.24	....	18.50	....	0.26	....
Nitrate of soda	2.20	1.12	18.71	....	18.90	....	0.19	....
Phosphate rock	1.15	0.57	....	30.82	....	31.00	....	0.18
Dried blood	5.85	6.85	17.63	....	17.44	....	0.19	....

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* The analytical data from which these tables were calculated was furnished by J. E. Breckenridge, H. D. Haskins, E. W. Magruder, J. H. Parkin, and Paul Rudnick.

per cent. The last two columns show the difference between the percentage of the active constituent as obtained on the prepared sample, and that which is actually contained in the material from which the sample was taken. It can readily be seen that this difference is much greater than the allowable error in determinations of this nature.

Table 2 is similar to Table 1, except that the samples for which results are shown in Table 2 were partially dried before grinding and preparing.

TABLE 2*.

Variation on active constituent shown by double moisture determinations.

MATERIAL	MOISTURE		ACTIVE CONSTITUENT BASIS OF ORIGINAL SAMPLE		ACTIVE CONSTITUENT BASIS OF PREPARED SAMPLE		DIFFERENCE	
	Original sample	Ground and prepared sample	Ammonia	Potassium oxid	Ammonia	Potassium oxid	Ammonia	Potassium oxid
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
Blood..........	8.25	3.50	16.68	....	17.54	....	0.86	....
Potassium chlorid	4.91	3.01	....	51.40	....	52.42	....	1.02
Pyrites ore......	2.78	0.65	....	47.70	....	48.74	....	1.04
Pyrites ore......	33.08	1.22	....	4.09	....	6.04	....	1.95
Gelatin refuse...	59.41	4.10	3.56	....	8.41	....	4.85	....

* The analytical data from which these tables were calculated was furnished by J. E. Breckenridge, H. D. Haskins, E. W. Magruder, J. H. Parkin, and Paul Rudnick.

SUMMARY.

Double moisture determinations should be made on many fertilizer materials. This is especially true when the moisture is above 10 per cent, and the percentage of the active constituent is fairly large.

REPORT ON TESTING CHEMICAL REAGENTS[1].

By C. O. EWING[2] (Bureau of Chemistry, Washington, D. C.), *Referee.*

No collaborative work was done on this subject. The report discussed, briefly, analyzed chemicals, the testing of acetic anhydrid, and the determination of alcohol in pharmaceutical preparations. In the latter discussion, attention was called to a possible source of error in the U. S. Pharmacopœia method for alcohol. To overcome this, all samples for analysis should be measured at exactly 15.56°C. instead of at "any

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[1] Abstract.

[2] Present address, United Drug Company, Boston, Mass.

definite temperature between 15°C. and 30°C.", as directed by the Ninth Revision of the U. S. Pharmacopœia.

RECOMMENDATIONS.

It was recommended—

(1) That work on the determination of alcohol in pharmaceutical preparations be continued.

(2) That the study of methods for the determination of the strength of acetic anhydrid be continued.

(3) That work on the testing of purity of immiscible organic solvents be continued.

Edgar T. Wherry and Elias Yanovsky[1] (Bureau of Chemistry, Washington, D. C.) submitted a paper on "The Identification of the Cinchona Alkaloids by Optical Crystallographic Measurements"[2].

REPORT ON MICROANALYTICAL METHODS.

By B. J. Howard (Bureau of Chemistry, Washington, D. C.),
Associate Referee.

At the 1916 meeting a new subject, microanalytical methods, was added to the work of the association.

For the purpose of obtaining ideas upon lines of work which might profitably be undertaken, letters were sent to each of the nine persons who originally signified a willingness to collaborate. Only seven replies were received, no two of which seemed at all similar. Among the subjects that were suggested as suitable for study were:

(1) General methods and structural characteristics of the ordinary plant substances with which the food control chemist has to deal most frequently.

(2) Microchemical methods for detecting alkaloids and synthetics such as cocain, novocain, tropacocain, stovain, propasin, anaesthesin, alypin, beta eucain, holocain, etc.

(3) Mixtures of various starches, or cocoa shells.

In microscopical, as well as in chemical work, there are two kinds of analysis, namely, qualitative and quantitative. The one most commonly used by the ordinary analyst is the qualitative method. The results obtained by this method are largely dependent upon the ability of the analyst to recognize diagnostic characteristics. Most of the literature upon microscopical methods deals with qualitative determinations.

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[1] Present address, Norwalk Tire & Rubber Co., Norwalk, Conn.
[2] *J. Am. Chem. Soc.*, 1918, **40**: 1063.

Comparatively little has been published on quantitative methods. Certain factors involved in these methods do not seem to have been recognized, or at least have not been investigated. The methods used in quantitative work may be classified either as estimation or as counting methods. In the former, the unknown is compared with substances of known composition. In this case, examinations are most commonly conducted by mounting the known substances in various mixtures upon one or more slides and comparing them with the unknown sample, mounted in a similar manner. To facilitate the comparison, certain microscopical devices are used, as, for instance, the comparison eye piece in which portions from each of two fields are brought into juxtaposition. By means of another device, two microscopes are so adjusted as to bring a field from each simultaneously into view. Another method, which is at times applicable in purely microchemical work, consists of determining the amount of substance present by a series of dilutions. In this way, the limit of reaction may be obtained and the approximate amount calculated from that.

The second system is based upon counting certain characteristic elements in the product. This would seem to be one step in advance of the pure estimation method mentioned above. A set of known mixtures is investigated, the characteristic elements from the substances are counted and a proportion is obtained from the results. A modification of this method is based upon the employment of a counting cell of definite capacity, such as a blood counting cell.

Experience has emphasized the importance of certain factors surrounding the question of efficient microanalytical work. Possibly there are few lines of work in which the personal element enters more largely, and yet it is the one most often overlooked. Some persons well qualified in some botanical lines seem to be quite unfitted for this type of analytical work, while others who have had less botanical, histological or microscopical training are able, after a certain apprenticeship, to do quite satisfactory work.

Chief among the various factors which influence the accuracy of quantitative work are the personal equation and slide variation. The personal equation includes eyesight, technique, training, experience, patience, memory, alertness to see and identify substances, and general aptitude for the work. By slide variation is meant those variations which occur in preparing the mounts for examination. These may arise from unevenness of sample and, in some cases, from error in weighing. In the first case, the amount of material used is so small that a representative portion is difficult to obtain, and for this reason a number of slides is often required. The number of slides required for the examination will probably vary with the substance under consideration; hence there is evident need of the inquiry, "How many slides are necessary to reduce to a minimum the experimental error due to this cause?" In some methods the sample is taken by weighing out a few milligrams of the substance, thus introducing a factor of uncertainty based upon the inaccuracies attending the weighing of such small quantities. This may in some cases cause a substantial percentage of error and constitutes another influencing factor which must be considered.

To illustrate these points reference may be made to some work conducted in the Microchemical Laboratory of the Bureau of Chemistry, which, however, was not planned primarily as an investigation of this association. In this case, the work consisted of examining flour samples for the amount of offal particles in the product. The work done brought out quite distinctly that there is a variation, due to the method employed, and also that there is a personal equation element, which varies with the individual doing the work. A study was also made of the daily variations which might be due to lighting or to the physical condition of the individual.

After some consideration, it was decided that an investigation along some of these lines would be of value to the association, and hence a number of samples were made up of wheat and corn products in varying proportions. These were sent to six different laboratories with the request that they make a determination of the amounts of the ingredients present in the samples. The investigators were asked to give a detailed account of methods used, when submitting their report.

In the reports received there is considerable variation. In each case, however, an effort has been made to obtain a ratio between the number of corn starch grains and the number of wheat starch grains in samples of known percentages.

Distilled water, 50 per cent glycerol, olive oil and gum tragacanth in alcohol, recommended by Wallis[1], have been used as mounting agents.

Known weights of known samples were taken by one analyst, but this was found impracticable owing to the great number of starch grains

At least ten fields were examined by each investigator for each sample In some cases these ten fields may have all been obtained from the same mount. In other cases ten mounts were made on a Thoma-Zeiss blood counting cell and all of the larger grains on the ruled area were counted. The results so far show a variation of from 3 to 32 per cent for any one analyst. This indicates that the method must be further developed in order to reduce the error to a minimum.

Microscopical methods can not, except in a few cases, be expected to give as close results as are obtained by normal quantitative chemical

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[1] *Analyst*, 1916, **41**: 357.

methods, but it seems that at present the work on microanalytical methods might well be devoted to investigations which have for their purpose the determination of the nature and extent of the various factors entering into these methods.

It is therefore recommended that during the coming year the studies on quantitative microanalytical methods be continued.

The appointment of the following committees was announced by the president:

Committee on auditing: W. A. Withers of North Carolina; W. D. Lynch of Washington, D. C.; and B. H. Silberberg of Washington, D. C.

Committee on nominations: F. P. Veitch of Washington, D. C.; Julius Hortvet of Minnesota; and L. L. Van Slyke of New York.

Committee on resolutions: A. S. Mitchell of Washington, D. C.; C. H. Jones of Vermont; and W. D. Bigelow of Washington, D. C.

Committee to invite the Secretary of Agriculture to address the convention: William Frear of Pennsylvania; J. A. LeClerc of Washington, D. C.; and W. B. Ellett of Virginia.

Committee to invite H. W. Wiley to address the convention: H. A. Huston of New York; B. B. Ross of Alabama; and R. E. Doolittle of Illinois.

The meeting adjourned at 12.30 p. m. to reconvene at 2 p. m.

FIRST DAY.

MONDAY—AFTERNOON SESSION.

No report on phosphoric acid was presented because of the death, during the year, of W. J. Jones, jr., the referee.

THE EFFECT OF MASS AND DEGREE OF FINENESS ON THE PERCENTAGE OF AVAILABLE PHOSPHORIC ACID IN PRECIPITATED PHOSPHATE.

By H. D. Haskins (Agricultural Experiment Station, Amherst, Mass.).

The purpose of this paper is to bring before the association the results of some studies made at the Massachusetts Agricultural Experiment Station on a high grade precipitated phosphate, which tend to show that the present official method for the determination of available phosphoric acid in fertilizers is not applicable to this class of materials.

The precipitated phosphate which served for the studies is prepared by treating the phosphoric acid obtained in the manufacture of glue with hydrated or slaked lime. The product is very finely divided, white, neutral in reaction, said to be dried at 150°F., and nearly chemically pure bicalcium phosphate.

The scope of the experiment was to study the effect of a 1- and 2-gram charge, using an ammonium citrate solution made neutral to cochineal as indicator (official) and ammonium citrate solution made neutral to

Table 1.

Analyses of precipitated phosphate material ground to pass a 1 mm. sieve.

NUMBER OF SAMPLE	MOISTURE	TOTAL PHOSPHORIC ACID	AMMONIUM CITRATE SOLUTION MADE NEUTRAL TO COCHINEAL AS INDICATOR (OFFICIAL)				AMMONIUM CITRATE SOLUTION MADE NEUTRAL TO LITMUS SOLUTION AS INDICATOR			
			Available phosphoric acid 2-gram charge	Insoluble phosphoric acid 2-gram charge	Available phosphoric acid 1-gram charge	Insoluble phosphoric acid 1-gram charge	Available phosphoric acid 2-gram charge	Insoluble phosphoric acid 2-gram charge	Available phosphoric acid 1-gram charge	Insoluble phosphoric acid 1-gram charge
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
1	5.12	42.92	29.70	13.22	38.84	4.08	31.01	11.91	36.80	6.12
2	4.15	42.92	31.77	11.15	39.78	3.14	32.89	10.03	37.72	5.20
3	4.49	42.92	31.01	11.91	39.60	3.32	32.59	10.33	36.95	5.97

TABLE 2.

Analyses of precipitated phosphate material ground to pass a 100 mesh sieve.

NUMBER OF SAMPLE	TOTAL PHOSPHORIC ACID	AMMONIUM CITRATE SOLUTION MADE NEUTRAL TO COCHINEAL AS INDICATOR (OFFICIAL)				AMMONIUM CITRATE SOLUTION MADE NEUTRAL TO LITMUS SOLUTION AS INDICATOR			
		Available phosphoric acid 2-gram charge	Insoluble phosphoric acid 2-gram charge	Available phosphoric acid 1-gram charge	Insoluble phosphoric acid 1-gram charge	Available phosphoric acid 2-gram charge	Insoluble phosphoric acid 2-gram charge	Available phosphoric acid 1-gram charge	Insoluble phosphoric acid 1-gram charge
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
1	42.92	32.64	10.28	39.83	3.09	34.35	8.57	37.97	4.95
2	42.92	33.28	9.64	40.27	2.65	35.09	7.83	38.17	4.75
3	43.02	40.47*	2.55*	40.26	2.76	34.65	8.37	38.27	4.75

* Two hundred mesh sieve; 1 gram charge.

litmus solution as indicator; also, to study the effect of the two solutions on the product ground to pass a 100 mesh sieve.

Unusual care was taken in standardizing the solutions of neutral citrate of ammonia. Considerable difference exists in the two solutions standardized by the two indicators, cochineal and litmus. The results obtained by the two solutions did not seem to be constant in all cases, a somewhat higher available phosphoric acid having been obtained by the citrate solution, standardized with litmus, in all cases where a 2-gram charge was employed. On the other hand, the citrate solution, standardized with cochineal, gave invariably higher results with a 1-gram charge, irrespective of the degree of fineness of the phosphate.

The available phosphoric acid obtained by both solutions was appreciably higher when used on the phosphate ground to pass a 100 mesh sieve. Finer grinding did not increase the availability of the phosphoric acid to any great extent.

The most striking results obtained in the study are noticed in the very much higher yield of available phosphoric acid in all cases where a 1-gram charge was used. This would indicate that in a product containing such a large proportion of available phosphoric acid (nearly 40 per cent) a 2-gram charge produces a highly saturated citrate solution, resulting in a correspondingly low phosphoric acid availability. It is believed that the subject is of sufficient importance to warrant recognition by the association.

RECOMMENDATION.

It is recommended—

That the referee on phosphoric acid for 1918 be instructed to make a study of the analysis of precipitated phosphate with a view to modifying the present official method, or to substituting therefor some suitable method for the estimation of available phosphoric acid in this class of materials.

E. O. Thomas (Paul-Gale-Greenwood Building, Norfolk, Va.), presented a paper giving some results obtained for "Insoluble Phosphoric Acid in Organic Base Goods"[1], using the official method[2].

No report was presented by the committee on basic slag, to cooperate with the committee on vegetation tests on the availability of phosphoric acid in basic slag.

REPORT ON NITROGEN.

By H. D. HASKINS (Agricultural Experiment Station, Amherst, Mass.), *Referee*, and I. K. PHELPS (Bureau of Chemistry, Washington, D. C.), *Associate Referee.*

Collaborators were asked to study the West Coast refraction method for the analysis of nitrate of soda and the use of sodium sulphate in place of potassium sulphate in the Gunning method. No samples were submitted but each collaborator was requested to use samples from his own collection. Determination of nitrogen in nitrate of soda by the Ulsch-Street, zinc iron, modified Kjeldahl and the ferrous sulphate-zinc-soda methods in addition to the West Coast refraction method was requested.

WEST COAST REFRACTION METHOD.

Moisture.—Weigh approximately 10 grams of the material (60 mesh) accurately, spread out on a tared watch glass and dry in a hot air oven at 160°C. for exactly 5 hours; cool in a desiccator and weigh. The loss in weight represents moisture.

Insoluble residue.—Dissolve approximately 50 grams of the material in 200–300 cc. of hot water and filter, preferably on a weighed Gooch crucible. Make the filtrate up to 1000 cc. and preserve it for the subsequent determinations of the other impurities. Dry the crucible and contents at 120–130°C. to constant weight. The net weight represents the total insoluble matter.

Sodium chlorid.—Titrate 100 cc. of the solution, equivalent to 5 grams of material, with N/10 silver nitrate solution. Before beginning the titration add 1–2 mg. of sodium carbonate to sharpen the end point with the potassium chromate indicator, (1 cc. of a saturated potassium chromate solution). The silver nitrate solution must be added slowly so that the red precipitate of silver chromate disappears as fast as it is formed; otherwise, there is danger of its being occluded in the silver chlorid. The end point is taken at the first permanent reddish tint. One cc. of N/10 silver nitrate equals 0.005846 gram of sodium chlorid. The report should read: "Total chlorids equivalent to ——— per cent of NaCl".

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[1] *J. Ind. Eng. Chem.*, 1917, 9: 865.
[2] *Assoc. Official Agr. Chemists, Methods*, 1916, 4.

Sodium sulphate.—In the determination of sulphates use exactly 100 cc. of the filtrate from the insoluble residue determination. Expel nitric acid by evaporation with an excess of hydrochloric acid. Precipitate the sulphuric acid with 10 cc. of 10% barium chlorid solution. Barium sulphate multiplied by 0.60859 equals sodium sulphate. The result should be stated in terms of sodium sulphate.

Potassium oxid.—Determine the percentage of potassium oxid according to the official method, reporting the results as "Potash equivalent to potassium nitrate".

RESULTS OF COLLABORATIVE WORK ON NITROGEN.

Table 1.

Determination of nitrogen in nitrate of soda.

ANALYST	ULSCH-STREET METHOD	ZINC IRON METHOD	MODIFIED KJELDAHL METHOD	FERROUS SULPHATE-ZINC-SODA METHOD	WEST COAST METHOD
	per cent	*per cent*	*per cent*	*per cent*	*per cent*
W. D. Richardson, Swift & Co., Chicago, Ill.	14.30	14.59	14.15	14.50	14.66
	14.53	14.57	14.29	14.57	14.66
	14.39	14.64	14.15	14.51	....
	14.39	14.59	14.29	14.44	
Average............	14.40	14.60	14.22	14.50	14.66
	15.41	15.41	15.41	15.41	15.34
	15.27	15.27	15.41	15.41	15.48
	15.26	15.44	15.41	15.41	15.35
	15.41	15.44	15.34	15.55	....
Average............	15.34	15.39	15.39	15.44	15.39
L. S. Walker, Agricultural Experiment Station, Amherst, Mass.	15.41	15.74	15.17	15.19	16.60
	15.45	15.63	15.17	15.27	16.58
	15.29	15.61	14.93	....	....
Average............	15.38	15.66	15.09	15.23	16.59
	15.21	15.41	15.19	14.88	15.33
	15.21	15.41	15.11	14.90	15.34
	15.31	15.29	14.91	....	....
Average............	15.24	15.37	15.07	14.89	15.34
	15.27	15.82	15.07	15.13	15.65
	15.31	15.74	14.95	15.11	15.63
	15.48	15.68	....	....	....
	....	15.80	....	....	....
Average............	15.35	15.76	15.01	15.12	15.64

It was estimated that the time required for the determination of the impurities in the case of the West Coast method was at least three times more than for the direct nitrogen determination by any one of the other methods. It does not seem to your referee that the method has sufficient merit to warrant further study, especially as the association has two official and one tentative method for the analysis of nitrate salts.

SUBSTITUTION OF SODIUM SULPHATE FOR POTASSIUM SULPHATE IN THE GUNNING METHOD.

Four collaborators have reported results on these methods and quite a variety of substances of difficult oxidation were included in the work.

TABLE 2.

Determination of nitrogen by the Gunning method.

ANALYST	MATERIAL	NITROGEN USING POTASSIUM SULPHATE	NITROGEN USING SODIUM SULPHATE
		per cent	*per cent*
J. W. Kellogg, State Department of Agriculture, Harrisburg, Pa.	Cottonseed meal	6.24	6.19
	Animal tankage	4.98	4.86
	Low grade fertilizer	0.19	0.13
	Chop feed	1.40	1.35
	Jersey peat	1.36	1.27
Average		2.83	2.76
W. D. Richardson	Cottonseed meal	7.29	7.32
	Castor pomace	5.43	5.37
	Tartar pomace	2.07	2.09
	Dried blood	14.00	13.99
	Peat	2.79	2.79
	Garbage tankage	2.71	2.67
	Leather scrap	7.10	7.12
	Base goods	3.33	3.36
	High grade tankage	9.13	9.16
Average		5.98	5.99
J. J. Vollertsen, Morris & Company, Chicago, Ill.	Cottonseed meal	5.68	5.65
	Leather tankage	7.22	7.24
	Garbage tankage	3.00	2.98
	Leather scrap	5.00	4.98
	Degreased garbage tankage	2.94	2.94
Average		4.77	4.76
L. S. Walker	Cottonseed meal*	6.33	6.32
	Castor pomace†	4.77	4.79
	Peat	0.94	1.03
	Millet straw and seed	1.33	1.37
	Nitrolene (treated leather)	7.43	7.37
	Wool waste†	1.63	1.66
	Barnyard manure†	1.05	1.03
	Goat manure	1.33	1.38
	Sheep manure	2.44	2.37
	Tannery waste	2.21	2.13
Average		2.95	2.95

* Average of 20 samples.
† Average of 2 samples.

DISCUSSION OF RESULTS.

Fifty-one samples have been tested in this study, including twenty-one different types of material, many of which are among the most difficult of oxidation. The average percentage of nitrogen obtained on the fifty-one samples by using potassium sulphate was 4.18 per cent; by using the sodium sulphate, 4.17 per cent. In view of these results, it does not seem necessary to accumulate further data on the subject.

RECOMMENDATIONS.

It is recommended—

(1) That work on the West Coast refraction method be discontinued.

(2) That the referee for 1918 study the Lunge nitrometer method, which is invariably used by the manufacturers of explosives, for the analysis of nitrate of soda.

(3) That the use of sodium sulphate in place of potassium sulphate in the Gunning method be made official.

REPORT ON THE STUDY OF THE EFFECT OF GLASS WOOL IN THE FERROUS SULPHATE-ZINC-SODA METHOD FOR NITRATES.

By I. K. Phelps (Bureau of Chemistry, Washington, D. C.).

From the very few reports which have been made by the collaborators on this method, it has been impossible to draw any definite conclusions. Furthermore, the opinions of various collaborators, obtained by correspondence, seem to be at variance. As this method is at present a tentative one, it is recommended that a further study be made before it is either rejected or accepted as official.

THE USE OF PERMANGANATE IN THE KJELDAHL METHOD MODIFIED FOR NITRATES.

By I. K. Phelps (Bureau of Chemistry, Washington, D. C.).

Although very few reports have been made on this study, it has been possible to draw certain definite conclusions regarding the addition of potassium permanganate to the products of boiling with sulphuric acid and mercury.

Two series of experiments were outlined. In the first series the directions of the official method were given[1]. Inasmuch as considerable

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 8.

latitude is allowed by these directions, a second series was conducted in which the directions for certain procedures were more specific. Accordingly, the following additional directions were given for this series:

(a) Allow the acid mixture and nitrate to stand for 2 hours.

(b) Allow the mixture to stand for 4 hours or more (note actual time) after the addition of the thiosulphate.

(c) Heat with a low flame for 15 minutes.

(d) Digest in the presence of mercury for 3 hours.

(e) Use 300 cc. of water instead of 200 cc. for diluting the sulphuric acid mixture.

At the end of the boiling with mercury and sulphuric acid, the directions specified the following procedure:

If permanganate is to be added, remove the flask from the flame and, holding it upright, add 0.5 gram of finely divided permanganate as directed under each of the following experiments:

(a) Omit the addition of the permanganate.

(b) Immediately upon the removal of the flask from the flame, add the permanganate as rapidly as possible.

(c) Beginning immediately upon the removal of the flask from the flame, gradually add the permanganate in such a manner that 1 minute is required to complete the addition.

(d) After allowing the contents of the flask to cool for 5 minutes, add the permanganate as rapidly as possible.

(e) After allowing the contents of the flask to cool for 5 minutes, gradually add the permanganate in such a manner that 1 minute is required to complete the addition.

The cooling, the addition of water, sulphid and sodium hydroxid, and the distillation were made in the usual manner, except as specified under the preceding directions.

The results of the first series were not considered because of the divergence in the procedure of the collaborators as allowed in the directions. In this connection it should be mentioned that certain procedures of the official method should be further studied and the directions made more specific.

It has been the experience of the referee that more accurate and more concordant results are obtained and the period of hydrolysis shortened if potassium sulphate is added with the mercuric oxid and if the use of permanganate is omitted. It seems desirable, therefore, that a critical study of such a method should be made. Furthermore, the experience of the referee indicates that it is desirable to study in more detail the effect of the time of standing of the nitrate with the sulphuric acid-salicylic acid mixture, before and after adding the thiosulphate; the preliminary heating with the low yellow flame; the vigorous heating before adding mercury and potassium sulphate; and the final vigorous heating in the presence of these catalysts. In this connection it might be pointed out to advantage that complete hydrolysis of many compounds is not assured when the sulphuric acid mixture becomes colorless. Moreover, it seems desirable to study the effect of substituting sodium sulphate for potassium sulphate.

In the following experiments 0.2 gram of sodium nitrate, containing 0.03296 gram of nitrogen, was allowed to stand for 2 hours with the sulphuric acid-salicylic acid mixture. After the addition of sodium thiosulphate, the mixture was allowed to stand for 4 hours. The preliminary heating with a low yellow flame required 15 minutes. After 5 minutes' vigorous boiling the mercuric oxid was added and the mixture boiled for 3 hours. The results of the second series are given in tabulated form.

Nitrogen in sodium nitrate.

ANALYST	WITHOUT PERMANGANATE	TREATMENT WITH PERMANGANATE			
		Immediate		After 5 minutes	
		Rapid	Minute to complete	Rapid	Minute to complete
	gram	*gram*	*gram*	*gram*	*gram*
J. J. Vollertsen, Morris & Company, Chicago, Ill.	0.03288	0.03206	0.03206	0.03330	0.03248
	0.03288	0.03206	0.03248	0.03288	0.03288
W. D. Richardson, Swift & Company, Chicago, Ill.	0.03313	0.03299	0.03292	0.03285	0.03292
	0.03306	0.03250	0.03229	0.03285	0.03292
L. J. Jenkins, Bureau of Chemistry, Washington, D. C.	0.03280	0.03252	0.03273	0.03266	0.03280
	0.03280	0.03266	0.03252	0.03280	0.03280
H. W. Daudt, Bureau of Chemistry, Washington, D. C.	0.03284	0.03266	0.03258	0.03274	0.03280
	0.03293	0.03275	0.03278	0.03276	0.03280

It is to be noted that, when the permanganate is added immediately after the flame is extinguished, whether rapidly or slowly, the amounts of nitrogen recovered are lower than when the permanganate is omitted. When 5 minutes are allowed to elapse before the addition of the permanganate, the results approach more closely those obtained when the permanganate is omitted.

These results agree so well with the results of William Frear[1], and are in such close accord with the experience of a large number of collaborators, whose opinions have been expressed in correspondence as well as in conversation, that the following recommendation is made:

RECOMMENDATION.

It is recommended—

That as the use of potassium permanganate in the Kjeldahl method modified for nitrates may cause the loss of nitrogen it should be omitted.

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[1] *J. Assoc. Official Agr. Chemists*, 1919, 3: 220.

INVESTIGATION OF THE KJELDAHL METHOD FOR DETERMINING NITROGEN.

By I. K. PHELPS (Bureau of Chemistry, Washington, D. C.), *Associate Referee on Special Study of the Kjeldahl Method*, and H. W. DAUDT[1] (Bureau of Chemistry, Washington, D. C.).

The hydrolysis of pyridin compounds and other refractory compounds was discussed in 1916[2]. Approximately 0.3 gram of pyridin zinc chlorid was hydrolyzed for each analysis, digesting for 2½ hours with a *boiling* mixture of 25 cc. of sulphuric acid, 0.7 gram of mercuric oxid and 10 grams of potassium sulphate. When sodium sulphate was substituted for an equal weight of potassium sulphate the results obtained were below the theory, the error equaling as much as 10 per cent of the total nitrogen. In order to investigate the effect of varying proportions of potassium sulphate, or of sodium sulphate and sulphuric acid in the presence of mercury, return condensers, constructed entirely of lead, were placed in the neck of the flask during hydrolysis. These served not only to prevent the vaporization of sulphuric acid, but also to retain the acid ammonium sulphate even when excessive quantities of potassium sulphate were employed.

It was found that, with the return condensers constructed of lead, amounts of potassium sulphate and sulphuric acid in the presence of mercury determine the completeness of the hydrolysis. For instance, in the presence of 0.7 gram of mercuric oxid, when 25 cc. of commercial "96 per cent C. P." acid and 10 grams of potassium sulphate were used, incomplete decomposition was obtained, but, when 15 cc. of sulphuric acid were used with amounts of potassium sulphate varying from 10 to 30 grams, excellent results were obtained. When more potassium sulphate was employed, the results were somewhat lower. Sodium sulphate seemed to give varying results.

Recent investigations have been made indicating the proportions of sulphuric acid and potassium sulphate or sodium sulphate which may be employed in hydrolyzing mixtures in open flasks without causing volatilization of ammonia from the acid ammonium sulphate. In these experiments the flasks rested in a 2½ inch perforation in an asbestos sheet $\frac{1}{16}$ inch thick and were supported 4 inches above the top of the burner. The source of heat was a Bunsen flame 7 inches in height, having a cold cone 3 inches in height. No losses in ammonia were observed when the mixture, containing 25 cc. of sulphuric acid and 10 grams of potassium or 8.2 grams of sodium sulphate, was heated for

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[1] Present address, Jackson Laboratory, E. I. Du Pont Co., Wilmington, Del.
[2] *J. Assoc. Official Agr. Chemists*, 1920, 3: 306.

2½ hours. When 15 cc. of acid with the same amounts of sulphate were used, the losses were very small in some cases and in others negligible. With larger amounts of either of the sulphates, losses occurred in each case becoming large with 20 grams or more of potassium sulphate. When 16.3 grams of sodium sulphate were used, the loss was smaller than that which occurred with the molecular equivalent (20 grams) of potassium sulphate.

Further investigations with the use of the lead condensers show the effect of water on the hydrolysis of pyridin zinc chlorid. By using 92.5 per cent sulphuric acid it was found impossible to hydrolyze 0.4 gram of pyridin zinc chlorid completely in 2½ hours, even with the mixtures of sulphuric acid, mercuric oxid and potassium sulphate found efficient in previous work. With 100 per cent sulphuric acid, the hydrolysis was found to be complete in all cases where suitable proportions of mercuric oxid, potassium sulphate and acid were maintained. In the presence of 0.7 gram of mercuric oxid and 10 grams of potassium sulphate, hydrolysis of 0.4 gram of pyridin zinc chlorid was complete in 2½ hours, when either 15 or 20 cc. of acid were used, but not when 25 cc. were used. In the presence of 0.2 gram of mercuric oxid, hydrolysis was complete only when 15 cc. of acid were used with 15 or 20 grams of potassium sulphate. With sodium sulphate, hydrolysis was complete only in the presence of 0.7 gram of mercuric oxid, 15 cc. of sulphuric acid and amounts of sodium sulphate varying from 8.2 grams to 16.3 grams. When 20 cc. of acid were used with 8.2 grams of sodium sulphate, the equivalent of 10 grams of potassium sulphate, hydrolysis was not complete. In no case was hydrolysis complete in the time stated (2½ hours) in the presence of 0.2 gram of mercuric oxid and sodium sulphate. Experiments on the hydrolysis of pyridin zinc chlorid in open flasks indicated that the hydrolysis of 0.4 gram was complete in 2½ hours with a boiling mixture of 0.7 gram of mercuric oxid, 10 grams of potassium sulphate and 25 cc. of sulphuric acid. With 15 cc. of acid the results were slightly lower than theory, owing to volatilization of nitrogen. This was indicated by the small amounts of sulphuric acid left after hydrolysis, as well as by the experiments cited earlier. When 8.2 grams of sodium sulphate were substituted for 10 grams of potassium sulphate, hydrolysis was incomplete with 25 cc., but complete with either 15 or 20 cc. of acid.

Upon investigation of the influence of the time necessary for hydrolysis, it was found that it varied with different proportions of acid and potassium or sodium sulphate. For instance, with condensers and with 0.7 gram of mercuric oxid and 15 cc. of sulphuric acid, the hydrolysis of 0.4 gram of pyridin zinc chlorid with 10 grams of potassium sulphate was far from complete in 1 hour, but complete in 1½ hours, while with 15 grams of potassium sulphate it was practically complete in 1 hour. Hydrolysis with 0.7 gram of mercuric oxid, 12.2 grams of sodium sulphate and 15 cc. of sulphuric acid was incomplete in 1½ hours, but complete in 2½ hours.

In a number of experiments conducted with open flasks in the presence of 0.7 gram of mercuric oxid and 10 grams of potassium sulphate, hydrolysis with 25 cc. of sulphuric acid required 2 hours, while with 20 cc. of acid only 1½ hours were required. With 8.2 grams of sodium sulphate, instead of 10 grams of potassium sulphate, and 15 or 20 cc. of sulphuric acid, 2 hours were required, while 1½ hours showed complete hydrolysis. It is to be noted that with the use of the open flasks another factor, the rate of volatilization of acid, is introduced. This effect depends mainly on the intensity of heating and to a less extent on the time of boiling.

The hydrolysis of certain organic compounds of various constitutions was reported in 1916[1]. In the presence of 0.7 gram of mercuric oxid, 10 grams of potassium sulphate and 25 cc. of sulphuric acid, weights of the compounds varying from 0.2 to 0.4 gram were hydrolyzed completely by heating in open flasks at the boiling point for 2½ hours. The use of sodium sulphate in the place of potassium sulphate has been applied to the compounds given below. Reliable results were obtained when these compounds were boiled for 2½ hours with mixtures of 0.7 gram of mercuric oxid, 8.2 grams of sodium sulphate and 20 cc. of sulphuric acid. Below are grouped the compounds studied.

Glucosamin hydrochlorid.
Tetramethylammonium derivative:
  Tetramethylammonium iodid.
Pyrol derivative:
  Isatin.
Pyrolidin derivatives:
  Atropin.
  Cocain.
Pyridin derivatives:
  Nicotin zinc chlorid.
  Nicotinic acid.
Piperidin derivative:
  β-Eucaine hydrochlorid.
Quinolin derivatives:
  Hydroxyquinolin.
  Cinchonidin.
  Strychnin.
  Brucin.
Isoquinolin derivatives:
  Papaverin.
  Narcotin.
  Morphin.
  Hydrastinin.
Purin derivative:
  Caffein.
Imidazole or glyoxalin derivative:
  Lophin.
Quinoxalin derivative:
  Quinoxalin hydrochlorid.
Quinazolon derivatives:
  2-Methyl 4-quinazolon.
  2-Methyl 3-phenyl 4-quinazolon.

When 25 cc. of acid were used with 8.2 grams of sodium sulphate and 0.7 gram of mercuric oxid, the hydrolysis in many cases was not complete

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **3**: 306.

as with nicotinic acid, nicotin zinc chlorid and hydroxyquinolin. With this mixture, the completeness of hydrolysis depends very markedly on the amount of acid volatilized.

The influence of reagents and apparatus was also investigated. Ammonia free water, redistilled from alkaline permanganate solution through a metal Kjeldahl connecting bulb soldered to a block tin condensing tube, was used in this work. The bulb was connected with the flask by means of a cork stopper entirely covered with tin foil. In all experiments where it was desired to avoid the influence of glass or of rubber stoppers, the above apparatus was used. It was noted that the pure reagents of commerce for the estimation of nitrogen by the Kjeldahl hydrolysis contribute small amounts of ammonia reacting substances. Rubber stoppers in the Kjeldahl flask during hydrolysis[1] are believed to contribute ammonia reacting substances. Again, rubber stoppers used in connecting the Kjeldahl flask to the condenser contribute ammonia reacting substances. The ammonia reacting substances contributed by a rubber stopper held in the neck of a flask during the acid hydrolysis are sufficiently large to be appreciable in ordinary routine Kjeldahl determinations, unless very closely defined conditions are followed. The error is, furthermore, variable and the use of stoppers unnecessary. Even the especially purified reagents contain traces of such substances. The glass, also, contributes a small amount of alkaline reacting substances. The magnitude of the error due to the alkaline reacting substances is, however, so small that all of these may be neglected except in work in which high precision is necessary. The conclusion is obvious that, in all routine work involving determinations by the Kjeldahl method, it is necessary to deduct from the result obtained the amount corresponding to the ammonia reacting substances contributed by reagents and apparatus in use in the particular experiments. The results recorded in this investigation were obtained even when greater precautions were taken than is customary in routine work. It is very obvious that under less carefully controlled conditions in routine work the errors, which are here called inappreciable, will become large enough to affect seriously the accuracy of the results obtained.

When hydrolysis is made in open flasks, the proportions of sulphuric acid and potassium or sodium sulphate which may cause loss of ammonia by volatilization have been indicated. The proportions of sulphuric acid, mercuric oxid and potassium or sodium sulphate giving complete hydrolysis of a refractory compound (pyridin zinc chlorid) have been shown. These proportions contain slightly more acid than those which may cause loss of ammonia by volatilization. This difference in proportions is so slight, however, that hydrolysis in open flasks of

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[1] *J. Ind. Eng. Chem.*, 1916, 8: 639.

refractory compounds must be conducted with closely controlled conditions. This is particularly true in the case of sodium sulphate because amounts of sodium sulphate in excess of those proportionate to the suitable amounts of potassium sulphate are necessary. The differences in the behavior of the two sulphates is probably due to differences in the tendencies of the acid sulphates to retain water. Consequently, the hydrolysis of very refractory compounds with sodium sulphate is not at present recommended, although with very closely controlled conditions excellent results can be obtained. The influence of the reagents and the apparatus on the accuracy of the modified Kjeldahl method has been indicated.

REPORT ON POTASH.

By T. D. Jarrell[1], (State College of Agriculture, College Park, Md.), *Referee.*

The following samples and instructions were sent to collaborators:

Sample No. 1.—Commercial potassium chlorid.

Sample No. 2.— A mixture of acid phosphate and sulphate of potash-magnesia (containing about 9% of potassium oxid).

Sample No. 3.—A mixture of acidulated garbage tankage, acid phosphate and commercial muriate of potash (containing about 7% of potassium oxid).

SAMPLE No. 1.

Determine the potash by the official[2] and the following method:

PERCHLORATE METHOD.

Dissolve the potash as in the official method. Acidify with about 5 cc. of hydrochloric acid. While the solution is hot, precipitate the sulphate by adding, drop by drop, in slight excess normal barium chlorid solution acidified with hydrochloric acid. Cool, make to volume and shake.

Allow the precipitate to settle and filter. Transfer an aliquot, corresponding to 0.25 gram of sample, to an evaporating dish, add 5 cc. of perchloric acid (sp. gr. 1.12); evaporate on a steam or sand bath until it fumes strongly, take up the residue with 5 cc. of water, add a second 5 cc. of perchloric acid and again evaporate the solution *carefully* until all free hydrochloric acid is driven off and dense white fumes of perchloric acid appear. If the solution goes to dryness and a hard mass remains, take up with a few drops of perchloric acid. When a water bath is used for the evaporation, finally place the dish on a hot plate and heat carefully until hydrochloric acid is driven off. After cooling, add 20 cc. of 95% alcohol and stir well. Allow to stand for 30 minutes. Decant the alcohol through a Gooch crucible having a fairly thick pad (about $\frac{1}{8}$ inch thick) of asbestos and wash twice by decantation with 95% alcohol containing 0.2% perchloric acid, made by adding 1 cc. of perchloric acid (sp. gr. 1.12 = 20%) to 100 cc. of 95% alcohol. Transfer the precipitate to a Gooch crucible with the 95% alcohol containing perchloric acid and wash until the entire filtrate amounts to 75 cc. Finally wash twice

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[1] Present address, Bureau of Chemistry, Washington, D. C.

[2] *Assoc. Official Agr. Chemists, Methods,* 1916, 12.

with alcohol-ether (1 part 95% alcohol to 1 part ethyl ether), using 3–5 cc. each time to wash out all perchloric acid. Dry for 30 minutes at 120–130°C., then weigh. Dissolve the potassium perchlorate from the Gooch crucible with about 200 cc. of hot water and dry to constant weight in an air oven. Allow to cool and weigh. The loss in weight is potassium perchlorate ($KClO_4$).

SAMPLE NO. 2 AND NO. 3.

Determine the potash by the official[1] and the following methods:

MODIFIED OFFICIAL METHOD.

This is the same as the official method except that the addition of 2 cc. of concentrated hydrochloric acid to the potash solution is omitted. After washing 2.5 grams on the filter paper with boiling water, add directly to the hot solution ammonium hydroxid and ammonium oxalate and proceed as in the official method.

PERCHLORATE METHOD.

Weigh 2.5 grams of the sample upon a 12.5 cm. filter paper and wash with successive small portions of boiling water into a 250 cc. graduated flask to a volume of about 200 cc. Allow to cool, make to the volume and shake. Do not add ammonium hydroxid or ammonium oxalate. Transfer 50 cc. of the solution to a porcelain or silica dish (do not use platinum), add an excess of a 3% solution of barium hydroxid and without filtering evaporate to dryness over a sand bath. Gently ignite the residue over a Bunsen burner *below redness* for about 5 minutes. Extract the residue with 25 cc. of boiling water, breaking up the material as much as possible. Filter into an evaporating dish of about 175 cc. capacity, and wash with boiling water until the filtrate amounts to about 150 cc. Add 5 cc. of perchloric acid, evaporate carefully on the sand bath until it fumes strongly, take up with 5 cc. of water, add a second 5 cc. of perchloric acid, evaporate, cool, and proceed as already outlined.

It is requested that the modified official method be tested thoroughly (i. e. omitting the addition of 2 cc. of concentrated hydrochloric acid to the potash solution) on some high potash content samples, using acid phosphate, kainit, commercial potassium chlorid, commercial potassium sulphate, and manure salts.

Since these mixtures can not be obtained on the market at present, it is suggested that the collaborator prepare them in his laboratory.

TABLE 1.

Collaborators' results of potash determination expressed as potassium oxid.*

ANALYST	SAMPLE NO. 1		SAMPLE NO. 2			SAMPLE NO. 3		
	Official method	Perchlorate method	Official method	Modified official method hydrochloric acid omitted	Perchlorate method	Official method	Modified official method hydrochloric acid omitted	Perchlorate method
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
T. D. Jarrell, College of Agriculture, College Park, Md.	50.22	50.70	9.67	9.73	9.41	7.58	7.63	7.18

* Results reported are averages.

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[1] *Assoc. Official Agr. Chemists, Methods,* 1916, 12.

TABLE I.—Continued.

Collaborator's results of potash determination expressed as potassium oxid.

ANALYST	SAMPLE NO. 1		SAMPLE NO. 2			SAMPLE NO. 3		
	Official method	Perchlorate method	Official method	Modified official method hydrochloric acid omitted	Perchlorate method	Official method	Modified official method hydrochloric acid omitted	Perchlorate method
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
E. F. Berger, Agricultural Experiment Station, E. Lansing, Mich.	51.45	51.07	9.68	9.63	9.65	7.55	7.56	7.58
Percy O'Meara, Agricultural Experiment Station, E. Lansing, Mich.	....	....	9.26	9.53	...	...	...	7.67
W. D. Richardson, Swift & Company, Chicago, Ill.	50.15	50.22	10.00	...	9.67	7.50	7.55	7.29
D. L. French, Dearborn Chemical Co., Chicago, Ill.	50.90	50.47	9.38	9.76	9.02	7.62	7.60	6.71*
R. F. Keeler, Agricultural Experiment Station, Geneva, N. Y.	49.90	49.83	9.62	...	9.30	7.44	...	7.14
G. J. Kuhlman, Department of Agriculture, Harrisburg, Pa.	50.68	49.44	...	...	...	7.38	7.47	...
J. T. Foy, Clemson Agricultural College, Clemson College, S. C.	50.91	49.90	9.96	10.02	8.82	7.67	7.60	7.03
Wm. Rodes, Agricultural Experiment Station, Lexington, Ky.	50.68	49.64	9.48	9.37	8.32*	7.55	7.58	7.14
C. G. Remsburg, State College of Agriculture, College Park, Md.	....	....	9.58	9.79	9.41	7.35	7.35	7.45
General Average	50.98	50.16	9.63	9.69	9.33	7.52	7.54	7.30

* Omitted from general averages.

In Tables 2 and 3 additional results of collaborators working upon special mixtures and raw materials are reported.

TABLE 2.

Comparative results of potash determination expressed as potassium oxid.

ANALYST	DESCRIPTION OF MIXTURE	OFFICIAL METHOD (AVERAGE RESULTS)	MODIFIED OFFICIAL METHOD. NO HYDROCHLORIC ACID (AVERAGE RESULTS)
		per cent	*per cent*
W. D. Richardson.	Kainit, acid phosphate and tankage.	7.83	7.98
	Manure salts, acid phosphate and tankage.	8.92	8.90
	Potassium chlorid, acid phosphate and tankage.	10.29	10.25
	Potassium sulphate, acid phosphate and garbage tankage.	12.09	11.98
E. F. Berger and Percy O'Meara.	Potassium chlorid, acid phosphate, tobacco stems and sheep manure.	4.47 4.22	4.41 4.22
	Potassium sulphate and acid phosphate.	8.09 8.08	7.96 7.96
	Potassium chlorid and acid phosphate.	12.32 12.26	12.30 12.15
T. D. Jarrell.	Sulphate of potash-magnesia and acid phosphate.	5.29* 7.18* 10.54*	5.38* 7.35* 10.67*
	Sulphate of potash-magnesia, kainit and acid phosphate.	8.15*	8.24*
	Sulphate of potash-magnesia and base goods.	7.47*	7.50*
	Potassium chlorid and acid phosphate.	6.64*	6.66*
	Potassium sulphate, dried fish and acid phosphate.	7.03*	7.00*
Average	....	8.29	8.29

* Average of three or more closely agreeing results.

TABLE 3.

A comparison of the official and modified official methods for potash.

(W. D. Richardson, Analyst.)

DESCRIPTION OF SAMPLE	POTASSIUM OXID	
	Official method	Perchlorate method
	per cent	*per cent*
Kainit, acid phosphate and tankage	8.09	8.25
	7.97	8.29
Manure salts, acid phosphate and garbage tankage	8.92	9.14
	8.90	9.16
Muriate of potash, acid phosphate and tankage	10.29	10.17
	10.25	10.23
Sulphate of potash, acid phosphate and garbage tankage.	12.13	12.33
	12.09	12.46
Commercial muriate of potash	53.90	54.03
	53.72	53.98
Kelp (dried)	15.71	15.37
	15.77	15.46
Commercial fertilizer	11.85	11.90
	11.70	12.06
Corn cob ash	13.85	14.00
	13.88	14.28
Refuse molasses ash	39.72	39.72
	39.58	39.72
Nebraska lake potash	21.71	21.83
	21.68	21.68

DISCUSSION BY REFEREE OF METHODS AND RESULTS.

MODIFIED OFFICIAL METHOD.

The following is a summary of the cooperative work since 1914, investigating the effect of adding hydrochloric acid to the potash solution.

TABLE 4.

Cooperative results from 1914 to 1917, expressed as potassium oxid.

YEAR	DESCRIPTION OF SAMPLE	WITH HYDROCHLORIC ACID	HYDROCHLORIC ACID OMITTED
		per cent	*per cent*
1914	Acid phosphate and kainit	6.11	6.10
1915	Acid phosphate, kainit and commercial potassium chlorid.	5.07	5.07
1915	Acid phosphate, dried blood and commercial potassium sulphate.	8.55	8.58
1916	Average of 48 samples of various mixtures.	3.66	3.65
1917	Acid phosphate and double manure salts.	9.63	9.69
1917	Acid phosphate, tankage and commercial potassium chlorid.	7.52	7.54

The cooperative results during the past four years on samples of many different mixtures have shown that no difference is caused by the addition or non-addition of hydrochloric acid to the potash solution.

In view of the data here reported, the referee concludes that the addition of 2 cc. of concentrated hydrochloric acid to the water extract of mixed fertilizers and boiling is an unnecessary operation, that its elimination is a desirable simplification and may be effected without sacrifice to accuracy.

PERCHLORATE METHOD.

The following is a summary of the results reported by collaborators, using the perchlorate and official methods:

TABLE 5.

Collaborators results, expressed as potassium oxid.

	SAMPLE NO. 1		SAMPLE NO. 2		SAMPLE NO. 3	
	Official method	Perchlorate method	Official method	Perchlorate method	Official method	Perchlorate method
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
General average	50.98	50.16	9.63	9.33*	7.52	7.30*
Maximum result	51.45	51.07	10.00	9.67	7.67	7.67
Minimum result	49.90	49.44	9.26	8.82	7.35	7.03
Extreme variation	1.55	1.63	0.74	0.85	0.32	0.64

* Results of one collaborator omitted from average.

The results which are presented in the table indicate that the perchlorate method is not as satisfactory as could be desired, yet there appears to be an improvement over the results obtained by collaborators in former years. Several chemists obtained excellent results by this method in comparison with the official method.

In the determination of potash by the perchlorate method it is extremely important that the details of manipulation be followed closely, and it is believed that the method of procedure, as given in the directions to collaborators, will give reasonable accuracy. Progress with this method has been relatively slow for the reason that collaboration from chemists experienced with the method has been difficult to obtain. So it seems reasonable to assume that the relatively wide differences in the results reported by several analysts may be attributed in a large measure to their inexperience with the method.

RECOMMENDATIONS.

It is recommended—

(1) That the official method for the preparation of potash solution[1] be revised to read as follows:

> Place 2.5 grams of the sample upon a 12.5 cm. filter paper and wash with successive portions of boiling water into a 250 cc. graduated flask until the filtrate amounts to about 200 cc. Add to the hot solution a slight excess of ammonium hydroxid and sufficient ammonium oxalate to precipitate all the lime present, cool, dilute to 250 cc. mix, and pass through a dry filter.

(2) That the work on the availability of potash be continued.

(3) That the study of the perchlorate method be continued.

A MODIFIED METHOD FOR THE DETERMINATION OF WATER-SOLUBLE POTASH IN WOOD ASHES AND TREATER DUST.

By H. D. Haskins (Agricultural Experiment Station, Amherst, Mass.).

It is a well established fact that products such as wood ashes, treater dust, or so-called lime-potash, manure ashes, etc., contain a much higher percentage of potash soluble in hydrochloric acid (strength 1 to 1), as well as weaker acid solutions (N/5 hydrochloric acid), than is dissolved by boiling water. It is conceivable that at least a portion of this water-insoluble potash may be loosely held in combination with silica as basic silicates, resulting from the process under which the wood ashes and treater dust are made.

The presence of more or less of these basic silicates was recognized

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 12.

by the earlier manufacturers of potash, who established the custom of allowing the ashes to remain in contact with water for a few days before subjecting them to the final leaching with hot water. Ashes thus treated usually gave a larger yield of potash than when leached without the soaking process.

Water-soluble potash was determined in a number of samples by the official method. Water-soluble potash was also determined by allowing the ashes to remain for two days in contact with just enough water to cover the ashes contained in a 4-ounce beaker. Subsequently, the ashes were washed with successive portions of boiling water to a volume of 200 cc. and, from this point, the manipulation was according to the official method. One sample of treater dust and 21 samples of ashes were analyzed by the two methods, the acid-soluble potash being determined in each case. The results are given in the following table:

Potash in wood ashes and treater dust.

DESCRIPTION OF SAMPLE	ACID-SOLUBLE POTASH	WATER-SOLUBLE POTASH (OFFICIAL)	WATER-SOLUBLE POTASH (MODIFIED METHOD)
	per cent	*per cent*	*per cent*
Dry treater dust	12.00	7.41	7.64
Wood ashes	3.39	1.92	2.16
Wood ashes*	7.08	3.95	4.31
Wood ashes	4.46	2.60	2.68
Wood ashes	5.40	2.69	2.81
Wood ashes	4.36	2.18	2.28
Wood ashes	1.40	0.70	0.79
Wood ashes	3.42	2.23	2.21
Wood ashes	3.07	1.52	1.51
Wood ashes	3.73	2.36	2.32
Wood ashes*	9.88	6.75	7.11
Wood ashes	0.47	0.11	0.13
Wood ashes	0.73	0.17	0.09
Wood ashes	3.64	2.48	2.21
Wood ashes	3.77	2.59	2.65
Wood ashes	2.93	2.38	2.38
Wood ashes	4.11	3.19	3.31
Wood ashes	4.23	3.00	3.14
Wood ashes	6.17	5.11	5.15
Wood ashes	3.11	2.26	2.20
Wood ashes	6.14	4.18	4.16
Wood ashes*	7.56	6.62	6.76

* Freshly made dry ashes.

This study indicates that freshly made dry ashes and dry treater dust, or cement potash, will yield appreciably more water-soluble potash if allowed to remain in contact with water for several hours previous to extracting with boiling water. In the case of ash products which contain considerable water (12 to 30 per cent), very little advantage results from the preliminary treatment with water. It would seem that, because of the present scarcity and high price of potash, it might be desirable for the association to make a further study of the subject with a view to adopting some modification of the present official method applicable to ashes and cement potash products.

REPORT ON WATER.

By J. W. SALE (Bureau of Chemistry, Washington, D. C.), *Referee.*

Three analytical methods were selected for the cooperative work, a rapid method for the determination of calcium and magnesium; a method for barium; and one for manganese. Numerous tests on the determination of free ammonia in water containing hydrogen sulphid were also conducted. This latter work was done in the Bureau of Chemistry, principally for the reason that sulphid waters change so rapidly in composition that it is not practicable to forward standard samples to collaborators.

Three samples of solutions of known composition, together with copies of the proposed methods, were forwarded to each of the collaborators. The methods of analysis, composition of standard samples, and results of the cooperative work are given below. The methods of analysis, submitted herewith, have been slightly modified to make them of general application.

INDUSTRIAL WATER.

CALCIUM AND MAGNESIUM.

REAGENTS.

(a) *Standard potassium permanganate solution, approximately N/5.*—Dissolve 6.322 grams of pure crystals in water and make up to 1 liter. Standardize against pure Iceland spar by the procedure given below.

(b) *Standard sodium thiosulphate solution, approximately N/5.*—Dissolve 49.6 grams of recrystallized sodium thiosulphate in 1 liter of water. Standardize against magnesium ribbon or some suitable compound of magnesium by the procedure given below.

(c) *Ammonium arsenate, crystals.*

(d) *Oxalic acid, crystals.*

(e) *Potassium iodid, crystals.*

(f) *Sulphuric acid (1 to 1).*

DETERMINATION[1].

Acidify and concentrate the sample as usual. Add about 0.5 gram of ammonium chlorid and precipitate the iron and alumina with ammonium hydroxid. Boil, filter and wash. To the filtrate, in a volume of about 100 cc., add 0.5 gram of oxalic acid, together with sufficient hydrochloric acid to clear the solution. Add 1–2 drops of methyl orange, heat to boiling and make slightly alkaline with dilute ammonia. Add immediately sufficient ammonium arsenate to precipitate the magnesium, then slowly add ammonia, with constant stirring, to the *hot* solution until the magnesium am-

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[1] *J. Ind. Eng. Chem.*, 1913, 5: 910.

monium arsenate comes down, or if the calcium oxalate is present in such quantity that this can not be observed, add about 10 cc. of strong ammonia water. Let cool and add 10–15 cc. more of strong ammonia. Let stand for 30 minutes with frequent shaking, filter and wash thoroughly with dilute ammonia water. Transfer the precipitate to a 300–500 cc. Erlenmeyer flask, using dilute sulphuric acid and water. Add 10 cc. of sulphuric acid, make up to about 75–80 cc. and titrate hot with permanganate solution. Let cool and add 25 cc. more of the acid. Add slowly 5 grams of potassium iodid and immediately titrate to a straw color with sodium thiosulphate. Stopper the flask and cover with black paper, or set in the dark for about 5 minutes. Eight minutes should elapse between the addition of the iodid and the last part of the titration. Without adding starch, complete the titration drop by drop. Read the end point and place the flask in the dark for 1 minute. If there is a return of color, discharge it. Usually there will be none if the last drops of thiosulphate have been added slowly. Apply a correction for iodin set free by the light, etc , by titrating as described above with 10 cc. and 20 cc. of a solution of ammonium arsenate (of which the exact concentration need not be known—about 20 grams to a liter is convenient). If 10 cc. required x cc. of thiosulphate and 20 cc. required y cc., then the correction is $(2x - y)$ cc.

MANGANESE.

REAGENTS.

(a) *Dilute nitric acid (1 to 4).*—Free from brown oxid of nitrogen by aeration.

(b) *Sulphuric acid (1 to 3).*

(c) *Dilute sulphuric acid.*—Dilute 25 cc. of concentrated acid to 1 liter with distilled water. Add enough permanganate solution to color faintly the dilute acid.

(d) *Standard manganous sulphate solution.*—Dissolve 0.2877 gram of pure potassium permanganate in about 100 cc. of distilled water, acidify the solution with sulphuric acid and heat to boiling. Add slowly a sufficient quantity of a dilute solution of oxalic acid to discharge the color. Cool and dilute to 1 liter. One cubic centimeter of this solution is equivalent to 0.1 mg. of manganese. The standard should be prepared by following the same procedure as is used for the sample. This solution is more permanent than a solution of potassium permanganate, which may, however, be used. To prepare it, dissolve 0.288 gram of potassium permanganate in distilled water and dilute the solution to 1 liter.

(e) *Sodium bismuthate.*—Pure dry salt.

DETERMINATION.

Remove chlorin by two or more evaporations with sulphuric acid from such a quantity of the sample as contains 1.0 mg. or less of manganese. Volatilize the sulphuric acid and ignite the residue gently at less than 500°C. Dissolve in 40 cc. of nitric acid, add about 0.5 gram of sodium bismuthate, and heat until the permanganate color disappears. Add a few drops of a solution of ammonium or sodium bisulphate to clear the solution and again boil to expel oxids of nitrogen. Remove the solution from the source of heat, cool to 20°C., again add 0.5 gram of sodium bismuthate, and stir. When the maximum permanganate color has developed, filter through an alundum or Gooch crucible containing an asbestos mat which has been ignited, treated with a solution of potassium permanganate and washed with distilled water. Wash the precipitate with dilute sulphuric acid until the washings are colorless. Transfer the filtrate to a colorimeter tube and compare the color of it with that of standards prepared from the potassium permanganate solution. To prepare the standards, dilute with sulphuric acid, (c), portions of 0.2, 0.4, 0.6 cc., etc., of the permanganate solution to the same volume as the filtrate.

BARIUM.

REAGENTS.

(a) *Ammonium dichromate solution.*—One hundred grams to the liter.

(b) *Ammonium acetate solution.*—Three hundred grams, neutralized by ammonia, to the liter.

(c) *Ammonium acetate solution.*—Twenty cubic centimeters of (b) diluted, to 1 liter. Reaction of acetate solution should be alkaline rather than acid.

DETERMINATION.

By weighing as barium chromate.

Acidify and concentrate the sample as usual. Add about 0.5 gram of ammonium chlorid and precipitate the iron and aluminium with ammonium hydroxid. Boil, filter and wash. To the filtrate add ammonium acetate [10 cc. of (b)] in excess (volume about 200 cc.). Heat to boiling and add, with stirring, about 5 cc. of the dichromate solution, (a). Allow to settle and cool. Decant the clear liquid through a filter, wash the precipitate by decantation with ammonium acetate, (c), until the filtrate is no longer perceptibly colored, which will require about 100 cc. of wash solution. Place the beaker under the funnel, dissolve the precipitate on the paper with warm dilute nitric acid, using as little as possible, and wash the paper. Add a little more acid to dissolve the precipitate in the beaker, follow with ammonia until the precipitate forming again no longer redissolves. Heat to boiling, add, with stirring, ammonium acetate [10 cc. of (b)], and ammonium dichromate solution [2 cc. of (a)], allow to cool slowly and wash the precipitate by decantation with (c). Dry the barium chromate, burn the filter separately, ignite moderately to constant weight. Weigh as $BaCrO_4$.

By titration.

Proceed as in the method just described to "* * * wash the precipitate by decantation with (c)", then proceed as follows:

Dissolve the precipitate in about 10 cc. of a mixture of hydrochloric acid (1 to 1) and hot water. Wash the filter and dilute the solution to about 400 cc.; add about 50 cc. of a freshly prepared 10% solution of potassium iodid. Mix carefully and titrate the liberated iodin after 3–4 minutes with standard thiosulphate (1 cc. of N/10 thiosulphate = 4.579 mg. of barium).

TABLE 1.

Composition of samples sent to collaborators.

CONSTITUENTS	CALCIUM AND MAGNESIUM SOLUTION (25 cc.)*	BARIUM SOLUTION (25 cc.)*	MANGANESE SOLUTION (10 cc.)†
	mg.	*mg.*	*mg.*
Calcium	80.0	61.2	6.1
Magnesium	56.0	214.2	17.1
Sodium	86.1	247.2	4.0
Strontium	...	50.8	...
Manganese	...	....	1.16
Barium	...	97.0	...

* Bases present in the form of chlorids.

† Manganese present in the form of sulphate; other bases present in the form of chlorids.

TABLE 2.

Results of cooperative work on water.

ANALYST	CALCIUM IN 25 CC.	MAGNESIUM IN 25 CC.	BARIUM IN 25 CC. (PRECIPITATE IGNITED)	MANGANESE IN 10 CC.
	mg.	*mg.*	*mg.*	*mg.*
J. C. Diggs, State Board of Health, Indianapolis, Ind.	84.3	35.5	92.7	1.3
	81.5	35.2	95.4	1.4
	84.2	36.0	91.6	...
	...	...	95.0	...
L. R. Taylor, State Water Survey Division, Urbana, Ill.	...	...	96.6	1.1
	...	...	96.9	...
	...	...	96.8	...
	...	...	96.8	...
Dearborn Chemical Company, Chicago, Ill.	79.4	55.1	97.2	...
	79.1	55.0	97.4	...
	79.3	54.3	97.3	...
W. F. Baughman, Bureau of Chemistry, Washington, D. C.	80.8	56.7	96.1	1.10
	80.8	57.0	95.8	1.05
	80.8	56.4	...	1.15
F. B. Furber, Bureau of Chemistry, Washington, D. C.	81.7	57.3	96.5	1.2
	81.7	57.3	96.8	1.2
	81.7	...	...	...
J. W. Sale, Bureau of Chemistry, Washington, D. C.	81.4	56.3	96.4	1.18
	81.4	58.1	96.9	1.10
	82.2	57.3	..	...
Maximum	84.3	58.1	97.4	1.4
Minimum	79.1	35.2	91.6	1.05
Average	81.3	52.0	96.0	1.20
Theory	80.0	56.0	97.0	1.16

In considering the results on calcium and magnesium, it should be borne in mind that this method was selected as a rapid volumetric method for use in examining waters for industrial purposes and as a possible substitute for the so-called soap method. Extreme accuracy in this method may be sacrificed if rapidity and reasonable accuracy are attained. The comments of the collaborators, with one exception, indicate that the method is worthy of consideration and that the results submitted, with the exception of one set of magnesium determinations, are sufficiently close to theory, considering the field in which the method is to be used. The remarkably low results on magnesium in the one instance may have been caused by adding an insufficient amount of ammonium arsenate reagent to precipitate all of the magnesium, by a too prolonged washing of the mixed precipitate, or by using an incorrect value for the standard thiosulphate solution. If the low results are due to either of the first two reasons suggested, the method could be changed by stating the minimum amount of arsenate reagent to be added, say 2–3 grams, as suggested by one of the collaborators, and by cautioning against prolonged washing of the mixed precipitate. In the referee's opinion, the indefiniteness of the end point in titrating the iodin with standard thiosulphate is the fundamental criticism to be made of the method. If this objection can not be overcome, the method should not be adopted. However, it is believed that very fair results can be obtained if the directions given regarding the titration are closely followed. With regard to the use of Iceland spar, the following directions were forwarded to the collaborators on July 17, 1917:

> It appears that Iceland spar is not always available for the standardization of the potassium permanganate solution in the determination of calcium. Furthermore, it has been found to contain impurities which may vitiate the results. It is suggested, therefore, that the phrase "or some suitable compound of calcium" be inserted in the third line under "Reagents (a)" after "* * * pure Iceland spar".

The referee does not feel warranted, in view of the data at hand, in recommending the adoption of this method until further cooperative work has been done upon it. It is suggested, however, that it be revised as indicated above and that the referee for 1918 be instructed to give it further consideration.

The methods for barium and for manganese tested this year, unlike the volumetric method for calcium and magnesium, are old, well-tested methods, in general use. The results on barium obtained by igniting the precipitate are generally slightly low, due probably to reduction of the precipitate on ignition and to the solubility of the barium chromate precipitate in the wash water. The following results, obtained by titrating the precipitate, were sent in by the collaborators:

COLLABORATOR	BARIUM IN 25 CC.
	mg.
W. F. Baughman	96.1 95.8
J. W. Sale	97.1 97.7

When it is considered that some of the analysts used this method for the first time, the results obtained are quite satisfactory.

The results on manganese by the well-known sodium bismuthate method are very satisfactory. It seems probable that even better results would have been obtained if the portions taken for analysis had contained less manganese. A milligram of manganese, in the form of potassium permanganate in 50 cc. of solution, gives a color that is too deep for the most accurate comparison with standards. The referee has had considerable experience with this method and personally prefers it to the persulphate method. It is recommended that this method be adopted by the association as an additional official method for manganese with the following change:

> "* * * filter through an alundum or Gooch crucible containing an asbestos mat which has been ignited, treated with a solution of potassium permanganate and washed with distilled water," instead of "* * * filter through an alundum or Gooch crucible containing an asbestos mat ignited and washed with potassium permanganate."

When ammonia is determined on a sample of water containing sulphids, it is necessary to modify the official method. Otherwise, the distillate will turn cloudy or black when nesslerized. In some laboratories, the sample of water containing hydrogen sulphid is acidified, freed from hydrogen sulphid by distillation, made alkaline and the ammonia determined as usual[1]. In other laboratories, the sulphids are precipitated by lead acetate, cadmium chlorid or another reagent and the ammonia determined either with or without the filtering off of the precipitate.

Since it is the general policy of the association to accept only those methods which it has tested, it seemed to the referee worth while to run a series of determinations comparing the different methods of determining ammonia in water containing sulphids before recommending a modification of the official method. Accordingly, such tests were made. R. H. Kellner, formerly of the Bureau of Chemistry, conducted the analytical work under the direction of the referee. The results obtained were as follows:

TABLE 3.

Determination of nitrogen as free ammonia in water containing sulphid.

TEST NUMBER	SODIUM CARBONATE 5N ADDED	SULPHURIC ACID N/5 ADDED	10 PER CENT SOLUTION OF CADMIUM CHLORID ADDED	10 PER CENT SOLUTION OF LEAD ACETATE ADDED	10 PER CENT SOLUTION OF COPPER SULPHATE ADDED	TREATMENT	HYDROGEN SULPHID ADDED	NITROGEN AS FREE AMMONIA	
								Added	Obtained
	cc.	*cc.*	*cc.*	*cc.*	*cc.*		*mg.*	*mg.*	*mg.*
1	5	..	...	...	...	Distilled.............	25.0	0.078	Cloudy
2	5	..	...	...	...	Distilled.............	323.5	0.078	Black precipitate
3	5	10	...	...	...	Acid solution aerated, made alkaline and distilled.	1.1	0.041	0.041
4	5	10	...	...	...	Acid solution aerated, made alkaline and distilled.	0.9	0.036	0.034
5	5	10	...	...	...	Acid solution aerated, made alkaline and distilled.	4.4	0.148	0.148

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[1] *J. Am. Chem. Soc.*, 1910, 32: 1256.

TABLE 3.—Continued.

Determination of nitrogen as free ammonia in water containing sulphid.

TEST NUMBER	SODIUM CARBONATE 5N ADDED	SULPHURIC ACID N/5 ADDED	10 PER CENT SOLUTION OF CADMIUM CHLORID ADDED	10 PER CENT SOLUTION OF LEAD ACETATE ADDED	10 PER CENT SOLUTION OF COPPER SULPHATE ADDED	TREATMENT	HYDROGEN SULPHID ADDED	NITROGEN AS FREE AMMONIA	
								Added	Obtained
	cc.	cc.	cc.	cc.	cc.		mg.	mg.	mg.
6	5	10	...	...	...	Acid solution distilled, made alkaline and redistilled.	0.9	0.044	0.042
7	5	10	...	...	...	Acid solution distilled, made alkaline and redistilled.	4.4	0.156	0.141
8	6	25	...	...	...	Acid solution distilled, make alkaline and redistilled.	323.5	0.086	0.081
9	6	25	...	...	...	Acid solution distilled, made alkaline and redistilled.	50.0	0.086	0.092
10	6	25	...	...	...	Acid solution distilled, made alkaline and redistilled.	25.0	0.086	0.092
11	6	25	...	...	...	Acid solution distilled, made alkaline and redistilled.	25.0	0.086	0.095
12	6	25	...	...	...	Acid solution distilled, made alkaline and redistilled.	280.0	0.116	0.120
13	6	25	...	...	...	Acid solution distilled, made alkaline and redistilled.	280.0	0.116	0.113
14	5	..	1.0	...	...	Distilled without filtering.	10.6	0.551	0.493
15	5	..	0.1	...	...	Distilled without filtering.	1.1	0.067	0.069
16	5	..	25.0	...	...	Distilled without filtering.	25.0	0.078	0.076
17	5	..	25.0	...	...	Distilled without filtering.	25.0	0.078	0.078
18	5	..	25.0	...	...	Distilled without filtering.	280.0	0.108	0.121
19	5	..	25.0	...	...	Distilled without filtering.	280.0	0.108	0.112
20	5	..	...	0.1	...	Distilled without filtering.	0.9	0.037	0.057
21	5	..	...	1.0	...	Distilled without filtering.	9.3	0.294	0.278
22	5	..	...	0.1	...	Filtered and distilled....	0.9	0.037	0.221
23	5	..	...	1.0	...	Filtered and distilled....	9.3	0.294	0.421
24	5	..	...	40.0	...	Distilled without filtering.	25.0	0.078	0.088
25	5	..	...	40.0	...	Distilled without filtering.	25.0	0.078	0.096
26	5	..	...	40.0	...	Distilled without filtering.	280.0	0.108	0.141
27	5	..	...	40.0	...	Distilled without filtering.	280.0	0.108	0.136
28	5	..	...	37.0	...	Distilled without filtering.	323.0	0.078	0.071
29	5	..	...	...	15	Distilled without filtering.	280.0	0.108	0.123
30	5	..	...	...	15	Distilled without filtering.	280.0	0.108	0.118

In each of these tests 500 cc. of boiled Washington city tap water, containing known quantities of free ammonia, were used. Either the free ammonia in the reagents was determined, and thus accounted for, or the reagents were rendered ammonia-free. The usual precautions of freeing the still of ammonia and of conducting the distillations in a room free from ammonia fumes were taken. The content of ammonia was obtained largely by adding a standard solution of ammonium chlorid to the sample.

Tests 1 and 2 show the necessity of some modification of the official method for samples containing sulphid. Tests 22 and 23 show that erroneous results may be obtained by filtering, even though the filter is carefully washed before use with ammonia-free water, as was done in these tests.

Tests 3 and 4 give satisfactory results, but the time consumed in aerating (1 to 2 hours) makes the method unduly tedious.

Tests 6 to 13, in which the acid solution was distilled, then made alkaline and redistilled, are satisfactory in every way. It was found that a 20 minute distillation sufficed to free the sample of sulphid. It may be mentioned that efforts to recover quantitatively the hydrogen sulphid from the distillate were fruitless.

Adding cadmium chlorid to the sample, then distilling without filtering, gave almost as accurate results and was less trouble than distilling off the hydrogen sulphid in the acid solution.

The use of lead acetate or copper sulphate, however, did not give quite such good results as did the other procedures.

It is believed that additional study should be given to the determination of free and albuminoid ammonia in water containing sulphid, particularly as to the effect of the reagent used on the quantity of albuminoid ammonia obtained, before any particular modification of the official method is recommended.

RECOMMENDATIONS.

It is recommended—

(1) That the method for the determination of barium, page 86, be adopted as official. (First presentation of the method for action.)

(2) That the method for the determination of manganese, page 85, be adopted as an additional official method. (First presentation of the method for action.)

(3) That further study be given to the rapid method for the determination of calcium and magnesium in industrial water, page 84.

(4) That further study be given to the determination of free and albuminoid ammonia in water containing sulphids, with a view to modifying the official method in this respect.

(5) That the referee for 1918 be instructed to continue the work on water along the lines suggested in this report, giving particular attention to the selection of methods for determining lead, copper, zinc and tin in waters, and to the calculation of the milligram equivalents of the radicals found in water with a view to their use in the interpretation of water analyses.

(6) That the methods listed below, recommended in 1916[1], for adoption as official, be adopted as official this year. (Second presentation of the methods for action.)

(a) Method for the determination of lithium, potassium and sodium[1].

(b) Method for turbidity, (**1**) and (**2**)[2].

(c) Method for color, **3** and **4**[2].

(d) Method for odor, **5**[2].

(e) The Schulze-Trommsdorf method for the determination of required oxygen, **22** and **23**[3].

(f) Method I and Method II for dissolved oxygen, **24**, **25**, **26**, **27**, **28** and **29**[4].

(g) Method for the determination of specific gravity, **30**[5].

(h) Method for the determination of hydrogen sulphid, **37**[5].

(i) Method for temporary hardness, **70**[6].

(j) Method for alkalinity, **71**, **72**, **73** and **74**[6].

(k) Method for total hardness, **75** and **76**[7].

(l) Method for permanent or non-carbonate hardness, **77**[7].

(7) That consideration be given to the Gutzeit method for the determination of arsenic with a view to having it printed in the methods for the analysis of water (as an additional official method). (Second presentation of the method for action.)

(8) That the official reduction method for the determination of nitrogen in the form of nitrate[8] be revised to read as follows:

NITRATE NITROGEN.

Reduction Method.

(For water of high chlorin content.)

REAGENTS.

(**a**) *Sodium or potassium hydroxid solution.*—Dissolve 250 grams of the purest hydroxid obtainable in 1250 cc. of distilled water. Add 2–3 strips of aluminium foil, (**b**), let stand about 12 hours. Concentrate the solution to 1 liter by boiling.

(**b**) *Aluminium foil.*—This reagent should be the purest obtainable. Cut into strips about 10 cm. long, weighing about 0.5 gram.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, 3: 522.

[2] *Assoc. Official Agr. Chemists, Methods*, 1916, 35.

[3] *Ibid.*, 39.

[4] *Ibid.*, 40-1.

[5] *Ibid.*, 41.

[6] *Ibid.*, 50.

[7] *Ibid.*, 51.

[8] *Ibid.*, 38.

DETERMINATION.

Place 100 cc. of the sample, or such a quantity as contains 0.1 mg. or less of nitrogen in the form of nitrate, in a 300 cc. casserole. Add 2 cc. of sodium hydroxid solution, (a). Concentrate by boiling to about one-third the original volume. Transfer to a 100 cc. test tube, using nitrogen-free water, diluting if necessary to a volume of about 75 cc. Prepare a blank (preferably several blanks, since the nitrogen impurity in aluminium is often distributed unevenly) by placing about 75 cc. of nitrogen-free water and 2 cc. of sodium hydroxid solution, (a), in a 100 cc. test tube. Place a strip of aluminium foil in each tube. Close the mouths of the test tubes with rubber stoppers connected by means of bent glass tubes with other test tubes containing about 50 cc. of slightly acidified ammonia-free water. These latter tubes serve as traps to prevent the escape of ammonia and at the same time permit free evolution of hydrogen. Allow the sample and blank to stand at room temperature for 12 hours or until reduction is complete, which is usually within 4 hours. Nesslerize the traps. If they show more than a few hundredths of a mg. of ammonia, the sample has probably frothed over, and the determination should be discarded. If the traps contain the equivalent of only 1 or 2 cc. of standard ammonia solution each, they should be disregarded.

Transfer the sample and blank to a distillation flask, using 250 cc. of ammonia-free water, distil, nesslerize and compare with standards, as in the determination of free ammonia.

Subtract the quantity of nitrogen found in the blank from that found in the sample. Calculate to mg. per liter of nitrate nitrogen (*N*).

No report on soils was made by the referee.

TECHNIQUE OF DETERMINATION OF SOIL PHOSPHORUS[1].

By H. A. Noyes[2] (Agricultural Experiment Station,
La Fayette, Ind.).

Considerable trouble was experienced in this laboratory previous to 1914 in obtaining reliable phosphorus determinations in samples of a silty clay loam. The official method[3], which gives the phosphorus soluble in hydrochloric acid (specific gravity 1.115), did not prove satisfactory. The sodium peroxid fusion method was not adopted because of irregularities in the character of the solutions made up from the same and different soils. The fusion methods used in metallurgical work were not chosen because of the many evaporations and dehydrations that are necessary in order to get rid of soluble silica. The amount of phosphorus that would not be dissolved out of ordinary soil, due to the occlusion of the phosphates by the silica as pointed out by Fry[4], is believed to be less than the errors made in carrying out fusion methods.

The method of Goss[5], with the modifications given here, has proved

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[1] Presented by C. B. Lipman.

[2] Present address, Mellon Institute of Industrial Research, Pittsburgh, Pa.

[3] *Assoc. Official Agr. Chemists, Methods*, 1916, 24.

[4] *J. Ind. Eng. Chem.*, 1913, 5: 664.

[5] H. W. Wiley. Principles and Practices of Agricultural Analysis. 2nd ed., 1906, 1: 465.

entirely satisfactory for the determination of the phosphorus variations of soils under investigation in this laboratory. The articles by Robinson[1], Rost[2], and Peters[3] have led to the belief that other workers might be interested in these modifications.

METHOD.

Place 10 grams (except for muck and peats) of the prepared air-dry sample in a 250 cc. graduated Kjeldahl flask, add 0.7 gram of mercuric oxid and digest by the regular Kjeldahl method, as for total nitrogen. Add a crystal (about 0.5 gram) of pure sodium nitrate or potassium nitrate to complete the oxidation. When it is partially cooled, add about 200 cc. of water, and when cooled to room temperature, make to volume.

The solution is filtered, through a good grade of folded filter, as follows: Shake the Kjeldahl flask and pour its entire contents on the filter. Pour back the filtrate until it comes through clear. The solid material settles down and soon prevents everything but the clear solution from passing through. Pipette out 25 cc. of the solution into a 250 cc. beaker, and add 15 grams of dry ammonium nitrate[4]. Heat the solution to boiling, stirring to insure solution of the nitrate. Add, with constant stirring, approximately 30 cc. of ammonium molybdate solution. Place the beaker in a water bath at 60–65°C. for 1 hour. Proceed from this point according to the official method[5].

The objects sought and attained by this technique are:

(1) The use of a representative amount of the soil sample.

(2) Uniformity of treatment of all samples not attained in fusion methods.

(3) A single speedy operation to remove the organic matter and prepare the solution.

(4) Conditions for the precipitation of the ammonium phosphomolybdate which may be duplicated.

The concentrations of the solutions used and the addition of the dry ammonium nitrate without preliminary neutralization always yield a clean yellow precipitate of ammonium phosphomolybdate.

Acknowledgment is made to S. D. Conner (Agricultural Experiment Station, La Fayette, Ind.) for cooperation in the testing out of this technique.

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[1] *J. Ind. Eng. Chem.*, 1916, **8**: 148.

[2] *Soil Science*, 1917, **4**: 295.

[3] *J. Ind. Eng. Chem.*, 1915, **7**: 39.

[4] *J. Assoc. Official Agr. Chemists*, 1917, **3**: 149.

[5] *Assoc. Official Agr. Chemists, Methods*, 1916, 2.

DETERMINATION OF MOISTURE IN FIELD SAMPLES OF SOIL[1].

By H. A. Noyes[2] and J. F. Trost (Agricultural Experiment Station, La Fayette, Ind.).

The amount of field soil to be used for making moisture determinations has been left largely to the individual analyst. It is obvious that differences in the structure of soils must bear a relation to the amount of sorting which will occur in the taking and transferring of specific amounts of soil for the determination of moisture.

Four different soils were studied—a fine gravel, a fine sand (Wabash sandy loam), a loam (Sioux silt loam), and a black sand, high in organic matter. The structural character of these soils when air-dry is brought out in Table 1.

Table 1.

Sieve mechanical analysis of soils.

SOIL	SIZE OF SOIL PARTICLES IN MILLIMETERS								TOTAL
	Over 5	Less than 5 over 3	Less than 3 over 2	Less than 2 over 1.5	Less than 1.5 over 1.0	Less than 1.0 over 0.75	Less than 0.75 over 0.5	Less than 0.5	
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
Gravel...	20.2	16.3	12.8	13.0	10.3	6.6	12.2	8.2	99.6
Sand.....	0.0	0.1	0.1	0.1	0.3	0.3	39.5	59.4	99.8
Loam....	4.0	5.5	5.7	8.5	12.7	8.7	21.5	32.5	99.1
Black sand.	0.0	0.0	0.5	7.4	12.2	14.5	36.7	28.0	99.3

The table shows that the relative proportions of the different sized particles varied considerably in the different samples.

Moisture determinations were made on each of these soils when they contained little more than hygroscopic moisture and also when they contained the moisture content found under average field conditions in early spring. A sample of the loam containing an intermediate amount of moisture was also analyzed. The samples were surface soil and were brought to the laboratory in sealed Mason jars. The jars were kept sealed except during the time that portions of the sample were being taken from them.

The average moisture content of the four soils under study was de-

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[1] Presented by C. B. Lipman.

[2] Present address, Mellon Institute of Industrial Research, Pittsburgh, Pa.

termined as follows: The cover was removed from the jar, the spatula inserted and the soil mixed. By means of the spatula, the approximate weight of soil desired was transferred to a weighed dish and the jar was resealed. Triplicate 2, 5, 7, 10, 15 and 20 gram portions of each sample were weighed quickly but accurately to milligrams. The samples were dried to constant weight in a drying oven regulated to run at 102°C. ± 2°. The average moisture content shown by the triplicate determinations is given in Table 2.

TABLE 2.

Effect of size of sample on moisture determination.

SOIL	WEIGHTS OF PORTIONS IN GRAMS							AVERAGE	VARIATIONS
	2	5	7	10	15	20	25		
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	
Gravel F*............	6.13	5.65	5.40	5.78	5.85	5.45	5.90	5.74	0.73
Gravel A†............	1.90	1.97	1.81	1.91	1.96	1.87	....	1.90	0.16
Sand F...............	12.87	12.88	13.24	13.53	13.92	13.16	....	13.27	1.05
Sand A...............	1.03	1.13	1.17	1.19	1.20	1.19	....	1.15	0.17
Loam F...............	20.76	22.03	21.00	21.48	21.56	21.78	....	21.43	1.27
Loam F...............	10.23	10.35	10.33	10.31	10.75	10.87	....	10.47	0.64
Loam A...............	2.87	3.13	3.14	3.25	3.38	3.40	....	3.19	0.53
Black sand F..........	30.73	31.52	31.46	32.12	32.05	32.21	....	31.68	1.48
Black sand A..........	1.43	1.65	2.10	2.16	2.37	2.47	....	2.03	1.04

* Denotes field samples.
† Denotes air-dry samples.

Table 2 shows the following:

(1) That the moisture content determined on different weights is not the same.

(2) That the average of all determinations made bears different relations to determinations made on the same weights of the different soils.

(3) That the amount of moisture in the different samples affected the results obtained.

Table 3 gives the variations between the triplicate determinations that were averaged to get the results reported in Table 2.

The following factors require that the weight of field soil taken for moisture determinations be large: (a) high moisture content; (b) variable proportions of coarse to fine material; (c) tendency of the soil particles to sort out; and (d) change in moisture during weighing (personal factor).

It was found advisable to ascertain, by testing, the weights of differ-

TABLE 3.

Effect of size of samples on variations between triplicate moisture determinations.

SOIL	WEIGHTS OF PORTIONS IN GRAMS								
	2	5	7	10	15	20	25	30	40
Gravel F*	1.49	1.41	0.45	0.20	0.47	0.38	0.65	0.47	0.09
Gravel A†	0.45	0.36	0.40	0.13	0.30	0.12	....	....	....
Sand F	1.19	0.52	0.35	0.31	0.15	0.20	....	....	....
Sand A	0.05	0.10	0.07	0.05	0.01	0.01	....	....	....
Loam F	1.43	1.13	0.19	0.82	0.37	0.25	....	....	....
Loam F	0.35	0.16	0.19	0.27	0.06	0.03	....	....	....
Loam A	0.05	0.07	0.08	0.04	0.09	0.05	....	....	....
Black sand F	0.35	0.07	0.68	0.43	0.28	0.11	....	....	....
Black sand A	0.40	0.31	0.30	0.05	0.12	0.08	....	....	....
Average	0.64	0.46	0.30	0.26	0.21	0.14	....	....	....

* Denotes field samples.
† Denotes air-dry samples.

ent types of soil that must be taken to have the moisture results agree to 0.1 per cent. This gives the relation between factor (d) and the others. It was noted that, although different individuals may not decide on the same weight of soil, due to a difference in individual working errors, they obtain the same moisture results when each one uses an amount large enough to make his duplicates check well.

SUMMARY.

(1) A quantity of field soil weighing less than 10 grams was found to be unsatisfactory for the accurate determination of the moisture present in a soil.

(2) The weight of a particular soil necessary for an accurate moisture determination depends on the soil, the amount of moisture present in it, and the technique of the person making the analysis.

(3) With all kinds of soil, the optimum amount required varies with the moisture content and physical condition of the soil, and therefore it is necessary to determine the weight of soil which the average analyst should use.

J. B. Rather[1] (Agricultural Experiment Station, Fayetteville, Ark.), submitted a paper on "An Accurate Loss-on-Ignition Method for the Determination of Organic Matter in Soils"[2].

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[1] Present address, Standard Oil Company, Chemical Laboratory, Brooklyn, N. Y.
[2] Ark. Agr. Expt. Sta. Bull. 140: (1917); J. Ind. Eng. Chem., 1918, 10: 439.

C. R. Wagner and W. H. Ross (Bureau of Soils, Washington, D. C.), presented by title a paper on "A Modified Method for the Determination of Fluorin, with Special Application to the Analysis of Phosphates"[1].

A STUDY IN SOIL SAMPLING.

By WILLIAM FREAR and E. S. ERB (Agricultural Experiment Station, State College, Pa.).

The official directions for soil analysis omit all mention of the method of soil sampling, and manuals for the guidance of soil analysts are reticent upon the procedure necessary to secure a sample. Reports of soil examinations discuss as significant small differences in composition, but furnish no data to establish within what limits analytical results vary for different portions of the same soil solution or for different samples of the same soil. In fact, the literature furnishes few data for guidance, and an inquiry, very limited in scope, showed that even those investigators who are directing extensive soil studies, involving large expenditure of time and money, are employing sampling methods quite different in detail.

The writers made a study which, it was hoped, would give information as to the precautions necessary to obtain representative samples of the surface soil upon which the general fertilizer series of plats of the Pennsylvania Agricultural Experiment Station are located. The soil is not of a single type, but is chiefly Hagerstown silty clay loam. The problem was, however, to represent the plats studied, not the individual soil types or subtypes. Earlier studies[2] have shown a marked lack of uniformity as to chemical composition in the soil adjacent to these plats.

Concretely, the question was how many subsamples must be taken from well and symmetrically distributed points over the respective plats in order that duplicate composites from the same one-eighth acre plat may agree satisfactorily with respect to the point of composition in question.

The plats sampled were Nos. 1 and 4, Tier II, of the series above-named.

The samples were taken in July and August, 1916, in three sets: I and II, by Erb and Kern, for one study; III, by G. J. Kuhlman, for another study. The sets differed in the following respects:

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[1] *J. Ind. Eng. Chem.*, 1917, 9: 1116.

[2] Annual Report of the Pennsylvania State College, 1908–1909, 215; 1909–1910, 163; 1910–1911, 313.

Set I.—Taken by excavation of holes 9 x 4 inches to subsoil (3–8 inches; average, $6\frac{1}{2}$ inches). Excavations, 11 to the series, taken in zigzag lines crossing the plat from side to side, beginning at the Northwest end and concluding at the Southwest end. Series A includes the excavations begun at the West corner of Plat I; Series B, those beginning at the North corner of that plat; Series C and D, the corresponding excavations for Plat IV. The subsamples from the excavations were weighed, air-dried, weighed and prepared separately for analysis, but only the corresponding fine soils (passing a sieve of $\frac{1}{20}$ inch mesh) were used in aliquot portions to make up series composites for analysis. The individual subsamples weighed from 2.25–5.75 kg. each, after air-drying.

Set II.—Taken by borings with a $\frac{7}{8}$ inch soil auger, using only the surface soil thus removed from each boring. The series in this set were lettered E to H, and correspond in distribution to the respective series, A to D, above described. Each series represents 40 borings, which were composited as they were taken. Weights of composites, air-dry, 4.5–4.9 kg.

Set III.—Taken and composited in the same manner as Set II, but distributed along the lines of an X over the respective plats, 64 borings to the plat. Series I includes the borings from Plat I; Series J, those from Plat IV.

All the results are based upon the air-dry soils, whose hygroscopic moisture content ranged from 0.72 to 0.95 per cent. The difference would affect only the acre weight determinations and these in amounts within the limits of error of the determinations.

ACRE-7 INCH WEIGHTS.

The acre-7 inch weights, calculated from the results obtained by the excavation method, with the correction described in the following paper[1], afford the following data. The computations were based upon apparent specific gravities for soils to the average depth of $6\frac{1}{2}$ inches. A slight, but nearly uniform error is thus introduced.

TABLE 1.

Surface soil acre-7 inch weight (corrected).

PLAT	SERIES	SERIES AVERAGES	DIFFERENCE	PLAT AVERAGES
		pounds	*pounds*	*pounds*
I	A	2,082,030.9	....	....
I	B	2,101,293.2	19,262.3	2,091,662.0
IV	C	2,025,964.7	....	....
IV	D	2,046,923.5	20,958.0	2,036,449.1
Plat difference				55,212.9

The probable error of these apparent specific gravity determinations, computed by the use of the Gauss formula, rarely affects the results to more than the third decimal place. Expressed in pounds to the acre-7

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[1] *J. Assoc. Official Agr. Chemists*, 1920, 4: 103.

inches, the probable errors of the single series of eleven subsamples each range from ± 9,300 pounds to ± 15,500 pounds, and for the twenty-two determinations to the plat, from ± 8,700 pounds to ± 9,800 pounds. The differences between series were therefore distinctly larger than the probable errors of the determinations. The extreme range of apparent specific gravity found for single subsamples (1.209 to 1.425) emphasizes the importance of the duplication of such determinations.

FRACTIONING THE SAMPLES.

The excavated subsamples were so large that, to save labor, their fractioning or parting before sifting out the fine soil was highly desirable. The accuracy of the usual simple parting method was first tested. The air-dry soils from two subsamples were rolled on an oilcloth fifty times, the fine soil was brushed radially to the central heap, the latter quartered, and each quarter pestled with a rubber pestle and sifted separately. The following results were obtained:

TABLE 2.

Mechanical analyses of subsample quarters.

DESCRIPTION OF SAMPLE	COARSE*	MEDIUM†	FINE‡
Subsample B_1:	*per cent*	*per cent*	*per cent*
First quarter	7.41	7.97	84.52
Second quarter	3.51	2.07	94.42
Third quarter	5.77	7.46	86.73
Fourth quarter	6.37	6.47	87.16
Subsample B_2:			
First quarter	2.99	2.45	94.56
Second quarter	2.38	1.76	95.86
Third quarter	2.05	1.54	96.41
Fourth quarter	3.11	2.67	94.22

* Left on 3 mm. sieve.
† Left on $\frac{1}{20}$ inch sieve.
‡ Passed $\frac{1}{20}$ inch sieve.

This method of mixing evidently failed to secure a uniform distribution of the larger particles through the aliquots. Probably no serious difference in the mechanical groups of the fine soil would have resulted from its use, but the gravel component might be very considerably affected. Since this point was of importance in the present study, the entire amount of each subsample was sifted.

SIFTING RESULTS FOR PLATS I AND IV.

The results from the sifting of the two lots of subsamples follow:

TABLE 3.

Mechanical analyses of the whole sample.

SAMPLE NUMBER AND DESCRIPTION	COARSE* (AVERAGE)	MEDIUM† (AVERAGE)	FINE‡ (AVERAGE)
	per cent	*per cent*	*per cent*
Plat I:			
Series A, excavation	4.45	5.90	89.65
Series B, excavation	4.70	4.18	91.12
Plat I:			
Average of 22 subsamples	4.57	5.04	90.39
Series E, 40 borings	3.42	5.49	91.09
Series F, 40 borings	2.36	3.55	94.09
Plat I:			
Average of 80 borings	2.89	4.52	92.59
Series I, 64 borings	7.03§	...	92.97
Plat IV.			
Series C, excavation	4.71	4.05	91.24
Series D, excavation	3.90	4.16	91.94
Plat IV:			
Average of 22 subsamples	4.31	4.11	91.58
Series G, 40 borings	2.50	4.50	93.00
Series H, 40 borings	3.53	3.51	92.96
Plat IV:			
Average of 80 borings	3.02	4.00	92.98
Series J, 64 borings	7.00§	...	93.00

* Left on 3 mm. sieve.
† Left on $\frac{1}{20}$ inch sieve.
‡ Passed $\frac{1}{20}$ inch sieve.
§ Coarse and fine combined.

COMPOSITION OF FINE SOIL.

Differences of the magnitude reported above must obviously affect the weight of fine soil found for a particular area and depth. Whether the chemical composition also of the fine soil is similarly affected remains to be considered.

The respective fine soils (series composites, in most cases) were separately analyzed, usually in duplicate, with respect to a number of the constituents. The nitrogen and phosphoric acid for all samples, and the loss on ignition for Set III samples, were determined by Walter Thomas; all the other determinations were made by E. S. Erb. The results, together with the year of analysis, are presented in the following table:

TABLE

Composition

(Air-dry basis

DETERMINATION	YEAR	SET I	
		Series A	Series B
		per cent	*per cent*
Hygroscopic moisture	1916	0.894	0.792
	1917	0.950	0.720
Loss on ignition	1916	4.532	4.532
	1917	....	....
Dry matter lost on ignition	1916	3.638	3.740
Nitrogen	1917	0.101	0.1055
Potassium oxid, total (Smith)	1916	3.799	3.918
Potassium oxid, soluble in hot hydrochloric acid (1.115 sp. gr.), 10 hours	1916	0.3687	....
Potassium oxid, soluble in N/5 hydrochloric acid, 40°C., 5 hours	1916	0.0140	0.0136
Phosphoric acid, soluble in N/5 nitric acid, 40°C., 5 hours	1916	0.0020§	....

* Recalculated to 0.95 per cent moisture basis.
† Recalculated to 0.82 per cent moisture basis.

All of these samples represent unusually large amounts of soil taken, an unusually large number of subsamples, and subsamples distributed with care, probably greater than usual; also, no error in parting or quartering the samples is involved in the case of Sets I and II. In contrast with these, each series of Set III represents, on the one hand, a greater number of subsamples than was used for the series of Sets I and II; but, on the other hand, a somewhat different distribution of subsamples and a mode of quartering that the writer's later work has shown to be inadequate for the purpose of a mechanical analysis.

Comparison of the respective series results for Sets I and II, shows that, despite unusual care, the sampling error remains greater than the analytical error. The series averages from the boring samples, Set II, agree as well with one another as do those obtained by excavation;

hence the one method represents the composition as well as the other. The importance of careful distribution of subsamples and preparation of composites is shown by the differences in results for the samples of Sets II and III.

4.

of fine soils.

averages.)

SET II		SET III	SET I		SET II		SET III
Series E	Series F	Series I	Series C	Series D	Series G	Series H	Series J
per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
0.792	0.785	....	0.791	1.088	0.875	0.864	....
0.950	0.950	1.35	0.850	0.820	0.820	0.870	1.50
4.733	4.679	....	4.967	4.867	4.970	4.867	....
....	....	5.666*	....	....	....	....	6.120†
3.941	3.894	4.716*	4.176	3.779	4.150	3.797	5.300†
0.109	0.1075	0.1000*	0.100	0.103	0.109	0.1005	0.1022†
3.891	3.678	....	3.495	3.596	3.496	3.585	....
....	....	....	0.4074	0.4067‡	....	....	....
0.0146	0.0150	....	0.0310	0.0303	0.0295	0.0294	....
0.0018§	....	0.0019§	0.0021§	....	0.0023§	....	0.0031§

‡ Represents average of different sets of heatings.
§ Represents grand composite for the set.

EXCAVATION METHOD FOR DETERMINING THE APPARENT SPECIFIC GRAVITY OF SOILS.

By William Frear and E. S. Erb (Agricultural Experiment Station, State College, Pa.).

Various methods have been proposed and used for determining the apparent specific gravity of soils. They may be divided into two classes: First, those in which, by means of a tubular or prismatic implement with a cutting edge, a block of soil of definite cross-section and depth is separated and removed from the body of the soil; second, those in which the measured block of soil is separated from the surrounding soil by trenching, is shaped to exact dimensions in place, and, with or without coating to preserve its integrity, is cut off at the desired depth and removed for weighing. Many cautionary measures have been given to secure a high order of exactness for the determination.

These methods are open to two serious objections. They are suited only to fairly homogeneous soils of fine texture. If the soil is composed in considerable part of gravel, or if it contains larger fragments of stone, cutting tubes or prisms can not be driven into the soil without disturbing its normal texture, and blocks of correct form and plane sides can not be secured for weighing. Second, these methods are so laborious that they can not well be used for repeated determinations made on the same soil to check against errors arising from variations in its uniformity of texture.

For use in a soil through which flinty fragments, of one or more inches or greater diameter, are irregularly but rather frequently scattered, the writers have therefore adopted a method quite different in principle from those of the classes above-mentioned. By this method, the soil is broken and removed from a roughly measured space and weighed before and after air-drying. The volume of the space from which the soil has been removed is measured by careful determination of the volume of dry sand required exactly to fill it. From the data for soil weight and excavation volume, with any necessary correction for change in the volume of the sand resulting from its transfer from the graduate to the excavation, the apparent specific gravity can be computed.

The following details have been employed in the use of the method:

(1) The soil was examined at a time when it was fairly dry, but sufficiently moist not to crumble too readily.

(2) The surface was cleared of stubble and smoothed by use of a sharp trowel or knife.

(3) By means of a rule and a knife, a rectangle was marked off upon the smoothed surface. For the soils studied, the dimensions 9 x 4 inches were chosen, so that sufficient space for the use of excavating tools might be secured. For coarser soils, larger excavations would be preferable.

(4) Two straight-edged pieces of wood were laid parallel, close to and on opposite sides of the lines marked on the surface, and a vertical cut 2 or 3 inches deep was made by use of a sharp knife or flat trowel. A helper kept the pieces of wood firmly in place during the cutting, and the trowel was withdrawn very carefully to avoid any displacement of the surface soil.

(5) The soil within the excavation was then removed by aid of a narrow trowel and transferred to a receiver. A piece of oil cloth was spread between the edge of the excavation and the receiver so as to catch and preserve any soil particles that might spill. The excavation was continued to approximately the desired depth by the use of a knife or trowel, in such manner as to leave undisturbed such of the larger stone fragments as were firmly fixed in the walls of the excavation, but so as to remove with the soil other fragments that came away loosely from the sides or bottom.

(6) The soil thus removed was promptly air-dried.

(7) The volume of the excavation thus made, was determined by filling it with sand

from a graduated cylinder. The sand was delivered uniformly by pouring from a height of 2 or 3 inches above the surface and along the major axis of the rectangle. From time to time as the filling proceeded, the central ridge of sand, formed in the manner described, was leveled and filled into the corners of the excavation and into the hollows in its sides that were caused by the breaking of stone fragments. This leveling and distribution was accomplished by means of a straight-edge, with which the sand was stroked as gently as possible to avoid unequal compression. Care was taken to deliver the last portions of the sand a very little at a time, so that no excess might in any case be used for the filling.

(8) To reduce as far as possible the error in the measurement of the sand, which was free from all but traces of loam, the liter cylinder was filled each time in precisely the same manner. The sand was delivered into a small funnel, set in the neck of the cylinder. When filled almost to the mark, the sand was leveled by gently rocking the cylinder without jarring it. The last portions of sand required were then allowed to trickle in from the hand.

(9) To secure the highest practicable exactness of measurement, the sand remaining in the large cylinder after the excavation was filled, was transferred in like manner to a 50 cc. cylinder to determine its volume.

(10) Finally, to determine what correction, if any, was necessary for a difference in the space occupied by the sand in the measuring vessel and in the excavation, a standard cylindrical brass half-peck measure (4409 cc.) was repeatedly filled with sand in the same manner in which the soil excavations were filled, and from the liter cylinder employed in the field measurements. The excavations were carried down to subsoil, which was found at depths varying from 3 to 7½ inches. The uncorrected volumes of the excavations ranged from 1658 to 4552 cc., with an average of 3467 cc. The depth of the measure was therefore approximately that of the average excavation. The quantities of sand severally required for 10 fillings of the half-peck occupied on the average 4459 ± 0.77 cc. in the glass graduates; that is, the sand was less compact in the half-peck in the proportion 4409 : 4459 ± 0.77 or as 1000 : 1013. The measures obtained as the volumes of the excavations were therefore divided by the factor 1.013 to correct for the relatively greater compactness of the sand in the measuring cylinder.

In the use of this method, it is necessary to determine the correcting factor corresponding to the filling material used and the conditions of filling maintained in each series of studies. The labor thus required is not great. On the other hand, the excavation can be made and its volume determined in little more than an hour, so that duplications of the determination at various points of the surface in question can easily be made. The importance of such duplication will be discussed in another paper.

NITROGENOUS COMPOUNDS IN SOILS[1].

J. K. PLUMMER (State Department of Agriculture, Raleigh, N. C.), *Associate Referee.*

The work outlined this year has been a continuation of that done by C. B. Lipman[2], the plan of which follows:

PLAN OF WORK.

The problem was to test out on the same material the official Kjeldahl method for nitrogen determination in soils[3]; the official method, as given under "Fertilizers"[4]; and the Hibbard method for nitrogen determination in fertilizers, as modified by C. B. Lipman for soils.

HIBBARD MODIFICATION OF THE GUNNING METHOD.

Place the soil in a 500 cc. or, better, in a 800 cc. long-necked Kjeldahl digestion flask, add 30 cc. of sulphuric acid and approximately 10 grams of a mixture prepared by grinding together and thoroughly mixing 10 parts of potassium sulphate; 1 part of ferrous sulphate; ½ part of copper sulphate. Immediately shake the mixture of acid, salt and soil so that no soil remains untreated by the acid. Then digest it, first with a low flame, and then with a strong flame for 1½–2 hours, depending on the amount of organic matter present. After digestion, dilute the mixture, transfer it to 1 liter copper distillation flasks, and distil into N/10 hydrochloric acid. Titrate the acid in the usual way, using either methyl orange or cochineal as an indicator.

METHODS TO BE TESTED.

(1) Official method under "Soils"[3].
(2) Official method under "Fertilizers"[4].
(3) Hibbard modification of the Gunning Method.

SPECIAL INSTRUCTIONS.

Try all of these methods on each soil as follows:

Ten gram portions of soil (20 cc. of sulphuric acid).

Twenty gram portions of soil (30 cc. of sulphuric acid).

The period of digestion should be 2½ hours in every case. Use methyl orange or cochineal as indicator. Take special care in neutralizing the acid before distillation. Preferably employ Greenbank's lye and make up by dissolving 1 part in 2 of water. Use N/20 hydrochloric acid amd N/20 ammonium hydroxid, or N/20 sulphuric acid and N/20 sodium hydroxid. Report the results in terms of cc. of acid, in mg. of nitrogen, and in per cent of nitrogen in air-dried or water-free soil.

Samples of Durham sandy loam (1 A) and Iredell loam (2 A) were washed free of nitrates. To portions of the original Durham sandy loam 0.02 (1 B) and 0.04 (1 C) per cent nitrogen, as sodium nitrate, was added. Portions of the original Iredell loam were similarly treated and the results appear in the table as 2 B and 2 C. All samples were oven-dried before being sent out. The following table gives the results obtained:

---

[1] Presented by C. B. Lipman.

[2] *J. Assoc. Official Agr. Chemists*, 1920, **3**: 326.

[3] *Assoc. Official Agr. Chemists, Methods*, 1916, 21.

[4] *Ibid.*, 8.

Comparative results of nitrogen determinations.

(Nitrogen expressed as per cent.)

SAMPLE		E. F. BERGER*			GEORGE WIBLE*			P. P. PETERSON†			S. LOMINETZ‡		
Number	Weight	Method			Method			Method			Method		
		1	2	3	1	2	3	1	2	3	1	2	3
	grams	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
1A	10	0.029	0.027	0.030	0.028	0.027	0.028	0.028	0.031	.....	0.017	0.015	0.025
1A	20	0 027	0.025	0 027	.....	.....	.....	0.028	0.032	0.028	0 021	0 025	0 019
1B	10	0.027	0.026	.....	0.027	0.025	0.029	0.022	0.029	.....	0.026	0.033	0.038
1B	20	0.026	0.026	.....	.....	.....	.....	.....	.....	0.025	0 029	.....	0.032
1C	10	0.047	0.059	0.046	0.050	0.059	0.046	0.053	0.059	.....	0.015	0.022	0.025
1C	20	0.047	0.052	0.046	.....	.....	.....	0.063	.....	0.042	0 020	0.026	0 023
2A	10	0.037	0.036	0.037	0.039	0.037	0.039	0.032	0.043	0.037	0.033	0.046	0.039
2A	20	.....	0.037	0.035	.....	.....	.....	0 039	.....	0.034	0.041	0.054	0.040
2B	10	0.034	0.037	0.039	0.039	0.037	0.038	0.039	0.045	.....	0.027	0.041	0.043
2B	20	0.037	0.036	.....	.....	.....	.....	0.039	0.036	0 039	0.032	0.038	0.031
2C	10	0.053	0.061	0.061	0.057	0.058	0.054	0.058	0.063	.....	0.041	0.054	0.048
2C	20	0.052	0.062	0.056	.....	.....	.....	0.057	.....	0.052	0.043	0.068	0.045

* Agricultural Experiment Station, E. Lansing, Mich.
† Agricultural Experiment Station, Moscow, Idaho.
‡ Agricultural Experiment Station, College Station, Texas.

CONCLUSIONS.

After a careful examination of the results obtained by the different analysts, it does not appear wise to offer any recommendation for the adoption of a new method to supplant the present official method. However, the results clearly show that there is little choice between the methods now in vogue for measuring small amounts of nitrogen in soils.

The official method to include nitrates does not recover the nitrogen which has been added in the form of sodium nitrate. Sometimes this method gives higher results than the other modifications tested, and sometimes not so high.

Taking the results as a whole, the Hibbard modification gives about as high figures as either of the other two methods. Considering the ease of manipulation of digestion and distillation, the Hibbard method seems to be preferable. Nitrates should be determined on another sample by either the colorimetric or reduction method.

No consistent difference is apparent whether ten or twenty grams of soil are taken for analysis.

REPORT ON THE LIME REQUIREMENT OF SOILS.

By W. H. MacIntire (Agricultural Experiment Station, Knoxville, Tenn.), *Associate Referee.*

The work outlined upon the problem of lime requirement has been along two lines: (1), an effort to ascertain the conceptions and viewpoints held by those who have given particular attention to the problem with a view to defining the term "Lime Requirement"; (2), a study of one or more representative types of the several procedures advanced.

It is well known to the members of this association that there exists a marked diversity of opinion as to the nature of the phenomenon causing the decomposition of calcium carbonate applied to soil. By some it is held that true, if peculiar, acids occur in soils, the hydrogen ion concentration of which may be determined. Others hold that the decomposition of carbonate is effected through physical absorption of the calcium ion, while a third conception is that the original basic silicates have undergone hydrolysis with subsequent leaching of the hydrolyzed products, thus leaving a complex mixture of what may be considered as true acid silicate salts.

It has been hoped first, to reach an agreement as to the terminology; second, to arrive at some definite conclusion as to what may be demanded of a method and what procedure most nearly fulfills the exactions decided upon and, furthermore, as to whether the procedure should be considered solely as a laboratory measurement of a physical or chemical phenomenon or whether the chemical data should be susceptible of interpretation into field practice. This, of course, involves the question of the possibility of correlating lime absorption measurements with plant response. This again raises the questions of pot studies *v.* field studies; selection of plant indicators; purity; hardness; porosity and solubility; and proper time for, and frequency of, application. Most of these considerations involve both the chemical and biochemical factors direct and indirect, as well as the economics of the problem.

To quote aptly, B. L. Hartwell, in correspondence with the associate referee, writes as follows:

> It seems to me that what we need is a definite criterion of what we are attempting to accomplish, by which we may judge of the merit of the rather confusing number of methods. The acquiring of more data without some definite standard is, I fear, unlikely to mean an advance. * * * It seems to me that we must ask the question—lime requirements for what? For what kind of a crop? And requirements for how long a time, etc.?

Queries as to the advisability of the utilization of pots have developed the fact that by many it is held that this method may be considered as indicative only. It has been observed by some that the mechanical preparation and handling incident to pot studies often produce an effect upon plant growth analogous to that produced by liming, so that some soils which respond to liming under field conditions fail to show a similar response or an equivalent response in pot studies because of the abnormality of the checks.

There also arises a number of considerations under field conditions which make the study of lime requirement an effort somewhat beyond the scope of this association. The amount and nature of initial soil components affected by treatment; the amount of carbon dioxid generated within the soil and the tendency of different soils to vary in the retention of this gas; soil type; plant adaptability; rainfall; and probably most important of all, the time factor, militate against extensive field studies. As a matter of fact, it is held by many that such a move would be a digression from the proper scope of the work of this association. While the possibility of cooperation with some organization such as the American Society of Agronomy has been suggested, it is maintained by some that such a course would not be feasible or advisable.

The economic phases of the problem are, in the final analysis, usually dependent upon local conditions and the recommendations advanced would most probably be based upon field trials rather than upon quantitative laboratory data. On the other hand, it is of great interest in studying soils under laboratory conditions to determine the lime absorption coefficient as a part of the laboratory inventory of a soil's composition or tendency toward reactions of various kinds. It is, however, to be admitted that in some cases indications of a tendency to absorb lime is not conclusive proof of the need of lime under field conditions. The fact that soil will decompose applied calcium carbonate is not positive proof of the lack of an abundance of calcareous silicates which may so readily yield to hydrolysis as to insure a sufficiency of lime to maintain a nutrient soil solution of alkaline reaction, *i. e.*, if calcium bicarbonate solution be considered as alkaline.

It is suggested that, for the present, the determination of the lime requirement of a soil be considered solely as a laboratory procedure, adopted to measure the amount of lime absorbed by a soil under uniform conditions to be set forth in such a method as may be later adopted.

It is further suggested that, as a preface to the statement of the details of the technique which may be later adopted, it should be stated that the procedure is intended and considered solely as a laboratory procedure supplementary to the determination of the chemical composition of the soil and that no correlation with practical or economic usage is intended or implied.

INVESTIGATION OF METHODS.

This work has consisted of studies of representative types of procedures. It was intended that a study of the several methods should be individual rather than comparative. In other words, instead of a quantitative comparision between methods, each method was used as its own criterion by which to judge the subsequent indications obtained by the same method. The plan was as follows:

The lime requirement indication for each soil was determined and the amount of calcium carbonate so indicated was applied in the form of carbon-dioxid-treated precipitated carbonate. The applications were made to the air-dry soil and thoroughly mixed, after which the soils were placed in stoppered bottles and wetted to a good condition. After about 48 hours contact, one set was permitted to dry spontaneously. A second set was kept under anaerobic condition for two weeks, while the third set was kept for a month or more. The dried soils were again thoroughly mixed and the lime requirements obtained.

The methods selected were as follows:

The Jones and Hopkins methods, as representative of the neutral salt solution treatments; the Veitch method; the electrometric procedure advanced by Sharp and Hoagland; the lowering of the freezing point method of G. J. Bouyoucos; and the Tacke. and MacIntire procedures, involving treatment with distilled water and carbonated water solutions of calcium carbonate, respectively; and the hydrogen ion colorimetric method advanced by L. J. Gillespie.

SOILS USED.

The work reported by L. T. Sharp and D. R. Hoagland, G. J. Bouyoucos, L. J. Gillespie and your referee was carried out upon ten soils collected by F. P. Veitch, the previous referee.

The following descriptions of the soils were furnished by Veitch:

L. & P. No. 32347. This sample was obtained from the Pennsylvania Agricultural Experiment Station, tier 2, plat 35. The sample was taken May 29, 1916, and at that time bore a thick, dark colored, well-developed stand of red clover. According to our tests, this soil is decidedly basic, both to phenolphthalein solution and red litmus paper, when allowed to stand 14–18 hours in contact with water. When allowed to stand 2 hours before filtering and making the determination, the soil is acid or neutral to phenolphthalein and very faintly basic to red litmus paper.

L. & P. No. 32348.—This sample is also from the Pennsylvania Agricultural Experiment Station, tier 2, plat 26. The sample was taken May 29, 1916, and, at the time, bore a thick, light-colored but poorly developed stand of red clover. The soil was neutral or faintly basic to phenolphthalein solution when allowed to stand 14–18 hours. It was faintly basic to red litmus paper. When allowed to stand but 2 hours, the soil was acid both to phenolphthalein and red litmus paper.

L. & P. No. 32349.—This sample was from the Pennsylvania Agricultural Experiment Station, tier 2, plat 6. It was taken May 29, 1916, and, at the time, bore a thin stand of red clover. Reaction when allowed to stand 14–18 hours: to phenolphthalein, basic; to red litmus paper, basic; when allowed to stand 2 hours: to phenolphthalein, acid; to red litmus paper, faintly basic.

L. & P. No. 32350.—This sample was from the Pennsylvania Agricultural Experiment Station, tier 2, plat 31. It was taken May 29, 1916. "Practically no red clover." Reaction when allowed to stand overnight: acid to phenolphthalein and red litmus paper.

L. & P. No. 32351.—This sample was from the Pennsylvania Agricultural Experiment Station, tier 2, plat 32. It was taken May 29, 1916. "No clover has grown on this end since 1908. All crops fail and only sheep sorrel, red tops and foxtail grow." Reaction acid to phenolphthalein and red litmus paper.

L. & P. No. 32352.—This sample was from the Rhode Island Agricultural Experiment Station, plat 23. It was taken during the summer of 1916. Reaction to phenolphthalein when allowed to stand 16 hours, acid; to red litmus paper, acid; and to blue litmus paper, acid.

L. & P. No. 32353.—This sample was from the Rhode Island Agricultural Experiment Station, permanent plat 25. It was taken in the summer of 1916. Reaction on standing 16 hours: to phenolphthalein, acid; to red litmus paper, neutral.

L. & P. No. 32355.—This sample was from the Rhode Island Agricultural Experiment Station, permanent plat 29. It was taken in the summer of 1916. Reaction when allowed to stand 14–18 hours: to phenolphthalein, basic; to red litmus paper, basic. Reaction when allowed to stand 2 hours: to phenolphthalein, acid; to litmus paper, faintly basic.

L. & P. No. 32361.—This sample was from W. H. MacIntire, Cornell University. "Surface soil clover field north of red barn, Dunkirk silt loam. Red clover, alsike and timothy. Limed some years ago, quite heavily. Distinctly acid to litmus paper tested moist in field." Reaction when allowed to stand 14–18 hours: to phenolphthalein, basic; to red litmus paper, basic. When allowed to stand 2 hours: to phenolphthalein, basic; to red litmus paper, basic.

L. & P. No. 32819.—This sample was from College Park, Md., uncultivated since 1888, frequently burned over since that time. Vegetation plantain, sedge, hen grass, briers, some vetch. Have never seen clover on it in 20 years. Reaction when allowed to stand 14–18 hours: to phenolphthalein, acid; to red litmus paper, faintly basic.

L. & P. No. 32820.—This sample was from flat land, College Park, Md. Frequently cultivated in the past 20 years but not in the past 3 years. Last put in corn, now overrun with weeds, plantain and briers; no clover. Reaction when allowed to stand 16 hours: to phenolphthalein, acid; to red litmus paper, basic.

As determined by Veitch, five of these soils were acid and five were alkaline to distilled water extractions run as blanks to the Veitch procedure.

The indications obtained in using the other six methods were that each of the ten soils was acid in character. In this connection, it might be stated that it is the firm conviction of Veitch that there exists no need of liming in practice in the case of soils which yield an alkaline distilled water extract. In detail, the reactions were as follows:

TABLE 1.

Qualitative reactions of the soils studied as indicated by the several procedures.

			PROCEDURES FOLLOWED					
L. & P. NO.	REFEREE NO.	TYPE OF SOIL	Veitch	Electrometric	Hydrogen ion concentration colorimetric	Lowering of freezing point	Tacke	MacIntire
32347	1	Very fine sandy loam	Alkaline	Acid	Acid	Acid	Acid	Acid
32348	2	Sandy clay loam	Alkaline	Acid	Acid	Acid	Acid	Acid
32349	3	Very fine sandy loam	Alkaline	Acid	Acid	Acid	Acid	Acid
32350	4	Sandy clay loam	Acid	Acid	Acid	Acid	Acid	Acid
32352	5	Sandy humus loam	Acid	Acid	Acid	Acid	Acid	Acid
32353	6	Very fine sandy humus loam	Acid	Acid	Acid	Acid	Acid	Acid
32355	7	Fine sandy humus loam	Alkaline	Acid	Acid	Acid	Acid	Acid
32361	8	Silt loam	Alkaline	...	Acid	Acid	Acid	Acid
32819	9	Fine sandy loam	Acid	Acid	Acid	Acid	Acid	Acid
32820	10	Sandy loam	Acid	Acid	Acid	Acid	Acid	Acid

HYDROGEN ION CONCENTRATIONS AND ELECTROMETRIC INDICATIONS OF LIME REQUIREMENT.

The ten samples collected by Veitch were sent to Sharp and Hoagland, University of California, Berkeley, Calif., who made separate determinations, the averages of which are given in Table 2. To quote:

TABLE

Hydrogen ion concentration of

L. & P. NO.	HYDROGEN ION CONCENTRATION ORIGINAL SOIL*		PURE CALCIUM CARBONATE ADDED TO 100 GRAMS OF SOIL	HYDROGEN ION CONCENTRATION OF TREATED SOIL AFTER 16 DAYS	
				Kept moist	
	Gram mols per liter	P_H†		Gram mols per liter	P_H
32347	$0.12x10^{-5}$	5.92	0.137	$0.92x10^{-7}$	7.04
32348	$0.27x10^{-5}$	5.57	0.156	$0.11x10^{-6}$	6.96
32349	$0.13x10^{-5}$	5.89	0.064	$1.00x10^{-7}$	7.00
32350	$0.19x10^{-4}$	4.72	0.285	$0.12x10^{-6}$	6.92
32352	$0.33x10^{-4}$	4.49	0.446	$1.00x10^{-7}$	7.00
32353	$0.36x10^{-5}$	5.44	0.267	$0.12x10^{-6}$	6.92
32355	$0.37x10^{-6}$	6.43	0.059	$0.50x10^{-7}$	7.30
32361	$1.00x10^{-7}$	7.00	0.000	$0.11x10^{-6}$	6.96
32819	$0.32x10^{-5}$	5.45	0.096	$0.67x10^{-7}$	7.17
32820	$0.24x10^{-5}$	5.62	0.095	$1.00x10^{-7}$	7.00

* Measured in suspensions of 10 grams of soil to 30 cc. of water.

† Neutral point taken as $0.8x10^{-7}$ P_H 7.10.

The hydrogen ion concentrations were determined in all cases in suspensions of soil in the proportion of 10 grams of soil to 30 cc. of water. The attainment of equilibrium was hastened by the shaking method. * * * The amounts of calcium hydroxid necessary to bring the soils to a neutral reaction were determined independently, as in the case of the hydrogen ion concentrations. Closely agreeing results were obtained and the averages of both are recorded in the table, expressed as grams of pure calcium carbonate necessary to neutralize 100 grams of air-dried soil under the experimental conditions. Titrations were made on 5-gram samples, using a standard calcium hydroxid solution. The time of titration was extended over several days, with shaking at intervals. The equivalent amounts of precipitated calcium carbonate were added to 100-gram portions of the soil and thoroughly mixed. The soils were then brought to an approximate optimum moisture content and again mixed. One-half of the total quantity of each soil was allowed to dry spontaneously; the rest was kept moist. The hydrogen ion concentrations of both sets were determined after 16 days and after 38 days. After 16 days the soils were in practically neutral condition, as shown in the table. At the end of 38 days, the soils were still close to the neutral point, but, in the majority of cases, a slight but distinct increase in the hydrogen ion concentration was noted. * * * In order to determine the quantity of calcium carbonate necessary to neutralize all of the soil acids present, it would be necessary to continue the titration over a longer period of time, or perhaps employ heat.

As a matter of fact, the method of treatment actually carried out, that is, a titration with calcium hydroxid covering a 3-day period, was more intense than any of the other procedures followed. In view of the foregoing findings, Hoagland wrote that he deemed it unnecessary to make determinations of possible residual carbonates. This, however, was done by the writer in studying the Tacke and the MacIntire pro-

2.

untreated and treated soil.

HYDROGEN ION CONCENTRATION OF TREATED SOIL AFTER 16 DAYS		HYDROGEN ION CONCENTRATION OF TREATED SOIL AFTER 38 DAYS			
Dried out		Kept moist		Dried out	
Gram mols per liter	P$_H$	Gram mols per liter	P$_H$	Gram mols per liter	P$_H$
0.82x10^{-7}	7.09	0.18x10^{-6}	6.75	0.82x10^{-7}	7.09
1.00x10^{-7}	7.00	0.21x10^{-6}	6.68	0.11x10^{-6}	6.96
0.12x10^{-6}	6.92	0.22x10^{-6}	6.66	0.11x10^{-6}	6.96
1.00x10^{-7}	7.00	0.41x10^{-6}	6.39	0.14x10^{-6}	6.85
0.14x10^{-6}	6.85	0.36x10^{-6}	6.44	0.31x10^{-6}	6.51
0.21x10^{-6}	6.68	0.22x10^{-6}	6.66	0.18x10^{-6}	6.75
0.73x10^{-7}	7.14	0.22x10^{-6}	6.66	0.15x10^{-6}	6.82
1.00x10^{-7}	7.00	1.00x10^{-7}	7.00	1.00x10^{-7}	7.00
0.82x10^{-7}	7.09	0.28x10^{-7}	7.55	0.12x10^{-6}	6.92
1.00x10^{-7}	7.00	0.22x10^{-6}	6.66	0.11x10^{-6}	6.96

cedures. As will be shown later, there were residual carbonates from the applications of calcium carbonate, as indicated by these two methods. It would seem possible, therefore, that in the hydrogen ion concentration studies there may have been still present part of the applied carbonate but not enough to come so quickly into solution of distilled water as to indicate alkalinity or neutrality.

HYDROGEN ION CONCENTRATION (COLORIMETRIC).

Each of the ten soils was examined as to hydrogen ion concentration by L. J. Gillespie, using the colorimetric method. Each sample was reported as being acid by this test. However, it is intended that the results be considered as qualitative only. The results as given by Gillespie are placed in Table 3.

Table 3.

Hydrogen ion exponents determined colorimetrically.

L. & P. NO.	REFEREE NUMBER	HYDROGENION EXPONENT*
32347	1	5.8
32348	2	5.4
32349	3	5.6
32350	4	4.7
32352	5	4.2
32353	6	5.1
32355	7	5.9
32361	8	6.4
32819	9	5.3
32820	10	5.4

* Exponent of 7 indicates neutrality; greater than 7, alkalinity; less than 7, acidity.

JONES, HOPKINS AND VEITCH PROCEDURES.

In the allotment of work, all three of these methods were assigned to each four laboratories which had voluntarily promised collaborative aid. It is to be regretted, however, that no report has been received from any one of the four laboratories. It is, therefore, impossible to give the indications as to the residual requirements which are to be expected subsequent to treatments as registered by these three methods.

FREEZING POINT METHOD.

In Table 4 are given the results submitted by G. J. Bouyoucos. The averages upon eleven soils studied by Bouyoucos show 2745 pounds of calcium oxid per 2,000,000 pounds of soil as the initial indications, as against 3139 pounds subsequent to intervals of two weeks and two months. This would seem to indicate either a very considerable analytical error or an average increase of over 14 per cent as a result of handling or of handling plus treatment. It would appear that no additions of calcium carbonate were made by Bouyoucos. If such were not the case, the results given would necessarily condemn the method for use in determining the "immediate lime requirement" because of its failure to record any corrective effect or reduction in the lime requirement as a result of applications of appreciable amounts of lime. The associate referee has written Bouyoucos in regard to this point and his reply is here given in toto.

TABLE 4.

Lime requirement determinations by the freezing point method.

L. & P. NO.	REFEREE NUMBER	TYPE	POUNDS OF CALCIUM OXID PER 2,000,000 POUNDS OF SOIL				
			Original	After 2 weeks		After 2 months	
				Dry	Moist	Dry	Moist
32347	1	Very fine sandy loam	3000	3600	3600	3200	2800
32348	2	Sandy clay loam	3000	2800	2400	2700	2200
32349	3	Very fine sandy loam	3000	2800	2400	2700	2200
32350	4	Sandy clay loam	3400	3300	3000	2800	2600
32352	5	Sandy humus loam	2000	2000	2200	2200	2200
32353	6	Very fine sandy humus loam	8000	7700	7700	7700	7200
32355	7	Fine sandy humus loam	4000	3300	3600	3300	3300
32361	8	Silt loam	3000	2200	2000	2200	2000
32819	9	Fine sandy loam	2000	2200	2200	2200	2200
32820	10	Sandy loam	1200	1200	1200	1200	500
32351	11		3700	3500	3400	3300	3000
		Average	3300	3145	3064	3045	2745

MICHIGAN AGRICULTURAL COLLEGE
Department of Soils

EAST LANSING, November 10, 1917.

DR. W. H. MACINTIRE,
Tennessee Agricultural Experiment Station,
Knoxville, Tenn.

Dear Dr. MacIntire:

I hasten to reply to your letter of the 8th instant and to inform you that in procuring the data which I submitted to you on the lime requirements of soils, as indicated by the freezing point method, I followed exactly the procedure which you outlined in your letter of May 31, 1917. In other words, the soils first received the required amount of precipitated calcium carbonate, as indicated by the freezing point method. The mixing of the soil and precipitated carbonate was done before the soil was moistened. Each soil sample was then moistened and afterward separated into two lots. One portion was kept at the optimum moisture content at which the mixing was done. After periods of 2 weeks and of 2 months, the lime requirement of this soil sample was again determined. The other portion of the soil samples was allowed to dry immediately and the lime requirement was determined also at the end of 2 weeks and 2 months.

In your letter of May 31st, you did not state whether the lime requirement of the moist soil should be determined in the moist condition or should be allowed to dry after it stood the required length of time. For uniformity, I allowed the soil to dry in the air and then determined the lime requirement.

It is true that the subsequent lime requirement of the soils was not much affected by the application of the first lime requirement. My further studies of the freezing point method have shown, however, that the soil takes up at once a large amount of lime, and then it continues to take up more lime slowly and gradually for a long time; and the process of air-drying the soil seems to hasten the lime absorption. This continued lime absorption by the soil, which of course you discovered, makes practically all the present lime requirement methods of little use from the practical standpoint. Truog's contentions, that active and latent acidity are definite and determinable quantities, are wrong, in my opinion. For instance, suppose I determine the active acidity of a soil and apply sufficient lime to correct this active acidity. Then, I send Truog a sample of this soil and ask him to ascertain the kind and amount of acidity of this soil. If he does not know that I have already corrected the active acidity, would he not call the acidity he finds active? Whereas, if he knew that I have corrected this already, he would call it latent.

It is very probable that before the soils would refuse to take up any more lime or make a very decided decrease in the subsequent lime requirement, four or five times the original amount of lime would have to be added, at least in some of the soils. It seems that before a soil refuses to take up any more lime, it must be completely satisfied with the lime and that the lime phase should begin to remain in the soil solution. When a soil is brought into contact with an excess amount of lime, it takes up almost instantaneously a large amount of lime and the remainder very slowly. Thus a soil may take up 10,000 pounds of calcium oxid per acre in 2 or 3 minutes and an additional amount of 10,000 pounds in 10 or 12 days. The additional absorption is so slow that it leads one to conclude that the final equilibrium is attained instantaneously. The final equilibrium of absorption, however, is greatly hastened by repeatedly air-drying the soil after it is treated with an excess of lime.

It may be of interest and probably of some use to you in preparing your report to know that in conducting an investigation to determine the rate of reaction between soils and salts, acids and bases, and the behavior of equilibrium, I found that in the case of the bases, which included calcium hydroxid, sodium hydroxid, potassium hydroxid and ammonium hydroxid, the reaction between the soils and the last three bases was almost instantaneous and the equilibrium remained constant for a long time, in many cases, 100 days. In the case of the calcium hydroxid, however, the equilibrium continued to change (or the concentration continued to decrease) slowly and gradually for a long time. According to these results, it would seem that the soils do not continue to take up sodium hydroxid, potassium hydroxid and ammonium hydroxid, but they do continue to take up calcium hydroxid.

Hoping that I have made the matter clear, I am,

Sincerely yours,
GEO. BOUYOUCOS.

TACKE AND MACINTIRE METHODS.

Four methods have been advanced for the determination of lime requirement by the use of calcium carbonate. The Tacke procedure and

TABLE 5.

Lime requirement by the Tacke method upon untreated and treated soils.

SOIL	CARBON DIOXID EVOLVED FROM ORIGINAL SOIL CC. N/20 NORMALITY	CARBON DIOXID EVOLVED SUBSEQUENT TO TREATMENT					
		Dried		Moist 2 weeks		Moist 4 weeks	
		Cc. N/20 normality	Per cent of original	Cc. N/20 normality	Per cent of original	Cc. N/20 normality	Per cent of original
1. Very fine sandy loam	15.50	5.35	34.5	6.60	42.6	6.40	41.3
2. Sandy clay loam....	16.05	9.25	57.6	6.60	41.1	5.70	35.5
3. Very fine sandy loam	13.20	7.05	53.4	5.45	41.3	5.45	41.3
4. Sandy clay loam....	27.90	9.10	32.6	7.00	25.1	5.85	21.0
5. Sandy humus loam .	48.80	17.60	36.1	16.20	33.2	17.05	34.9
6. Very fine sandy humus loam........	36.40	13.05	35.8	13.60	37.3	12.60	34.6
7. Fine sandy humus loam............	27.45	11.70	42.6	10.40	37.9	11.00	40.1
8. Silt loam..........	10.50	6.20	59.0	5.00	47.6	4.65	44.3
9. Fine sandy loam....	13.80	4.70	34.1	5.25	38.0	4.20	30.4
10. Sandy loam........	16.15	9.00	55.7	4.70	29.1	4.20	26.0
Average.........	22.58	9.30	44.1	8.08	37.3	7.71	34.9

the Süchting modification of this procedure involve the reaction which transpires between a distilled water solution of calcium carbonate and an acid soil. The modification is based upon the assumption that the continued evolution of carbon dioxid over long periods of contact between soil and carbonate is due to the action of calcium carbonate upon soil organic matter and seeks to eliminate this objection. In studies upon the Tacke method, the associate referee has found that this continued evolution of carbon dioxid is rather a function of the speed of reaction, which is depressed because of the limited solubility of calcium carbonate. As a matter of fact, clay subsoils almost devoid of organic matter demonstrate this continued evolution in a more marked degree than do many surface soils relatively high in their organic matter content. The Süchting modification is based upon the erroneous assumption that a strong hydrochloric acid solution is less active than a weak calcium carbonate solution upon soil organic matter and that all of the carbon dioxid evolved from the action of acid on soil, plus residual carbonate from that applied, is to be accredited to the carbonate alone. The Tacke method was deemed to be less free of objection and simpler than the Süchting technique and it was accordingly used as more representative of this type or procedure. The other two methods which employ a solution of calcium carbonate are that of Hutchinson and MacLennan which directs the treatment of a soil with a cold bicarbonate

TABLE 6.

Data showing the speed of the reaction involving the evolution of carbon dioxid in the Tacke procedure.

SOIL	CARBON DIOXID EVOLVED							
	During first 3 hours		During second 3 hours		During third 3 hours after standing overnight		Total	
	Cc. N/20 normality	Per cent calcium carbonate	Cc. N/20 normality	Per cent calcium carbonate	Cc. N/20 normality	Per cent calcium carbonate	Cc. N/20 normality	Per cent calcium carbonate
1. Very fine sandy loam..	15.5	0.194	7.0	0.088	8.3	0.104	30.8	0.386
2. Sandy clay loam........	16.05	0.201	7.0	0.088	9.65	0.121	32.7	0.410
3. Very fine sandy loam..	13.2	0.165	3.85	0.048	6.35	0.079	23.4	0.292
4. Sandy clay loam........	27.9	0.349	8.4	0.105	7.7	0.096	44.0	0.550
5. Sandy humus loam........	48.8	0.610	11.1	0.139	10.0	0.125	69.9	0.874
6. Very fine sandy humus loam........	36.4	0.455	11.05	0.138	9.5	0.119	56.95	0.712
7. Fine sandy humus loam.	27.45	0.343	14.2	0.178	15.15	0.189	56.8	0.710
8. Silt loam....	10.5	0.131	3.75	0.047	6.45	0.081	20.7	0.259
9. Fine sandy loam........	13.8	0.173	4.65	0.058	7.45	0.093	25.9	0.324
10. Sandy loam .	16.15	0.202	4.30	0.054	6.8	0.085	27.25	0.341
Average.....	22.58	0.282	7.53	0.094	8.74	0.109	38.84	0.486

solution and total pressure of carbon dioxid, instead of partial; and that of the writer which directs the evaporation of soil and calcium bicarbonate, followed by the estimation of residual calcium carbonate by means of acidulation and agitation under reduced pressure at room temperature. A study of the Hutchinson and MacLennan method by the associate referee and by F. W. Bouson of the associate referee's laboratory has shown that the carbonated water effects either a forcing back of the reaction responsible for the decomposition of the carbonate or else a hydrolysis of the native silicates, thus, in a number of instances, yielding a solution of alkalinity greater to titration than that of the original bicarbonate solution, and this upon soils shown by the the Veitch, Tacke and MacIntire procedures to be distinctly acid. In view of the foregoing facts, the Tacke and Tennessee Agricultural Experiment Station methods were used as being less characterized by glaring faults.

The results secured upon the Tacke method are given in Table 5.

TABLE 7.

Lime requirement by the MacIntire method upon untreated and treated soils.

SOIL	CALCIUM CARBONATE DECOMPOSED BY ORIGINAL SOIL		CALCIUM CARBONATE DECOMPOSED BY SOIL SUBSEQUENT TO TREATMENT					
			Dried		Moist 2 weeks		Moist 4 weeks	
	Cc. N/20 normality	Per cent calcium carbonate	Cc. N/20 normality	Per cent calcium carbonate	Cc. N/20 normality	Per cent calcium carbonate	Cc. N/20 normality	Per cent calcium carbonate
1. Very fine sandy loam..	17.70	0.221	8.60	0.107	6.40	0.080	3.20	0.040
2. Sandy clay loam........	24.80	0.310	7.30	0.091	5.50	0.069	3.10	0.038
3. Very fine sandy loam..	20.30	0.253	5.40	0.068	6.40	0.080	1.50	0.019
4. Sandy clay loam........	33.10	0.414	18.10	0.226	14.50	0.181	5.50	0.069
5. Sandy humus loam........	81.30	1.016	11.70	0.146	5.50	0.069	7.90	0.099
6. Very fine sandy humus loam........	56.60	0.708	16.80	0.210	12.40	0.155	17.70	0.221
7. Fine sandy humus loam .	44.80	0.560	17.70	0.221	13.20	0.165	11.50	0.144
8. Silt loam....	25.00	0.313	14.20	0.178	10.40	0.130	9.40	0.118
9. Fine sandy loam........	23.00	0.288	2.90	0.036	2.60	0.033	....	
10. Sandy loam .	22.80	0.285	....	....	7.70	0.096	...	..
Average.....	34.94	0.437	11.41	0.143	8.46	0.106	7.48	0.094

The indications obtained by this method were secured by the use of the following technique:

> Twenty grams of air-dried soil and 5 grams of C. P. precipitated carbonate of lime, previously treated with carbonated water, were placed in 300 cc. Erlenmeyer flasks and aspirated for 15 minutes under 5 inches reduced pressure. Distilled water, carbon dioxid-free, was then added and the agitation and aspiration continued for a 3-hour period. In order to obtain additional data as to the completion of the carbon dioxid evolution and as to the speed of reaction, the agitation and aspiration were continued for a second period of 3 hours and again for 3 hours after being allowed to stand overnight.

These results are given in terms of N/20 acid, in order to convey some idea of what dependence may be placed in differences of the magnitude obtained, and also in terms of percentages of the initial indication, in the cases of the repetitions after treatments.

The data of Table 5 bring out the following points:

Applications of the amounts indicated initially do not prevent further lime requirement indications after spontaneous drying and after inter-

TABLE

Estimation of residual calcium carbonate from amounts applied

10-GRAM CHARGES OF COMPOSITE SOIL SAMPLES	AFTER APPLICATION OF AMOUNTS INDICATED BY THE TACKE PROCEDURE							
	Blank		Increase over blank after spontaneous drying		Increase over blank after remaining wet 2 weeks		Increase over blank after remaining wet 1 month	
	A*	B†	A*	B†	A*	B†	A*	B†
1, 2, 3 and 4....	4.15	0.104	0.85	0.021	1.25	0.031	1.30	0.033
5, 6, 7 and 8....	3.05	0.076	1.25	0.031	1.55	0.039	2.40	0.060
Average......	3.60	0.090	1.05	0.026	1.40	0.035	1.85	0.047

* Results expressed in cc. N /20 normality.

vals of two weeks and one month under moist conditions. That this is in part due to incomplete decomposition of the applied carbonate is indicated by the analyses for combined carbon dioxid, Table 8, upon two composite samples, each composite being made from equal amounts of each of four soils. The blanks on the composites of the original soils do not represent absolute carbonate determinations upon the acid soils, but they include the atmosphere of the apparatus, as well as the result of the slight action of the liberating acid upon soil organic matter.

It is of interest to note that in the case of both of the composites where applications were made according to the indications of the Tacke method and also those obtained by the Tennessee Agricultural Experiment Station method the residual carbonate appeared to be higher where moist conditions were maintained than where spontaneous drying was effected. Such a result would not be expected. It would seem that this finding must be attributed either to consistent analytical error, or to the possible retention of the carbon dioxid from the carbonate decomposition within the closed containing bottle, thus either retarding the absorption of calcium carbonate or effecting the reversal of the reaction by hydrolysis of calcareous soil components in the manner previously mentioned as characteristic of the Hutchinson and MacLennan method.

The average initial lime requirement of the ten soils studied was represented by a carbon dioxid evolution equivalent to 22.58 cc. of N/20 acid for the three-hour period of contact while the subsequent lime requirements after treatment, as indicated by the initial determination, were 44.1, 37.32, and 34.94 per cent of the originals, for the spontaneous drying, two weeks and four weeks under moist condition, respectively. However, the average requirement indication during the second three-hour period was 33 per cent greater than that of the first three-hour

8.

according to indications of the Tacke and MacIntire procedures.

10-GRAM CHARGES OF COMPOSITE SOIL SAMPLES	AFTER APPLICATION OF AMOUNTS INDICATED BY THE MACINTIRE PROCEDURE							
	Blank		Increase over blank after spontaneous drying		Increase over blank after remaining wet 2 weeks		Increase over blank after remaining wet 1 month	
	A*	B†	A*	B†	A*	B†	A*	B†
1, 2, 3 and 4....	4.15	0.104	0.05	0.001	3.85	0.096	3.55	0.089
5, 6, 7 and 8....	3.05	0.076	4.25	0.106	5.65	0.141	4.05	0.101
Average......	3.60	0.090	2.15	0.054	4.75	0.119	3.80	0.095

† Per cent calcium carbonate.

period, while this indication plus the third three-hour period results gave an indication equivalent to 72 per cent of the initial indication. These data upon the same soils, which were studied by the lime water titration and hydrogen ion concentration method, show that the longer period of contact allowed in the Tacke procedure would have indicated amounts of calcium carbonate which would probably have more than attained the condition of reaction found in the studies carried out by Sharp and Hoagland.

The determinations by the Tennessee Agricultural Experiment Station method were analogous to those obtained by the Tacke procedure, although the former method gave a lime requirement 50 per cent higher than that recorded by the Tacke procedure during a three-hour period. The average of the subsequent lime requirements showed 32, 24 and 21 per cent, respectively, of the average initial indication for the spontaneous drying and moist periods of two weeks and one month. This residual lime requirement does not necessarily mean, however, that insufficient calcium carbonate was applied, but it is in part accounted for by the fact that the reaction between the soil and the carbonate had not been complete, as evidenced by the appearance of minute particles of carbonate and by the increase in the amounts of carbonate carbon dioxid. This method differs from the Tacke procedure in that the residual carbonate enters quantitatively into the repetition of the method upon the carbonate treated soil and decreases from actual to apparent the amount of carbonate absorbed in the subsequent trials.

Since the foregoing was written, the associate referee has received additional data from L. J. Gillespie upon soils, the history of which was unknown to him. Four samples of soil which had been treated with the amounts of calcium carbonate indicated by the Tacke procedure and by the MacIntire procedure, and then permitted to remain wet two weeks, and four spontaneous dryings of the latter method only, were examined in order to ascertain their subsequent or residual hydrogen ion concentration. The results are incorporated in the following table:

TABLE 9.

Relation of the amounts of calcium carbonate to the attainment of neutrality, as determined by hydrogen ion concentration.

REFEREE NUMBER	HYDROGEN ION CONCENTRATION			
	Originally	Moist		Dry
		Tacke	MacIntire	MacIntire
1	5.8*	7.1	7.4	7.3
2	5.4	7.2	7.4	7.2
4	4.7	7.0	7.3	7.2
8	6.4	7.3	7.4	7.2

* Exponent of 7 expresses neutrality; greater than 7, alkalinity; less than 7, acidity.

The foregoing data demonstrate that the treatments indicated by both the Tacke and MacIntire procedures will effect neutrality or rather slight alkalinity as measured by hydrogen ion concentrations. The alkalinities recorded by hydrogen ion concentration determinations are practically the same for both methods. However, the applied treatments indicated by the MacIntire procedure were about 50 per cent greater than those of the Tacke procedure.

It is of interest to note that a comparison of the hydroxyl ion concentrations of the spontaneous drying treatments, with those of the treatments which remained moist, exhibits a slight but constantly greater alkalinity in the case of the moist treatments. This is in accord with the finding by Bouyoucos of lesser residual lime requirements in the case of the moist contacts as compared to spontaneous drying. The hydroxyl ion concentrations above referred to are also in accord with the data of Tables 5 and 9. The data of Table 5 show a higher subsequent lime requirement after maintenance of moist condition, as compared with spontaneous drying; while the data of Table 9 indicate a greater residual calcium carbonate content for the moist maintenance as compared with spontaneous drying after applications as registered by both the Tacke and the MacIntire methods.

It has been pointed out by a number of those interested in the lime problem, in its relation to soils, that the term "Lime Requirement" is somewhat vague and indefinite, and that it suggests the query "Lime requirement for what?" It is certainly true that it does not definitely indicate whether a chemical equilibrium is thus designated or reference is made to plant response. It is positively demonstrated by the foregoing, and also by other studies, that a soil exhibiting alkaline reaction by one test may still effect considerable decomposition of calcium carbonate, when soil and carbonate are permitted contact under controlled conditions. It is, therefore, believed that the term "Lime Absorption Coefficient" is more definite and to be preferred.

As previously stated, the Hopkins, Jones and Veitch methods were assigned to several laboratories but no report was received upon these procedures. If it were not for its tediousness, the Veitch method would be very much more acceptable. Nevertheless, it is based upon a principle which precludes a large variation in the active mass of the applied alkali. The Hopkins method was formerly included in the official methods of this association. It has been shown, however, that it is not permissible to assume that molar equivalent amounts of the various basic ions are absorbed when presented to soils in the form of neutral salt solutions. For this reason, the method is not now in general use, in so far as the writer has been able to learn. There is a method, however, which offers very attractive features. This is known as the Jones method. It presents to the soil a *lime* salt, and it has the advantage of being very rapid and susceptible of close duplication and requires no setting up of apparatus. Irrespective of the merits of the other methods, it is the belief of the associate referee that the Jones method offers the greatest possibilities for obtaining the coefficient of lime absorption.

RECOMMENDATIONS.

It is recommended—

(1) That the work of the ensuing year be directed along lines which will fully develop the optimum conditions for carrying out the Jones method.

(2) That future work on the problem be done by a referee to be designated as referee on the problem of "Lime Absorption Coefficient".

Messrs. B. L. Hartwell, F. R. Pember and L. P. Howard (Agricultural Experiment Station, Kingston, R. I.) presented a paper on "Lime Requirements as Determined by the Plant and by the Chemist"[1].

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[1] *Soil Science*, 1919, **7**: 279.

THE DETERMINATION OF CALCIUM IN THE PRESENCE OF PHOSPHATES[1].

By J. F. **Breazeale** (Shula Vista, Calif.), *Referee on Inorganic Plant Constituents.*

It is well known that, when dilute oxalic acid is added to solid calcium phosphate [$Ca_3(PO_4)_2$], the calcium phosphate is converted into calcium oxalate, not partially but, practically speaking, wholly. The fact that calcium oxalate is more insoluble than calcium phosphate may be shown readily by precipitating the calcium from a saturated solution of calcium phosphate by means of oxalic acid. The phosphates of magnesium, iron and aluminium, on the other hand, are readily soluble in oxalic acid.

DETERMINATION OF CALCIUM.

Method I.

(When little or no manganese is present.)

Start with a dilute hydrochloric acid solution of an ash, containing calcium, magnesium, an excess of phosphates and some iron, take an aliquot, heat to boiling, add a few drops of methyl orange as an indicator, and make slightly alkaline with ammonia. This will precipitate the phosphates of calcium, magnesium and iron. Add a saturated solution of oxalic acid until the solution is slightly acid. If too much ammonia has been added, boil off the excess before adding the oxalic acid so as to avoid having so much ammonium oxalate in the solution. The oxalic acid will dissolve the phosphates of iron and magnesium and convert the calcium phosphate into calcium oxalate. Again make the solution slightly alkaline with ammonia, and then slightly acid with oxalic acid. While this procedure is not always necessary, the precipitation seems to be better, especially when the solution contains small amounts of calcium oxid and relatively large amounts of ammonium and sodium salts. The final filtration must be done in an oxalic acid solution. Keep the solution hot and allow it to stand until the calcium oxalate settles in its characteristic way. This requires 10–60 minutes. The calcium oxalate, when precipitated in acid solution, is more crystalline and filters more readily than when precipitated in the presence of ammonia. Filter, wash with hot water, ignite and weigh as calcium oxid, or dissolve the precipitate in sulphuric acid solution and titrate with standard potassium permanganate.

In the calcium determination, as ordinarily conducted in the presence of magnesium (precipitating with ammonia and ammonium oxalate), when the calcium oxalate precipitate is large, a little magnesium is often occluded with the calcium oxalate[2]. Except when there is a large amount of lime this is not a serious error, but it can be overcome easily by dissolving the calcium oxalate in dilute hydrochloric acid and reprecipitating with ammonia and ammonium oxalate. In the method above described, when there is a large amount of lime, and it is desired

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[1] Presented by G. H. Baston.

[2] *Proc. Am. Acad. Arts Sci.*, 1901, **36**: 377; *J. Am. Chem. Soc.*, 1909, **31**: 917.

to ignite the calcium oxalate precipitate and to weigh the lime as calcium oxid, it is advisable to redissolve the calcium oxalate and again precipitate with ammonia and oxalic acid. However, if the magnesium is not desired in the filtrate and the calcium is to be titrated with potassium permanganate, a second precipitation is not necessary as the magnesium is occluded as phosphate and not as oxalate.

Method II.

(When manganese is present.)

The above directions apply to the determination of calcium in the ash of plants and similar materials when little or no manganese is present. In the presence of an appreciable amount of manganese, a slight modification is necessary, as manganese oxalate will precipitate in the presence of oxalic acid in much the same way as calcium oxalate.

Precipitate an aliquot of the original solution with oxalic acid, as before described, filter and wash a few times to remove all of the phosphates. Dissolve the precipitate, containing calcium and manganese oxalates, in hydrochloric acid, using 10 cc. or more of acid in order to have plenty of ammonium chlorid in the solution when reprecipitation takes place. Make the solution alkaline with ammonia and add a little ammonium oxalate. The calcium oxalate will be reprecipitated, while the manganese will remain in solution. Filter, wash, dissolve in dilute sulphuric acid and titrate with standard potassium permanganate. A characteristic property of manganese is that it is precipitated as the white hydroxid [$Mn(OH)_2$], which soon darkens upon exposure to the air and forms manganese trioxid. Ammonia behaves similarly if no ammonium salts are present. In the presence of ammonium chlorid, however, the precipitation is held back in much the same way as that of magnesium. Manganous hydroxid is dissolved by ammonium chlorid in direct proportion to the concentration of ammonium ions in solution. Therefore, have plenty of ammonium chlorid present when the precipitation is made and filter as soon as possible. Excellent results have been obtained with small amounts of lime in the presence of 0.5 per cent of manganese sulphate. If manganous hydroxid begins to form in the solution before the filtration, it can be detected readily by its dark color. In such a case, dissolve the precipitate in hydrochloric acid and again make alkaline with ammonia. In this way, more ammonium chlorid will be brought into the solution and the precipitation will be held back.

With ordinary care, in a precipitation with oxalic acid alone, such as has been before described, there is little danger of any calcium being held in solution by the oxalic acid.

SOLUBILITY OF CALCIUM OXALATE IN OXALIC ACID AT ROOM TEMPERATURES.

A test solution, made slightly acid with hydrochloric acid, was prepared, containing 0.1000 per cent of calcium, 0.0497 per cent of magnesium and 0.2900 per cent of phosphoric acid. Aliquots containing 0.0100 gram each of calcium were withdrawn, neutralized with ammonia, solid oxalic acid added in the cold, as indicated in Table 1, and the solutions made up to 50 cc. The solutions were allowed to stand overnight at room temperature, filtered, washed with cold water and the calcium oxalate titrated with potassium permanganate.

TABLE 1.

Solubility of calcium oxalate in cold solutions of oxalic acid.

OXALIC ACID	CALCIUM RECOVERED (0.0100 GRAM ADDED)
per cent	*gram*
1.0	0.0100
2.5	0.0100
5.0	0.0098
10.0	0.0099

These determinations were repeated several times with results almost as good as those given. Evidently, there is little or no solubility of the calcium oxalate in the oxalic acid under these conditions.

SOLUBILITY OF CALCIUM OXALATE IN HOT SOLUTIONS OF OXALIC ACID.

Aliquots of the test solution, before described, were withdrawn, brought to the boiling point, neutralized with ammonia, and solid oxalic acid added. The solutions were kept boiling hot for 1 hour, filtered, washed with hot water and the calcium oxalate titrated with potassium permanganate.

TABLE 2.

Solubility of calcium oxalate in hot solutions of oxalic acid.

OXALIC ACID	CALCIUM RECOVERED (0.0100 GRAM ADDED)
per cent	*gram*
0.1	0.0098
0.5	0.0100
1.0	0.0097
2.0	0.0098
3.0	0.0096
5.0	0.0090
10.0	0.0087
20.0	0.0081
30.0	0.0078

These determinations were also repeated several times with this and lower concentrations of calcium. As might be expected, the solubility of the calcium oxalate is about the same in concentrations of 50 parts per million of calcium as it is in 100 parts per million, approaching a solubility of about 20 parts per million at a concentration of 30 per cent of oxalic acid.

SOLUBILITY OF CALCIUM OXALATE IN SOLUTIONS OF AMMONIUM SALTS IN THE PRESENCE OF OXALIC ACID.

As the precipitation of calcium usually takes place in the presence of more or less ammonium salts, the solubility of calcium oxalate in ammonium nitrate, ammonium chlorid and ammonium sulphate was determined. The aliquots, containing 0.0100 per cent of calcium with increasing amounts of ammonium salts, were brought to the boiling point, made alkaline with ammonia, and then made slightly acid with oxalic acid. They were kept hot for 1 hour, then filtered and titrated.

TABLE 3.

Solubility of calcium oxalate in solutions of ammonium nitrate in the presence of oxalic acid.

AMMONIUM NITRATE	CALCIUM RECOVERED (0.0100 GRAM ADDED)
per cent	*gram*
1.0	0.0099
5.0	0.0095
10.0	0.0100
20.0	0.0098
50.0	0.0097

TABLE 4.

Solubility of calcium oxalate in solutions of ammonium chlorid in the presence of oxalic acid.

AMMONIUM CHLORID	CALCIUM RECOVERED (0.0100 GRAM ADDED)
per cent	*gram*
1.0	0.0098
5.0	0.0099
10.0	0.0100
20.0	0.0100

TABLE 5.

Solubility of calcium oxalate in solutions of ammonium sulphate in the presence of oxalic acid.

AMMONIUM SULPHATE	CALCIUM RECOVERED (0.0100 GRAM ADDED)
per cent	*gram*
1.0	0.0099
5.0	0.0097
10.0	0.0096
20.0	0.0094

Repeated experiments show that there is little or no solubility of the calcium oxalate in either ammonium nitrate or ammonium chlorid at any concentration encountered in ordinary work. With ammonium sulphate, there seems to be a slight solubility at high concentrations. In dissolving the original ash it is advisable, therefore, to use either hydrochloric or nitric acid in preference to sulphuric.

Another point which should be borne in mind in precipitating calcium oxalate in the presence of large amounts of either ammonium or sodium salts, is that the presence of such salts has a decided tendency to retard the precipitation of calcium. This is particularly true of ammonium sulphate. In such cases, it is advisable first to make the solution slightly alkaline with ammonia, then slightly acid with oxalic acid. If the calcium oxalate is not precipitated on stirring, the solution should be made slightly alkaline again with ammonia and then reacidified with oxalic acid. This procedure will immediately precipitate quantities of calcium oxalate that would otherwise perhaps not be precipitated at all, or would require several hours for complete precipitation. This is usually necessary only in the presence of large amounts of ammonium or sodium salts.

SOLUBILITY OF CALCIUM OXALATE IN SOLUTIONS OF SODIUM SALTS IN THE PRESENCE OF OXALIC ACID.

The effect of sodium salts upon the precipitation of calcium oxalate was determined in the same way as has been described for the ammonium salts. The procedure that would be followed in any ordinary calcium determination was followed in each case. The results were not intended as definite solubility measurements but more as a measurement of the probable error one might expect in every day work.

TABLE 6.

Solubility of calcium oxalate in solutions of sodium nitrate in the presence of oxalic acid.

SODIUM NITRATE	CALCIUM RECOVERED (0.0100 GRAM ADDED)
per cent	*gram*
1.0	0.0097
5.0	0.0100
10.0	0.0100
20.0	0.0099
40.0	0.0099

TABLE 7.

Solubility of calcium oxalate in solutions of sodium chlorid in the presence of oxalic acid.

SODIUM CHLORID	CALCIUM RECOVERED (0.0100 GRAM ADDED)
per cent	*gram*
1.0	0.0100
5.0	0.0099
10.0	0.0101
20.0	0.0098
30.0	0.0098

TABLE 8.

Solubility of calcium oxalate in solutions of sodium sulphate in the presence of oxalic acid.

SODIUM SULPHATE	CALCIUM RECOVERED (0.0100 GRAM ADDED)
per cent	*gram*
1.0	0.0099
5.0	0.0100
10.0	0.0100
20.0	0.0099

While not indicated in Table 8, there is often a slight solubility of calcium oxalate in the highest concentrations of sodium sulphate. These tables, however, show that the solubility of calcium oxalate in sodium salt solutions is negligible under the conditions of ordinary work.

SOLUBILITY OF CALCIUM OXALATE IN SOLUTIONS OF PURE SODIUM SALTS.

Contrary to what might be expected from the results just given, the solubility of calcium oxalate in solutions of sodium salts, when no oxalic acid or ammonium oxalate is present, is considerable. Pure calcium oxalate was prepared and an excess of the salt added to 100 cc. portions of neutral solutions of sodium nitrate and sodium sulphate. The solutions were shaken at room temperature until equilibrium was established. The excess of calcium oxalate was then filtered off and the amount that had gone into solution was determined by acidifying the filtrate with sulphuric acid and titrating with permanganate. With the sodium chlorid solutions, however, this procedure could not be followed, as the higher concentrations of the salt affected the permanganate titration. Therefore, definite amounts of the calcium oxalate were added to the sodium chlorid solutions and these brought to equilibrium. The calcium oxalate remaining undissolved was then filtered off, washed, dissolved in dilute sulphuric acid and titrated. These solubility determinations are shown in Tables 9, 10 and 11.

TABLE 9.

Solubility of calcium oxalate in solutions of pure sodium nitrate at room temperature.

SODIUM NITRATE	CALCIUM OXALATE DISSOLVED IN 100 CC.
per cent	*gram*
0.0	trace
1.0	0.0024
5.0	0.0048
10.0	0.0054
20.0	0.0068
40.0	0.0072
60.0	0.0066

TABLE 10.

Solubility of calcium oxalate in solutions of pure sodium chlorid at room temperature.

SODIUM CHLORID	CALCIUM OXALATE DISSOLVED IN 100 CC.
per cent	*gram*
0.0	trace
1.0	0.0019
5.0	0.0048
10.0	0.0058
20.0	0.0067
30.0	0.0058

TABLE 11.

Solubility of calcium oxalate in solutions of pure sodium sulphate at room temperature.

SODIUM SULPHATE	CALCIUM OXALATE DISSOLVED IN 100 CC.
per cent	*gram*
0.0	trace
1.0	0.0029
5.0	0.0058
10.0	0.0106
20.0	0.0154

The calcium oxalate was found to be equally as soluble in slightly ammoniacal solutions of sodium salts as in the neutral solutions.

SOLUBILITY OF CALCIUM OXALATE IN SOLUTIONS OF PURE SODIUM SALTS AT THE BOILING POINT OF THE SOLUTION.

Increasing concentrations of sodium nitrate, sodium chlorid and sodium sulphate were prepared, as shown in Tables 12, 13 and 14, and made slightly alkaline with ammonia. The reason for adding the ammonia was that the salts were often found to be slightly acid. These solutions were then brought to the boiling point and 0.0800 gram of pure, freshly precipitated calcium oxalate added to each. The solutions were kept boiling for 1 hour, filtered, the residue of calcium oxalate washed, dissolved and titrated. The difference between the amount of calcium oxalate remaining undissolved in the sodium salt solutions and that in the water represents the solubility of calcium oxalate.

TABLE 12.

Solubility of calcium oxalate in boiling solutions of sodium nitrate.

SODIUM NITRATE	CALCIUM OXALATE DISSOLVED BY 100 CC.	CALCIUM OXALATE PRECIPITATED FROM FILTRATE WITH 0.5 PER CENT OF OXALIC ACID
per cent	*gram*	*gram*
0.0	0.0000	0.0000
1.0	0.0019	0.0019
5.0	0.0054	0.0070
10.0	0.0067	0.0083
20.0	0.0093	0.0105
40.0	0.0125	0.0128
60.0	0.0125	0.0128

TABLE 13.

Solubility of calcium oxalate in boiling solutions of sodium chlorid.

SODIUM CHLORID	CALCIUM OXALATE DISSOLVED IN 100 CC.	CALCIUM OXALATE REPRECIPITATED FROM FILTRATE WITH 0.5 PER CENT OF OXALIC ACID
per cent	*gram*	*gram*
0.0	0.0000	0.0000
1.0	0.0054	0.0045
5.0	0.0086	0.0067
10.0	0.0093	0.0086
20.0	0.0099	0.0093
30.0	0.0099	0.0077

The filtrates from these precipitates, with the calcium oxalate held in solution by the sodium salts, were then evaporated to a volume of 100 cc. and 0.5 per cent of oxalic acid added to each. In the higher concentrations of the sodium salts, the method of precipitation referred to before was adopted; that is, the solutions were first acidified slightly with oxalic acid, made slightly alkaline with ammonia, then distinctly acid with oxalic acid. The amount of calcium oxalate reprecipitated in the filtrates is also shown in the tables.

TABLE 14.

Solubility of calcium oxalate in boiling solutions of sodium sulphate.

SODIUM SULPHATE	CALCIUM OXALATE DISSOLVED IN 100 CC.	CALCIUM OXALATE REPRECIPITATED FROM FILTRATE WITH 0.5 PER CENT OF OXALIC ACID
per cent	*gram*	*gram*
0.0	0.0000	0.0000
1.0	0.0048	0.0038
5.0	0.0150	0.0147
10.0	0.0237	0.0221
20.0	0.0355	0.0320

By reference to the above tables it will be seen that calcium oxalate is especially soluble in pure sodium salts and that this solubility is almost completely overcome by the addition of a common ion, oxalic acid.

TESTS OF THE OXALIC ACID METHOD.

A stock solution of pure salts was prepared, containing 0.1002 per cent of calcium, 0.1688 per cent of magnesium and 0.5000 per cent of phosphoric acid with sodium and potassium salts and iron added as impurities. This was an imitation of a plant ash. Aliquots of this stock solution were drawn off by C. A. Jensen, Riverside, Calif., and given to the writer in two sets, in amounts unknown to him, for test determinations. The first set contained relatively large amounts, while the second had very small amounts of calcium. These two sets were run

TABLE 15.

Test analyses of solutions containing relatively large amounts of calcium.

CALCIUM ADDED	CALCIUM FOUND BY ANALYSIS
gram	*gram*
0.0368	0.0366
0.0109	0.0103
0.0011	0.0010
0.0022	0.0020
0.0478	0.0487
0.0043	0.0040
0.0327	0.0339
0.0010	0.0008
0.0120	0.0115

when the work upon the method was first begun, and before very much technique had been developed. They represent the degree of accuracy that might be expected from any analyst. The results are given in Tables 15 and 16, without any duplication and without any determination being left out.

TABLE 16.

Test analyses of solutions containing relatively small amounts of calcium.

CALCIUM ADDED	CALCIUM FOUND BY ANALYSIS
gram	*gram*
0.0004	0.0003
0.0005	0.0005
0.0007	0.0006
0.0004	0.0003
0.0010	0.0008
0.0005	0.0003
0.0007	0.0005
0.0007	0.0007

By keeping the solution down to a small volume, an accurate determination of calcium can be made, in amounts as low as 0.0005 gram, in the presence of an excess of phosphates. The same degree of accuracy is noted in relatively large amounts. The accuracy in the lower concentrations makes the method well adapted to the analysis of the ash of such material as wheat seedlings, when the calcium in 100 seeds or 100 plants, which is a convenient quantity to use, runs about 0.0050 gram.

It often happens that an analyst can get good results with his own method, when other analysts find it unsatisfactory. To test this point, samples of the imitation ash solution, described above, were sent to

TABLE 17.

Comparative analyses by cooperating chemists.

ANALYST	CALCIUM ADDED	CALCIUM FOUND
	gram	*gram*
B. F. Robertson, Clemson Agricultural College, Clemson College, S. C.	0.0501	0.0498
C. S. Lykes, Clemson Agricultural College, Clemson College, S. C.	0.0501	0.0497
J. H. Crandall, Riverside, Calif.	0.0501	0.0498
W. P. Kelley, Riverside, Calif.	0.0501	0.05075

the analysts mentioned in Table 17, with the request that 50 cc. aliquots be taken and analyzed for calcium by the oxalic acid method. The results are given in Table 17.

It is not claimed that this method is advisable in all cases of plant analysis, but, with the ash of cereals and seedlings, especially when calcium alone is desired, it is rapid and accurate.

After the calcium has been removed by the oxalic acid, iron, alumina and magnesium may be determined in the filtrate. Very few ashes contain enough iron or aluminium to amount to anything in the gravimetric determination, so ordinarily they may be left out of consideration. The procedure is as follows:

Neutralize the excess of oxalic acid with ammonia, evaporate the filtrate to dryness, drive off all ammonium salts, take up with a little hydrochloric acid and precipitate the magnesium as phosphate in the regular way. In case iron or aluminium is present, evaporate, drive off the ammonium salts, take up with a little hydrochloric acid, and precipitate with ammonia.

REPORT ON INSECTICIDES AND FUNGICIDES[1].

By O. B. WINTER (Agricultural Experiment Station, E. Lansing, Mich.), *Referee.*

The cooperative work on insecticides included a study of the determination of lead, copper and zinc in products containing arsenic, copper, zinc, lead, calcium and magnesium (Bordeaux-lead arsenate, Bordeaux-zinc arsenite, etc.); and a comparison of the Gyory bromate method with the official iodin method for determining arsenic when present in a hydrochloric acid solution as arsenious oxid. Lime sulphur solutions and London purple also received consideration.

BORDEAUX-LEAD ARSENATE AND BORDEAUX-ZINC ARSENITE.

GENERAL PROCEDURE FOR THE ANALYSIS OF A PRODUCT WHICH MAY CONTAIN ARSENIC, ANTIMONY, LEAD, COPPER, ZINC, CALCIUM, MAGNESIUM, ETC.

For the determination of lead, copper and zinc in a product which may contain arsenic, antimony, lead, copper, zinc, iron, calcium, etc., a composite product was prepared by thoroughly mixing samples of lead arsenate, zinc arsenite, and dry Bordeaux, each of which had been passed through a No. 40 sieve and well mixed.

The lead arsenate used in preparing this mixture was obtained from one of the large chemical companies in Michigan. This sample was

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[1] Presented by A. J. Patten.

found to contain 63.72 per cent of lead oxid. The zinc arsenite was prepared by adding a solution of arsenious oxid to a solution of zinc sulphate, both solutions being slightly acid, and then neutralizing with sodium hydroxid solution[1]. The amount of zinc oxid in this preparation was determined and found to be as follows:

1. Determined as zinc sulphate and calculated to the oxid	30.50 per cent
2. Precipitated as the sulphid and burned to the oxid	30.90 per cent
3. Determined as pyrophosphate and calculated to the oxid	30.60 per cent
Average	30.67 per cent

The dry Bordeaux was prepared from equal parts by weight of C. P. calcium oxid and copper sulphate in the following manner: The copper sulphate was dissolved in water, the lime slaked and diluted, and the milk of lime added to the copper sulphate solution while stirring. This mixture was allowed to stand for some time, filtered by means of suction and the precipitate dried and ground. An electrolytic determination showed that the mixture contained 20.22 per cent of copper.

The Bordeaux-lead arsenate was prepared by mixing one part by weight of the dry Bordeaux with two parts of the lead arsenate, passing the whole through a No. 20 sieve and mixing again. Theoretically, this compound should contain 42.48 per cent of lead oxid and 6.74 per cent of copper. The Bordeaux-zinc arsenite was prepared by mixing three parts by weight of the zinc arsenite with four parts of the dry Bordeaux, passing the mixture through a No. 20 sieve and remixing. Theoretically, this should contain 13.14 per cent of zinc oxid and 11.56 per cent of copper. The Bordeaux-lead arsenate with Bordeaux-zinc arsenite was prepared by mixing equal parts of the above mixtures, passing the mixture through a No. 20 sieve and remixing. Theoretically, this composite sample should contain 21.24 per cent of lead oxid, 9.15 per cent of copper and 6.57 per cent of zinc oxid.

The methods sent to the collaborators for determining the lead, copper and zinc in the above composite sample are as follows:

GENERAL PROCEDURE FOR THE ANALYSIS OF A PRODUCT CONTAINING ARSENIC, ANTIMONY, LEAD, COPPER, ZINC, IRON, CALCIUM, MAGNESIUM, ETC.

(Applicable to such preparations as Bordeaux-lead arsenate; Bordeaux-zinc arsenite; Bordeaux-Paris green; Bordeaux-calcium arsenate, etc.)

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[1] *J. Am. Chem. Soc.*, 1906, **28**: 1163.

LEAD OXID (PbO).

Weigh 1 gram of the dry powdered sample, transfer to a beaker, add 5 cc. of hydrobromic acid (sp. gr. 1.31) and 15 cc. of hydrochloric acid (sp. gr. 1.19) and evaporate to dryness to remove arsenic; repeat this treatment; then add 20 cc. of the hydrochloric acid and again evaporate to dryness. Dissolve in 25 cc. of 2N hydrochloric acid, dilute to 100 cc. and pass in hydrogen sulphid until precipitation is complete. Filter, and wash the precipitate thoroughly with N/2 hydrochloric acid, saturated with hydrogen sulphid. Save the filtrate and washings for the determination of zinc. (Antimony may be present in samples containing zinc and should be removed by digesting the sulphids with sodium sulphid.) Transfer the filter paper containing the sulphids of lead and copper to a porcelain casserole or evaporating dish and completely oxidize all organic matter by heating with a few cc. of concentrated sulphuric acid, together with a little fuming nitric acid; then completely remove all nitric acid by heating on the hot plate with sulphuric acid to copious evolution of white fumes, cool, and determine lead as the sulphate, as directed for lead arsenate[1]. From the weight of lead sulphate calculate the amount of lead oxid present, using the factor: $PbSO_4 \times 0.73600 = PbO$.

COPPER.

Evaporate the filtrate and washings from the lead sulphate precipitate to fuming, add a few cc. of nitric acid to decompose the alcohol, and continue the evaporation until about 3 cc. of concentrated sulphuric acid remain. Determine the copper by Low's titration method as directed under Bordeaux mixture[2], or by electrolysis as follows:

Take up the sulphuric acid solution with water, add 1 cc. of concentrated nitric acid, and filter if necessary. Make the volume to about 150 cc. and electrolyze as usual.

ZINC OXID (ZnO).

Method I.

Evaporate the filtrate and washings from the precipitate of copper and lead sulphids to a small volume, add 1–2 cc. of concentrated nitric acid, boil for a few minutes, then evaporate to dryness, add a few cc. of concentrated sulphuric acid and heat to fuming, take up with water, remove by filtration any calcium sulphate that may have separated out and wash with cold water. Neutralize with concentrated sodium hydroxid solution (using phenolphthalein as indicator) and then add about 15 grams of solid sodium hydroxid. Transfer to a beaker (the volume should now be about 150 cc.), heat to boiling and electrolyze, using a rotating nickel cathode, which should be placed below the anode, and a current of about 3.5 amperes. About 10 minutes before the electrolysis is completed the electrodes and the sides of the beaker should be washed down with a jet of water. The solution should be cooled to about 25°C. just before the end of the electrolysis by applying water and ice to the outside of the beaker. When the deposition is completed, which should take about 45 minutes, lower the beaker, quickly rinse the cathode with cold water, remove, and rotate for a moment in each of two vessels containing cold water, then in alcohol and, finally, in ether dried over sodium. Heat in an oven, or by holding some little distance above a free flame for a few moments, and weigh as metallic zinc. Calculate the zinc oxid as follows: $Zn \times 1.24476 = ZnO$.

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 68.

[2] *Ibid.*, 70.

Method II.

Evaporate the filtrate and washings from the sulphid precipitation of the copper and lead to a small volume, add 1–2 cc. of concentrated nitric acid, boil a few minutes, then evaporate to dryness, add a few cc. of concentrated sulphuric acid and heat to fuming. Take up with water, remove any calcium sulphate that may have precipitated out by filtration and wash with cold water. Neutralize with ammonium hydroxid (using methyl red as an indicator), add 4 cc. of 5% sulphuric acid per 100 cc. of solution (the volume should be about 150 cc.) and pass hydrogen sulphid gas into the solution at room temperature for about 40 minutes. Allow to stand for 30 minutes with occasional stirring, filter through a tared Gooch crucible, wash with cold water, dry, burn to the oxid, preferably in a muffle at 850–930°C., and weigh as zinc oxid.

The following results were received on this sample:

TABLE 1.

Bordeaux-lead arsenate with Bordeaux-zinc arsenite.

ANALYST	LEAD OXID (PbO)	COPPER (Cu)	ZINC OXID (ZnO)	
			Method I	Method II
	per cent	*per cent*	*per cent*	*per cent*
1. L. E. Sayre, University of Kansas, Lawrence, Kans.	20.51	9.10	....	6.50
	20.49	9.04	....	6.75
Average	20.50	9.07	....	6.62
2. D. K. French, Dearborn Chemical Co., Chicago, Ill.	20.76	9.54	....	6.47
	20.75	9.55	....	6.38
	21.08	9.57	....	6.25
	20.51	9.55	....	6.42
Average	20.78	9.55	....	6.38
3. J. J. T. Graham, Bureau of Chemistry, Washington, D. C.	21.51	9.52	6.27	6.48
	21.55	9.46	6.40	6.52
	21.62	9.54	6.34	6.34
	21.47	9.50	6.35	6.50
	21.59	....	....	6.40
Average	21.55	9.51	6.34	6.45
4. E. F. Berger, Agricultural Experiment Station, E. Lansing, Mich.	21.23	9.19	....	6.40
	....	....	....	6.56
	....	....	.....	6.50
Average	21.23	9.19	....	6.49
5. O. B. Winter, Agricultural Experiment Station, E. Lansing, Mich.	21.26	9.10	5.14	6.55
	21.26	9.20	5.38	6.70
	....	9.35	....	6.63
Average	21.26	9.22	5.26	6.63
General average	21.11	9.37	5.98	6.49
Theory	21.24	9.15	6.57	6.57

DISCUSSION.

A careful study of the table shows the following:

1. That there is considerable variation in the average results by the different analysts.
2. That the different determinations made by the same analyst agree very closely.
3. That the general average in each case agrees well with the calculated amount present with the exception of Method I under zinc oxid (ZnO).

The greatest variations are found in the results for zinc oxid by Method I, the next for lead oxid, then for copper, and the least for zinc oxid by Method II. It is unfortunate that only two analysts determined the zinc oxid by Method I, and one of these, Analyst 5, admits that he had not mastered the method. Analyst 3 obtained results which agree with each other very closely indeed, and they are only slightly low. This makes it appear that the method is efficient, and undoubtedly should have further consideration, even though the results appear very unsatisfactory in the 1916 "Report on Insecticides"[1]. The variations in the lead oxid and copper are rather puzzling. In the results by Analyst 2, they nearly balance each other—the lead oxid being low and the copper high. This indicates difficulty in separation. Analyst 1 finds them both low, and Analyst 3 finds them both high, and in no case does the zinc oxid vary sufficiently from the theoretical to make up the difference. Considering that both these methods are standard methods, and that the variations are not entirely explainable by difficulty in making the separations, they look promising and deserve future study. The results for zinc oxid by Method II are quite satisfactory, and should also receive further consideration.

LEAD ARSENATE

The sample of lead arsenate previously referred to in this report was used to compare the Gyory bromate method[2] with the official iodin method for the titration of arsenious oxid in a hydrochloric acid solution. The directions sent out for this work are as follows:

TOTAL ARSENIC PENTOXID.

REAGENTS.

Solutions required:

Starch solution.—Prepare as directed under Paris green[3].

Standard arsenious oxid solution.—Prepare as directed under Paris green[3].

Standard iodin solution.—Prepare as directed under Paris green[3] but calculate in terms of arsenic pentoxid (As_2O_5).

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **3**: 331.

[2] *Z. anal. Chem.*, 1893, **32**: 415.

[3] *Assoc. Official Agr. Chemists, Methods*, 1916, 63.

Standard potassium bromate solution.—Dissolve 1.41 grams of pure potassium bromate in water and dilute to 1 liter. This is approximately a N/20 solution. Standardize by titrating against a hot arsenious oxid solution containing about 10% free hydrochloric acid, using methyl orange as indicator. The end point is most easily determined if the indicator is not added until near the end of the titration.

DETERMINATIONS.

Iodin method.—Determine as directed under lead arsenate[1].

Potassium bromate method.—Proceed as directed under the iodin method until the distillation is completed, taking an amount of sample equal to the arsenic pentoxid equivalent of 500 cc. of the standard potassium bromate solution. Dilute the distillate to 1 liter, take a 200 cc. aliquot, add about 30 cc. of concentrated hydrochloric acid, heat nearly to boiling and titrate with the standard potassium bromate solution, using methyl orange as indicator.

The number of cc. of the standard potassium bromate solution used represents directly the total per cent of arsenic present in the sample expressed as arsenic pentoxid (As_2O_5).

The results received are shown in Table 2.

DISCUSSION.

The results in Table 2 again show considerable variation between the average results by the different analysts. However, since this table shows a comparison, the merits of the bromate method are readily ascertained by noting the results obtained by the different analysts when determinations are made by each of the two methods, and by comparing the general averages. With the exception of Analyst 2, the results compare very favorably, as do also the general averages. Therefore the bromate method should have further consideration.

The results obtained by Graham when the bromate method was run without heating and without the addition of hydrochloric acid are very satisfactory, and since these factors save time and expense, this modified method should be studied.

LONDON PURPLE.

In 1916 the referee suggested[2] that methods be studied for removing the coloring matter without loss of arsenic from London purple in making the arsenic determinations. Some work has been done along this line and a few of the methods look very promising. Animal charcoal, blood charcoal, sponge charcoal, sugar charcoal, kaolin, fullers' earth, aquadag, etc., were used as adsorbents for removing the coloring matter, and the organic matter was entirely destroyed by heating with a mixture of zinc oxid and sodium carbonate. Of the adsorbents, blood charcoal was found to be the only one which would remove the color entirely,

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 68.

[2] *J. Assoc. Official Agr. Chemists*, 1920, **3**: 357.

TABLE 2.

Lead arsenate:*

ANALYST	ARSENIC PENTOXID	
	Iodin method	Bromate method
	per cent	*per cent*
1. L. E. Sayre	32.20	32.34
2. D. K. French	32.69	33.39
	32.38	32.97
	32.48	32.97
	32.75	33.02
	32.47	32.99
Average	32.55	33.07
3. J. J. T. Graham	32.56	32.70
	32.63	32.81
	32.67	32.81
	32.57	32.81
Average	32.61	32.78
4. E. F. Berger	33.10	33.19
	33.00	....
	33.15	....
Average	33.08	33.19
5. O. B. Winter	32.87	32.80
	32.93	32.92
Average	32.90	32.86
6. W. F. Walsh, Agricultural Experiment Station, Geneva, N. Y.	32.75	32.52
	32.52	32.72
	32.75	32.66
	32.53	32.64
	32.42	32.51
	32.42	32.42
	32.70	32.53
	32.64	32.65
	32.47	32.53
	32.53	32.18
	32.42	32.18
	32.76	32.14
	32.50	32.27
	32.50	32.18
	....	32.18
	....	31.95
Average	32.56	32.39
General average	32.63	32.62

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* Additional results on the iodin method, with an average of 32.42 per cent of arsenic pentoxid, were received from M. R. Miller, Berkeley, Calif., after the report was completed, and are not included in this tabulation.

although some of the others, especially animal charcoal and sugar charcoal, removed part of it.

Several different procedures were followed in an attempt to remove the coloring matter by means of blood charcoal and then to determine the arsenic present as arsenious oxid. In all cases the results were lower than by the official method. Since the loss was approximately twice as great when these same methods were used in trying to determine the total arsenic as when only the arsenic present as arsenious oxid was determined, the loss must have been due to the adsorption of arsenic by the charcoal.

The only procedure for removing the coloring matter by absorbents which gave satisfactory results was the official distillation method[1], modified by placing about 5 grams of blood charcoal in the distillation flask with the sample before beginning the determination. This removed the color entirely without any loss of arsenic, as shown by the following results:

TABLE 3.

Determination of total arsenic as arsenic trioxid in London purple.

ANALYST	OFFICIAL METHOD	OFFICIAL METHOD MODIFIED
	per cent	*per cent*
Commercial analyst	38.96	....
Referee	39.00	....
Referee	....	38.95

This method is simple, has a sharp end point and should receive cooperative study.

The destruction of the organic matter in London purple by burning the sample with an intimate mixture of four parts of zinc oxid and one part of dry sodium carbonate was carried out as follows[2]:

> Thoroughly mix the sample in a shallow porcelain crucible with the zinc oxid-sodium carbonate mixture, and then cover with a layer of the latter. Place the crucible uncovered in a muffle, heat gradually at first and finally for about 15 minutes at full heat. (The mass will not sinter.) Cool, transfer to a distillation flask and proceed according to the official distillation method for total arsenic[1].

By this method the referee found 38.98 per cent of arsenic trioxid in the same sample of London purple referred to in Table 3. Roark reports for an analysis of a sample of London purple by this method, 38.22 per cent of total arsenic as (As_2O_3) in comparison with 38.15 per cent by the official method.

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 63.

[2] *J. Assoc. Official Agr. Chemists*, 1920, 3: 355.

The technique of this method is simple and it gives accurate results. Therefore, it should also receive cooperative study in order to compare it with the method previously described.

LIME-SULPHUR SOLUTIONS.

At the last meeting of this association the zinc chlorid method for analyzing lime sulphur solutions was made tentative, and the referee was directed to continue the study of the different methods which have been proposed to the association for the analysis of such solutions. Considering the amount of cooperative work that has been done on this problem, with nothing particularly new to work on, the referee hardly felt justified in sending out samples to burden the already busy chemists with collaborative work. In the past, solutions of various kinds have been sent out for analysis—namely, solutions especially prepared to contain, if possible, nothing but polysulphids, thiosulphate and sulphate of calcium; solutions especially prepared to contain other compounds than those just mentioned; and ordinary commercial solutions. It seems that results which have been presented to the association in former reports are just as valuable now as they were when presented, and just as indicative as would be the results on samples that might have been sent out this year.

For these reasons, the referee's work has consisted of a study of the reports of the past, even though they have already been discussed, and an attempt has been made to contribute something on the chemical composition of a "straight"[1] lime-sulphur solution. He also gave considerable consideration to Chapin's method[2] for analyzing lime-sulphur solutions.

Evidently, there are still varying opinions regarding the methods used for analyzing lime-sulphur solutions. The two methods which have been before the association for some little time are known as the "Iodin Titration Method" and the "Zinc Chlorid Method". Of these two methods, it is generally conceded that the iodin titration method consumes less time, which is an important factor in commercial work. It is also held by some that in the determination of the monosulphid sulphur equivalent—a determination that is made by the iodin titration method and not by the zinc chlorid method as recommended to the association last year—data are obtained which are exceedingly useful in evaluating the solution as an insecticide. Furthermore, some laboratories have not the time to make complete analyses of all the lime-sulphur samples received and it is desirable to have a uniform

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[1] In this paper a "straight" lime-sulphur solution is interpreted as a solution that has been prepared from ordinary commercial lime and sulphur to which no foreign substances such as common salt, etc., have been added, and which has stood a few days at least.

[2] *J. Ind. Eng. Chem.*, 1916, 8: 151, 339.

standard method adopted which will give the required information in a comparatively short time. By means of the iodin titration method, it is possible to determine the monosulphid sulphur equivalent, the thiosulphate sulphur, and the total sulphur in a very short time; and from these data, the complete analysis of a "straight" lime-sulphur solution may be calculated. However, this method has been criticised from two general standpoints—first, that the method is deficient in itself (errors may arise from difficulty in reading end points while titrating with iodin, from using dilute solutions, from occlusion of compounds in solution by precipitates, etc.); second, that lime-sulphur solutions may contain compounds which interfere with the determinations to be made.

It is the aim of this report to emphasize again the fact that the first of these criticisms is unwarranted—a fact which has been clearly demonstrated by the collaborative work of this association, and that the second criticism has very little foundation in fact in so far as "straight" lime-sulphur as found on the market is concerned.

In considering the first of the above criticisms, let us assume that the sulphur present in a lime-sulphur solution exists only as the polysulphids, thiosulphate, and sulphate of calcium. Roark[1] made a resumé of the objections to the "iodin titration method" under these conditions.

More recently Chapin[2] questioned the "selective action whereby iodin reacts more readily with calcium polysulphid than with calcium thiosulphate", which action makes it possible to determine the monosulphid equivalent, and the thiosulphate in the same solution.

Now, if we may assume that all of the samples of lime-sulphur solutions reported in the past on which the iodin titration method has been run, except a few which are known to have been especially prepared (Samples 1 and 2, Report on Insecticides, 1916[3]), are "straight" lime-sulphur solutions and contain only polysulphids, thiosulphate and sulphate of calcium, some of the above objections may be answered by work which has already been before this association. These reports are as follows: Samples 1 and 2, Report on Insecticides, 1912[4], Samples 1 and 2, Report on Insecticides, 1913[5], and Sample 3, Report on Insecticides, 1916[3]. The results show that, when considering the determination of the monosulphid equivalent, the variations in the individual determinations by the different analysts are very small indeed—the maximum by two analysts being 0.10 per cent, the minimum by five analysts 0.00 per cent, and the average by all the analysts 0.03 per cent; that

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[1] *J. Assoc. Official Agr. Chemists*, 1915, **1**: 80.
[2] *J. Ind Eng. Chem.*, 1916, **8**: 157, 339.
[3] *J. Assoc. Official Agr. Chemists*, 1920, **3**: 338.
[4] U. S. Bur. Chem. Bull. 162: (1913), 33.
[5] *J. Assoc. Official Agr. Chemists*, 1915, **1**: 62.

the general average in Samples 1 and 2, 1912 (the only samples in which the theoretical is given), is identical with the theoretical; and that the variation of the average of the determinations of each chemist, from the general average, with very few exceptions, is remarkably small—averaging about 0.03 per cent.

The results for the thiosulphate sulphur are not quite so satisfactory. However, on examining the 1913 and 1916 reports, the ones in which the iodin titration and zinc chlorid methods are compared, it will be noticed that with the exception of two analysts in the 1916 report (and the results of one of these analysts by the zinc chlorid method varies considerably from the theoretical in the 1913 report), the two methods appear equally accurate.

The effect of moderate dilution should also be satisfactorily answered by these reports. When results checking as closely as those shown for monosulphid equivalent can be obtained, certainly the variation in the dilution of the samples titrated can have no appreciable effect.

If the reports referred to be studied carefully, it will readily be seen that every objection to the iodin titration method, with one exception, is fully answered in that the two methods agree as closely as can be expected of any two methods; that the general average for the iodin titration method, in the report where the theoretical is given, is slightly closer to the theoretical than those by the zinc chlorid method; that in this same report a smaller percentage of the determinations made by the iodin titration method are discarded from the general averages on account of being too widely different from the theoretical, than by the zinc chlorid method; and that the variations in the averages of the results by the different analysts from the general averages in the other two reports, are no greater, in general, when made by the iodin titration method than by the zinc chlorid method. It would be impossible to obtain the results reported, if any of the above objections (except one) were valid.

The objection alluded to above, which is not answered by a study of these reports, refers to the difficulty in filtering the solution in order to remove the free sulphur immediately after titrating for the thiosulphate. In reply to this, it may be stated that this filtration may be made within a very short time by acidifying the solution with a few drops of dilute hydrochloric acid and warming gently—as was shown by Averitt[1], and his work has been substantiated in the writer's laboratory.

The following samples represent commercial and laboratory made preparations, ranging in age from freshly boiled to ten years, and the

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[1] *J. Ind. Eng. Chem.*, 1916, 8: 623.

results given in this table show a comparison between the thiosulphate when determined by the iodin titration and the zinc chlorid methods:

TABLE 4.

Determination of thiosulphate.

SAMPLE NUMBER	IODIN METHOD	ZINC CHLORID METHOD
	per cent	*per cent*
1	1.29	1.19
2	1.32	1.32
3	1.03	1.03
4	6.27	6.14
5	3.19	3.19
6	3.76	3.72
7	5.67	5.63
8	5.07	4.94
9	0.71	0.85
10	13.39	13.31
11	1.10	1.11

Some work was also done to determine the effect of diluting the aliquot in making the monosulphid equivalent determination and the following conclusions were drawn:

1. Moderate dilution, such as is necessary for the analysis of a lime-sulphur solution, has no appreciable effect.

2. Excessive dilution, which is entirely unnecessary, gives rise to rapid decomposition of the solution, and causes difficulty in determining the end point while titrating with iodin.

3. The amount of dilution has no appreciable effect until it reaches the point of rapid decomposition.

Considering the fact that in the more recent articles in which the iodin titration method has been attacked, the emphasis has been placed on the assumption that lime-sulphur solutions contain an appreciable amount of compounds which interfere with this method, rather than on the accuracy of the method itself, it seems that the iodin titration method may be considered an accurate method for the analysis of such solutions, provided the solutions do not contain an appreciable amount of other compounds than those mentioned above.

Since the cooperative work of this association shows that the iodin titration method is accurate and agrees very closely with the zinc chlorid method for the analysis of straight lime-sulphur solutions; since the ordinary solutions as found on the market are straight lime-sulphur solutions, and if an occasional one is not, this fact can be readily detected and the solution treated accordingly; since this method is more practical for commercial analyses and is now used in many experiment station laboratories for their control work; and since it is desired that these analysts continue the use of the method and have their results considered official, the method should be made a tentative one. Therefore, it seems advisable to make those determinations official where the two methods are practically the same, retain as tentative the remaining determinations by the zinc chlorid method and also make tentative the remaining determinations by the iodin titration method.

The following indicates those determinations which are recommended to be made official, and those iodin titration method determinations which are recommended to be made tentative:

LIME-SULPHUR SOLUTIONS.

TOTAL SULPHUR.—OFFICIAL[1].

MONOSULPHID EQUIVALENT.—TENTATIVE[2].

Dilute 10 cc. of the solution prepared for total sulphur to about 30 cc. with recently boiled distilled water and titrate with N/10 iodin until the yellow color just disappears. A small crystal of nitroprussid of sodium may be used if difficulty is experienced in determining the end point. From the number of cc. of N/10 iodin calculate the monosulphid equivalent as follows: cc. N/10 I × 0.0016 = grams of sulphur as monosulphid equivalent.

THIOSULPHATE SULPHUR.—TENTATIVE[2].

Continue the above titration carefully, letting the iodin act as its own indicator until a small drop produces a slight permanent coloration. From the number of cc. of N/10 iodin used in this second titration, calculate the thiosulphate sulphur as follows: cc. N/10 I × 0.0064 = grams of sulphur as thiosulphate.

SULPHID SULPHUR.—TENTATIVE[2].

Direct determination.—Allow the solution from the thiosulphate determination to stand several hours with an occasional stirring, or acidify with a few drops of dilute hydrochloric acid and warm gently with stirring, filter and wash thoroughly with warm water. Place the filter paper with the sulphur in a small vessel and dissolve the sulphur in about 15 cc. (1 to 3) sodium hydroxid by heating gently on a steam or water bath for 1–1½ hours (do not boil). Keep the flask covered and shake gently a few times during the digestion to remove the sulphur from the sides. Oxidize and complete the determination as under total sulphur.

Indirect method.—The difference between the total sulphur and the sum of the thiosulphate sulphur and sulphate sulphur is the sulphid sulphur.

SULPHATE SULPHUR.—TENTATIVE[2].

To the filtrate from the thiosulphate determination, add several drops of hydrochloric acid, and precipitate in the cold with 5 cc. of 5% barium chlorid solution, allow to stand overnight, filter, calculate the sulphur from the weight of the barium sulphate and report as sulphate sulphur.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **3**: 353–4.
[2] U. S. Bur. Chem. Bull. **162**: (1913) 37.

TOTAL LIME, CaO.—OFFICIAL[1].

SUGGESTIONS FOR FUTURE WORK.

1. No satisfactory method was found for removing the color in order to determine the arsenious oxid in London purple. The end point can be read quite accurately by the present official method, but it is probable that a method may be found which will take less time and prove just as accurate.

2. It is the belief of several of the adherents to the iodin titration method for the analysis of lime-sulphur solutions, that the sulphid sulphur may be determined as accurately by simply weighing the sulphur as by the much more tedious methods now in use, as was shown by Averitt[2]. Some work done in the writer's laboratory along this line looks very promising. If this method can be made an accurate one, the entire analysis of a lime-sulphur solution (omitting the determination of the sulphate sulphur) can be made in a very short time. It appears that this work deserves consideration.

RECOMMENDATIONS.

It is recommended—

(1) That further work be done on the Gyory method for titrating arsenious oxid in hydrochloric acid solution with a solution of potassium bromate.

(2) That further work be done on the determination of lead, copper and zinc in the analysis of such preparations as Bordeaux-lead arsenate, Bordeaux-zinc arsenite, etc.

(3) That work be done on the determination of total arsenic in London purple by first destroying the color by heating the sample with a mixture of zinc oxid and sodium carbonate.

(4) That work be done on the removal of coloring matter from London purple by the use of an adsorbent.

(5) That the methods given by Roark[3] for total sulphur and for total lime be made official.

(6) That methods for determining the monosulphid equivalent, thiosulphate sulphur, sulphid sulphur, and sulphate sulphur under the iodin titration method, as given on page 146, be made tentative.

A paper on "The Determination of Arsenic in Insecticides by Potassium Iodate"[4] was presented by G. S. Jamieson (Bureau of Chemistry, Washington, D. C.).

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **3**: 355.

[2] *Ibid.*, 1915, **1**: 95.

[3] *Ibid.*, 1920, **3**: 354–5.

[4] *J. Ind. Eng. Chem.*, 1918, **10**: 290.

DRUG SECTION.

REPORT ON DRUGS.

By W. O. EMERY (Bureau of Chemistry, Washington, D. C.), *Referee.*

Considerable progress can be reported in the development and adaptation of certain reactions and procedures which, it is expected, will eventually be available for cooperative investigation. For example, some time last year the attention of the referee was directed to the desirability of a procedure whereby it would be possible to determine, without undue complication of apparatus, monobromated camphor as it occurs in migraine tablets. The evaluation of this rather elusive compound *per se* is a matter of no great difficulty. When in admixture with other therapeutic agents and materials, however, as in the various forms of migraine combinations, this bromin derivative of camphor requires a special treatment, since it does not lend itself readily to quantitative isolation from the vehicular and other components of tablets by means of organic solvents.

Without going into all the details of the method as finally adopted, it may be stated that any convenient aliquot of the powdered sample, containing 100 to 150 mg. of monobromated camphor, is treated in a 100 cc. round-bottomed flask, in connection with a reflux condenser, with 30 cc. of 50 per cent alcohol and 20 to 25 grams of 1.5 to 2 per cent sodium amalgam, the mixture warmed gently for 30 minutes over a wire gauze, and subsequently without the condenser for 1 hour or more on the steam bath. The liquid is then strongly acidified with nitric acid, and, after the amalgam is completely spent, the halogen is determined gravimetrically in the usual way, or volumetrically according to Volhard. Recoveries of monobromated camphor are entirely satisfactory, as demonstrated fully by controls.

Considerable attention likewise has been given to an examination of salvarsan substitutes appearing on the American market. The method followed in estimating the arsenic was the one suggested by C. R. Smith[1], and depends on the interaction of arsine and mercuric chlorid, the resulting precipitate of calomel being filtered off and weighed or titrated as directed by the author.

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[1] U. S. Bur. Chem. Circ. 102: (1912).

REPORT ON MEDICINAL PLANTS.

By ARNO VIEHOEVER (Bureau of Chemistry, Washington, D. C.),
Associate Referee.

The report is divided into four parts:

I. A method for the determination of volatile oil in mustard seed and substitutes.
II. Methods for the hydrolysis of linamarin and the subsequent determination of hydrocyanic acid.
III. The effect of abnormal conditions on trade in crude drugs.
IV. The value of weights of unit volumes in the analysis of crude drugs and spices.

PART I.

The method for the determination of volatile oil in mustard seed and substitutes deals with the hydrolysis of the glucosides present in such products and the estimation of the volatile oil, using silver nitrate solutions of known strength. The method proposed has been published[1]. While uniform results have been obtained in the writer's laboratory with this method, higher results may be obtained by the addition of alcohol before maceration. Raquet[2] also found high results when the maceration was carried on in an aqueous alcoholic solution, as first suggested by Roeser[3]. Raquet concludes that the addition of alcohol prevents bacterial action; he thus explains the higher amount of volatile oil obtained. In order to confirm further the influence of alcohol when added before maceration, samples of Japanese mustard seed (*Brassica cernua* Thunberg), from a uniform lot, were submitted to collaborators. The suggestions were to try out both the method and the modification of adding alcohol, as outlined below:

METHOD FOR THE DETERMINATION OF VOLATILE OIL IN MUSTARD SEED AND MUSTARD SUBSTITUTES.

Place 5 grams of the ground seed (No. 20 powder) in a 200 mil flask, add 100 mils of water, stopper tightly, and macerate for 2 hours at about 37°C. Then add 20 mils of U. S. P. alcohol (95%), and distil about 60 mils into a 100 mil volumetric flask containing 10 mils of 10% ammonium hydroxid solution, taking care that the tip of the condenser dips below the surface of the ammonium hydroxid solution. Add 20 mils of N/10 silver nitrate solution to the distillate, set aside overnight, heat to boiling on a water bath (in order to agglomerate the silver sulphid), cool, make up to 100 mils with water, and filter. Acidify 50 mils of the filtrate with about 5 mils of concentrated nitric acid and titrate with N/10 ammonium thiocyanate, using 5 mils of 10% ferric ammonium sulphate solution for an indicator. Each mil of N/10 silver nitrate consumed equals 0.004956 gram of allylisothiocyanate.

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[1] U. S. Dept. Agr., S. R. A., Chemistry, **20**: (1917) 59.
[2] *Répert. pharm.*, 1912, **24**: 145.
[3] *J. pharm. chim.*, 1902, 6th ser., **15**: 361.

In this method the alcohol is added after maceration. In Roeser's method, abstracted in Leach[1], the alcohol is added before the maceration and the mustard macerated for 2 hours. To determine the advisability of the addition of alcohol before maceration, it was suggested that 15 mils of U. S. P. alcohol (95 per cent), be added to the mixture before maceration, and the remaining 5 after maceration, proceeding otherwise according to the method on page 149.

The results obtained by the collaborators are given in Table 1.

TABLE 1.

Determination of volatile mustard oil in Japanese mustard seed (Brassica cernua Thunberg).

ANALYST		1 HOUR AT 25°C.		2 HOURS AT 37°C.	
	No alcohol used either in maceration or distillation.	No alcohol used in maceration; 20 cc. of alcohol added just before distillation	15 cc. of alcohol used in maceration; 5 cc. of alcohol added just before distillation	No alcohol used in maceration; 20 cc. of alcohol added just before distillation	15 cc. of alcohol used in maceration; 5 cc. of alcohol added just before distillation
	per cent	*per cent*	*per cent*	*per cent*	*per cent*
J. H. Bornmann, U. S. Food and Drug Inspection Station, Transportation Building, Chicago, Ill.	0.64	0.69	0.79	0.66	0.80
	0.50	0.68	0.76	0.65	0.80
	....	0.65	....	0.59	0.79
	....	0.65	....	0.59	0.79
	....	....	....	0.58	....
V. B. Bonney, U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, San Francisco, Calif.	....	....	....	0.81	1.02
	....	....	....	0.83	1.04
R. Rippetoe and N. Smith, Schieffelin & Co., New York, N. Y.	....	....	....	0.768	....
Samuel Ginsburg, U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, New York, N. Y.	....	....	....	0.70	0.78
	....	....	....	0.71	0.81
	....	....	....	0.71	0.82

As will be seen, the amount of volatile oil obtained by adding the alcohol before maceration is up to 30 per cent higher than when the alcohol is added after maceration.

COMMENTS BY ANALYSTS.

J. H. Bornmann.—The objection to Roeser's method, as outlined in Leach, is that too small a volume of liquid remains in the flask after distillation. It is difficult to

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[1] A. E. Leach. Food Inspection and Analysis. 3rd ed., 1913, 457.

avoid scorching the mass unless the steam bath is used. In these experiments an asbestos board, having a hole 2½ inches in diameter, was used. This prevents superheating the upper part of the flask and scorching the mustard. There is some indication that the rate of distillation influences the amount of mustard oil recovered. The rate employed was about 3 cc. per minute. When the rate was slower, slightly lower results were obtained. It seems necessary to add the alcohol in the maceration. The results obtained by 1 hour maceration at 25°C. agree very closely with those obtained by macerating for 2 hours at 37°C. It seems advisable to change the method so as to include the addition of alcohol in the maceration, and to change the time and temperature to 1 hour at room temperature.

V. B. Bonney.—I did not have any trouble with any of the methods used, although it was my first experience with the assay of mustard seed.

J. R. Rippetoe and N. Smith.—The method seems to be very good and is simple to operate.

Samuel Ginsburg.—I believe that in our work we should adopt the modified method, adding alcohol before maceration.

PART II.

This part deals with the proper hydrolysis of the glucoside present in beans of the lima type (*Phaseolus lunatus*) and the subsequent determination of hydrocyanic acid obtained from such beans. Since the glucoside "linamarin" occurs not only in *Phaseolus lunatus*, but also in flax (*Linum usitatissimum*), cassava (*Manihot utilissima*), and other plants, work on this subject may be justified. A sample of foreign grown beans (Red Rangoon) was submitted to the collaborators and the results obtained with the methods described below are given in Table 2.

METHODS FOR THE HYDROLYSIS OF LINAMARIN AND THE SUBSEQUENT DETERMINATION OF HYDROCYANIC ACID.

Macerate a 20 gram sample (No. 20 powder) at room temperature in a stoppered flask with 100 cc. of water for 2 hours. Add 100 cc. of water and distil with steam. The hydrocyanic acid contained in the distillate may be determined according to the Volhard, Liebig, Liebig-Denigès, or Viehoever and Johns methods.

Liebig Method.

Receive the distillate containing the hydrocyanic acid in a solution of 0.5 gram of potassium hydroxid in 20 cc. of water. When the distillate amounts to 150 cc., pour it out in a beaker and titrate with silver nitrate solution, which should be added drop by drop under continuous stirring. The end point[1] is the first permanent turbidity produced, viewed with black paper background. In case 0.0185 normal silver nitrate solution is made up, 1 cc. of this solution equals 1 mg. of hydrocyanic acid, and the result, representing the number of cc. used, can be read from the burette.

Volhard Method.

Receive the distillate containing the hydrocyanic acid in 20 cc. of N/50 silver nitrate solution acidified with 1 cc. of nitric acid. Filter the distillate through a Gooch filter

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[1] Potassium iodid, about 0.2 gram, added before titration, improves the end point (Drehschmidt-Denigès), especially in the presence of ammonia (10 per cent), adding about 1 to 2 cc. (Denigès). The procedure thus modified is known as Liebig-Denigès method.

and titrate the excess of silver nitrate with N/50 potassium thiocyanate, using ferric alum indicator. The number of cc. of silver nitrate consumed multiplied by 2.7 = mg. hydrocyanic acid per 100 grams.

Notes on the methods.—The distillation should not be continued too long, otherwise some other products of a reducing nature may be formed. Alkaline silver nitrate solution, used in the Liebig method, is more readily reduced by many substances, such as aldehydes, and other organic bodies, than acidified silver nitrate solution.

In the Volhard method it is necessary to filter off the silver cyanid before the titration, since the reaction is reversible and the end point uncertain. In the Liebig method, continuous stirring during the titration is necessary, otherwise the silver oxid precipitated by the potassium hydroxid colors the liquid yellow, obscuring the end point more or less. In the event of the presence of volatile chlorids, only the Liebig method can be applied.

Viehoever and Johns Method.

This method[1] is based on the precipitation and colorimetric determination of Prussian blue. Since the formation of Prussian blue necessitates the presence of hydrocyanic acid, this formation is a definite proof of such presence, and the method is regarded as an accurate means for the determination of hydrocyanic acid. The method will be recommended for such cases where it is desired to check the results obtained by other methods and in such instances where other methods cannot be used on account of interfering substances present in the distillate.

TABLE 2.

Determination of hydrocyanic acid in red Rangoon beans (Phaseolus lunatus Linne).

(Expressed as mg. per 100 grams.)

ANALYST	LIEBIG METHOD	VOLHARD METHOD	LIEBIG-DENIGÈS METHOD
C. B. Gnadinger, U. S. Food and Drug Inspection Station, Transportation Building, Chicago, Ill.	57.0 58.6	46.6 46.0	50.2 49.1 49.1
V. B. Bonney.	45.85 45.30	45.20 44.90	....
C. O. Ewing, Bureau of Chemistry, Washington, D. C.	....	44.8 44.6	44.0 44.5
Arno Viehoever.	....	45.1 45.1	44.5 45.0
R. Rippetoe and N. Smith.	58.1	44.2	....

COMMENTS BY ANALYSTS.

C. B. Gnadinger.—Philipp says of the Liebig method[2] "* * * large excesses of alkali hydroxid and ammonia defer the end point of the titration on account of the slight solubility of silver cyanid in these alkaline hydroxids. When a few drops of potassium iodid are added to the solution this source of error may be wholly avoided. In the presence of this salt the end of the reaction is indicated by a turbidity due to

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[1] *J. Am. Chem. Soc.*, 1915, **37**: 601.

[2] A. H. Allen. Commercial Organic Analysis. 4th ed., 1913, **7**: 481.

silver iodid, a compound which is not soluble in dilute alkali hydroxid and ammonia solutions. The use of iodid allows ammonia to be used instead of fixed alkali neutralizing free hydrocyanic acid."

This modification was tried. The distillate was received in a flask containing 20 cc. of 5% ammonium hydroxid. When the distillation was completed, 10 drops of 15% potassium iodid solution were added and the solution titrated with N/50 silver nitrate to the appearance of turbidity.

In all determinations, during the 2 hours' maceration, the flask was closed with a stopper carrying the inlet and outlet tubes for the steam distillation; the tubes were closed with corks. The additional 100 cc. of water was added through the steam-inlet tube after the flask had been attached to the condenser. In this way, loss of hydrocyanic acid while adding the water and attaching the flask to the condenser was avoided.

V. B. Bonney.—I have analyzed several import samples of Rangoon beans, using the Volhard method, without experiencing any difficulty.

J. R. Rippetoe.—I consider the Liebig method unreliable, due to the obscure end point, and the Volhard method satisfactory.

PART III.

The following report is a brief review of conditions observed in the past year in the interstate and import trade of crude drugs and spices:

INTERSTATE TRADE.

As a result of surveys of some of the crude drugs on the market, among other findings the following were observed:

(1) In a considerable number of instances spurious unofficial aconites had been substituted for aconite (*Aconitum napellus*), the chief adulterant being Japanese aconite (*Aconitum fischeri*). Some unofficial Indian aconites were also found. The substitutes do not contain aconitin but other alkaloids.

(2) *Chimaphila maculata* was found substituted for pipsissewa (*Chimaphila umbellata*).

In one instance false unicorn root (*Chamaelirium luteum*) was substitued almost entirely for true unicorn root (*Aletris farinosa*).

(4) *Aspidium aculeatum* and an *Osmunda* species, probably *Osmunda cinnamomea*, were found to be substituted for male fern or aspidium (*Dryopteris Filix-mas* or *marginalis*). The samples which consisted of true *Aspidium* were old and did not comply with the requirements of the United States Pharmacopoeia.

(5) Many domestic drugs were not carefully collected. They were frequently found to contain large amounts of dirt and other foreign material and often were mixed with other plants. For example, samples of pennyroyal leaves (*Hedeoma pulegioides*) have been found which were extremely dusty and contained as much as 20 per cent and more of sand; unicorn root (*Aletris farinosa*) has been found to contain as high as 15 per cent of sand. Pipsissewa leaves were found to consist

almost entirely of stems; 10 per cent of Koellia mutica was found in a sample of pennyroyal leaves.

IMPORT TRADE.

The following findings may be cited to show some of the striking examples of adulterations detected in imported crude drugs during the past year:

Inula britannica for arnica flowers (*Arnica montana*), *Solanum nigrum* for belladonna (*Atropa belladonna*), *Xanthium strumarium* for stramonium (*Datura stramonium*), *Lippia berlandieri* and *Origanum vulgare* for marjoram (*Marjoram hortensis*), *Heteropteris pauciflora*, *Ipecacuanha fibrosa* and *Ionidium* species for ipecac (*Cephaelis ipecacuanha*), *Ballota hirsuta* for horehound (*Marubium vulgare*), *Foeniculum piperitum* for fennel (*Foeniculum vulgare*), *Rheum rhaponticum* for rhubarb (*Rheum officinale*), etc. Up to 20 per cent of *Tephrosia apollinea*, containing a poisonous substance, were found in Tinnevelly senna (*Cassia angustifolia*). A fungus growth, resembling closely the sclerotium known as "ergot", has been found in caraway (*Carum carvi*) and cumin (*Cyminum cuminum*).

On the basis of certain studies the importation of some supplies obtained from new geographical or new botanical sources was encouraged; for instance, the importation of chamomile flowers and valerian root from Japan, as well as that of *Hyoscyamus muticus* for the manufacture of the alkaloid hyoscyamin.

PART IV.

Attention is called to the value of weights of unit volumes in the analysis of crude drugs and spices. While the weight of a certain volume is extensively used in grain standardization and, to some degree, by the trade in the judgment of pepper, recent findings in regard to areca nuts, nutmegs, fennel, etc., suggest that the weight of say 500 to 1000 cc. of crude drugs can often be used for an immediate judgment of authenticity or of inferiority due to worm infection, moldiness, immaturity, presence of foreign matter, etc.

This method, which obviously can be used only for drugs of rather uniform sizes, consists in filling a graduated cylinder up to the mark with the material of questionable quality, determining its weight, and repeating the same procedure with a sample of the drug of good quality. The sizes of the cylinders used vary from 100 to 1000 cc., depending on the size of the material. The few data given below indicate the application of this method:

TABLE 3.

Weights of unit volumes.

MATERIAL	VOLUME	WEIGHT
	cc.	*grams*
Fennel seed (*Foeniculum vulgare*)	100	35
Bitter fennel (*Foeniculum piperitum*)	100	26.7
Areca nuts (cleaned)	1000	582.5–597.5
Areca nuts (rejections)	1000	520.5–527.5
Black mustard (normal product)	500	370.0
Black mustard (rain damaged)	500	345.0
Charlock (adulterant of black and brown mustard)	500	350.0
White mustard	500	384.0
Chinese colza (adulterant of white mustard)	500	352.0

TABLE 4.

Examination of commercial samples of nutmegs.

NUMBER OF SAMPLES EXAMINED	QUALITY	WEIGHT PER BUSHEL*
		pounds
125	Good	40–50
16	Passable	30–40
5	Rejections	20–30
4	Rejections	0–20

* Determined with Boerner weight per bushel tester, described in U. S. Dept. Agr. Bull. 472: (1916).

RECOMMENDATIONS.

It is recommended—

(1) That the methods for the hydrolysis of linamarin and the subsequent determination of hydrocyanic acid be adopted as tentative methods.

(2) That the method for the determination of volatile oil in mustard seed and substitutes, especially with regard to the nature of the influence which alcohol exerts if added before maceration, receive further study.

(3) That new sources of supplies of proper substitutes for drugs not now obtainable be further investigated.

(4) That further work be done to determine the value of a more extended use of weights of unit volumes in the analysis of crude drugs and spices.

REPORT ON ALKALOIDS.

By H. C. Fuller (Institute of Industrial Research, Washington, D. C.), *Associate Referee.*

The referee has done some further work on the method for the determination of atropin in tablets, and recommends that the directions for drying the alkaloidal residue be changed so that they will read as follows: "Dry in vacuo to a constant weight, and weigh as atropin."

Quite a study has been made of methods for the quantitative separation and estimation of quinin and strychnin, with special reference to determining a method which will be accurate for the analysis of the class of mixtures which are ordinarily made in practice. In most of these mixtures, the quantity of quinin is greatly in excess of that of strychnin. Consequently, the problem becomes one of determining a minute quantity of strychnin in the presence of a large amount of quinin.

A number of methods have been considered, and have been tried out by the referee, but it is apparent that they must receive further study before it is safe to send them out to the collaborators.

No collaborative work was undertaken because of the above conditions.

RECOMMENDATIONS.

It is recommended—

(1) That the method for the determination of atropin in tablets, with the change mentioned above, be made provisional.

(2) That further work be done on the methods for separating quinin and strychnin, and that a method be submitted to the collaborators, which has a reasonable certainty of yielding concordant results.

(3) That the members of the association submit to the Associate Referee on Alkaloids suggestions for the study of important matters which may have developed in their practice.

No report on synthetic products was made by the associate referee.

No report on balsams and gum resins was made by the associate referee.

No report on enzyms was made by the associate referee.

The meeting adjourned at 5.20 p. m. for the day.

SECOND DAY.

TUESDAY—MORNING SESSION.

No report on saccharine products was made by the referee.

REPORT ON MAPLE PRODUCTS.

By J. F. **Snell** (Macdonald College, Quebec, Canada), *Associate Referee.*

Three samples of maple sirup and three of maple sugar were sent out. Sugar No. 4 was made in the laboratory from Sirup No. 1; Sugar No. 5, from Sirup No. 2; and Sugar No. 6, from Sirup No. 3. Dry basis results from the sugars should be comparable with those from the corresponding sirups.

PREPARATION OF THE SAMPLE.

The directions to collaborators called for a study of the desirability of adding water to all samples, whether sirup, sugar or other maple products, boiling to 104°C. (219°F.) and filtering after such concentration was completed. A comparison of the merits of filter paper, muslin, and cotton wool as filtering media was also requested.

The reboiling and filtering of sirups before analysis was recommended by C. H. Jones in 1905[1] and suggested to the association at its twenty-second annual convention[2]. The suggestion was endorsed by Bryan at the twenty-ninth convention[3]. Its whole-hearted adoption would simplify the directions materially.

S. F. Sherwood favors filtering after the product has been evaporated to the proper density and regards filter paper as a satisfactory medium. The experience of the writer is that filtration of the concentrated sirup through paper is too slow, since the sirup becomes more concentrated during the filtration. Muslin is rather an indefinite and perhaps too coarse a medium. The associate referee prefers cotton wool (absorbent cotton) loosely packed in the point of a funnel. This is also open to the charge of indefiniteness but the writer has found it satisfactory.

In one series of experiments, where different portions of the same hot sirup were poured upon filters of the three classes, 100 cc. ran through the muslin, and 50 cc. through the cotton wool, in 2 minutes, while it

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[1] Vt. Agr. Expt. Sta. Rept., 1905, 327.
[2] U. S. Bur. Chem. Bull. 99: (1906), 45.
[3] *Ibid.*, 162: (1913), 59.

took 14 minutes for 50 cc. to run through the filter of double S. & S. No. 597 paper. In another instance, 50 cc. ran through both the muslin and the cotton wool filter in 4 minutes but nearly 24 hours were required for the same quantity to run through the double filter paper. In still another instance, the quantities of sirup filtering through the three media in 1 minute were, respectively:

Muslin (double)	40 cc.
Cotton wool	25 cc.
Filter paper (single)	5 cc.

In almost every instance the portions of sirup filtered through the paper gave a higher refractometer reading than either of the other portions.

The few comparative experiments made upon the analytical values of samples filtered through the different media reveal no material differences. Thus, in Sirups Nos. 1 and 2, results for total ash (dry basis) are as shown in Tables 1 and 2.

TABLE 1.

Determination of total ash (dry basis) in Sirup No. 1.

ANALYST	COTTON WOOL	FILTER PAPER S. & S. NO. 597
	per cent	*per cent*
G. J. Van Zoeren, Macdonald College, Quebec, Canada	0.88	0.88*
	0.88	0.88*
	0.85	0.85*
	0.81	0.85*
Average	0.86	0.87
J. F. Snell	0.83	0.81†
	0.92	0.83†
	0.90	....
	0.84	....
	0.88	....
	0.86	....
Average	0.87	0.82

* Single.
† Double.

TABLE 2.

Determination of total ash (dry basis) in Sirup No. 2.

(Analyst, J. F. Snell.)

MUSLIN	COTTON WOOL	FILTER PAPER S. & S. NO. 597 (DOUBLE)
per cent	*per cent*	*per cent*
3.48	3.94	3.54
3.56	3.59	3.48
....	3.73	....
....	3.60	....
....	3.50	....
Average....3.52	3.67	3.51

The amount of work done upon this point is not sufficient to justify any recommendation other than a continuation of study.

MOISTURE BY REFRACTOMETER.

TABLE 3.

Determination of moisture by refractometer[1].

SIRUP NUMBER	ANALYST	
	Van Zoeren	Snell
	per cent	*per cent*
1	32.63	32.66
2	34.96	34.96
3	32.66	32.70

These determinations were made with the one instrument, a Féry refractometer made by Adam Hilger, London, England.

Results by one observer on ten samples using the Abbé and the Féry instruments were reported by Snell and Scott[2] in 1914 and showed close agreement.

Sherwood is of the opinion that the refractometer method is quite accurate in the case of maple sirup. A. H. Bryan[3] also pointed out the advantages of this method of estimating dry substance in maple sirup as well as in most other liquid saccharine products. In twelve out of thirteen samples he obtained higher results for moisture by the refractometer method than by drying 3 to 5 grams on 10 to 15 grams of sand in a flat-bottomed dish at 70°C. in a vacuum oven until the loss in 5 hours did not exceed 3 mg.

MOISTURE BY DRYING.

The method of drying on sand in a vacuum oven at 70°C., prescribed for honey and tentatively adopted for all maple products, is open to criticism on the grounds of indefiniteness as to the measure of the vacuum, the times for weighing and the degree of constancy to be attained; and the inappropriateness for maple products, which ordinarily contain but little levulose. The Laboratory of the Canadian Department of Inland Revenue follows the practice of drying at 100°C. to constant weight, having the sugar finely powdered and spread upon a watch glass, the sirup on asbestos fiber or sand. The two tentative methods of the association[4] for massecuites, molasses and other liquid and semi-liquid products, *i. e.*,

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 126.
[2] *J. Ind. Eng. Chem.*, 1914, **6**: 216.
[3] *J. Am. Chem. Soc.*, 1908, **30**: 1443.
[4] *Assoc. Official Agr. Chemists, Methods*, 1916, 121.

drying upon pumice at 70°C. (in the absence of levulose, 100°C.), and drying upon quartz sand at 100°C., may possibly be suitable for maple products.

None of these methods is regarded as quite satisfactory. With saccharine products, absolute constancy of weight is rarely, if ever, attainable by any method of oven drying and in none of the methods referred to are the conditions so closely defined as to yield concordant results in the hands of different analysts. In the case of sirups, the refractometer method forms a satisfactory substitute, not only giving much more accordant results, but also effecting great economy of time and labor. From the work of West[1] on sorghum sirup, it would appear that Danne's calcium carbide method possesses similar advantages. Both of these methods, however, require special apparatus, and the recognition of a direct drying method is perhaps unavoidable as a concession to the laboratory of modest resources. Such a method must, however, be much more closely defined than any of those at present in use. Provision must be made not merely for uniform temperature throughout the oven, but for uniform ventilation as well. In the determination of moisture in evaporated apples, J. A. Dawson (Laboratory of the Inland Revenue Department, Vancouver, B. C.) reports results differing by one to two units of percentage, depending upon whether the dish stood at the front or back of the shelf of an electrically heated oven of the Freas type. He also states that, in such an oven set for 100°C., he has observed variations from 97 to 103°C. in a calibrated thermometer laid horizontally on the shelf with its bulb in the back left corner. Such observations are suggestive of the need for further improvement of the apparatus available for the determination of moisture by loss of weight by drying.

The points indicated for study under this head were:

(1) Comparison of drying at 70°C. in a vacuum oven and at 100°C. in a water-jacketed or electrically heated oven.

(2) Quantity of sample to be used.

(3) Spreading material—sand, pumice or asbestos—and in the case of sugars, omission of spreading material.

EXPERIMENTS AT 100°C.

Dawson made preliminary experiments upon a standard 65 per cent by weight solution of cane sugar, prepared by shaking together at 30°C., 70 grams of water and 130 grams of commercial extra fine granulated pure cane sugar, previously ground to pass a 40-mesh sieve, and dried for 16 hours at 100 to 110°C. The sugar used gave, after the drying, a polarimeter reading of 99.8° Ventzke. The solution obtained

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[1] *J. Ind. Eng. Chem.*, 1916, 8: 31.

had a refractive index of 1.4509 at 28°C., as measured by the Abbé refractometer. By Geerlig's tables such a solution contains 64.52 per cent of sugar. The results obtained by drying 2 grams of this solution, plus 10 cc. of water, in a glass crystallizing dish 5.5 cm. in diameter and 3 cm. high in a Freas electric oven, set for 100°C., for exactly 4 hours, were as follows:

TABLE 4.

Moisture determined by different methods.

(Analyst, J. A. Dawson.)

SAMPLE NUMBER	SPREADING MATERIAL OMITTED	SAMPLE PLUS 15 GRAMS ACID-WASHED IGNITED SAND	SAMPLE PLUS 5 GRAMS IGNITED CHRYSOTILE ASBESTOS
	per cent	*per cent*	*per cent*
1	30.84	34.32	36.19
2	30.89	34.24	(35.46)
3	29.86	34.42	36.10
4	30.53	34.33	35.92

The sand used was fine enough to pass a 20-mesh, but not a 40-mesh sieve. It was stirred at the beginning of the experiment and after 1 and 2 hours.

In the case of asbestos No. 2, it was observed that the spreading material was not in contact with more than half of the bottom of the dish and evidently some of the solution was not distributed over the fiber.

Dawson infers from his results that drying with sand for 4 hours with stirring gives approximately accurate results, though possibly an additional 2 hours would be better, and that drying with asbestos for 4 hours gives results about 0.5 per cent higher than true results. He hopes to continue his investigation.

Van Zoeren, interpreting the term "to constant weight" literally, endeavored to realize an absolute constancy, or, at least, one not exceeding a change of 0.01 per cent of the original weight per hour. Finding in his experiments on Sirup No. 1 at 100°C. that such constancy was not obtainable within less than 40 hours at 100°C., he made his first weighings on the other sirups after 30 hours, his second after 40 hours, and his third after 50 hours. These experiments were made with a small electrically heated oven of Sargent's make, an oven exhibiting much greater variations of temperature than the Freas oven. Aluminium dishes 7.5 cm. in diameter and 1.8 cm. in depth were used in some cases, and dishes of 6.0 cm. × 1.5 cm. in others. The sand was washed with hydrochloric acid and ignited. The asbestos was tremolite, such as is ordinarily used in Gooch crucibles. In all cases, 5 grams of sirup were weighed in a sugar dish and transferred to the tared dish with a small quantity of distilled water. The results at 40 hours are given in Table 5. With one exception, they show lower percentages of dry matter than were obtained by the refractometer but, considering the long period of heating, the differences are remarkably small.

TABLE 5.

Determination of dry matter in sirups.

(Analyst, G. J. Van Zoeren.)

SAMPLE NUMBER	DRIED 40 HOURS AT 100°C. UNDER ATMOSPHERIC PRESSURE. SPREADING MATERIAL			DRIED 100 HOURS AT 70°C. UNDER REDUCED PRESSURE. SPREADING MATERIAL			DRY MATTER BY REFRACTOMETER
	Tremolite asbestos	Sand	Pumice stone	Asbestos	Sand	Pumice stone	
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
1............	66.84	66.62	67.69	67.42	67.36	68.29	67.37
	66.79	66.69	67.76	67.78	67.34	68.24	....
Average.....	66.81	66.66	67.73	67.60	67.35	68.27	67.37
2............	64.89	65.18	64.80	64.90	65.19	66.12	65.04
	64.98	64.71	64.69	64.87	64.76	64.32	....
Average.....	64.94	64.95	64.75	64.89	64.98	65.22	65.04
3............	67.05	67.01	66.80	67.32	66.99	67.82	67.34
	67.25	67.05	67.52	67.95	67.11	67.46	....
Average.....	67.15	67.03	67.16	67.64	67.05	67.64	67.34

TABLE 6.

Mean variations of individual results by drying from refractometer results.

SAMPLE NUMBER	AT 100°	AT 70°	SPREADING MATERIAL	AT 100°	AT 70°
1............	±0.54	±0.38	Asbestos	−0.28	±0.23
2............	±0.21	±0.42	Sand	±0.43	±0.18
3............	±0.29	±0.30	Pumice	±0.34	±0.70

The drying experiments conducted by the writer on sirups at 100°C. were confined to a single series in which a reboiled and cotton-wool filtered sample of Sirup No. 1 was dried on the three spreading materials and samples of Sirup No. 2, also reboiled and refiltered, were dried on asbestos. All the portions were dried at once in the same Sargent oven that Van Zoeren used, and the weight of sample and method of weighing were the same as his. For Sirup No. 2 and for the second of each pair of duplicates on Sirup No. 1, 7.5 cm. aluminium dishes were used. In all cases, the drying was more rapid in the 7.5 cm. than in the 6 cm.

dish. Weighings were made after 5, 7, 8, 9, 10, 11, 14 and 16 hours. In the final 2 hours of drying the loss of weight per hour was less than 0.1 per cent in all the 7.5 cm. dishes and in all 6 cm. dishes except the one in which Sirup No. 1 was dried on asbestos. In that dish and the two pumice dishes, the percentages of residue at the end of the 16 hours were decidedly higher than those deduced from the refractometer observations. The other asbestos portion and the two portions dried on sand gave results according closely with the refractometric indication (Table 7). On the other hand, the two sirups prepared from Sample No. 2 and dried on asbestos gave results for dry matter which, even at the end of 5 hours' drying, were lower than those derived from the refractometer readings. Considering the inadequate control of conditions, the discordance of the results obtained, and the fact that the sirups used were not the original collaborative samples, it does not appear worth while to burden the report with the details.

TABLE 7.

Determination of dry matter in a prepared sample of Sirup No. 1 (16 hours at 100°C. under atmospheric pressure).

(Analyst, J. F. Snell.)

MEDIUM	SIZE OF DISH	RESIDUE
	cm.	*per cent*
Asbestos	6.0	67.21
Asbestos	7.5	66.96
Pumice	6.0	67.56
Pumice	7.5	67.30
Sand	6.0	66.70
Sand	7.5	66.62
By refractometer	...	66.79

One point, however, must be mentioned. Among the fifty weighings made after the ninth hour of drying, no less than twelve showed an increase of weight, instead of a decrease. As the dishes were always covered during the weighings and as other dishes cooled in the same desiccator showed no similar increase, and, furthermore, as similar results were later obtained with the sugars, the only conclusion to be reached is that simultaneously with the elimination of water some chemical change (possibly an oxidation) resulting in an increase of weight is taking place. This is a question that deserves further study.

EXPERIMENTS AT 70°C.

The experiments at 70°C. were carried out in the Freas oven, a vacuum desiccator connected with a filter pump through a large safety-bottle being used as the vacuum chamber. In the experiments conducted by the writer, a slow current of air, dried by passing through sulphuric acid, was allowed to flow through the chamber, and the pressures, as measured by a mercury manometer connected between the safety bottle and the vacuum chamber, were from 60 to 160 mm. Van Zoeren used the same oven and pump but a larger desiccator and admitted no current of air. He did not measure the pressures.

Van Zoeren's results at the end of 100 hours are given in Table 5. The average results are closer to the refractometer indications than are those obtained by drying 40 hours at 100°C. But when the individual results are examined, as is done in Table 6, there is but little choice between the two methods.

The only experiment conducted by the writer with sirup at 70°C. was made with a reboiled and cotton-wool filtered portion of Sample No. 3. This gave 66.04 per cent of total solids by the refractometer, and the residues after 18 hours' heating (which did not result in absolute constancy) were 66.18 and 66.32 per cent in two 7.5 cm. dishes, and 66.40 per cent in a 6 cm. dish.

With sugars, constant weight was realized by both experimenters in about 48 hours but results were not concordant. These results, as well as those obtained with the sugars at 100°C.. are omitted on account of such discordance.

WINTON LEAD NUMBER.

The points indicated for study in reference to the Winton method were:

(1) The advisability of substituting 25 grams of cane sugar sirup for the few drops of acetic acid in the blank determination[1].

(2) The advisability of reducing the results to the dry matter basis[2].

The results of the collaborative work are shown in Table 8. The basic acetate solution was prepared from Horne's salt. In the experiments conducted by the writer the acetic acid blank was treated with a few drops of 2N acetic acid. On diluting, slight clouding ensued, but the weights of lead sulphate obtained by precipitation after settling were practically identical with those obtained from the cane sugar blanks, which remained quite clear. Van Zoeren, who added enough acetic acid to prevent precipitation, obtained 2.2 mg. less lead sulphate from these blanks than from the cane sugar blanks.

The results may be interpreted as slightly favorable to the use of the cane sugar sirup. This has also a logical advantage over acetic acid, in that the substance added is identical with that which, in the case of the

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[1] *J. Ind. Eng. Chem.*, 1913, 5: 997.
[2] *Ibid.*, 1914, 6: 221.

TABLE 8.

Collaborative results on Winton lead number.

SAMPLE NUMBER	ACETIC ACID BLANK		CANE SUGAR BLANK	
	Van Zoeren	Snell	Van Zoeren	Snell
1	1.82	About 0.01 per cent	1.92	1.89
	1.84		1.94	1.89
	1.83		1.93	1.88
	....		....	1.88
	....		....	1.89
4	1.73		1.79	2.07
	1.81		1.91	2.08
	1.77		1.85	2.08
2	2.90	lower than with the cane sugar blank	2.99	2.29
	2.82		2.91	2.31
	2.86		2.95	2.47
	....		....	2.47
	...		....	2.39
5	2.23		2.33	2.50
	2.22		2.30	2.47
	2.23		2.31	2.49
3	2.36	......	2.45	2.43
	2.38	......	2.48	2.40
	2.37	......	2.47	2.42
6	2.41	......	2.56	2.39
	2.38	......	2.47	2.36
	2.39	......	2.52	2.38

maple sirup, prevents the precipitation of the basic acetate. If it were adopted, the directions would be to make a blank determination, using 25 cc. of a pure cane sugar sirup (sp. gr. 1.320) in place of the maple sirup.

The question of the advisability of abandoning the reduction of Winton lead numbers to the dry basis is one which does not permit of solution upon the basis of work on a few samples. The reduction to dry basis was not a part of the original method of Winton and Kreider[1] and, as has been shown by Snell and Scott[2], the range of variation of wet basis Winton numbers in genuine maple sirups is narrower than that of the dry basis numbers. The wet basis number is therefore a sharper criterion for the detection of adulteration than the dry basis.

Sherwood favors the retention of the reduction to dry basis on the grounds that the difference of range is not great; analyses of maple products published by the Bureau of Chemistry and certain other investigators and frequently used as bases of comparison are stated on the

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[1] *J. Am. Chem. Soc.*, 1906, 28: 1204.
[2] *J. Ind. Eng. Chem.*, 1913, 5: 997.

dry basis, and to discontinue the calculation of the Winton number to dry basis would tend to confusion. Opinions have not been expressed by other collaborators.

The directions of the Winton lead method should be closely followed to insure correct results. The sugar solution must not be warm nor can it be poured into the lead subacetate. The following parallel results on Sample No. 5 illustrate this point:

TABLE 9.

Determination of lead number on Sample No. 5.

DESCRIPTION OF METHOD	LEAD NUMBER
Regular method	2.49
25 cc. of subacetate solution, measured into a flask, sirup added and washed in with water	2.69
The diluted sirup heated to 60°C. and the subacetate added	2.26

CANADIAN LEAD NUMBER.

The Laboratory of the Canadian Inland Revenue Department has for many years used this value as the chief criterion for the discrimination of genuine and adulterated maple products. The directions for its determination, as approved by A. Valin of that laboratory, are as follows:

> Weigh the quantity of sirup containing 25 grams of dry matter, transfer to a beaker, add 50–75 cc. of water, boil gently for 2–3 minutes, transfer to a 100 cc. flask, cool and make up to the mark.
>
> Pipette 20 cc. of this solution into a large test tube, add 2 cc. of lead subacetate solution (sp. gr. 1.26) and mix. Allow to stand 2 hours, filter through a tared Gooch, wash four or five times with boiling water, dry at 100°C. and weigh. Multiply the weight of the dry precipitate by 20.

The points indicated for study were:

(1) The advisability of weighing 25 grams of sirup instead of the quantity of sirup containing 25 grams of dry matter.

(2) The necessity for boiling the diluted sirup if all samples have been boiled in the preparation for analysis.

(3) The advisability of substituting 1.25 for 1.26 as the density of the subacetate solution.

(4) The advisability of defining the volume of wash water and allowing more license as to temperature[1].

(5) Variations between duplicates.

The collaborative results are given in Table 10. Van Zoeren and the writer used a subacetate solution (sp. gr. 1.25) prepared from Horne's

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[1] *J. Ind. Eng. Chem.*, 1913, **5**: 996.

salt. They also used a 100 cc. beaker in place of the large test tube. The subacetate solution was added from a burette. The writer washed the solution as directed without close attention to the quantity of wash water. Van Zoeren always washed the solution with exactly 100 cc. of boiling water. It should be noted that the mat of asbestos in the Gooch crucible in this determination needs to be heavier than for most other precipitates. Inattention to this detail and to the exact volume of the wash water may possibly account for the inferior agreement of the writer's duplicates as compared with those of the other collaborators.

TABLE 10.

Collaborative results on Canadian lead number.

SAMPLE NUMBER	COLLABORATORS			USING 25 GRAMS SIRUP AND CALCULATING TO DRYNESS
	Valin	Van Zoeren	Snell	Van Zoeren
1	2.20	2.51	2.55	1.96
	2.24	2.57	2.04	1.93
	2.20	2.49	2.28	1.74
	2.18	....	....	1.68
Average	2.20	2.52	2.29	1.83
4	2.22	2.52	2.24	1.69
	2.22	2.48	2.23	1.72
	2.08	....	....	....
	2.22	....	....	....
Average	2.19	2.50	2.24	1.71
2	3.22	4.04	4.30*	3.76
	3.24	4.01	3.94	3.56
	3.16	4.07	3.59	3.40
	3.16	3.92	3.73	3.46
Average	3.19	4.01	3.75	3.54
5	2.84	3.12	3.12	2.83
	2.74	3.15	3.35	....
	2.80	....	....	....
	2.70	....	...	....
Average	2.77	3.14	3.24	2.83
3	3.22	3.31	3.52	3.16
	3.06	3.36	3.42	3.08
	3.10	3.32	....	3.18
	3.20	3.32	....	3.16
Average	3.14	3.33	3.47	3.15
6	3.16	3.32	3.33	3.07
	3.14	3.29	3.23	3.17
	3.08	....	....	....
	3.06	....	....	....
Average	3.11	3.31	3.28	3.12

* Not included in average.

Valin admits that it would be more convenient to weigh 25 grams of sirup instead of 25 grams of dry matter, but states that the Laboratory of the Canadian Inland Revenue Department follows the other procedure in order to have a uniform standard for sirup and sugar. In the process of this association in which the sugar is converted into sirup for analysis, this object is attained in another way, and the only objection to adopting the procedure with 25 grams of sirup is that the results so obtained would not, even after reduction to a dry basis, be comparable with those which the Laboratory of the Canadian Inland Revenue Department now publishes. On the other hand, they would be comparable with the results which that laboratory has published upon a large number of genuine sirups[1]. This is not made clear in the publication itself, but in answer to an inquiry, the Chief Analyst advised the associate referee under date of February 17, 1913, that the lead determinations were made on 5 grams of the sirup, and the results were later calculated to a dry basis.

The wide difference which usually exists between dry basis results obtained with the use of a definite weight of sirup and those obtained with the quantity of sirup containing that definite weight of dry matter has been pointed out by Snell and Scott[2] and is further illustrated by results obtained by Van Zoeren on the present collaborative samples, particularly on Samples 1 and 4 (Table 10). In all cases, his results, using 25 grams of sirup, are lower than those using 25 grams of dry substance. This is consistent with the work of Snell and Scott and is what one would expect in view of the solvent action of sugar upon the precipitate. It is notable, however, that in four instances out of six, the results with 25 grams of sirup are equal to or greater than Valin's results obtained with the use of 25 grams of dry substance. This is doubtless only a detail of the more general fact that Valin's results as a whole are lower than those of the other collaborators. Whether this is due to a difference in the lead subacetate solutions used, or to some variation in the procedure, is not known.

The only results bearing on the question of the necessity of boiling the diluted sirups when they have been previously boiled in the preparation of the sample are those of the associate referee on Sirup No. 2. A portion of this sirup, prepared for analysis by boiling to 104°C. and filtering through cotton wool, gave 3.60 and 3.50 as the Canadian lead number without reboiling, and 3.22 and 3.44 when the sirup was reboiled after dilution.

Valin is of the opinion that the use of subacetate solution of specific gravity of 1.25 instead of 1.26 would make no difference in the results.

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[1] Can. Lab. Inland Rev. Dept., Bull. 228: (1911).

[2] *J. Ind. Eng. Chem.*, 1913, 5: 997.

Since the former strength is now commonly used in clarifying for the polariscope, its use is to be recommended on the score of convenience.

Further study on the Canadian lead method is strongly recommended since this method is so simple and possesses the advantage that in adulterating with refined sugar the values fall off more rapidly than the percentage of maple sirup[1].

ASH VALUES.

Van Zoeren and the writer have done considerable work on the ash values but as no results from collaborators were received our results are reserved for later publication.

OTHER METHODS.

The electrical conductivity method[2] should be further tested before it receives official recognition. It is a very simple, rapid test. In the writer's laboratory, it has given results of a more restricted range in genuine sirups than any of the recognized methods. For collaborative purposes, it might perhaps be well to dilute the sirups to a more definite sugar content than is directed for the rapid test, but whether the increase of accuracy would compensate for the loss of time is a point that would require study.

The volumetric lead method[3] has not given satisfactory results with the present samples. With sirups of high quality, there is sometimes room for difference of opinion as to the plotting of the graphs.

RECOMMENDATIONS.

It is recommended—

(1) That work on the preparation of the sample be continued with a view to revision of the directions.

(2) That collaborative work be done on the Winton lead number.

(3) That the Canadian lead number and conductivity value methods be further studied collaboratively with a view to their adoption.

(4) That work on the determination of moisture and ash be resumed when these topics are under study in reference to other saccharine products.

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[1] *J. Ind. Eng. Chem.*, 1913 5: 995.
[2] *Ibid.*, 1916, 8: 331.
[3] *Ibid.*, 1916, 8: 241.

REPORT ON HONEY.

By SIDNEY F. SHERWOOD[1] (Bureau of Chemistry, Washington, D. C.), *Associate Referee.*

The report includes a study of honeydew honeys, mixtures of honeydew and glucose, and normal honey and glucose with the object of ascertaining methods of differentiating between honeydew and normal honeys and mixtures of these products containing glucose (this problem is discussed at length by Browne[2]). It will be noted that honeydew exhibits a high plus polarization at 20°C. and at 87°C., both before and after inversion.

Since it was observed that normal honey usually gives a very slight precipitate with basic lead acetate, while honeydew usually gives a very copious precipitate (glucose giving a very slight or no precipitate), it was thought that a study of the application of the Winton lead number[3] might prove this determination to be of value. The application of this determination to typical honeydew honeys gave values at first of 2.81 to 3.06. However, in one case, a value of 0.32 was found and, as this is lower than the values found in certain normal honeys, the investigation of the lead number was discontinued.

Since König and Karsch[4] called attention to the fact that after precipitating the dextrins with absolute alcohol, natural honeys exhibit levorotation while honeys containing 25 per cent or more of glucose exhibit dextrorotation, and since this method was found of confirmatory value by Browne[2], it has been applied to various honeydew honeys and mixtures of honey with glucose with the following results:

Polarization of honeydew honeys before precipitation with alcohol.*

SAMPLE NUMBER	DESCRIPTION OF SAMPLE	DIRECT 20°C.	INVERT	
			20°C.	87°C.
1	Honeydew	+ 6.80	+ 5.80	+26.00
2	Honeydew	+ 5.70	+ 2.00	+23.40
3	Honeydew	+13.40	+ 9.20	+30.60
4	Honeydew	+14.70	+10.40	+31.60
5	Honeydew No. 1 + 20 per cent glucose	......	......	......
6	Honeydew No. 2 + 20 per cent glucose	......	......	......

* All polarization figures are stated on a basis of 20 grams of the original honey in 100 cc. of water; readings in a 200 mm. tube.

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[1] Present address, Bureau of Plant Industry, Washington, D. C.
[2] U. S. Bur. Chem. Bull. **110**: (1908).
[3] *J. Am. Chem. Soc.*, 1906, **28**: 1204.
[4] *Z. anal. Chem.*, 1895, **34**: 1.

Polarization[*] *of honeydew honeys and mixtures of honeydew honeys and glucose after precipitation with alcohol.*

SAMPLE NUMBER	DESCRIPTION OF SAMPLE	DIRECT 20°C.	INVERT	
			20°C.	87°C.
1	Honeydew	−2.70	−3.52	+2.09
2	Honeydew	−3.30	−4.24	+1.98
3	Honeydew	−2.20	−2.80	+1.98
4	Honeydew	−2.00	−2.70	+2.42
5	Honeydew No. 1 + 20 per cent glucose	+0.75	+0.28	+3.19
6	Honeydew No. 2 + 20 per cent glucose	+0.30	−0.33	−2.86

[*] All polarization figures are stated on a basis of 26 grams of the original honey in 100 cc. of water; readings in a 200 mm. tube.

In view of the slight values for the plus polarizations of the mixtures and of the small variation between these values and the values for the honeydew honeys, it is thought that this method, in the case of the addition of small amounts of glucose, is of minimum value only.

Investigation of the precipitate thrown down by alcohol was begun but, owing to lack of time, was discontinued. It is believed that a study of this precipitate may prove of value in connection with the problem.

No report on sugar house products was made by the associate referee.

No report on food preservatives was made by the referee.

REPORT ON COLORING MATTERS IN FOODS.

By W. E. Mathewson (Bureau of Chemistry, Washington, D. C.), *Referee.*

The work was restricted to the consideration of tests for some of the more common natural coloring substances. The qualitative differentiation of the various natural coloring matters found in food products can scarcely be carried out satisfactorily until much more work has been done, but many tests are in use that have been found reliable and convenient. The Committee on Editing Methods of Analysis arranged some of the best known of these tests in the form of a table[1]. This table is somewhat incomplete, chiefly where tests are concerned in which no definite color change takes place.

It was requested that the collaborators make tests with such samples of coloring matters as were available, so that for every case a definite

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 166.

statement might be given in the table, concerning the behavior of the reagents and coloring matters with each other. Attention was also called to tests depending on the treatment of an acetic anhydrid solution of the coloring matter with concentrated sulphuric acid and with other reagents; and to the work of Palmer and Thrun[1] relative to the behavior of the natural coloring matters of butter and oils with ferric chlorid and other reagents.

The following comments were received from Leonard Feldstein, U. S. Food and Drug Inspection Station, Tabor Opera House Building, Denver, Colo.:

> The results obtained in the cooperative work on food colors at this laboratory are as follows:
>
> Hydrochloric acid with alkanet produces no change. Sodium hydroxid with annatto causes fading of the yellow color and the production of a light brown color.
>
> Ferric chlorid decolorizes alkanet, leaving a turbid dark solution. It produces no change with carotin, and turns anthocyans (cherries) deep purple.
>
> Alum solution produces a slight purple color with alkanet; turns cochineal and anthocyans (cherries) purple-red, and has no action on annatto, carotin or caramel.
>
> Uranium acetate with Brazil wood produces a deep purple-red color; no change is noticed with annatto or caramel.
>
> Acetic anhydrid and sulphuric acid produce a green fluorescence in the red color of Brazil wood; a yellow color is produced with cochineal. No action is noticed with caramel.
>
> It would appear that reference should be made in the tentative methods to the tests for the detection of carotin in butter since the action of ferric chlorid and carotin, whether alone or in the presence of fats and oils, appears to be different from that in aqueous solution. Water seems to retard the reducing action of carotin on ferric chlorid; at least no change is visible. When a crystal of ferric chlorid is added to dry carotin extract, the ferric chlorid is changed. If water is then added to the mixture, a wine colored solution is produced.

REPORT ON METALS IN FOODS.

By DAVID KLEIN[2] (Division of Foods and Dairies, Illinois Department of Agriculture, 1410 Kimball Building, Chicago, Ill.), *Referee.*

TIN.

A study was made of a volumetric method for tin, the essential features of which were suggested by W. B. D. Penniman, Baltimore, Md. Broadly outlined, the method consists of extracting the tin with hydrochloric acid, the tin is precipitated from this solution by zinc, the mixed metal residue is dissolved in hydrochloric acid in the absence of air, and this solution is titrated with standard potassium iodate. The advantages over the provisional methods are the elimination of the acid digestions

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[1] *J. Ind. Eng. Chem.*, 1916, 8: 614

[2] Present address, The Wilson Laboratories, Chicago, Ill.

and of the sulphid precipitation. Furthermore, potassium iodate solution maintains a constant strength, and is, therefore, preferable to the variable iodin solution used in the provisional methods.

The oxidation of stannous chlorid by potassium iodate is an interesting reaction, which may be assumed to occur in the following steps:

$$\begin{array}{llcl}
(1) & 4\,KIO_3 + 12\,SnCl_2 + 28\,HCl & = & 12\,SnCl_4 + 4\,HI + 12\,H_2O + 4\,KCl \\
(2) & KIO_3 + 6\,HCl & = & ICl + 4\,Cl + 3\,H_2O + KCl \\
(3) & 4\,HI + 4\,Cl & = & 4\,HCl + 4\,I \\
(4) & KIO_3 + 4\,I + 6\,HCl & = & 5\,ICl + 3\,H_2O + KCl \\
\hline
 & 6\,KIO_3 + 36\,HCl + 12\,SnCl_2 & = & 12\,SnCl_4 + 6\,ICl + 18\,H_2O + 6\,KCl \\
 & KIO_3 + 6\,HCl + 2\,SnCl_2 & = & 2\,SnCl_4 + ICl + 3\,H_2O + KCl
\end{array}$$

If equation (1) goes to completion before the action represented by equation (2) begins, then it will be possible to use starch as an end point indicator, for, as soon as reaction (1) is completed, further addition of potassium iodate will liberate free chlorin. This, in turn, will displace the iodin from the hydriodic acid of equation (1), according to equation (3). Continued addition of potassium iodate will convert the liberated iodin into iodin monochlorid. This action can be traced by the decolorizing of chloroform, since iodin monochlorid does not impart color to chloroform. Thus two end points may be used. However, it has been found that the chloroform end point is not always satisfactory. With certain lots of zinc, the pink color of the chloroform was not discharged, even when a large excess of potassium iodate was added. At times the coloring matter of the original food material was carried along, and imparted a decided color to the final solution. In such cases, the chloroform end point was unreliable. For these reasons, the chloroform end point was discarded.

The sensitiveness of the starch end point is dependent upon the concentration of the iodin liberated in equation (3), since an appreciable concentration of iodin is necessary to develop the color in starch[1]. Where a small amount of tin is being titrated, it is conceivable that the amount of hydriodic acid formed would be insufficient to liberate the minimum quantity of iodin necessary for the development of the blue color with starch. This condition should be remedied by increasing the concentration of hydriodic acid, as by the addition of potassium iodid. Then the sensitiveness will be dependent only upon the concentration of the chlorin derived from equation (2). These theoretical considerations have been fully verified experimentally. For example, no blue color is formed, when solutions containing as much as 4 mg. of tin in 125 cc. of liquid are titrated with potassium iodate (1 cc. = 0.001 gram of tin). On the other hand, when potassium iodid was added, a sharp end point was obtained with as little as 0.0001 gram of tin.

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[1] *J. Am. Chem. Soc.*, 1908, **30:** 45.

TABLE 1.

Effect on starch end point of the addition of potassium iodid.

ZINC POWDER	ZINC USED	CONCENTRATED HYDROCHLORIC ACID USED	TIN ADDED	POTASSIUM IODID ADDED	TIN RECOVERED	CORRECTED FOR BLANK
	grams	*cc.*	*gram*	*mg.*	*gram*	*gram*
E	6	25	0.0030	50	0.0035	0.0029
E	6	25	0.0000	50	0.0006	0.0000
E	6	25	0.0010	50	0.0016	0.0010
D	6	25	0.0000	100	0.0001	0.0000
D	6	25	0.0003	100	0.0004	0.0003
E	6	20	0.0050	100	0.0055	0.0049
E	6	25	0.0100	100	0.0104	0.0098
E	6	25	0.0100	100	0.0104	0.0098

TABLE 2.

Effect of varying conditions of temperature and concentration of acid on the amount of tin precipitated.

VOLUME	TIN ADDED	TIN RECOVERED	CORRECTED FOR BLANK	REMARKS
cc.	*gram*	*gram*	*gram*	
100	0.0000	0.00095	0.0000	Precipitated hot, 70–80°C., stood overnight.
100	0.0000	0.0010	0.0000	
85	0.0200	0.0202	0.0193	
85	0.0200	0.02035	0.0195	
85	0.0050	0.0057	0.0048	Precipitated hot, stood 1 hour, 70–80°C.
150	0.0400	0.0404	0.0395	Boiled 1 hour.
150	0.0400	0.0404	0.0395	
150	0.0400	0.0408	0.0399	
150	0.0400	0.0408	0.0399	
150	0.0400	0.0408	0.0399	
150	0.0400	0.0408	0.0399	
150	0.0400	0.0409	0.0400	
150	0.0400	0.0406	0.0397	Precipitated near 100°C.
150	0.0400	0.0404	0.0395	
150	0.0400	0.0404	0.0395	
150	0.0400	0.0409	0.0400	
150	0.0400	0.0410	0.0401	Diluted with 100 cc. of air-free solution before titration.
150	0.0400	0.0410	0.0401	
250	0.0000	0.0006	0.0000	0.10 gram of ferric chlorid diluted to 100 cc. air-free solution before titration.
250	0.0000	0.0005	0.0000	
250	0.0400	0.0400	0.0395	0.10 gram of ferric chlorid. Boiled 1 hour.
250	0.0400	0.0406	0.0400	
250	0.0400	0.0405	0.0400	
125	0.0010	0.0000	0.0000	Titrated without potassium iodid.
125	0.0020	0.0000	0.0000	
125	0.0030	0.0000	0.0000	
125	0.0050	0.0060	0.0051	

The experiments in Table 2 show the efficiency of zinc in precipitating tin from an aqueous solution acidified with hydrochloric acid, under different conditions of concentration and temperature of precipitation. Twelve grams of the same lot of zinc powder were used for each experiment. Where ferric chlorid is indicated, it means that tin was precipitated in its presence. In each case 24 cc. of hydrochloric acid were used.

PROPOSED VOLUMETRIC METHOD.

REAGENTS.

(a) *Standard tin solution.*—Dissolve 1 gram of tin, hammered into a thin ribbon and cut into narrow strips, in 150 cc. of concentrated hydrochloric acid. Make up to 1 liter with water.

(b) *Standard potassium iodate solution.*—Dissolve 0.6010 gram of the pure salt in water and make up to 1 liter. One cc. of this solution is equivalent to 1 mg. of tin.

(c) *0.5% potassium iodid solution.*

(d) *Air-free wash solution.*—Dissolve 20 grams of sodium bicarbonate in 2 liters of boiled water and add 40 cc. of concentrated hydrochloric acid. This solution should be freshly prepared before use.

(e) *Starch paste.*—Preferably prepared as directed by Treadwell-Hall[1].

(f) *Zinc (20 mesh powder).*—Its efficiency in completely precipitating tin from solutions containing organic matter must be determined for each lot. A blank should be run on all lots.

DETERMINATION.

Fifty grams of the sample are usually sufficient. Digest at 70°C. for an hour with an equal bulk of concentrated hydrochloric acid. If the sample contains much sugar, the digesting liquid should not contain more than 10 per cent of acid. Dilute sufficiently (about one-half) to prevent hydrolysis of the filter paper, transfer the mass to a large Büchner funnel and filter by suction. Return the filter paper and residue to the original beaker, redigest with half the volume of concentrated acid originally used. Filter as before, wash the residue with hot dilute hydrochloric acid, unite the filtrates and proceed as follows:

Neutralize the filtrate with strong ammonia in a 1000 cc. beaker, add 20–24 cc. of concentrated hydrochloric acid and heat to 70–80°C. on the water bath, then add 12 grams of zinc. Allow the action to run almost to completion or until only a very small amount of hydrogen is evolved. Remove the beaker from the water bath and add ammonium hydroxid until zinc hydroxid just persists, or to alkalinity if no precipitate forms. Filter the remaining zinc and precipitated tin through a Caldwell crucible containing an asbestos pad. Wash the metallic residues with hot water made slightly ammoniacal.

Transfer the detachable asbestos pad and residue to a 300 cc. Erlenmeyer flask, the crucible being wiped out with a moist piece of asbestos. Attach the flasks in duplicate, as described below, to a large carbon dioxid generator[2] or, more conveniently, to a tank of liquid carbon dioxid. Pass the gas through a scrubber containing water, and divide into two branches by means of a Y tube. A stream is led into each flask by means of rubber tubing and a bulbed glass tube so adjusted in the 2-holed stopper that its lower end shall be near the surface of the liquid which is to be subsequently added.

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[1] F. P. Treadwell. Analytical Chemistry. Quantitative Analysis. Translated from the German by William T. Hall. 5th ed., 1904, 2: 513.

[2] A slight modification of the apparatus described in *J. Assoc. Official Agr. Chemists*, 1915, 1: 258.

The carbon dioxid leaves the flask by a second bulbed tube, the opening of which is near the top of the flask. This glass tube is connected by a long rubber tube to a second glass tube, preferably a reversed adapter, which is immersed in a cylinder containing water. A 50 cc. dropping funnel is passed through the stopper of each flask. After the flasks are connected, displace the air by a vigorous stream of carbon dioxid, then run in, cautiously to avoid excessive foaming, 50 cc. of concentrated hydrochloric acid through the dropping funnel. As soon as the action is over, heat the flasks to boiling while a steady stream of carbon dioxid is being passed through the apparatus. A convenient means of heating the flasks is a hot plate, made from an ordinary skillet and a Jewel burner. As soon as all the metallic particles are dissolved, remove the hot plate and substitute a cooling bath of tap or ice water.

The stream of carbon dioxid should be momentarily increased, in order to prevent back suction. When cool, disconnect the flasks one at a time, and wash the tubes, stopper and sides of the flasks with air-free wash solutions. If the contents of the flasks are dark and make the end point uncertain, dilute with air-free wash solution, add 4–5 cc. of potassium iodid and 20 cc. of starch solution. Titrate at once with standard potassium iodate to a strong blue color.

The method was applied to various food products, with results indicated in the following tables. Considerable difficulty was experienced with sugar solutions, owing to the formation of a black gummy precipitate during the precipitation of the tin by the zinc. This was obviated in a large measure by reducing the amount of hydrochloric acid. When the product is highly colored, it often happens that the coloring matter is carried through the entire procedure, and may mask the blue color of the end point. However, the addition of potassium iodid permits of liberal dilution of the solution, and the production of a satisfactory end point.

EXPERIMENTAL RESULTS.

SUGAR.

TABLE 3.

Determination of added tin in simple sugar solution.

SUGAR	CONCENTRATED HYDROCHLORIC ACID	TOTAL VOLUME	TIN	
			Added	Found
grams	*cc.*	*cc.*	*gram*	*gram*
20	20	440	0.07932	0.0783
20	20	440	0.07932	0.0780
20	6.7	140	0.03966	0.03936
20	20	420	0.03966	0.03936
20	20	420	0.03966	0.03990
20	20	420	0.03966	0.0396

POTTED CHICKEN.

Fifty grams portions of the same lot of thoroughly mixed material were used.

TABLE 4.

Determination of tin in canned potted chicken by the proposed method with a study of extraction details.

FIRST EXTRACTION TIN FOUND	SECOND EXTRACTION TIN FOUND	THIRD EXTRACTION TIN FOUND	TOTAL TIN FOUND	ADDED TIN	RECOVERED TIN	REMARKS
gram	*gram*	*gram*	*gram*	*gram*	*gram*	
0.00471	0.00048	0.00006	0.00525	......	......	First extraction 200 cc. of hydrochloric acid (1 to 1); second and third extractions 100 cc. of the same solution.
0.00426	0.00036	0.00006	0.00468	......	......	
0.00456	lost	0.00012	......	......	......	
0.0430	0.0008	......	0.0438	0.03966	0.0388	First extraction 200 cc. of hydrochloric acid (1 to 1); second extraction 100 cc. of the same solution.
0.0433	0.0008	......	0.0441	0.03966	0.0391	
0.0430	0.0007	......	0.0437	0.03966	0.0387	
......	......	......	0.02436	0.01938	0.01936	Two extractions only, each with 100 cc. of hydrochloric acid (1 to 1). Tin was determined in the combined filtrates.
......	......	......	0.05436	0.04963	0.04936	

To 50 gram portions of the same material, potted chicken, varying amounts of tin, 2 to 20 mg., were added.

TABLE 5.

Determination of tin in canned potted chicken by the tentative volumetric method.*

TIN ADDED	FOUND	DIFFERENCE	TIN ADDED	FOUND	DIFFERENCE
gram	*gram*	*gram*	*gram*	*gram*	*gram*
0.02000	0.02493	0.00493	0.01000	0.01578	0.00578
0.02000	0.02438	0.00438	0.00500	0.00921	0.00421
0.02000	0.02370	0.00370	0.00500	0.00978	0.00478
0.02000	0.02395	0.00395	0.00200	0.00663	0.00463
0.01000	0.01431	0.00431	0.00200	0.00626	0.00426

* *Assoc. Official Agr. Chemists, Methods*, 1916, 173.

PINEAPPLE.

To a 50 gram sample of the juice of canned pineapple a known volume of standard tin solution was added. The sample was then digested with 50 cc. of concentrated hydrochloric acid and 250 cc. of water.

TABLE 6.

Determination of tin in canned pineapple.

TOTAL FOUND	ADDED	RECOVERED
gram	*gram*	*gram*
0.04938	0.03966	0.03870
0.04953	0.03966	0.03885
0.01068	0.0000	0.0000
0.04953	0.03966	0.03885

RASPBERRIES.

Red raspberries were poured from a large can, well lacquered on the inside, upon a colander, and the free sirup, 60 per cent of the whole, separated from the berries. The large amount of sugar present and the strong color of the material carried down during the precipitation, made it a very unpromising material from which to precipitate tin with zinc.

Measured volumes of standard tin solution were added to 50 gram samples and digested with hydrochloric acid. The tin was precipitated with 12 grams of zinc powder.

The action was allowed to go on until the originally red solution became colorless. The following table gives the per cent of strong hydrochloric acid in the solution used in digesting the samples, volume of solution from which the tin was precipitated, the tin added and recovered.

TABLE 7.

Determination of tin in canned raspberries.

SIRUP				STRAINED BERRIES			
Hydrochloric acid	Volume of solution	TIN		Hydrochloric acid	Volume of solution	TIN	
		Added	Recovered			Added	Recovered
per cent	*cc.*	*gram*	*gram*	*per cent*	*cc.*	*gram*	*gram*
10	400	0.0150	0.0142	28	230	0.0300	0.0299
10	400	0.0300	0.0299	41	295	0.0450	0.0462
10	400	0.0450	0.0439	30	245	0.0450	0.0466
15	400	0.0150	0.0141	26	245	0.0450	0.0448
15	400	0.0450	0.0430	26	245	0.0450	0.0441
				26	245	0.0000	0.0000
				26	245	0.0000	0.0001

The following results were obtained in the course of routine examination of several canned products:

TABLE 8.

Comparison of tin recoveries by the tentative and proposed methods.

SUBSTANCE	TENTATIVE VOLUMETRIC METHOD	PROPOSED METHOD
	parts per million	*parts per million*
Pumpkin	259	250
Sweet potatoes	228	213
Sweet potatoes	278	266
Cider	...	244
Cider	...	244

All of the above experimental work was performed by J. Zavodsky, Division of Foods and Dairies, State Department of Agriculture, Chicago, Ill.

SUMMARY.

1. A critical study has been made of a method for the determination of tin suggested by Penniman, Baltimore, Md.

2. This method yields results comparable with those obtained by the tentative methods.

3. The new method avoids the objectionable nitric-sulphuric acid digestion, and the hydrogen sulphid precipitation. It requires less time than either of the tentative methods.

ARSENIC.

Some preliminary experiments were conducted in an endeavor to get a satisfactory method for arsenic in gelatin. In view of the unsatisfactory results last year with acid digestion, further attempts along similar lines were abandoned for the present. Instead, attention was devoted to the possibility of adapting the arsenic trichlorid distillation method to gelatin and other products. This work has been interrupted very frequently and not enough has been done to warrant any statement regarding the feasibility of this method.

RECOMMENDATIONS.

It is recommended—

(1) That the Penniman method for tin be made the subject of collaborative work during 1918.

(2) That the Gutzeit method as modified during 1916 be made the subject of collaborative work on baking powder materials during 1918.

(3) That a study be made of methods for the determination of arsenic in gelatin and similar products.

(4) That a study be made of methods for the determination of zinc, copper, and aluminium in foods.

No report on fruits and fruit products was made by the referee.

REPORT ON CANNED FOODS.

By W. D. Bigelow (National Canners Association, 1739 H Street, Washington, D. C.), *Referee on Canned Vegetables.*

No systematic work was done on this subject. The laboratory of the referee gave further attention to tomato products which formed the subject of the last report[1]. The results are confirmatory of those reported a year ago.

During the last year the referee has given careful consideration to the possible scope of the subject of canned foods in this association

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[1] *J. Assoc. Official Agr. Chemists*, 1920, 3: 453.

and has consulted with many others regarding it. As the result of this deliberation, it does not appear that the subject of canned foods lends itself to the work of this association in the same way as many of the other subjects that have been studied. The methods employed for the examination of canned foods are largely the same as are employed for fresh foods and those preserved by other methods. The majority of topics, therefore, that might otherwise come within this field have been classified under other topics in the methods of this association. Those methods which are peculiar to canned foods, such as methods for the examination of the can, the consistency, character, and quality of the product, do not lend themselves to cooperative work and probably are not available for study in the association. If it is the desire of the association, a description of these methods as far as they have been formulated will be presented at a future meeting.

REPORT ON CEREAL PRODUCTS.

By J. A. LeClerc[1] (Bureau of Chemistry, Washington, D. C.), *Referee.*

The work outlined by the referee followed closely the 1916 recommendations of Committee C[2]. In addition to this, the collaborators were asked to make an ash determination, comparing the official method with the calcium acetate method, in which the amount of calcium acetate was reduced to a minimum, *i. e.*, 2.5 mg. per 5 grams of flour.

The moisture determinations were confined to a comparison of the official method of drying in a vacuum water bath with the so-called calcium oxid method, the drying being done, in the latter case, in a vacuum desiccator.

The gluten determinations consisted in comparing the results obtained by using ordinary tap water, distilled water containing 0.1 per cent of sodium chlorid, and distilled water.

The soluble carbohydrates were determined by the Bryan, Given and Straughn method[3], and the results compared with those obtained by hydrochloric acid extraction, using 0.5, 1, and 2 per cent hydrochloric acid, respectively.

The cold water extract was studied by comparing the extraction at 10°C. for 45 minutes with extraction at 10°C. for 1½ hours and with an extraction at 5°C. for 45 minutes.

The quantitative determination of chlorin was studied by two methods—the gasoline extraction method which was used last year, and the extraction in a Johnson fat extractor with alcohol-free ether.

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[1] Present address, Miner-Hillard Milling Co., Wilkes-Barre, Pa.
[2] *J. Assoc. Official Agr. Chemists*, 1920, 3: 532.
[3] *Assoc. Official Agr. Chemists, Methods*, 1916, 109.

TABLE 1.

Determination of moisture and ash.

ANALYST	MOISTURE		ASH	
	Official method*	Vacuum method (calcium oxid)	Official method†	Calcium acetate method‡
	per cent	*per cent*	*per cent*	*per cent*
F. C. Atkinson, American Hominy Co., Indianapolis, Ind.	12.70§	12.40	0.464	0.484
	12.80§	12.45	....	....
R. M. Bohn, Bureau of Chemistry, Washington, D. C.	11.40	12.35	0.442	0.452
	11.43	12.52	0.446	0.462
C. D. Garby, Bureau of Chemistry, Washington, D. C.	12.90	12.85	0.448	0.432
	12.91	12.77	0.448	0.434
K. J. Osterhout, Bureau of Chemistry, Washington, D.C.	11.82	12.12	0.476	0.472
	11.69	11.77	0.464	0.476
	....	....	0.470	....

* *Assoc. Official Agr. Chemists, Methods*, 1916, 79.
† *Ibid.*, 187.
‡ U. S. Bur. Chem. Bull. **107**, rev.: (1912), 21.
§ Dried in hydrogen.

TABLE 2.

Determination of soluble carbohydrates and cold water extract.

ANALYST	SOLUBLE CARBOHYDRATES				COLD WATER EXTRACT		
	Bryan, Given and Straughn method*	0.5 per cent hydrochloric acid	1 per cent hydrochloric acid	2 per cent hydrochloric acid	45 minutes 10°C.	1½ hours 10°C.	45 minutes 5°C.
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
R. M. Bohn	1.39	1.53	1.45	1.59	4.09	5.22	5.08
	1.39	1.43	1.40	1.47	4.24	5.28	5.25
C. D. Garby	1.24	1.60	1.37	1.39	5.50	5.45	4.71
	...	1.54	1.31	1.31	...	5.54	4.83
K. J. Osterhout	1.28	1.63	1.63	1.82	5.32	5.84	5.23
	1.41	...	1.58	1.83	5.20	6.00	5.16
F. C. Atkinson	...	...	...	...	...	6.02	5.00
				...	5.42	..	..

* *Assoc. Official Agr. Chemists, Methods*, 1916, 109.

TABLE 3.

Determination of gluten.

ANALYST	TENTATIVE METHOD*		DISTILLED WATER CONTAINING 0.1 PER CENT OF SODIUM CHLORID		DISTILLED WATER	
	Wet	Dry	Wet	Dry	Wet	Dry
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
R. M. Bohn............	29.07	10.67	29.53	10.43	22.60	8.93
	29.13	10.87	30.40	10.73	23.33	8.80
	....	....	29.13	10.77	24.00	9.27
C. D. Garby............	27.55	10.13	31.01	10.58	21.88	8.44
	27.35	10.12	31.01	10.50	22.35	8.75
K. J. Osterhout.........	29.4	30.1	25.7	10.60	10.30	9.80
	29.5	29.7	25.1	....	10.40	9.50

* *Assoc. Official Agr. Chemists, Methods*, 1916, 189.

TABLE 4.

Determination of chlorin.

ANALYST	GASOLINE EXTRACTION	ETHER EXTRACTION IN JOHNSON EXTRACTOR
	parts per million	*parts per million*
F. C. Atkinson..............................	100	...
R. M. Bohn..............................	105	105
	81	105
K. J. Osterhout..........................	96	100
	98	125

CONCLUSIONS.

From these results the following conclusions may be drawn:

Moisture.—The use of calcium oxid would seem to give results which are as good as those obtained by the official method. In most cases, a slightly larger amount of moisture was obtained by the calcium oxid method than by the official method, which would indicate that the calcium oxid in a vacuum desiccator absorbs practically all of the moisture.

Gluten.—The results obtained would seem to indicate that distilled water containing 0.1 per cent of sodium chlorid gives approximately the same amount of gluten as ordinary Washington tap water. On the other hand, the use of distilled water alone causes a very large loss of gluten. Inasmuch as the tap water varies to a very large extent in different cities, it might be best to recommend that the washing of gluten should be done by the use of distilled water containing a certain proportion of sodium chlorid or other salts.

Soluble carbohydrates.—The results in this case would seem to indicate that the use of 1 per cent hydrochloric acid is to be preferred as a medium of extraction.

Cold water extract.—The results of these analyses would show that a 45 minute extraction at 10°C. gives practically the same results as the 45 minute extraction at 5°C.; 1½ hours' extraction at 10°C. gives considerably higher results. Therefore, it might be wise to advocate that the extraction be carried on at a temperature of from 5 to 10°C., instead of limiting it to 10°C. alone.

Chlorin.—Three collaborators obtained quite concordant results in the determination of chlorin by the two methods. The results, therefore, are very encouraging, but, inasmuch as these results are so few in number, it is recommended that this work be continued another year.

Ash.—The results of the ash determination by the use of a minimum amount of calcium acetate are remarkably close to those obtained by the official method. If it is more convenient to use calcium acetate in the determination of ash in flour, it should be allowed in view of these results.

RECOMMENDATION.

It is recommended—

That the work of this year be repeated.

No report on wines was made by the referee.

REPORT ON SOFT DRINKS.

By W. W. Skinner (Bureau of Chemistry, Washington, D. C.), *Referee.*

The Referee on Soft Drinks, after correspondence with several persons who had signified a desire to participate in work of this character, decided that collaborative work was inadvisable until methods could be suggested with a reasonable prospect of satisfactory results being obtained. The referee is of the opinion that valuable time and energy may be wasted to no purpose in collaborative work on methods which have not as yet been subjected to a critical study and investigation by some one, expert in the particular line of work to which the methods apply. It seems to the referee that if collaborative work is to be maintained on a sound basis, then collaborators should be expected merely to test the application of well-defined methods rather than to assume the role of investigator in the development of new methods.

With this in mind, therefore, no collaborative work was planned. Work, however, was undertaken by one investigator under the direction of the referee, with the hope of developing quantitative methods for the determination of ginger and of capsicum in ginger ale and other ginger drinks. Many difficulities have been encountered in this work and while no satisfactory method has so far been developed, the preliminary work has yielded interesting information which has a direct bearing on the controversy regarding the use of capsicum in ginger ale. The referee is not in a position to report definitely on this matter, but can submit only a report of progress with the suggestion that the work be continued.

ADDRESS BY THE HONORARY PRESIDENT.

H. W. Wiley (Good Housekeeping, Bureau of Foods, Sanitation and Health, Washington, D. C.).

I think we are getting on in the world. I remember the earlier days when we used to meet at Cabin John Bridge, and all could get on one trolley car, and those, by the way, were very happy days, which I remember with great pleasure. Those were the days before I had turned prohibitionist, and it was not considered bad form to say "Prosit". That is one Latin word now that is taboo. Most people think it is German, but that is because they do not understand Latin. After the Cabin John Bridge days, we went first to a small hotel until we graduated, and for a long while we went to the Raleigh. Now we have come to the Willard. That is what we call "high life". Now I promise you that, if this country ever has the great good fortune to make me President of the United States, I will invite you to meet in the White House.

I know that very few of your members came to this town with the idea of insulting me. One of them did, but I am not going to name him. In the English Parliament to name a man is the highest disgrace that can be heaped upon him. I will tell you, however, what he said: "Have you written out and committed to memory your annual extemporaneous address to the Association of Official Agricultural Chemists?" Well, now, in one sense that was a compliment. When a man can stand up and make an extemporaneous address that has all the ear marks of the midnight oil, it shows the possibilities of the human intellect. Now you are going to get another one of that kind. If I remember, my last extemporaneous address was on the subject of colloidal chemistry, and I can only say that great progress has been made in the application of colloidal chemistry in the experience of man, because, if ever things were mixed up more completely in the history of the world than now, I do not know when it was. We have nothing but colloids afloat in a sea of blood—itself a sea of colloids. I am not going to make another address on colloidal chemistry because I am sure I would not be able to equal the address I formerly made, but I do want to call attention to the condition of affairs to which this disorganized condition of the world has brought us, and it emphasizes what has always been known to be. and now is recognized by everybody to be the fundamental industry of the world—the industry of agriculture. This is the industry which you are aiming to promote. and it is one which today has supreme importance, more even than munitions and supplies of guns, more even than the man power of the world. The problem of agriculture looms up as the great predominant problem of today. Some of its aspects are not, perhaps, peculiarly chemical, and I am going to take the liberty this morning of disgressing somewhat from the path of chemistry, and call attention to some of the problems in agriculture which, although partly chemical, are not entirely so.

First of all, the problem of feeding the world is a problem peculiarly agricultural, and also, to a considerable extent, a problem which is peculiarly chemical. The food problem today is paramount in importance as an agricultural problem, both from the point of production and the point of distribution, and the agricultural chemist has a great deal to do with both. The production of food, as we all know, is a strictly agricultural-chemical problem. Given the area, given the climate, the amount of food produced is a problem of scientific agriculture. The better the principles of scientific agriculture are understood and practised by the farmer, the greater will be the yield of his fields. other things remaining equal. That being the case, the production of the crop is largely a matter of cultivation and plant food. The war has cut us off from one of the essential ingredients of plant food, without which the ration of the plant is unbalanced, namely, potash. The problem is to supply, if possible from other sources, the potash which we formerly received from Germany. Of course, there are no deposits of potash which compare in extent and inexhaustibility to those which are within the confines of the German Empire, and yet there are sources of potash which, if they could be utilized, might supply our need. There is enough potash locked up in the feldspar of this country to supply the nations of the earth for an unnumbered series of years, if it could be unlocked, and chemists are working upon that problem. The deposits of potash which we already have are being exploited, as you know, and a considerable amount of potash is now available which a few years ago was untouched. Even the dust from cement and other factory chimneys is being saved for the potash it contains. The sea is to be utilized as far as possible, and the kelp is being harvested to a somewhat considerable extent in furthering this purpose. We are getting some potash, but at a price the farmer can ill afford to pay, or perhaps can not pay at all at the present time. It therefore seems to me that the application of potash to our fields in any considerable quantity, representing the needs of the crop, is altogether a difficult problem at the present time. Now that has taught some of us a good lesson. Many have learned that the soil contains sufficient potash for the crop, and that is a good lesson to learn, for, when the old supply again becomes available, the farmer will have learned that some soils are sufficiently rich in potash, so that it is not altogether necessary to purchase additional quantities. It is hoped that to this extent the shortage of potash may prove a blessing in disguise.

All other plant foods, as you know, have greatly increased in price. Take, for instance, the acid phosphate which is used as a basic fertilizer, and used more extensively than any other kind. This is a substance that helps unlock the imprisoned potash. The price has more than doubled since the war began. A few years ago, I was able to get acid phosphate by the carload at from nine dollars and fifty cents to ten dollars, laid down at my station, and now I pay twenty dollars, and perhaps I shall have to pay more before the war is over. This has led to the use of natural phosphate rock reduced to a fine powder; and, where the soil is inclined to be acid, that treatment apparently is giving very favorable results. I, myself, have tried it on my farm—I have treated about forty or fifty acres of some acid soil with natural phosphate rocks, and have had good results therefrom. But it does not do any good, or at least very little good, to put such a product on a soil which has been properly limed, and which is in a condition to bear leguminous crops. It is difficult to secure any visible good effect. In so far as nitrogenous fertilizers are concerned, the price has also gone up one hundred per cent or more, so that now fish scrap and tankage can not be purchased at a price which the farmer feels he is able to pay. Therefore, my experience is that all forms of plant food should be put on a strictly economic ration by the farmer. To this statement the reply is made that the farmer is receiving proportionately more for his crops and can pay the increased price without suffering any hardship. That is true in so far as it is applicable. If the farmer's crop is one hundred per cent more profitable than it was before, he can pay one hundred per cent more than he did for his plant foods. If, on the other hand, his crop is not that much more valuable, then he is working at a loss. This is one of the great problems of agriculture today—the feeding of the crop. It is driving us again into a path which we may in the future follow as a matter of choice. To supply nitrogenous fertilizers on the farm, it is advisable to grow and plow under leguminous crops. That is being done to a very large extent in this country, and now, to a larger extent than before. The supply of nitric acid from the air, while promising, is yet commercially infinitesimal. The price of lime has also greatly increased. I have just joined the large and increasing body of agriculturists who are not using burned lime any more. The farmer who has a large amount of manure from his stables can not afford to use burned lime on his land, for if you have any experience you know how wasteful it is. It would be interesting to drive about in the country where freshly burned lime has been spread on stable manure and smell the ammonia in the air, as you can do at any time, even several days after the application. So we are growing beans and other leguminous crops, not only for the hay, but for the actual benefit which we get by ploughing the crop under. In this way we have been able, so far, to keep our crops almost, if not quite, up to normal. There has been very little shortage in the magnitude of the crop by reason of the increased cost of production. That part of the problem seems to have worked itself out most satifactorily.

We come now to the most important problem of all, and that is man power. How are we to fill the place of our farmer soldiers? There are two ways of overcoming that difficulty. One is to secure a larger number of laborers, and the other is to go to work yourself. I think the best advice I can give is to go to work yourself. I am somewhat of a believer in Tolstoy's theory, that it is a crime for any man to eat anything which he, by his own labor on his own farm, has not produced. If every one could be brought to that way of thinking, the scarcity of farm labor would be quickly overcome. I go right out into the field and work, and I feel sure that I have fulfilled Tolstoy's theory. I believe that every bit of food which I and my family shall eat during the next year has been produced directly or indirectly by my own hands. I do not sit down to my table and feel that I am robbing any one.

DEVOLUTION.

Those who live in the city must move out. The great curse of this country is concentration in cities, and the great curse of our industries is not that they are industries, but that they are centered in cities. I am of the opinion that cities should not allow any productive industries, but should devote their energies to banking, transportation, and exchange. They should distribute commodities or make them available, but when they make anything out of a raw material, and dig into the soil and make something out of it, that is wrong. Every time you send a shoe factory or a machine factory to an agricultural region, you benefit not only those who work in that factory, but the farmers round about. We must devolute our cities, and this you see going on all the time.

Every one is going out to the country and getting a little place if he can. Do you realize that if you have one or two acres you can grow almost enough on it to supply your family? I know a banker in this city who has a half-acre of land, and he grows almost enough on that half-acre to supply his family with food. He can sell enough of the excess of the articles he raises to pay for the food he can not raise on his land. He understands how to get the most out of the soil. His wife does her own work in the house. These industrious people are doing a large part in the great struggle in which we are engaged.

MAN POWER.

Now as to man power. I remember James Whitcomb Riley's story of the "Old Man and Jim". That is one of the best stories I ever read and one of the most pathetic, for that matter. What I am going to call attention to is the worthlessness of Jim on the farm. While he never did anything on the farm, he was the old man's pride. The other sons of the farmer were his right-hand men, but although "Jim was the wildest boy he had" the old man was "all wrapped up in Jim". But when the war broke out—the contest between the States that is called the Civil War—Jim enlisted and made an ideal soldier, for "his fightin' was good as his farmin' bad". If a great many people I know in the country made as good soldiers as they make bad farmers, it would be a blessing to have a lot more of them drafted into the Army. As it happened in my part of the community, the worthless farmers were not selected, and those who were industrious and of some account were taken. I should like a selective draft, too. I should like to make all of the boys in the country who will not work go into the Army. Instead of hanging around the railroad station they learn discipline and industry. When I go to my little place in the country I see dozens of young men hanging around the station. I suppose they want to see me—I do not know what else attracts them. I will give them the benefit of the doubt. I should like to see a selective draft applied in such a way that they would ask each farmer who are good workmen and who are not. In that way, they might select an army of men whose "fighting would be as good as their farming was bad". Our man power is going to be utilized to a great extent on the field of battle, and I am one of those—I may be misguided—who believe that every single activity and power of this country should be put behind the President in this great war. I do not care whether I agree with his policies or not. That is of no consequence. He is our President, he is the representative, the supreme authority of this great nation, and the man—I do not care what his political creed is—who does not stand behind the President is not fit to be called a citizen of the United States. So I am asking no exemptions for the people in the country from military service—not a single one.

MOBILIZATION OF THE WOMEN.

But what are we going to do? First, we ourselves are going to work. Second, we must call upon the women of this country. They are ready to help. You know there are hundreds and hundreds of things that a woman can do even better than a man. I saw three women gathering apples in Virginia last October, and doing it just as well and better even than the men could do. We should not have had our apple crop gathered without the help of the women.

Then there is another great branch of industrial agriculture where the women are better than the men, and that is the dairy industry. Three or four years ago I was in Duluth and wanted to see some of the dairies there. They took me to a dairy owned and operated entirely by women as being the star dairy of that vicinity. If we could release the men in the dairy industry today and replace them with women, we should come very near making up for all the losses we have so far experienced. There are dozens of things a woman can do on the farm. I saw a woman not long ago in the corn field. It was ensilage cutting time and she said to me, "Let me go out in the field and I will show you that I can cut ensilage as well as a man". She took a cutting knife and went out into the field, where she took one row while the man took two, and she finished her row and kept up with him, and never flinched. That shows the spirit of the women who want to do something for the country. They are always good workers and would make good soldiers. No doubt some of them would fight mighty well. I know they can fight if necessary, but perhaps we will not want to organize a "battalion of death" as they did in Russia. If the pinch comes they can go into the trenches. They can fight for their country and they will do it if necessary. So we are not going, in my opinion, to let our crops fail for man power, for we can have woman power.

JUSTICE TO WOMEN.

It seems to me that the men of this country ought to be generous enough, when the women want to go with them to the polls, to give them the rights of citizenship in this country. What a glorious thing it would be if, as a war measure, just as Lincoln enfranchised the slaves at the time of the Civil War, our President would say to Congress: "Enfranchise the women of this country. They are giving all of their energies to this war—give them their independence and their rights as American citizens." What a splendid hour it would be in this country, what a blow to the terrorism and the autocracy of the German Empire if all our women were enrolled in this great army! So I say we are going to call upon the women in this great problem of food production, and we are not going to call upon them in vain.

FOOD ADMINISTRATION.

The next great point is distribution. First, plant food; second, man power; and third, distribution. That is one of the most important of the triumvirate. We are not going to solve the distribution problem in the old-fashioned way—that is evident. We thought perhaps that in this generation we had discovered the idea of directing the distribution of food. I discovered lately in reading from an old author, a man who wrote two thousand years ago, Lucius Apuleius, that the Food Administrator is no new thing. Lucius Apuleius wrote a book, "The Golden Ass". Lucius was transformed into an ass. Some men are born asses, others have asininity thrust upon them, while still others acquire assdom. Lucius was metamorphosed into an ass, but before this metamorphosis took place he traveled into Thessaly, where he met an old schoolmate of his who was evidently a man of very distinguished powers. He had a great retinue of servants, and Lucius was astonished to meet his old friend in such an imposing environment. But listen to Lucius:

" 'Holy Moses', said I. 'Who is this I see? It surely beats the band to see you in this gorgeous uniform all spangled with decorations. And such a crowd of attendants! You must be the Mayor of this town, old friend Pythias'. He replied, 'Not quite so bad as that. I am only the Food Administrator of this municipality. Is there anything in particular you would like to have for your supper?' 'Thanks awfully,' I replied, 'I have already bought fish'. When Pythias saw my basket (for they had no delivery system in this market) he took it and made a careful inspection of its contents. 'How much did you pay for these minnows?' he asked. 'The horrid fish profiteer', I replied, 'wanted a whole silver plunk for this bunch of flappers but finally he let me have them for two dimes'. Taking me by the hand, Pythias led me into the Central Market and said 'Show me the scoundrel who cheated you so egregiously'. 'That is he, crouching in the corner', said I, pointing my finger at the mercenary wretch, who seemed to shrink up as soon as he saw the Food Administrator looking at him. Pythias rushed up to the Shylock of the Billingsgate and gave him a good tongue lashing. 'You food shark', he said, 'How dare you play such a shabby trick on this old chum of my college days? You must be trying to make a desert of this fair country by your exhorbitant prices. Your license is revoked. You shall never have another fish to sell if you take more than a nickel a pound. You'll know what a Food Administrator is before I get through with you'. With that, Pythias seized my basket of fish and poured them on the floor of the market and jumped on the measly minnows with all four feet. And Pythias patted himself on the back and said, 'See how I conserve the food and punish the violators of my regulations'. So I, blinded by the mighty power and diligence of the Food Administrator, went my way minus both my money and my supper."

A great many people in this country, since the Food Administration took its place, seem to be in the same fix. It is not anything new—this regulating of the food supply—and so I say the distribution of our food supply is the crowning work of the agricultural problem. What do we do with it today? I do not want to be personal in this matter, but I must speak the truth. It has been a very unfortunate habit of mine to tell the truth even if it is not very popular. The American stomach is the biggest garbage can in the world. We throw into it unnecessarily a large amount of food, which would feed another hundred million people. Now there is no doubt of that fact. The first thing we have to do is to stop making our stomachs garbage cans, and we can do that by eating only those foods that are necessary to keep us well and strong. I am not an advocate of any method of regulating nutrition that diminishes the vitality of the human organism. I want our soldiers to be well fed, I want our citizens to be well fed, and we have plenty of food to do that if we do it wisely, and at the same time we can save immense quantities from the garbage cans of this country for the benefit of our allies, so that they also may be well nourished and able to fight their battles. Napoleon said, and as far as human wisdom is concerned I think he is the greatest man that ever lived, "Soldiers fight on their bellies". He knew the value of the commissariat. When his soldiers had climbed to the top of the Alps he had stored cheese and bread and wine with the St. Bernard monks (the prohibition law had not gone into effect on the Alps) and every soldier as he reached the summit had an abundant supper. They felt that their commander was looking after their physical wants. And so, refreshed and enthusiastic, they descended to the plains of Lombardy and fought at Lodi and Marengo. We must realize that nutrition, and proper nutrition, is the basis of all our military success. Hence, I am heartily in sympathy with our Food Administration. I believe in the great principle of conservation of food by force and not by argument. There is no use in arguing with a man who has the means to sit down to a "square meal". I do not want you to stop with mere argument in this food administration work. I want to see bread cards and meat cards and sugar cards, so that not even the President may get more than his share. Let us make this food administration work more effective by making it more urgent and less persuasive. I am perfectly willing to argue with you in times of peace as to what you should eat and what you should drink and wear; I was once opposed to prohibition because I did not believe it was my business to say to you what you should guzzle. But now I believe we should say to you what you shall eat and what you shall drink in this time of stress. It is a necessity of war. I would not allow any one to buy food except in proportion to the number in the family. Then they have to divide it wisely. How much do we need? You know as much about that as I do. A man of your size and my size needs 3000 calories a day, and if he goes out into the field and earns a part of his food he may need 500 more. If he sits down and writes letters to you to persuade you not to eat anything he ought not to have more than 1800 or 2000 at most.

I tell you if the Kaiser could only look in upon this country and see the number of typewriters at work he would slink away in terror. We are fighting this war by typewriters instead of by machine guns. The President hit it off beautifully in his Buffalo address when he said, "I am glad to get away from Washington where so many people know so many things that are not so". One of the things that is not so is that you can win this war by writing on a typewriter. Every one of those people operating typewriting machines should be making shot and shell—they carry messages that can be understood by the Germans. I should like to see the battle cry of freedom engraved on a bayonet point and the Battle Hymn of the Republic engraved on a shell. Then the Germans would understand what we mean!

THE NEED OF EFFICIENCY.

Now my plea to the farmers of this country is for efficiency, to get all the plant food that you can, to get the man power and the woman power that you can, and you can get plenty of it if you will recognize woman's ability. Women are human beings, and are entitled to the right to labor, just as much as they are to the right to vote and to fight.

What we want to do is to put all of our energy into effective work and not into teaching and persuading. The time for that is past. When a German soldier has his bayonet against your breast that is no time to argue with him. I want to urge men and women to action. When this war broke out, I asked to serve in the Quartermaster's Department. I did not care in what capacity—only to help in some way, as I believed I was capable of doing. What reply did I get? Something like this: "Dear Sir: Your application to serve in the Quartermaster's Department has been received and placed on file. I call your attention to Regulation 18072 X which forbids the employment of any one in the Quartermaster's Department who has passed the age of 65." It was no fault of mine that I was over sixty-five years—I have entered my seventy-fourth year. I surely could do service of some kind for my country. Are we going to let regulations stand in the way of our fighting this great war? Let every man fight and help if he can for freedom and democracy.

My friends, this is my extemporaneous address. If you think it has been written out and committed to memory, well and good. I think it is good enough to have been written out and committed to memory myself. I have been with you now for thirty-five years; you are my professional brethren. Some of you are older than I, at least in appearance, but I doubt if there is one here with a younger heart or with a more flexible artery than I have, who speak to you. I want you to have a strong heart and a flexible artery. Then you can serve your country, and your country now, more than ever before in its history, needs your help.

The meeting adjourned at 12.30 p. m. to reconvene at 2 p. m.

SECOND DAY.

TUESDAY—AFTERNOON SESSION.

REPORT ON DISTILLED LIQUORS.

By J. I. PALMORE (Bureau of Chemistry, Washington, D. C.), *Referee.*

No report was made other than to mention an experiment undertaken to test a method for the preservation of aldehyde-free alcohol. This experiment will be continued over several years and a full account of it will be included in a subsequent report.

No report on beers was made by the referee.

No report on vinegars was made by the referee.

No report on flavoring extracts was made by the referee.

No report on meat and meat products was made by the referee.

A NEW METHOD FOR THE ESTIMATION OF HISTIDIN.

By. W. E. THRUN and P. F. TROWBRIDGE[1] [2] (Agricultural Experiment Station, Columbia, Mo.).

The following experiments were made to determine whether or not bromin absorption determinations would be of value in conjunction with the Van Slyke analysis. Two 5 cc. aliquots (equivalent to 0.0323 gram of protein nitrogen) of the solution of the bases of a coagulable protein sample were treated with 10 cc. of bromate solution (equivalent to 18.71 cc. of thiosulphate) and 50 cc. of water, 10 cc. of bromid solution, and 5 cc. of hydrochloric acid. Bromination was allowed to proceed for 15 minutes. It took 17.0 cc. of thiosulphate (equivalent to 0.008465 gram of bromid) to titrate the excess of bromin. The bromin absorbed was 0.01477 gram. Deducting 0.00259 gram due to cystin (cystin nitrogen = 0.0009 gram found by determining sulphur in another aliquot), assuming that cystin consumes 10 atoms of bromin per molecule, there is a consumption of 0.0119 gram of bromin by the histidin in solution. One molecule of histidin absorbs 2 atoms of bromin accord-

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[1] Present address, Agricultural Experiment Station, Agricultural College, N. Dak.

[2] Associate Referee on the Separation of Nitrogenous Compounds in Meat Products.

ing to Siegfried and Reppin[1]. Calculating from the above data, 9.68 per cent of histidin nitrogen is obtained. To this must be added 1.17 per cent as a correction for the solubility of histidin in the presence of phosphotungstic acid as determined by Van Slyke, making it 10.86 per cent of histidin nitrogen. This is somewhat higher than was obtained by the Van Slyke method, 9.08 per cent. By the same method another sample had an average of 4.73 per cent of histidin nitrogen; by the Van Slyke method, 4.97 per cent. The method gave also a value of 2.15 per cent of histidin nitrogen for another sample, while the Van Slyke method gave 1.90 per cent.

The accuracy of this method may be considerably increased by using more dilute solutions for the titrations, so as to measure larger volumes. A factor for bromin absorption for histidin may be necessary, since histidin seems to absorb somewhat more than 2 atoms of bromin per molecule.

Attention may be called to the fact that this method requires only two determinations of which the cystin nitrogen is very accurate, while the Van Slyke method requires three determinations. Amyl alcohol may interfere by absorbing bromin, but in the Van Slyke analysis the solution of the bases, after removal of the phosphotungstic acid, is evaporated under a vacuum and boiled off. It is not known whether the purin and pyrimidin which are precipitated by phosphotungstic acid will absorb bromin under the conditions employed in these experiments. If further proof of the value of this method for the determination of histidin is obtained, a Van Slyke apparatus will no longer be necessary for a determination of the hexon bases.

No report on meat extracts was made by the associate referee.

REPORT ON EDIBLE FATS AND OILS[2].

By R. H. **Kerr** (Bureau of Animal Industry, Washington, D. C.), *Referee.*

The work consisted of a study of the modified method for the detection of the adulteration of lard with fats containing tristearin. No samples were sent out for cooperative work but it was requested that each collaborator make his own mixtures using such fats as he believed to be used for the adulteration of lard, or for whose detection he considered a method of detection desirable, and to examine them by the method to be studied. After completing his tests, he was requested to

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[1] *Z. physiol. Chem.*, 1915, 95: 18.

[2] Presented by H. S. Bailey.

send the sample mixtures to the referee, withholding the report of his results and the composition of the samples. Samples were submitted by three collaborators: P. Rudnick of Armour & Co.; J. J. Vollertsen of Morris & Co.; and C. H. Robinson, Central Experimental Farm, Ottawa, Canada. Mixtures were also made up in the Meat Inspection Laboratory of the Bureau of Animal Industry, Washington, D. C., and tested by the referee and also by two other analysts, to whom the composition of the mixtures was unknown.

COMPOSITION OF MIXTURES MADE BY THE REFEREE.

Sample 1.—Pure lard + 3 per cent oleo stearin.
Sample 2.—Pure lard + 3 per cent hydrogenated cottonseed oil.
Sample 3.—Pure lard + 5 per cent hydrogenated lard.
Sample 4.—Pure lard.
Sample 5.—Pure lard + 5 per cent hydrogenated whale oil.
Sample 6.—Pure lard + 5 per cent soft beef tallow.
Sample 7.—Pure lard + 1 per cent hydrogenated cottonseed oil.
Sample 8.—Pure lard + 10 per cent hydrogenated lard.
Sample 9.—Pure lard + 10 per cent coconut oil.
Sample 10.—Pure lard.
Sample 11.—Pure lard.

TABLE 1.

Cooperative work on the determination of melting point on mixtures made by the referee.

ANALYST AND SAMPLE NUMBER	GLYCERIDES (A)	ACIDS (B)	DIFFERENCE	A+2 (A−B)
	°C.	*°C.*	*°C.*	*°C.*
R. H. Kerr				
Sample 1	62.8	59.4	3.4	69.6
Sample 2	62.4	62.0	0.4	63.2
Sample 3	63.6	59.0	4.6	72.8
Sample 4	64.2	58.6	5.6	75.4
Sample 5	61.2	58.4	2.8	66.8
Sample 6	62.4	58.2	4.2	70.8
Sample 7	63.0	60.6	2.4	67.8
Sample 8	62.8	59.6	3.2	69.2
Sample 9	64.2	58.8	5.4	75.0
Sample 10	64.4	58.8	5.6	75.6
Sample 11	64.2	58.8	5.4	75.0
R. M. Mehurin, Bureau of Animal Industry, Washington, D. C.				
Sample 1	62.4	59.0	3.4	69.2
Sample 2	61.7	61.4	0.3	62.3
Sample 3	63.4	58.8	4.6	72.6
Sample 4	64.2	59.4	4.8	73.8
Sample 5	61.0	57.8	3.2	67.4
Sample 6	63.2	59.6	3.6	70.4
Sample 7	62.6	59.0	3.6	69.8
Sample 8	62.4	59.2	3.2	68.8
Sample 9	64.1	59.8	4.3	72.7
Sample 11	64.0	59.8	4.2	72.4

TABLE 1.—Continued.

ANALYST AND SAMPLE NUMBER	GLYCERIDES (A)	ACIDS (B)	DIFFERENCE	A+2 (A−B)
	°C.	°C.	°C.	°C.
E. H. Ingersoll*, Bureau of Animal Industry, Washington, D. C.				
Sample 1	62.6	58.8	3.8	70.2
Sample 2	62.8	61.2	1.6	66.0
Sample 3	63.0	58.0	5.0	73.0
Sample 4	64.2	57.8	6.4	77.0
Sample 5	60.6	56.2	4.4	69.4
Sample 6	62.4	58.8	3.6	69.6
Sample 7	62.6	59.4	3.2	69.0
Sample 8	62.5	59.0	3.5	69.5
Sample 9	64.4	58.2	6.2	76.8
Sample 11	63.9	58.0	5.9	75.7

* Since deceased.

COMPOSITION OF MIXTURES PREPARED BY P. RUDNICK.

Sample 1.—Lard + 5 per cent completely hydrogenated cottonseed oil.
Sample 2.—Pure lard.
Sample 3.—Lard + 5 per cent hydrogenated soy bean oil.
Sample 4.—Lard + 5 per cent hydrogenated corn oil.
Sample 5.—Lard + 5 per cent soft oleo stock.
Sample 6.—Pure lard.
Sample 7.—Pure lard + 5 per cent hydrogenated corn oil.

TABLE 2.

Cooperative work on the determination of melting point on mixtures prepared by P. Rudnick.

ANALYST AND SAMPLE NUMBER	GLYCERIDES (A)	ACIDS (B)	DIFFERENCE	A+2 (A−B)
	°C.	°C.	°C.	°C.
P. Rudnick				
Sample 1	62.7	61.8	0.9	64.5
Sample 2	63.9	57.5	6.4	76.7
Sample 3	63.6	57.5	6.1	75.8
Sample 4	63.5	62.7	0.8	65.1
Sample 5	62.8	57.7	5.1	73.0
Sample 6	63.3	57.2	6.1	75.5
Sample 7	62.2	60.9	1.3	64.8
R. H. Kerr				
Sample 1	63.2	62.4	0.8	64.8
Sample 2	64.2	58.6	5.6	75.4
Sample 3	63.4	58.0	5.4	74.2
Sample 4	64.6	63.0	1.6	67.8
Sample 5	63.4	58.8	4.6	72.6
Sample 6	64.2	58.6	5.6	75.4
Sample 7	62.0	61.2	0.8	63.6

COMPOSITION OF SAMPLES PREPARED BY J. J. VOLLERTSEN.

Sample 1.—Pure lard + 0.5 per cent vegetable stearin.
Sample 2.—Pure lard + 1.0 per cent vegetable stearin.
Sample 3.—Pure lard + 2.0 per cent vegetable stearin.
Sample 4.—Pure lard + 0.5 per cent beef stearin.
Sample 5.—Pure lard + 1.0 per cent beef stearin.
Sample 6.—Pure lard + 2.0 per cent beef stearin.

TABLE 3.

Cooperative work on the determination of melting point on mixtures prepared by J. J. Vollertsen.

ANALYST AND SAMPLE NUMBER	GLYCERIDES (A)	ACIDS (B)	DIFFERENCE	A+2 (A−B)
	°C.	*°C.*	*°C.*	*°C.*
C. Kumli, Morris & Co., Chicago, Ill.				
Sample 1	61.5	57.0	4.5	70.5
Sample 2	60.8	57.8	3.0	66.8
Sample 3	59.7	58.8	0.9	61.5
Sample 4	62.6	57.9	4.7	72.0
Sample 5	61.8	56.6	5.2	72.2
Sample 6	60.8	55.8	5.0	70.8
R. H. Kerr				
Sample 1	63.0	58.8	4.2	71.4
Sample 2	62.2	59.6	2.6	67.4
Sample 3	63.2	61.8	1.4	66.0
Sample 4	63.4	58.6	4.8	73.0
Sample 5	62.8	57.6	5.2	73.2
Sample 6	62.0	57.8	4.2	70.4

COMPOSITION OF SAMPLES PREPARED BY C. H. ROBINSON.

Sample 1.—Lard + 1 per cent hydrogenated soy bean oil.
Sample 2.—Lard + 2 per cent hydrogenated soy bean oil.
Sample 3.—Lard + 3 per cent hydrogenated soy bean oil.
Sample 4.—Lard + 4 per cent hydrogenated soy bean oil.
Sample 1.—Lard + 1 per cent hydrogenated cottonseed oil.
Sample 2.—Lard + 2 per cent hydrogenated cottonseed oil.
Sample 3.—Lard + 3 per cent hydrogenated cottonseed oil.
Sample 4.—Lard + 4 per cent hydrogenated cottonseed oil.
Sample 5.—Lard + 5 per cent hydrogenated cottonseed oil.
Sample 1.—Lard + 1 per cent hydrogenated vegetable stearin.
Sample 2.—Lard + 2 per cent hydrogenated vegetable stearin.
Sample 3.—Lard + 3 per cent hydrogenated vegetable stearin.
Sample 4.—Lard + 4 per cent hydrogenated vegetable stearin.

TABLE 4.

Cooperative work on the determination of melting point on mixtures prepared by C. H. Robinson.

ANALYST AND SAMPLE NUMBER	GLYCERIDES (A)	ACIDS (B)	DIFFERENCE	A+2 (A−B)
	°C.	°C.	°C.	°C.
C. H. Robinson				
Lard	63.6	57.3	6.3	76.2
Sample 1	63.2	60.1	3.1	69.4
Sample 2	62.5	60.5	2.0	66.5
Sample 3	63.1	61.0	2.1	67.3
Sample 4	62.5	61.1	1.4	65.3
Sample 1	62.9	57.9	5.0	72.9
Sample 2	62.8	59.2	3.6	70.0
Sample 3	62.3	58.7	3.6	69.5
Sample 4	62.1	59.2	2.9	67.9
Sample 5	62.5	59.8	2.7	67.9
Sample 1	63.2	59.8	3.4	70.0
Sample 2	62.5	60.5	2.0	66.5
Sample 3	62.4	60.7	1.7	65.8
Sample 4	63.4	62.1	1.3	66.0
R. H. Kerr				
Sample 1	63.0	59.8	3.2	69.4
Sample 2	62.2	60.4	1.8	65.8
Sample 3	62.4	61.2	1.2	64.8
Sample 4	62.6	61.2	1.4	65.4
Sample 1	62.8	59.0	3.8	70.4
Sample 2	62.4	59.6	2.8	68.0
Sample 3	62.0	59.8	2.2	66.4
Sample 4	62.2	60.6	1.6	65.4
Sample 5	62.6	59.8	2.8	68.2
Sample 1	62.8	59.8	3.0	68.8
Sample 2	62.6	61.0	1.6	65.8
Sample 3	63.0	61.4	1.6	66.2
Sample 4	65.0	62.4	2.6	70.2

TABLE 5.

Determination of melting point on lards known to be pure.

SAMPLE NUMBER	GLYCERIDES (A)	ACIDS (B)	DIFFERENCE	A + 2 (A − B)
	°C.	°C.	°C.	°C.
9347	64.4	59.8	4.6	73.6
9405	64.6	58.8	5.8	76.2
9406	64.4	59.4	5.0	74.4
9415	64.4	59.0	5.4	75.2
9322	63.4	57.0	6.4	76.2
9436	63.8	57.8	6.0	75.8
9577	64.0	57.4	6.6	77.2
9594	63.6	57.6	6.0	75.6
9550	63.6	57.6	6.0	75.6
9553	63.6	57.6	6.0	75.6

TABLE 5.—Continued.

SAMPLE NUMBER	GLYCERIDES (A)	ACIDS (B)	DIFFERENCE	A+2 (A−B)
	°C.	*°C.*	*°C.*	*°C.*
9570	63.6	57.6	6.0	75.6
9674	63.6	57.6	6.0	75.6
9675	63.6	57.6	6.0	75.6
9687	63.8	57.6	6.2	76.2
9697	63.8	58.2	5.6	75.0
9747	64.0	58.2	5.8	75.6
10025	64.0	57.8	6.2	76.4
10028	64.0	58.4	5.6	75.2
10187	64.2	59.0	5.2	74.6
2135	63.4	57.8	5.6	74.6
10536	63.6	56.8	6.8	77.2
10537	63.4	57.2	6.2	75.8
10538	63.4	57.2	6.2	75.8
10543	64.0	57.6	6.4	76.8

TABLE 6.

Determination of melting point on a series of mixtures containing graded proportions of oleo stearin added to the same lard.

SAMPLE NUMBER	ADULTERANT	GLYCERIDES (A)	ACIDS (B)	DIFFERENCE	A+2 (A−B)
		°C.	*°C.*	*°C.*	*°C.*
1	None	64.4	57.6	6.8	78.0
2	0.5 per cent oleo stearin	64.0	58.4	5.6	75.2
3	1 per cent oleo stearin	63.6	58.6	5.0	73.6
4	2 per cent oleo stearin	63.0	59.0	4.0	71.0
5	3 per cent oleo stearin	62.4	59.0	3.4	69.2
6	5 per cent oleo stearin	62.2	59.4	2.8	67.8
7	5 per cent ox marrowfat	63.4	58.4	5.0	73.4

The results given above show clearly that the figure 71 for the sum A + 2 (A − B) is too low to provide against quite material adulterations. The examination of the samples of pure lard shows that this figure may be increased to 73 without danger of condemning pure lard. It is recommended therefore that the method be adopted as a tentative method in the following form:

METHOD FOR THE DETECTION OF ADULTERATION OF LARD WITH FATS CONTAINING TRISTEARIN.

Weigh out 5 grams of the filtered fat into a glass-stoppered cylinder graduated to 25 cc., add warm acetone until the 25 cc. mark is reached. Shake the cylinder until the contents are thoroughly mixed; then allow the cylinder and its contents to stand in a suitable place in which a temperature of 30°C. is maintained. After 18 hours, remove the cylinder and carefully decant the supernatant acetone solution from the crystallized glycerides, which are usually found in a firm mass at the bottom of the cylinder. Then add warm acetone in three portions of 5 cc. each from a small wash bottle, care being taken not to break up the deposit while washing and decanting the first two portions. Actively agitate the third portion in the cylinder and, by a quick movement, transfer with the crystals to a small filter paper. Wash the crystals with five successive small portions of the warm acetone by means of the wash bottle and remove by suction the excess acetone. Transfer the paper with its contents to a suitable place, where it should be spread out, and any large lumps of the glycerides broken up by gentle pressure. When dry, thoroughly comminute the mass and determine the melting point of the crystals[1]. A melting point below 63.0 is regarded as evidence of adulteration, and a melting point below 63.4 is regarded as suspicious.

After the melting point of the crystallized glycerides has been determined, transfer them to a 50 cc. beaker, add 25 cc. of approximately N/2 alcoholic potassium hydroxid and heat on the steam bath until saponification is complete. Pour the solution into a separatory funnel containing 200 cc. of distilled water, acidify, add 75 cc. of ether and shake. Draw off the acid layer and wash at least three times with distilled water. Transfer the ether solution to a clean, dry 50 cc. beaker, drive off the ether on the steam bath and finally dry the acids at 100°C. After the acids have stood for at least 2 hours after drying, determine the melting point in the same manner in which the melting point of the crystals was determined. If the melting point of the glycerides plus twice the difference between the melting point of the glycerides and the melting point of the fatty acids is less than 73°C., the lard is regarded as adulterated.

REPORT ON DAIRY PRODUCTS.

By JULIUS HORTVET (State Dairy and Food Commission, St. Paul, Minn.), *Referee.*

The work included:

(1) A further study of modifications of the official Roese-Gottlieb method applied to malted milk, dried milk and plain ice cream.

(2) A further study of the Harding-Parkin method for fat determinations in comparison with the present official and provisional methods.

(3) A further study of the tentative Schmidt-Bondzynski modified method for the determination of fat in cheese.

In accordance with the plan of work above outlined, the instructions sent out to the collaborators included full descriptions of the methods, following in the main the original published authors' texts or the texts as found in the last compilation of tentative and official methods of analysis[2]. No material changes were incorporated and such additional directions as seemed to be required were given in separate paragraphs. Directions were given regarding the preparation, weighing, and preliminary treatment of samples. Much attention has been given by the referee to these preliminary details and experience has led to the conclusion that no uniform rule of procedure can be laid down which will be suitable for all classes of dairy products. Before the application of

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[1] U. S. Bur. Animal Ind. Circ. 132: (1908).

[2] *Assoc. Official Agr. Chemists, Methods*, 1916, 287.

the regular analytical method, therefore, it has been found necessary to subject each given sample to such mode of preliminary treatment as has been shown by experience to yield most reliable results. The collaborators were also given general instructions relative to the following topics: (a) the condition of the sample as to fitness for analysis; (b) the various comparative tests to be made on each sample submitted; (c) the number of determinations to be made in duplicate and the averages to be reported; (d) the importance of experience and preliminary trials of the various methods before attempting determinations for the purposes of collaborative work. In the 1916 report of the referee[1], considerable attention was given to a discussion of the acid extraction modification of the official Roese-Gottlieb procedure, and it was then recommended that this modification be given further study. Accordingly, the plan of work has been so arranged as to secure from the collaborators comparative results showing the relative merits of the two procedures which have been somewhat in controversy for many years. Whatever may be the relative theoretical advantages of either mode of treatment of material before proceeding with the extractions, the aim has been at least to obtain practical comparative results under as favorable uniform conditions as possible by as many analysts as could be engaged to assist in the work.

The referee is indebted to W. D. Strack, Chief Chemist for Borden's Condensed Milk Company, New York, N. Y., for obtaining uniform sets of samples and forwarding them direct to the collaborators. Each set of samples included: (1) a plain ice cream, preserved with 15 grains of mercuric chlorid, in a tightly covered glass container; (2) a dried skimmed milk enclosed in a pasteboard carton; (3) a malted milk powder in a well-sealed bottle. In the study of the modified Schmidt-Bondzynski method, each collaborator was provided with a sample of Parmesan, a type of Italian cheese made from partly skimmed milk. Also, several weeks later, uniform samples of an American Pineapple cheese were distributed, and, although this type of cheese is essentially a Cheddar cheese made from whole milk, there was little difficulty experienced in preparing uniform samples and getting them to the collaborators in condition satisfactory for analysis.

The collaborators were directed to carry out the work described in the following instructions:

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **3**: 436.

PREPARATION OF SAMPLES.

DRIED MILK AND MALTED MILK.

Thoroughly mix the entire sample before weighing a portion for analysis. Weigh out a 1 gram sample and transfer to the Röhrig tube with the help of a camel's hair brush and glazed paper. The mixing and weighing should be done as rapidly as possible for the reason that these powders very readily take up moisture from the air.

(*a*) *Alkaline method.*—Add 10 cc. of water to the sample in the Röhrig tube, place the tube in a water bath at 60°C. and mix the contents by frequent shaking until no lumps remain. Add 2 cc. of concentrated ammonium hydroxid, mix thoroughly, heat again in the water bath at 60°C., and proceed as directed in the official Roese-Gottlieb method.

(*b*) *Acid method.*—Heat the sample with 10 cc. of hydrochloric acid (sp. gr. 1.125) in the Röhrig tube, by means of a water bath at 80°C. for 15-25 minutes, being certain that the curd is well dissolved. Cool, add 10 cc. of alcohol, and proceed as in the official Roese-Gottlieb method.

ICE CREAM.

Allow the sample to soften at room temperature. Stir thoroughly with a spoon or mix by pouring from one beaker to another. Another good method of obtaining a uniform sample is to mix with an egg beater just before weighing. Owing to the fact that melted butter fat readily separates out and tends to rise to the surface, it is not advisable to soften the ice cream by heating on a water bath or over a flame. Weigh out 4 grams of the sample in a small dry beaker, add 3 cc. of water, thoroughly mix with a glass rod, and pour into the Röhrig tube, washing out the remaining portion with 3 cc. of water. Add 2 cc. of concentrated ammonium hydroxid, mix thoroughly, heat in a water bath at 60°C., and proceed as directed in the official Roese-Gottlieb method. The method of weighing may be simplified by pouring the mixed sample into a 25 cc. graduated cylinder. Weigh the cylinder, pour about the right amount into a tube, weigh, and determine the amount of the sample by difference.

CHEESE.

By means of a cheese sampler, draw three plugs, one from the center, one from a point near the outer edge, and one from a point between the other two. Reject the rind and grind the plugs in a sausage machine or cut them very finely and mix thoroughly. Proceed as directed in the Schmidt-Bondzynski method.

DESCRIPTION OF METHODS.

ROESE-GOTTLIEB METHOD.—OFFICIAL[1].

Notes on the method.—If an emulsion occurs in the Röhrig tube, the addition of a little alcohol, followed by shaking, will usually break it up. Also, the use of a tube having a larger caliber will tend to overcome this difficulty. If a Röhrig tube is not available, prepare an apparatus consisting of a large test tube provided with a blow-off device, as used in the Werner-Schmidt method. Care should be taken to make the final weighing of the ether-freed residue of fat under exactly the same conditions as those under which the dish was first weighed.

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 289.

HARDING-PARKIN METHOD[1].

In applying the method to malted milk, weigh out a 1 gram sample, transfer to a Werner-Schmidt extraction tube, add 8 cc. of acetic acid (25% by volume) and warm the contents of the tube to about 50°C. in a water bath. When the protein has dissolved, add 12.5 cc. of redistilled carbon tetrachlorid and shake the tube vigorously for 2 minutes, then add 25 cc. of 95% alcohol and shake thoroughly; add 25 cc. of petroleum ether, shaking vigorously for 2 minutes, and an additional 15 cc. of the same ether, and continue shaking 1 minute longer. Close the tube and let stand until separated. Insert the blow-off device and blow out the ether layer cautiously through a filter into a weighing flask, taking care that none of the carbon tetrachlorid is blown off. Place 5 cc. of petroleum ether in a small evaporating dish and gently draw into the tube by suction applied to the blow-off device. After the ether has mixed with the layer in the jar, blow off and filter as before. Add 5 cc. of carbon tetrachlorid to the contents of the jar, thoroughly shake, then add 30 cc. of petroleum ether with repeated thorough shaking. Let stand and allow time to separate and wash once, as in the first blow-off. Repeat the above operation, using 5 cc. of carbon tetrachlorid and 30 cc. of petroleum ether, wash the filter paper with small portions of petroleum ether, evaporate off the ether slowly and heat the flask in an oven at a temperature of 100°C. and weigh.

In applying the method to ice cream, weigh out a 5 gram sample. Add 5 cc. of acetic acid, 12 cc. of carbon tetrachlorid, 20 cc. of alcohol and 30 cc. of petroleum ether, and proceed as directed above.

SCHMIDT-BONDZNSKI METHOD, MODIFIED[2].

GENERAL INSTRUCTIONS.

Condition of samples.—Carefully note the condition of each sample. The ice cream should not be separated or fermented, should be perfectly smooth, containing no small lumps of fat or curded matter. The cheese should not show evidence of having been overheated or of separation of melted fat.

Comparative tests.—On the sample of dried milk, make the official Roese-Gottlieb determination, both after the regular alkaline method of preparation and after the acid method of preparation, as directed under "Preparation of Samples". On the sample of malted milk, make the same determinations as on the sample of dried milk and also make the determination by the Harding-Parkin method. On the sample of ice cream, make the official Roese-Gottlieb determination and also make the determination by the Harding-Parkin method. On the sample of cheese, make the determination by the Schmidt-Bondzynski method, modified.

Duplicate determinations.—In all cases, so far as possible, make two or more determinations by each method on all samples as directed. Report the individual results so obtained and calculate the averages of results which are in reasonable agreement.

Experience.—In the case of all methods make preliminary trials. Repeat as many times as seems necessary in order to become adequately prepared for the regular determinations to be made on selected samples. The importance of ample experience can not be overestimated, especially in connection with the Roese-Gottlieb method and the Harding-Parkin method.

Supplementing the foregoing instructions, at a later date, the collaborators were directed to make moisture determinations on all samples, except the sample of ice cream, according to methods of the association

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[1] *J. Ind. Eng. Chem.*, 1913, **5**: 131.

[2] *Assoc. Official Agr. Chemists, Methods*, 1916, 297.

for dairy products[1]. It was also directed that the results obtained by the fat determinations be reported on the dry basis, as well as on the original whole samples. It so happens, however, that no special method is given for malted milk and milk powders. A general method is described[2] which is applicable to milk and condensed milk products and there is also given the tentative method for cheese[3].

A special effort was made to distribute uniform samples of each kind of product among the collaborators. There was no difficulty in the case of the malted milk for the reason that the sampling was made from the same uniformly mixed batch and immediately put up in well-sealed containers. The dried milk samples were similarly made up, although they were enclosed in pasteboard cartons which, in several instances, reached the collaborators in a somewhat damaged condition. However, even in this case there was no evidence reported of serious change in composition, and the material was promptly placed in stoppered bottles when received. The plain ice cream was thoroughly worked up to uniform consistency before being placed in tightly covered glass jars, and each sample was furthermore preserved with a small quantity of corrosive sublimate. The samples of Parmesan and American Pineapple cheese were prepared in a satisfactory, uniform condition and great care was taken to put them up in suitable containers so that they might reach the collaborators without change or deterioration. Practically no serious complaints were received regarding the condition of the samples. Owing to the wide discrepancies shown among the moisture determinations on all kinds of samples, it has been decided to omit the results expressed on the dry basis. There are no circumstances to justify the belief that these differences could be due to actual variations in moisture content. For example, the results obtained on the malted milk show wide variations of from approximately 3.5 to 4.75 per cent. Similar results are noted among the other samples, particularly in the case of the Parmesan cheese. The method adopted for preparing and bottling the cheese samples was sufficient to assure the referee that no wide variations ought to be shown in the moisture results obtained by the various collaborators. The same statements can not be made with equal confidence regarding the samples of dried milk owing to the nature of the containers in which they were forwarded. Nevertheless, a careful inspection of the results for butter fat does not lead to any clue consistent with the wide variations reported among the results for moisture. These results vary from a trifle below 3.0 to somewhat over 7.5 per cent. This feature of the work has really no essential relation

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 293, 296.
[2] *Ibid.*, 293, **33.**
[3] *Ibid.*, 296, **53.**

to the plan of study adopted for the present year and was simply thrown in as an afterthought. Nevertheless, it is worth while calling attention to the fact that the methods for determining moisture on certain classes of dairy products are not in very satisfactory shape. An inspection of the tables simply indicates that there is here an opportunity for profitable study during the coming year. There is no moisture method adapted to such products as malted milk and dried milk, and some questions have been raised regarding the method for cheese[1].

TABLE 1.

Moisture determinations.

	MALTED MILK	DRIED MILK	CHEESE	
			Parmesan	Pineapple
	per cent	*per cent*	*per cent*	*per cent*
	3.55	5.76	10.25	23.71
	4.78	2.88	10.15	24.18
	3.90	4.75	10.45	23.68
	4.11	6.91	10.10	24.02
	3.71	7.65	10.15	24.21
	3.76	7.52	9.88	24.58
	3.58	5.66	9.07	23.16
	3.89	6.41	10.65	24.02
	3.40	6.60	9.94	24.09
	3.77	....	10.31	....
	3.78	....	10.23	....
Maximum	4.78	7.65	10.65	24.58
Minimum	3.40	2.88	9.07	23.16

A few comments on this subject have been submitted by collaborators as follows:

In the moisture determinations, it was found impossible to obtain constant weight upon the dried and malted milk samples by drying in the water bath. Moisture was determined by drying in a vacuum oven at 95°C. to constant weight. There was apparently no oxidation or darkening under these conditions. The time required was approximately 2 hours.

In determining moisture on the Parmesan cheese, the weight was found to gain after 4½ hours' heating. In the case of Pineapple cheese, the weight gained after 7½ hours' heating. The final weighing was made at the expiration of 9 hours.

In determining moisture on cheese, about 1 gram of material was weighed into a dish with a glass rod, 2 cc. of water added and the sample thoroughly mixed. The moisture was quickly evaporated on a hot plate at 180°C.; then the dish was placed in a vacuum oven at a temperature of 100°C. for 20 minutes. Heating was continued at 20 minute intervals to constant weight. Some difficulty was experienced in determining moisture on the Parmesan cheese.

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[1] *Assoc. Official Agr. Chemists, Methods,* 1916, 296, 53.

The results obtained by the various methods for the determination of fat have been compiled and arranged for purposes of comparison in Table 2.

Table 2.

Fat determinations on dairy products.

ANALYST	MALTED MILK			DRIED MILK		ICE CREAM		CHEESE	
								Par-mesan	Pine-apple
	Roese-Gottlieb Alkaline Method	Roese-Gottlieb Acid Method	Harding-Parkin Method	Roese-Gottlieb Alkaline Method	Roese-Gottlieb Acid Method	Roese-Gottlieb Method	Harding-Parkin Method	Schmidt-Bondzynski Method	Schmidt-Bondzynski Method
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
M. L. Jones, Sears, Roebuck & Co., Chicago, Ill.	9.00	9.26	8.88	1.07	1.10	7.69	7.75	23.07	....
	8.98	9.26	8.84	1.07	1.12	7.69	7.81	23.04	....
	9.00	9.28	8.85	1.05	1.11	....	....	....	....
Average..........	8.99	9.27	8.86	1.06	1.11	7.69	7.78	23.06	....
E. C. Thompson, Borden's Condensed Milk Co., New York, N. Y.	8.99	9.02	9.02	1.10	1.02	7.74	7.78	22.05	36.02*
	9.00	9.04	9.04	1.10	1.07	7.75	7.81	22.20	35.89*
	9.01	9.00	....	1.12	1.05	7.75	7.84	....	....
Average..........	9.00	9.02	9.03	1.11	1.05	7.75	7.81	22.13	....
H. E. Otting and E. E. Dysart, The John Wildi Evaporated Milk Co., Columbus, Ohio.	9.07	9.16	8.99	1.19	1.02	7.93	7.83	22.85	38.96*
	9.12	9.16	8.99	1.24	1.07	7.93	7.81	22.82	39.52
	9.16*	9.13	9.02	1.24	1.03	7.93	7.82	23.03	39.46
	9.07	9.13	9.00	1.16	1.13	7.92	7.80	....	39.03*
	....	9.19	9.00	....	....	7.92	....	....	39.88*
	....	....	....	....	....	7.90	....	....	39.55
Average..........	9.09	9.15	9.00	1.21	1.06	7.92	7.82	22.90	39.51
Raymond Hertwig, U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, San Francisco, Calif.	8.94	9.40	8.89	0.99	1.11	....	....	23.13	39.63
	8.97	9.42	8.94	1.03	1.15	....	....	23.31	39.77
	....	....	8.98	....	....	....	....	....	....
Average..........	8.96	9.41	8.94	1.01	1.13	....	....	23.22	39.70
E. W. Thornton, State Department of Agriculture, Raleigh, N. C.	8.99	9.34	9.50	1.31	1.56	7.90	7.73	22.57	38.67
	8.94	9.51	9.52	1.23	1.50	7.93	7.74	22.58	38.86
	8.92	9.52	....	1.20	1.58	7.73	....	22.45	....
	8.98	....	....	1.21	1.54	7.99	....	22.50	....
	....	....	....	....	1.64	7.64*	....	....	....
	....	....	....	....	1.51	7.80	....	....	....
Average..........	8.96	9.46	9.51	1.24	1.56	7.87	7.74	22.53	38.77

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* Not included in calculations.

TABLE 2.—Continued.

ANALYST	MALTED MILK			DRIED MILK		ICE CREAM		CHEESE	
								Parmesan	Pineapple
	Roese-Gottlieb Alkaline Method	Roese-Gottlieb Acid Method	Harding-Parkin Method	Roese-Gottlieb Alkaline Method	Roese-Gottlieb Acid Method	Roese-Gottlieb Method	Harding-Parkin Method	Schmidt-Bondzynski Method	Schmidt-Bondzynski Method
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
Miss Kathryn Holden, Hires Condensed Milk Co., Philadelphia, Pa.	....	....	....	0.926	0.927	7.86	7.89	22.59	39.74
	....	....	....	0.956	0.901	7.85	7.86	22.65	39.83
	....	....	....	0.987	....	7.84	....	....	39.66
Average..........	....	....	....	0.96	0.91	7.85	7.88	22.62	39.74
J. H. Bornmann, U. S. Food and Drug Inspection Station, Transportation Building, Chicago, Ill.	8.98	9.17	8.74	0.89*	1.01	7.69	7.35*	22.75*	39.42
	8.82	9.23	8.84	0.89*	1.00	7.63	7.44*	23.30	39.35
	....	....	....	....	....	....	....	23.26	....
Average..........	8.90	9.20	8.79	....	1.01	7.66	....	23.28	39.39
William Brinsmaid, Division of Foods and Dairies, State Department of Agriculture, Chicago, Ill.	7.39*	7.78*	6.43*	1.31	1.51	....	....	19.58*	36.55*
	7.39*	6.87*	7.11*	1.26	1.52	....	....	20.15*	36.12*
Average..........	....	....	....	1.29	1.52	....	....	....	....
J. T. Keister, Bureau of Chemistry, Washington, D. C.	9.028	9.480	8.990	1.107	1.256	7.856	7.848	22.92	39.76
	9.133	9.375	9.934*	1.150	1.293	7.837	7.780	22.96	39.75
	9.017	9.453	8.966	1.189	1.276	....	7.890	23.12	39.62*
	8.910	9.540	9.075	1.146	....	....	7.752	....	39.755
	....	9.560	....	....	....	....	7.820	....	39.76
Average..........	9.022	9.482	9.010	1.15	1.28	7.85	7.82	23.00	39.76
Hugo Ringstrom, State Dairy and Food Department, St. Paul, Minn.	9.20*	9.10	....	....	....	....	....	22.96	39.30
	9.14	8.98	....	....	....	....	....	22.84	39.37
	9.05	9.02	....	....	....	....	....	22.72	39.42
Average..........	9.10	9.03	....	....	....	....	....	22.84	39.36
Walter Egge, State Dairy and Food Department, St. Paul, Minn.	....	8.86	9.05	....	....	....	....	22.61	....
	....	9.03	8.90	....	....	....	....	22.58	....
	....	8.96	9.01	....	....	....	....	22.65	....
	....	8.96	9.03	....	....	....	....	....	....
	....	8.93	8.92	....	....	....	....	....	....
	....	8.97	....	....	....	....	....	....	....
Average..........	....	8.95	8.98	....	....	....	....	22.61	....
Maximum..........	9.10	9.48	9.51	1.29	1.56	7.92	7.88	23.28	39.76
Minimum..........	8.90	8.95	8.79	0.96	0.91	7.66	7.74	22.13	38.77
Differences..........	0.20	0.53	0.72	0.33	0.65	0.26	0.14	1.15	0.99

* Not included in calculations.

It was intended to include among the reported results only such figures as were believed by the analysts to be obtained by a proper procedure and concerning which there could be no assignable reason for doubt. It was, furthermore, contemplated that these averages would represent fairly the results obtained upon the various samples. In computing these averages, attention has been given to the customary rule of discarding values which are not in reasonable agreement. If two results have been found to agree within reasonable limits, it has been considered fair to report the mean of those results. If more than two results have been obtained by repeated determinations, only such values as agree within reasonable limits have been used in computing the averages. The comparisons are therefore made strictly among the averages, although it is interesting to note the variations which exist among the large number of individual determinations. In the case of the uniform samples of malted milk, the smallest variation between maximum and minimum results is found in the column headed "Roese-Gottlieb Alkaline Method". The acid method, on the other hand, fails to yield results which are in equally satisfactory agreement. The maximum and minimum difference indicated among the results obtained in the alkaline method is 0.20 per cent, whereas in the case of the acid method the corresponding figure is 0.53. The results obtained by the Harding-Parkin method are less satisfactory, although there are no serious criticisms upon this method, except that the method is rather time-consuming and involves a few cumbersome details. The results obtained upon the sample of dried milk, while hardly as satisfactory as could be wished, are nevertheless not especially disappointing when it is considered that the product was a skimmed milk powder containing about 0.1 per cent of butter fat. No decided preference seems to be indicated in the preliminary treatment of the sample in favor of either the alkaline or the acid method. The results obtained on the ice cream samples are very satisfactory, especially those obtained by the official Roese-Gottlieb procedure. The maximum and minimum variations cover a range of only 0.26 per cent. The results reported by the Harding-Parkin method are equally satisfactory and show even a somewhat smaller difference between the maximum and minimum values, 0.14 per cent. Barring the objection pointed out by a few of the collaborators regarding the time consumed in carrying out this latter procedure, it may be said that there is no decided preference regarding the relative merits of the Harding-Parkin method as compared with the official Roese-Gottlieb method applied to ice cream. An inspection of the results obtained on the two samples of cheese leads to the conclusion that the Schmidt-Bondzynski method is capable of yielding satisfactory results, especially when it is considered that there were somewhat greater difficulties to be encountered in preparing and forwarding uniform samples of products of this class. The variations among the maximum and minimum values have a range of about 1.0 per cent, being in the case of the Parmesan cheese 1.15 per cent, and in the case of the American Pineapple 0.99 per cent. Considering, also, the high fat content of the cheese samples, it may be concluded that these results are approximately as good as may be expected in practical work. One or two of the collaborators have suggested criticisms regarding the tentative method for cheese, and appear to prefer the long discarded treatment employed in the application of the Werner-Schmidt method, which consisted in the digestion of the sample with dilute hydrochloric acid. There are also a few more or less positive differences of opinion expressed regarding the difficulties arising on account of the formation of foam. While there are certain analysts who encounter difficulties in the application of the alkaline method, there are others who apply the same criticism to the acid procedure. However, the consensus of opinion seems to favor the alkaline mode of preparing the sample and the practical difficulties indicated do not appear to be formidable. Such annoyances as troublesome emulsions, foams, etc., can be overcome easily by proper exercise of ingenuity and ordinary analytical technique. On the whole, it is gratifying to conclude that the results reported are very satisfactory.

RECOMMENDATIONS.

It is recommended—

(1) That the Roese-Gottlieb method, as applied to malted milk products and plain ice cream, be adopted as official.

(2) That the Harding-Parkin method for fat in ice cream be adopted as a tentative method.

(3) That the Schmidt-Bondzynski modified method for the determination of fat in cheese be adopted as official.

(4) That a further study be made of the Roese-Gottlieb method as applied to dried milk products having a high as well as a low content of butter fat.

(5) That a study be made of methods for the determination of moisture in milk products, including condensed and sweetened condensed milk, dried milk and malted milk, and that the tentative method for the determination of moisture in cheese[1] be subjected to further study with a view to its adoption as an official method.

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 296, 53.

REPORT ON THE SEPARATION OF NITROGENOUS SUBSTANCES IN MILK AND CHEESE.

By L. L. VAN SLYKE (Agricultural Experiment Station, Geneva, N. Y.), *Associate Referee.*

Last year it was recommended that study be continued leading to the adoption of methods for the determination of the non-casein proteins and the products of protein decomposition in milk.

It was also recommended that collaborative work on the subject of enzym reactions of milk be undertaken. Before such enzym work can be done to advantage, much serious study must be made of the tests that are in use. The methods now used are largely empirical, and the interpretations based on the results of their use are far from satisfactory. A beginning was made in the study of the methylene blue reaction. The work, however, has been carried only far enough to reveal how much remains to be learned about the details of the action of this reagent in milk before it can be utilized as an intelligible and dependable means for practical use in relation to milk. The demands of this field are sufficient to require a separate referee to carry on research work, preparatory to collaborative investigations.

The referee did not reach the special subject of non-casein proteins in milk. In his judgment further work remained to be done with casein and its separation from milk before taking up details involving the isolation of the other milk proteins and the study of their properties in pure form.

The progress made by the referee on the preparation of pure casein[1] was then presented.

No report on spices and other condiments was made by the referee.

No report on cacao products was made by the referee.

REPORT ON COFFEE.

By H. A. LEPPER (Bureau of Chemistry, Washington, D. C.), *Referee.*

The Stahlschmidt method for the determination of caffein in tea has been before the association since 1911 and was slightly modified and used for this determination on coffee in 1915[2]. This modified method was recommended for official adoption this year.

In view of the fact that very few collaborators offered their services in 1915 and that the method had little trial on coffee, some preliminary

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[1] *J. Biol. chem.*, *1918*, 35: 127.
[2] *J. Assoc. Official Agr. Chemists*, 1917, 3: 21.

analyses were made by this method on several samples of coffee, yielding the results shown in Table 1. The nitrogen determinations reported by the referee were made by the Nitrogen Laboratory, Bureau of Chemistry, Washington, D. C.

TABLE 1.

Comparison of the Stahlschmidt and Fendler and Stüber methods for the determination of caffein in coffee.

SAMPLE NUMBER	DESCRIPTION OF SAMPLE	STAHLSCHMIDT		FENDLER AND STÜBER	
		Gravimetric	N × 3.464	Gravimetric	N × 3.464
		per cent	*per cent*	*per cent*	*per cent*
1	Santos medium roast.........	1.33	1.17	1.23	1.14
		1.33	1.17	1.23	1.16
		1.53	1.16	....	....
2	Santos dark roast............	1.43	1.24	1.19	1.14
		1.52	1.24	1.18	1.18
		1.38	1.14	1.20	1.14
		1.42	1.16	....	....
		1.37	1.14	....	....
	Average................	1.41	1.18	1.21	1.15
3	Coffee "caffein-free".........	0.25	0.09	0.09	0.05
		0.23	0.09	0.09	0.05
		0.27	0.12	....	....
		0.24	0.12	....	....
	Average...............	0.25	0.11	0.09	0.05

These results on pure coffee compare favorably with those reported in 1915, showing a variation averaging 0.24 per cent in the values obtained by weighing the residue and by calculating from the nitrogen determinations. However, the values on a commercial sample of coffee (No. 3), labeled as coffee having 95 per cent of the caffein removed, are not so favorable. The results show that the caffein obtained contains impurities greater in weight than the alkaloid itself. Moreover, the amount of sample required by this method gave a weight of residue of about 5 mg., which, being less than one-half caffein, furnished a rather small sample for the determination of nitrogen. The errors incident to the nitrogen determination become relatively larger than with larger residues of crude caffein. As the so-called caffein-free coffees are becoming more plentiful and more widely used, it seemed desirable to the referee to have a method better adapted to these products and at the same time one which will give a purer crude caffein. The following method was chosen from among several[1]:

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[1] J. Burmann. *Bull. soc. chim.*, 1910, **7**: 239; C. A., 1910, **4**: 1777.
C. Virchow. *Chem. Ztg.*, 1910, **34**: 1037; C. A., 1911, **5**: 542.
F. Adam. *Arch. Chem. Mikros.*, 1910, **3**: 212; C. A., 1911, **5**: 1470.
G. Costes. *Ann. chim. anal.*, 1912, **17**: 246; *Analyst*, 1912, **37**: 401.

DETERMINATION OF CAFFEIN IN COFFEE[1].

By G. Fendler and W. Stüber.

Pulverize the coffee to pass without residue through a 1 mm. sieve. Treat a 10 gram sample with 10 grams of 10% ammonium hydroxid and 200 grams of chloroform in a glass-stoppered bottle and shake continuously by machine or hand for 30 minutes. Pour the entire contents of the bottle on a 12.5 mm. folded filter, covering with a watch glass. Weigh 150 grams of the filtrate into a 250 cc. flask and evaporate on the steam bath, removing the last chloroform with a blast of air. Digest the residue with 80 cc. of hot water for 10 minutes on the steam bath, with frequent shaking, and let cool. Treat the solution with 20 cc. (for roasted) or 10 cc. (for unroasted) of 1% potassium permanganate and let stand 15 minutes at room temperature. Add 2 cc. of 3% hydrogen peroxid (containing 1 cc. of glacial acetic acid in 100 cc.). If the liquid is still red or reddish, add hydrogen peroxid, 1 cc. at a time until the excess of potassium permanganate is destroyed. Place the flask on the steam bath for 15 minutes, adding hydrogen peroxid in 0.5 cc. portions until the liquid ceases to become lighter. Cool, and filter into a separatory funnel, washing with cold water. Extract four times with 25 cc. of chloroform. Evaporate the chloroform extract from a weighed flask with the aid of an air blast and dry at 100°C. to constant weight (30 minutes is usually sufficient). Weigh the residue as caffein and calculate on 7.5 grams of coffee. Test the purity of the residue by determining nitrogen and multiplying by the factor 3.464.

Fendler and Stüber, in considering more than twenty methods and studying those of C. C. Keller[2], J. Katz[3], and Lendrich and Nottbohm[4] found the last two to be trustworthy but long and tedious. They also found, as did the referee for 1910[5] and 1911[6], that the Lendrich-Nottbohm method gave an exceptionally pure caffein but low results unless the extraction was carried on for at least 6 hours. The exceptional purity of the caffein can be attributed to the action of potassium permanganate on the crude caffein residue, a procedure of purification employed originally by Lendrich and Nottbohm. Fendler and Stüber after a study of the extraction, separation of the fat, purification and drying of the caffein, have retained the potassium permanganate purification in principle and have included it in a composite of the Katz and Lendrich-Nottbohm methods.

Analyses were made on the same samples by this method by the Stahlschmidt method and the results are given in Table 1. The averages on pure coffee show a variation of 0.05 per cent between the weighed caffein and the calculated nitrogen value, indicating a much purer product than that obtained by the other method. The results on the "caffein-free" sample also show a very much purer caffein. Besides the advantage of yielding a residue only slightly contaminated, the Fendler-Stüber

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[1] *Z. Nahr. Genussm.*, 1914, **28**: 9; *C. A.*, 1914, **8**: 3599.
[2] Ber. pharm. Ges., 1897, **7**: 105.
[3] *Ibid.*, 1902, **12**: 250.
[4] Z. Nahr. Genussm., 1909, **17**: 241.
[5] U. S. Bur. Chem. Bull. **137**: (1911), 106.
[6] *Ibid.*, **152**: (1912), 163.

method is easier of manipulation and much quicker than the Stahlschmidt method and, at the same time, uses a sample three times as large.

Three samples of coffee—A, "caffein-free" coffee purchased on the market; B, Santos medium roast; and C, Rio medium roast—were ground to pass a 1 mm. sieve without residue, were thoroughly mixed and sent to twelve collaborators with the request that they be examined by the Stahlschmidt[1] and the Fendler-Stüber methods. Although the latter method was tried in its original form, it is believed that an improvement tending to easier, quicker, and more accurate manipulation is

TABLE 2.

Results of collaborative work on Sample A.

ANALYST	CAFFEIN			
	Stahlschmidt		Fendler and Stüber	
	Gravimetric	N × 3.464	Gravimetric	N × 3.464
	per cent	*per cent*	*per cent*	*per cent*
H. J. Wichmann, U. S. Food and Drug Inspection Station, Tabor Opera House Building, Denver, Colo.	0.30 0.31	....	0.109 0.094	....
G. N. Watson, reported by L. E. Sayre, School of Pharmacy, University of Kansas, Lawrence, Kans.	0.22 0.22	0.189	0.117 0.085	0.095 ....
E. C. Merrill, Bureau of Chemistry, Washington, D. C.	0.237	....	0.18	....
Leicester Patton, U. S. Food and Drug Inspection Station, Federal Building, Buffalo, N. Y.	0.50	0.09	0.09	0.07
E. M. Bailey, reported by J. P. Street, Agricultural Experiment Station, New Haven, Conn.	0.23	0.13	0.08	0.05
C. E. Shepard, reported by J. P. Street.	....	....	0.08	0.06
C. B. Gnadinger, reported by G. W. Hoover, U. S. Food and Drug Inspection Station, Transportation Building, Chicago, Ill.	0.25	0.10	0.09	0.06
C. K. Glycart, reported by G. W. Hoover.	0.11	0.05*	0.04 0.06	0.04*
H. A. Lepper......................	0.23 0.25	0.07 0.07	0.11 0.11	0.05 0.05

* Purified by iodin method. U. S. Bur. Chem. Bull. **137**: (1911), 191.

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 332.

possible. It is suggested that the filtrate from the first chloroform extract be caught in a tared flask, with stopper, and the entire filtrate weighed to the nearest tenth of a gram, thereby eliminating the necessity of weighing 150 grams of volatile chloroform solution with the coincident concentration by evaporation. The results received from nine collaborators are submitted in Tables 2, 3 and 4.

TABLE 3.

Results of collaborative work on Sample B.

ANALYST	CAFFEIN			
	Stahlschmidt		Fendler and Stüber	
	Gravimetric	N × 3.464	Gravimetric	N × 3.464
	per cent	*per cent*	*per cent*	*per cent*
H. J. Wichmann	1.39	....	1.26	....
G. N. Watson	1.092	1.041	1.19	1.179
	1.15	....	1.205	....
E. C. Merrill	1.02	....	1.14	....
Leicester Patton	1.52	1.38	1.21	1.09
	1.55	1.33	1.26	1.19
E. M. Bailey	1.30	1.18	1.19	1.12
C. E. Shepard	....	....	1.24	1.16
C. B. Gnadinger	1.34	1.16	1.21	1.16
C. K. Glycart	1.31	1.08*	1.07	1.01*
H. A. Lepper	1.39	1.14	1.23	1.10
	1.34	1.12	1.24	1.14

* Purified by the iodin method. U. S. Bur. Chem. Bull. 137: (1911), 191.

DISCUSSION OF RESULTS.

On pure coffee, Samples B and C, the Fendler-Stüber method, judging from the nitrogen determination, gives a purer caffein than the Stahlschmidt method and in all cases but one gives a percentage of alkaloid equal to that obtained by the other procedure. The results on the "caffein-free" coffee, Sample A, also show a purer caffein obtained by the Fendler-Stüber method but in all cases a slightly less amount. It is possible that, in the method of manufacture of this type of product, nitrogen-containing bodies are affected in a manner whereby they contaminate the crude residue of caffein in the Stahlschmidt method but are eliminated in the purification process of the Fendler-Stüber method. The analysts who commented on the methods were unanimous in their preference for the Fendler-Stüber method.

TABLE 4.

Results of collaborative work on Sample C.

ANALYST	CAFFEIN			
	Stahlschmidt		Fendler and Stüber	
	Gravimetric	N × 3.464	Gravimetric	N × 3.464
	per cent	*per cent*	*per cent*	*per cent*
H. J. Wichmann	1.35	....	1.24	....
G. N. Watson	1.088 1.084	1.051	1.169 1.168	1.148 ....
E. C. Merrill	1.06	....	1.21	....
Leicester Patton	1.50 1.50	1.21 1.38	1.15 1.26	1.09 1.13
E. M. Bailey	1.33	1.18	1.16	1.10
C. E. Shepard	....	....	1.20	1.15
C. B. Gnadinger	1.35	1.14	1.21	1.14
C. K. Glycart	1.20	1.02*	1.11	1.08*
H. A. Lepper	1.22 1.23	1.09 1.09	1.21 1.21	1.14 1.14

* Purified by iodin method. U. S. Bur. Chem. Bull. 137: (1911), 191.

RECOMMENDATIONS.

In view of the concordant satisfactory results obtained by the collaborators and the many advantages of the Fendler-Stüber method, it is recommended—

(1) That the Gorter method for the determination of caffein in coffee[1] be dropped.

(2) That the Stahlschmidt method be not made official this year.

(3) That the Fendler-Stüber method be adopted tentatively.

(4) That the Fendler-Stüber method be tried next year on other coffees, including raw coffee, with a view to its adoption as official.

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 332.

REPORT ON TEA.

By E. A. Read (Bureau of Chemistry, Washington, D. C.), *Referee*.

According to the 1916 annual report of the supervising tea examiner of the Treasury Department practically no colored tea (0.00197 per cent) was offered for entry into this country during that year. The problems in connection with adulteration could be well included under microscopical methods, otherwise, the examination of tea seems to be entirely chemical. It is therefore suggested that the subjects, coffee and tea, be combined as in previous years and assigned to a chemist as referee.

REPORT ON BAKING POWDER.

By. H. E. Patten[1] (Bureau of Chemistry, Washington, D. C.), *Referee*.

Following the recommendations of the referee for 1916, a further study of the Wichmann, and of the newly proposed Chittick, and Corper-Bryan methods for the determination of lead in baking powder has been conducted. The results obtained in 1916 demonstrated that none of the available methods for lead was satisfactory. Consequently, the collaborators were advised to vary conditions, make preliminary runs and do independent research, as well as to analyze samples of baking powder containing known quantities of lead.

In addition to the work on lead, collaborators have greatly improved the method of determining fluorin, so that it is now possible to present a rapid, accurate method of great range as to quantity determined which has the added value that silico-fluorids, as well as fluorids, give up their fluorin.

In connection with the lead investigation, experiments were made to determine the hydrogen ion concentration at which calcium citrate is held back from rapid precipitation in so-called neutral ammonium citrate solution, which is of general analytical interest, as well as the hydrogen ion concentration at which lead sulphid is precipitated, and the iron held up in solution under the conditions of the Wichmann modification of the Seeker-Clayton method.

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[1] Present address, Provident Chemical Works, St. Louis, Mo.

WORK ON COLLABORATIVE SAMPLES.

The samples were made up in 100 pound lots from standard baking powder ingredients as used in the trade, and the lead when added was in the form of lead sulphate. The proportions of ingredients and kinds of baking powder making up the various samples are given in Table 1.

TABLE 1.

Data on preparation of collaborative samples of baking powder.

SAMPLE NUMBER	KIND OF BAKING POWDER	COMPOSITION
1701	Monocalcium phosphate powder	Soda .27 Phosphate .37 Starch .36
1702	Monocalcium phosphate powder + known lead.	2.656 grams of lead sulphate per 80 pounds = 50 parts per million added.
1711	Sodium aluminium sulphate baking powder.	Soda .27 Sodium aluminium sulphate .26 Starch .47
1712	Sodium aluminium sulphate baking powder + known lead.	2.656 grams of lead sulphate per 80 pounds = 50 parts per million added.
1721	Combination phosphate, sodium aluminium sulphate baking powder.	Soda .27 Sodium aluminium phosphate .21 Phosphate 8 Starch .44
1722	Combination phosphate, sodium aluminium sulphate baking powder + known lead.	2.656 grams of lead sulphate per 80 pounds = 50 parts per million added.

It will be noted that Nos. 1701, 1711 and 1721 are the samples containing no added lead. Their lead content was obtained by analysis. The lead added amounted to 50 parts per million parts of baking powder, calculated as metallic lead.

NOTE: *In the following methods, lead-free reagents must be used.*

Gravimetric Method for the Determination of Lead in Baking Powder.

(Proposed by J. R. Chittick, Jaques Manufacturing Company, Chicago, Ill.)

PREPARATION OF REAGENTS.

Sulphuric acid (1 to 5).—Mix 500 cc. of 95% sulphuric acid, C. P., with 2500 cc. of water, let stand overnight, and filter.

Acid-alcohol-water mixture.—Mix 80 cc. of 95% sulphuric acid, C. P., with 3000 cc. of water. Then add 800 cc. of redistilled 95% alcohol (methyl or ethyl), stir thoroughly, let stand overnight, and filter.

Glacial acetic acid.—Redistil C. P. glacial acetic acid and store in bottles made from lead-free glass.

Alkaline ammonium acetate solution.—Mix 350 cc. of redistilled glacial acetic acid with 650 cc. of water; dilute 500 cc. of ammonium hydroxid, C. P. (sp. gr. 0.90), with 500 cc. of water; then mix the two solutions. Store in bottles made from lead-free glass.

Potassium chromate solution.—Dissolve 65 grams of C. P. potassium chromate in 100 cc. of water, heating gently. Allow the solution to come to room temperature and filter.

DETERMINATION.

Weigh 100 grams of the thoroughly mixed sample and place in a 2 liter lipped beaker˙ Add, in small portions, 750 cc. of dilute sulphuric acid (1 to 5). When frothing has ceased, mark the volume of the mixture on the side of the beaker. Heat on the hot plate to boiling and continue boiling for 3–4 minutes; then heat on the steam bath until the starch is hydrolyzed, which requires 20–30 minutes. The mixture will have a yellow color. (See Note 1, below.)

Remove and add, while stirring, C. P. calcium sulphate which has first been finely powdered in a mortar and rubbed with water to a thin paste.

> Monocalcium phosphate baking powder does not need the addition of calcium sulphate, since of itself it forms sufficient insoluble residue. To combination baking powders containing in part monocalcium phosphate, add 10 grams of calcium sulphate. To all other baking powders, add 15 grams of calcium sulphate.

Cool and make up to the original volume with water. Add, while stirring, 1 liter of filtered 95% alcohol, either methyl or ethyl, cover and let stand overnight.

By means of a siphon which can be controlled by a pinch cock, transfer the clear supernatant liquid to a dense 50 cc. conical alundum filter, using suction. To the moist residue remaining in the beaker, add 100 cc. of the acid-alcohol-water mixture. Stir well and let settle. Pour this liquid on the filter. Repeat this operation, using a fresh 100 cc. portion of the acid-alcohol-water mixture. Wash the beaker, residue and filter with 70% alcohol, passing the washings through the filter until the filtrate is nearly free from acid. Discard the filtrate and washings. Wash the filtering flask. Dry the alundum filter and its residue. Transfer the bulk of the residue from the filter to the original beaker. Extract the lead sulphate from the residue by using 100 cc. portions of alkaline ammonium acetate solution and by heating to boiling; pass the washings through the alundum filter, using suction. Five extractions are necessary.

Transfer the filtrate, which will measure about 500 cc., to a lipped beaker (See Note 4, page 220). Neutralize with glacial acetic acid, using litmus paper as an indicator; then add 10 cc. of glacial acetic acid in excess. Heat nearly to boiling and add 25 cc. of saturated potassium chromate solution. Cover and let stand overnight at room temperature. Filter through a tared Gooch crucible. Wash well with cold water and dry at 125°C. for at least 45 minutes. Cool in a desiccator and weigh.

The weight of lead chromate multiplied by the factor 0.641 gives the weight of the lead.

NOTES.

1. During the hydrolysis of the starch, do not heat the mixture to a brown color, as this greatly interferes with the filtration.
2. The calcium sulphate is added as a diluent and carrier for the lead sulphate.
3. The alcohol used should be redistilled and kept in glass. Either methyl or ethyl alcohol is efficient.

4. If desired, an aliquot (10 cc.) of the alkaline ammonium acetate solution containing the lead may be used colorimetrically[1].

5. A practically complete solution of the calcium sulphate by the alkaline ammonium acetate solution is advisable for the complete solution of the lead sulphate.

6. The Gooch crucible should be prepared with a good felt of purified asbestos fiber and dried at 125°C. for at least 45 minutes.

ELECTROLYTIC METHODS.

In 1915 the referee made a few preliminary experiments on the conditions for depositing lead from very dilute acid solutions[2] and from solutions of baking powders by use of the electric current. A brief summary follows:

In depositing from clear nitric acid solution upon the anode, using known quantities of lead, no deposit was found for quantities lower than 24 parts per million. With 24 parts per million in solution, 60 per cent of the total lead was deposited after 18 hours' electrolysis with a $C. D._{100} = 1.8$ amperes.

From a dilute phosphoric acid solution, containing 50 parts per million of lead, 95 per cent of the total amount was deposited (in electrolyzing overnight) on the cathode with a $C. D._{100} = 0.2$ amperes.

By adding dilute phosphoric acid to baking powder, a pasty mixture was obtained from which, with varying currents, lead was not deposited. Upon the addition of a small amount of nitric acid, which partly hydrolyzed the starch, a deposit was obtained. This was so crusted over with starch and salts, however, that it could not be handled.

When the baking powder was completely neutralized with nitric acid and the starch hydrolyzed until a practically clear solution was obtained, no deposit was secured upon the anode with prolonged electrolysis and varying current density.

T. J. Bryan (Calumet Baking Powder Company, Chicago, Ill.) informed the referee that he had modified the method of H. J. Corper (Calumet Baking Powder Company, Chicago, Ill.) and could recommend it. Consequently, the referee discontinued his experiments and directed attention to this Corper-Bryan method. After the apparatus and operating conditions had been standardized by the referee and G. H. Mains (Bureau of Chemistry, Washington, D. C.), the method was submitted to the collaborators of 1917.

Trials using the original directions failed to give concordant results. Experiments by A. H. Fiske of Rumford Chemical Works, Providence, R. I., and N. V. S. Malmstrom of Wilckes, Martin and Wilckes, Camden, N. J., and by David Klein and A. K. Epstein of the State Department

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 346.
[2] *Cf.* E. F. Smith. Electro-Analysis. 5th ed., 1911, 104–8.

of Agriculture, Chicago, Ill., showed the variations to be largely due to high acidity during electrolysis, and that by suitably modifying the method to control the acidity good results could be obtained.

The following revised directions for the Corper-Bryan electrolytic method were then sent out for use with the collaborative samples described above.

Electrolytic Method for Determination of Lead in Baking Powders.

(Bryan's modification of the Corper method.)

APPARATUS.

The necessary apparatus is shown in Fig. 1 and consists of:

(*a*) *A source of direct current.*—This source may be either a commercial D. C. 110-volt or 220-volt circuit, an A. C. 110-volt or 220-volt circuit rectified to D. C. or a storage battery of at least 3 cells. The potential of the current at the source must be at least 6 volts.

(*b*) *A means of varying the current.*—This is accomplished by a variable rheostat (series resistance) or by a variable shunt resistance.

(*c*) *A means of measuring the current.*—A D. C. ammeter in the circuit, as shown in Fig. 1, should be graduated at least to 0.1 ampere divisions and have a range from 0 to at least 5 amperes.

(*d*) *A decomposition bath.*—A tall 500 cc. beaker serves very well for the bath. The anode consists of a piece of platinum foil 25 mm. square, welded to a small piece of platinum wire (1—2 cm. long) which, in turn, is fused into a glass tube. By partly filling the tube with mercury and inserting the wire from the circuit, contact is made with the anode foil. The cathode consists of 30 cm. of No. 22 platinum wire with one end fused in a glass tube, and the remainder of the wire wound into a spiral about 30 mm. in diameter and in a plane perpendicular to the axis of the glass tube. Details are shown in Fig. 1. Both anode and cathode tubes should be tested to be sure that mercury does not leak around the fused-in wires. If it does, the determination will be spoiled.

(*e*) *A means of starting or stopping the current.*—A single throw single blade switch, C, placed near the deposition bath forms a convenient means of opening or closing the circuit.

PREPARATION OF SAMPLE.

Place 100 grams of baking powder in a tall 500 cc. beaker. Slowly add 75 cc. of concentrated hydrochloric acid (sp. gr. 1.18), stirring continuously. The acid must not be added so rapidly that the mixture foams over the top of the beaker. A thick paste results, to which add, slowly with stirring, 75–100 cc. of luke warm water. When the action has ceased, place on a steam bath and warm until the starch is completely hydrolyzed. (Test for starch with iodin solution.) Upon the completion of hydrolysis, add concentrated ammonia carefully from a burette until the first trace of precipitate of calcium phosphate is formed. Then add 5 cc. of hydrochloric acid (sp. gr. 1.18), and make up to 400 cc. with water. (The 400 cc. level should be previously marked on the beaker.) Warm if necessary to completely dissolve the precipitate. The mixture is then ready for electrolysis.

ELECTROLYSIS.

Insert the cathode in the solution until the spiral is about 2 cm. from the bottom of the beaker; then insert the anode above the cathode, so that there is 1 cm. between the spiral and the plate. The electrodes may be conveniently supported in the proper position by a ringstand and clamps. Cover the beaker during electrolysis with a couple of split electrolytic watch crystals or a heavy card cut to fit around the electrodes. The temperature during electrolysis should be room temperature or higher, but must not exceed 50°C. Make connections to the electrodes and close the switch with the full resistance in the electrolyzing circuit. (With the variable rheostat type, the total resistance of the rheostat must be large enough to cut the current down to approximately 0.1 ampere.) Then cut resistance out of the circuit until the ammeter shows 0.3 ampere flowing. Electrolyze for 8 hours (or overnight).

CLEANING THE ELECTRODES.

Upon completion of the electrolysis, considerable quantities of salts will frequently be found adhering to the lead deposit on the cathode. The quantity of salts can be lessened by additions of water during electrolysis to keep the 400 cc. level constant. Carefully remove the beaker without jarring the electrodes, while the current is still on. Replace this beaker immediately by one of the same size, filled with water to which 2 cc. of concentrated hydrochloric acid have been added. Allow the current to run until the salts have been removed from the electrodes; then, without interrupting the current, replace the beaker with one filled with water, and continue the current for 30 minutes.

TREATMENT OF LEAD DEPOSIT.

Remove the beaker of water, open the switch, and wash the cathode in a 50 cc. beaker containing sufficient alcohol to cover the lead deposit. Dry in air, and dissolve the lead from the cathode with 3–4 cc. of concentrated nitric acid in a 50 cc. beaker, heating gently to aid solution. Wash the cathode with dilute nitric acid and remove. Evaporate the contents of the beaker to drive off the excess of nitric acid, being careful not to overheat. Take up the residue with 20 cc. of 20% acetic acid by warming. Filter, and precipitate with 2 cc. of saturated potassium dichromate solution. Allow to stand overnight, filter on a tared Gooch, wash with water, dry for 30 minutes at 125°C., cool and weigh as lead chromate. Multiply the weight of lead chromate by the factor 0.641 to obtain the weight of lead.

Set-Up for Making Simultaneous Determinations with the Electrolytic Method.

A very convenient installation has been devised by A. H. Fiske for plant control work by means of which it is possible to make several determinations at a time, or only one, as desired. It also serves to make sure that the current is not interrupted during the washing of the lead deposit.

The general scheme of wiring is given in Fig. 2. It can be adapted to any source of direct current. Fundamental to this scheme of wiring is the rocking three point double throw switch (as sold in the trade) which will not break circuit on three points until the second three points are engaged. This insures an uninterrupted flow of current. The idea here is to connect the electrolytic cells in series with the variable resistance, L, and the ammeter, A, while the lead is being deposited from the original solutions of baking powder. The rocking switches, S, will all be in the lower position making contact with the dead point, O, during this operation. The resistance, L, is regulated to secure the required current through the series of cells. When the electrolysis is completed the cells must be washed in turn without breaking the current in the other cells which still contain the solution. The switch, S, of the first cell is thrown to the upper position, and the first cell will now be in parallel with the group of other cells. The beaker of the first cell may be removed and replaced with one of water and the electrodes washed according to the directions for the single determination with the Corper-Bryan

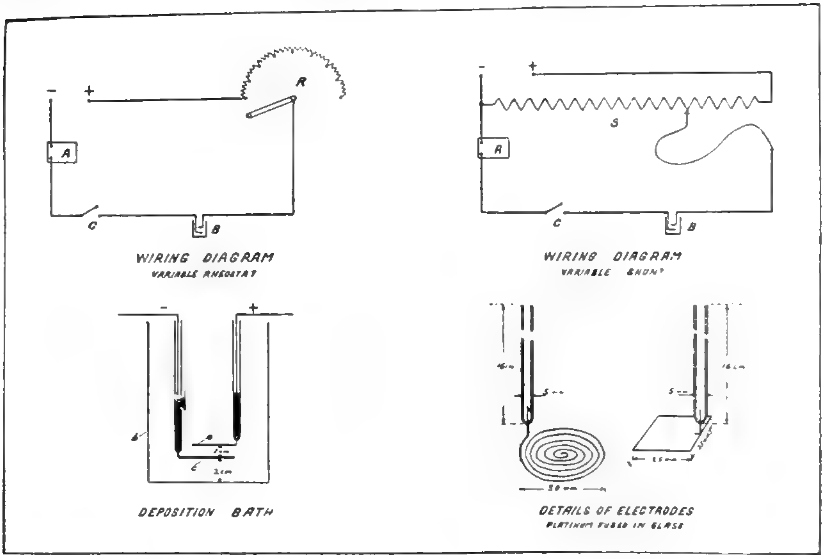

FIG. 1. APPARATUS FOR ELECTROLYTIC DETERMINATION OF LEAD.

Wiring Diagrams:
A = Ammeter.
B = Deposition bath.
C = Switch.
R = Variable rheostat.
S = Variable shunt.

Deposition Bath:
a = Anode (plate).
b = Beaker.
c = Cathode (spiral).

Details of Electrodes:
Spiral = 30 cm. of No. 22 wire.
Plate = Foil—25 mm. square.

method. The placing of this cell in parallel gives an increased voltage across the remaining cells and a resulting increased current in them which should be regulated by the variable resistance.

The other cells may in turn and in a similar manner be removed from the series circuit and the electrodes washed by using the first replacement beaker with water and 2 cc. of hydrochloric acid, and the second with water alone. Following that the cathode is removed from the holder, washed in alcohol in a small beaker, and then the

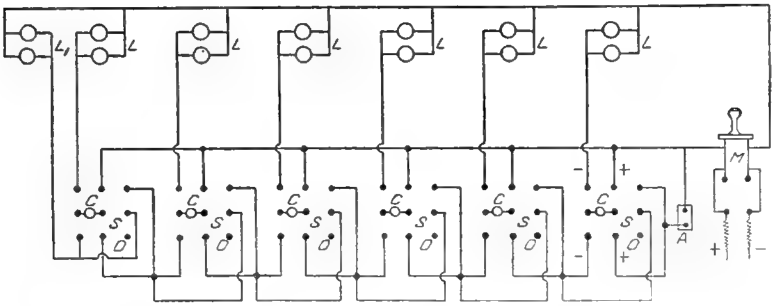

FIG. 2. ELECTROLYTIC SET-UP FOR PLANT CONTROL WORK.

A—Ammeter.
C—Electrolytic cell.
L—Electric bulbs or other resistance.
M—Main line switch.
O—Point in switch S intentionally left dead.
S—Triple point double throw switch.

lead is dissolved with concentrated nitric acid heated gently. The lead is determined gravimetrically after precipitation as the chromate, or for plant control work may be determined colorimetrically by comparison with standard solutions.

In Fig. 3 is shown a simple hinged arm for supporting the electrodes and enabling their shift from electrolytic cell to the beaker for resolution of the lead to be conveniently made. The clamps, K, are simply wooden clothespins with a spring clamp; they are screwed to the long wooden arm, T, which is hinged to W, a wall support of wood. A flexible insulated copper cable is connected to the electrodes, Q, R, by a two point hard rubber dashlight connector, or other suitable means. During resolution of the lead from the cathode, the anode may be placed in the clamp, K′, out of the way.

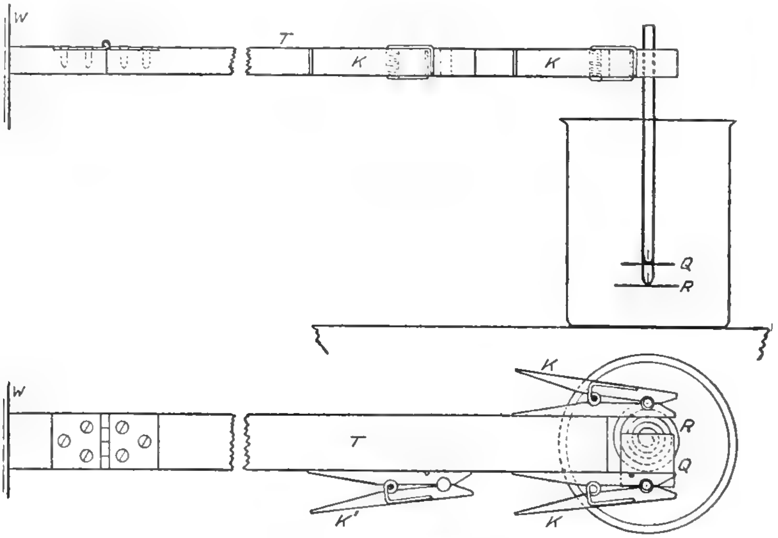

FIG. 3. ELECTRODE DETAILS.

K—Electrode clamp.
K′—Extra anode clamp.
Q—Anode.
R—Cathode.
T—Wooden arm electrode support.
W—Wall or other support.

RESULTS OF COLLABORATORS USING CHITTICK METHOD.

In Table 2 are given the results obtained for 1917 on Samples 1701, 1702, 1711, 1712, 1721 and 1722 of baking powder. The determinations of Exner, Malmstrom and Collins, Epstein, and Strunk are very consistent, on the whole, with themselves and with each other. Epstein's values on Sample No. 1702 evidently were due to misnumbering the sample, as he hits almost exactly on the lead content of the control sample. Holbrook and Burkhardt are low but consistent. In ten determinations, Lyman approximates the actual lead content but his results, in the main, are not near the correct value. Clarke's values are consistent but much too low. Exner has modified his former method and submits results for these samples obtained by it, as shown in the last column of the table; but they are very much higher than his own results (using the Chittick method) which came very close to the actual value of the lead content. Consequently, further details of the Exner method will not be given at this time.

The actual directions sent out to collaborators for the Chittick method are not those published in this report, since it was thought wise to publish this method in as perfect form as possible at the first writing and save the necessity for correction later, as well as confusion in the minds of analysts who may wish to use the method. Those who tried the method as first sent out will recognize at once the need of these changes.

TABLE 2.

Chittick method for lead in baking powders.

SAMPLE NUMBER	TOTAL LEAD PRESENT	LEAD FOUND							
		E. F. Exner*	R. A. Holbrook† and C. W. Burkhardt†	J. O. Clarke‡	A. Malmstrom§ and W. R. Collins§	E. R. Lyman**	D. Klein and A. K. Epstein	A. C. Strunk††	E. F. Exner‡‡
	parts per million	*parts per million*	*parts per million*	*parts per million*	*parts per million*	*parts per million*	*parts per million*	*parts per million*	*parts per million*
1701	6	....	....	....	6	...	....	....	....
1702	56	60.6	14.7	16.6	60	301	3.8	79	119
		....	22.0	25.6	..	305	5.1	....	96
		....	41.0	....	..	232	....	....	....
1711	4.8	....	....	....	..	...	....	....	....
1712	54.8	56.4	41.0	35.2	..	118	30	48.4	84
		....	38.0	30.7	..	67	55	....	80.8
		....	....	....	..	182	....	....	....
1721	3.2	....	....	....	..	...	....	....	....
1722	53.2	55.2	40.0	19.2	..	65	48.7	48.4	82
		....	43.6	21.8	..	60	....	....	83
		....	....	....	..	30	....	....	....

* Carleton College, Northfield, Minn.

† Victor Chemical Works, Chicago, Ill.

‡ State Department of Agriculture, Atlanta, Ga.

§ Wilckes Martin Wilckes Company, Camden, N. J.

** Since deceased.

†† Jaques Manufacturing Company, Chicago, Ill.

‡‡ Using Exner's method, modified.

RESULTS ON THE CORPER-BRYAN ELECTROLYTIC METHOD FOR LEAD IN BAKING POWDER.

In Table 3 are given the findings of various collaborators using the Corper-Bryan method. Where the conditions of this method have been adjusted to reduce the high acidity originally mentioned in the method, very consistent and accurate results were obtained.

TABLE 3.

Electrolytic method for lead in baking powders.

(Corper-Bryan Method.)

SAMPLE NUMBER	TOTAL LEAD PRESENT	LEAD FOUND							
		D. Klein and A. K. Epstein	A. Malmstrom and W. R. Collins	C. B. Morey* and P. F. Webster*	R. A. Holbrook and R. H. McCreary†	A. Malmstrom and W. R. Collins	A. F. Seeker‡ and A. L. Burns§	W. S. Allen**	A. H. Fiske and N. V. S. Mumford
	parts per million	*parts per million*	*parts per million*	*parts per million*	*parts per million*	*parts per million*	*parts per million*	*parts per million*	*parts per million*
1701	6	....	..	....	..	..	0	..	..
1702	56	49.3	67	....	39	69	..	Negative results	52
	....	....	67	....	49	..	..		59
	....	....	85	....	..	..	..		..
1711	4.8	....	..	4.8	..	..	..	..	..
	....	....	..	4.8	..	..	..	..	..
	....	....	..	5.1	..	..	..	..	..
1712	54.8	50.6	52	42	24	53	..	..	..
	....	48.0	49	44	24	54	..	..	..
	....	....	53	....	..	..	..	..	..
1721	3.2	....	..	3.2	..	..	12	..	..
1722	53.2	45.4	51	23	33	..	..	..	..
	....	48.0	50	26	29	48	..	..	..
1074††	20	....	..	19.5	..	..	..	..	..
	....	....	..	20.5	..	..	..	..	..

* Larkin Company, Buffalo, N. Y.

† Victor Chemical Works, Chicago, Ill.

‡ Since deceased.

§ U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, New York, N. Y.

** General Chemical Company, Laurel Hill, N. Y.

†† Special baking powder used by Morey and Webster.

OTHER EXPERIMENTAL WORK.

INVESTIGATION OF THE WICHMANN METHOD.

In accordance with the findings for 1916, the referee and G. H. Mains made some experiments and outlined a method of investigation with a view to modifying the Wichmann method. The results obtained by the collaborators in this case were neither full nor satisfactory, but the method of working and the preliminary experiments have merit and are included here.

OUTLINE OF EXPERIMENTS TO BE CARRIED ON WITH A VIEW TO MODIFYING THE WICHMANN METHOD.

After consideration of the difficulty encountered in the Wichmann method, that phosphate made from rock bears relatively large amounts of iron and thus renders difficult the separation of lead and iron, in the precipitation as sulphids, an attempt was made to hold up the iron during the hydrogen sulphid precipitation. The hydrogen ion concentration, expressed in P_H value ($P_H = \log \frac{1}{C_H}$) was determined for the point at which the iron was just held up in hydrochloric acid solution. The accompanying notes give a description of this work.

Having determined that at $P_H = 3.3$ iron will be held up and lead sulphid deposited when hydrogen sulphid is run in, it is desirable to determine whether or not under these conditions all of the lead present is precipitated as the sulphid.

The following suggestions for the work are made in order to secure comparable results:

1. Make up a baking powder containing 27 parts by weight of sodium bicarbonate, sufficient monocalcium phosphate of the best quality and with lead content and neutralizing power carefully determined exactly to neutralize the soda, and enough starch to make a total of 100 parts. The lead content of the sodium bicarbonate and of the starch should be determined. A determination of the per cent of iron present in the phosphate should likewise be made.

2. Take 100 gram portions of this completed baking powder mixture, place in 1.3 liter beakers, add 200 cc. of 10% hydrochloric acid to each, and heat on the steam bath until the starch is completely hydrolyzed. To different portions then add enough lead nitrate solution (0.320 gram per liter) to give, together with the amount of lead determined in the baking powder ingredients, the following quantities of lead per 100 grams of baking powder: (a) 0.001 gram; (b) 0.002 gram; (c) 0.005 gram; (d) 0.010 gram; (e) 0.02 gram or 10, 20, 50, 100 and 200 parts per million, respectively.

Dilute the baking powder solution containing added lead to 1 liter, and add 200 cc. of neutral ammonium citrate solution (lead-free), and cool to room temperature.

3. Test the solution for its P_H value as follows:

Place a 3 cc. portion of the solution in a test tube, and add 4 drops of the indicator tetrabromphenolsulphonephthalein. The concentration of the indicator is 0.1 gram in 250 cc. of alcohol[1]. This indicator turns from green to purple through a P_H range of 3.3–4.5, showing the first faint tinge of purple at 3.3. If the sample in the test tube shows no purplish tint, add dilute ammonium hydroxid to the main solution, a few drops at a time, with constant stirring, and remove 3 cc. portions to test after each

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[1] H. A. Lubs and W. M. Clark. *J. Wash. Acad. Sci.*, 1915, 5: 609.

addition, until the value P_H = 3.3 is reached. If this value is exceeded and the test tube sample shows more than a tint of purple color, dilute hydrochloric acid may be added a few drops at a time until the correct shade is obtained.

4. Having brought the solution to a P_H value of 3.3 proceed with the precipitation of the lead sulphid, without the addition of mercuric chlorid as in the Wichmann method. Allow the lead sulphid to settle out overnight. Continue with the regular procedure through the weighing of the lead chromate.

5. Repeat this procedure (3) and (4), with a second series of solutions exactly as before, except that 15 cc. of saturated mercuric chlorid solution are added to each portion, and the treatment of the combined mercury and lead sulphids carried out according to the Wichmann method. This is to test the efficacy of the use of mercury to hasten the lead separation as recommended in this method.

Whether or not at the acidity given, P_H = 3.3, the mercury and lead will be clearly precipitated by hydrogen sulphid is to be determined. In case it is found that some of the lead is retained in solution in the first series of solutions, with no mercury present, the second series should still be run, as it may be found that with mercury present during the precipitation a more complete separation of lead and iron may be effected.

Following this outline, A. H. Fiske and A. L. Thayer, working on a baking powder containing 0.55 per cent of iron, have obtained the results given in Table 4. It is evident that under the conditions of the Wichmann method accurate results are not obtained, and that the addition of mercuric chlorid does not increase the accuracy.

TABLE 4.

Wichmann method.

(A. H. Fiske, Collaborator; A. L. Thayer*, Analyst.)

LEAD PRESENT	LEAD RECOVERED			
	Without mercury		With 15 cc. of saturated mercuric chlorid solution added	
	Lead found	Recovery	Lead found	Recovery
parts per million	*parts per million*	*per cent*	*parts per million*	*per cent*
10	4	40	22	220
20	0	0	20	100
50	7	14	28	56
100	90	90	24	24
200	50	25	101	50.5

* Rumford Chemical Works, Providence, R. I.

EXPERIMENTS ON THE SOLUBILITY OF LEAD CHROMATE.

The question was raised by some collaborators as to the solubility of lead chromate under the conditions of precipitation which obtain in the quantitative determination of lead in this form. Consequently, at the request of the referee, experiments were carried out as follows by A. H. Fiske:

Lead chromate was prepared by mixing solutions of lead nitrate and potassium dichromate which had been prepared from commercially C. P. material. The amounts mixed were approximately chemically equivalent. The precipitate was washed several times with hot water, redissolved in nitric acid and reprecipitated with ammonia in the presence of acetic acid. It was washed again several times by decantation with hot water and dried in the oven at 115°C.

Great care was used to filter the solutions to make sure that no insoluble materials were present in the precipitate. The precipitation was done in flasks and the greatest care taken in every respect to avoid contamination.

After cooling the precipitate in the desiccator and allowing it to stand in a weighing bottle in the balance case for 24 hours, charges of the material were weighed out into beakers and then warmed with 100 cc. of 20% acetic acid and 10 cc. of saturated dichromate solution. These proportions were used because it is recommended in the directions for Bryan's modification of the Corper method that 20 cc. of 20% acetic acid be used to dissolve the lead material and the solution then be precipitated with 2 cc. of saturated potassium dichromate solution. Thus, in each experiment, there is *five times as much* solution as was used in the precipitation of the lead chromate by the Corper-Bryan method.

The beaker containing the solution and the lead chromate was heated on the steam bath for 2 hours with occasional stirring and was then allowed to cool overnight.

The following morning, the temperature of the solution was taken, it was filtered on a weighed Gooch containing an asbestos mat, dried in the oven at 115°C. for 2 hours, and weighed.

The loss in weight represents the amount of lead chromate dissolved in the solution.

Irregularities in the results are probably due to the occlusion of soluble material in the original charge of lead chromate, because this work is usually supposed to be within a limit of error of 0.0002 gram.

A tabular statement is given below of the loss of the lead chromate:

TABLE 5.

Difference between amount of lead chromate weighed out and the amount recovered.

LEAD CHROMATE	TEMPERATURE	LEAD CHROMATE	TEMPERATURE
gram	*°C.*	*gram*	*°C.*
0.0014	26	0.0030	23
0.0011	26	0.0008	23
0.0011	21	0.0008	23

Average difference in weight of lead chromate 0.0014

It will be noted from the above that the loss of the lead chromate in precipitation, using 20 cc. of 20% acetic acid and 2 cc. of saturated dichromate solution, must be very small if it is one-fifth of the average of the above.

J. R. Davies (Calumet Baking Powder Company, Chicago, Ill.) carried out the following experiments:

Lead chromate, made by the action of potassium dichromate on lead acetate, thoroughly washed and dried, analysis showing 64.7 per cent of lead, was taken in amounts from 2.8–4.4 mg. This was brought into solution with 0.3 cc. of concentrated potassium hydroxid solution, and sufficient water was added to make a total volume of 30 cc., with the addition of the required amount of acetic acid and potassium dichromate. This dilute solution of lead chromate in alkali was brought to the boiling point, when the dichromate solution and 5 cc. of acetic acid, plus that required to neutralize the potassium hydroxid used, were added simultaneously in order to approach the same conditions as obtained in the regular procedure. After the solution had stood for 18 hours at room temperature, the regular procedure was followed, filtering through tared Gooch crucibles, washing and drying the precipitate at 115°C. for 3 hours.

The results appear in Table 6. The greatest loss was 0.2 mg., the greatest increase 0.1 mg. In the collaborative work of two or three years ago, a difference was obtained of at least 0.2 mg. in the weight of the same prepared crucible which had been dried and weighed repeatedly. It is evident that the solubility of lead chromate under these conditions is nil.

TABLE 6.

Solubility of lead chromate in potassium dichromate.

(J. R. Davies, Analyst.)

LEAD CHROMATE ADDED	LEAD CHROMATE RECOVERED	LOSS	GAIN	POTASSIUM DICHROMATE	DICHROMATE IN SOLUTION
mg.	*mg.*	*mg.*	*mg.*	*cc.*	*per cent*
4.1	4.1	0.0	...	0.5	0.246
4.4	4.3	0.1	...	0.5	0.246
3.8	3.6	0.2	...	1.0	0.492
3.6	3.5	0.1	...	1.0	0.492
3.4	3.2	0.2	...	2.0	0.984
3.4	3.3	0.1	...	2.0	0.984
3.4	3.3	0.1	...	3.0	1.47
3.3	3.4	...	0.1	3.0	1.47
3.2	3.0	0.2	...	5.0	2.46
3.2	3.2	0.0	...	5.0	2.46
3.2	3.3	...	0.1	7.0	3.44
3.1	3.0	0.1	...	7.0	3.44
3.1	3.2	...	0.1	8.0	3.93
3.0	3.1	...	0.1	8.0	3.93
2.9	3.0	...	0.1	10.0	4.92
2.8	2.8	0.0	...	10.0	4.92

There is a statement[1] to the effect that if potassium dichromate is added to a neutral or very slightly acid solution of a lead salt, there is formed a basic lead chromate. As a precautionary method, the large amount of acetic acid was used. If a condition occurs where the basic chromate is formed, a large error will naturally result, the results showing a loss of lead.

NEW METHODS SUBMITTED.

ELECTROLYTIC METHOD FOR LEAD DETERMINATION.

R. A. Holbrook and R. H. McCreary have worked out an electrolytic method using a phosphoric acid solution and a rotating cathode. The time of electrolysis is much shortened, and they have obtained results

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[1] *J. pharm. chimie.*, 1914, **10**: 265.

on this year's collaborative samples comparable with those obtained with the Corper-Bryan method. The method seems well worthy of study. The details as given by the authors follow:

The method consists in separating the lead from the solution of the sample by electrolysis, dissolving the lead from the electrode, precipitating and weighing as $PbCrO_4$. No attempt is made to decompose the starch.

The electrical apparatus is a motor generator set, capable of generating 20 amperes at 6 volts, with field rheostat for control of voltage. A voltmeter and ammeter are necessary. The cell is an ordinary 800 cc. beaker, and the electrodes consist of a platinum wire anode of about 18 B. & S., gauge, with 2½ inches submerged in electrolyte, and a gold cathode made of a disk 2 inches in diameter and soldered or rivetted on a gold spindle. Arrangements are made to rotate the disk at about 3000 revolutions per minute. A better cathode for this purpose would be of platinum, not more than 1¼ inches in diameter.

DETAILS OF ELECTROLYSIS

Weigh 100 grams of baking powder into a 800 cc. beaker and decompose by stirring in, a little at a time, 50 cc. of phosphoric acid (sp. gr. 1.75). When the reaction has subsided, add 400 cc. of water. The mixture is then ready for electrolysis.

The electrodes are mounted so that the spindle of the cathode is at the bottom of the beaker and the disk ½ inch from the bottom, and the anode is adjusted to clear the disk of the cathode by ½ inch. The cathode is revolved and the current is turned on. With 6–7 volts potential drop across the cell, a current of 1.2 amperes is maintained. This corresponds to approximately 3.4 amperes per 100 square cm. of cathode surface exposed to electrolyte, since a part of the disk is not covered, which is due to the vortex caused by the high speed of rotation. No heat is applied.

After 30 minutes' electrolysis, quickly disconnect the cathode, dip in a beaker of water to wash off the electrolyte and place in a beaker just large enough to take the disk. Add enough strong nitric acid to cover the electrode, apply heat until the deposit is all dissolved, rinse off the electrode with a stream from a wash bottle and evaporate the solution to a volume of about 3 cc. Dilute this solution with 10 cc. of water and neutralize by adding ammonium hydroxid until a slight precipitate appears or until the solution is neutral to methyl orange, add 5 cc. of glacial acetic acid, boil the solution and, if there is a precipitate, filter, washing through with hot water. Bring the filtrate to the boiling point and precipitate the lead by adding 2 cc. of saturated potassium dichromate solution. Allow the precipitated lead chromate to settle overnight and filter off on a dried and weighed porcelain Gooch crucible with an asbestos pad, wash with cold water, dry at 110°C. for 1 hour, cool and weigh.

The above method is applicable to monocalcium phosphate using 100 grams of sample, 25 cc. of phosphoric acid (sp. gr. 1.75) and 400 cc. of water. The time of electrolysis should exceed 1 hour. This amount of phosphate is not entirely soluble but good results can be obtained if the sample is as finely ground as in baking powder materials.

METHOD OF DETERMINING FLUORIDS IN BAKING POWDER.

During 1917 a method of broad range and great accuracy was elaborated by C. H. Wagner and W. H. Ross[1] (Bureau of Soils, Washington, D. C.) for the determination of fluorids. Some preliminary experiments

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[1] *J. Ind. Eng. Chem.*, 1917, **9**: 1116.

have shown this method to be well applicable to baking powders, and it deserves study and consideration.

ACKNOWLEDGMENT.

The referee wishes to express his appreciation of the assistance rendered by G. H. Mains and A. J. Johnson (Bureau of Chemistry, Washington, D. C.), and likewise to the various collaborators mentioned in the tables.

RECOMMENDATIONS.

It is recommended—

(1) That the modified Corper-Bryan method for the electrolytic determination of lead in baking powder (page 221) be adopted as a tentative method. (First reading.)

(2) That a study be made of the electrolytic method, proposed by Holbrook and McCreary (page 230), in which a rotating cathode is used and which operates directly in an aqueous phosphoric acid suspension of the powder without preliminary hydrolysis of the starch.

(3) That a study be made of the electrolytic separation and determination of zinc in baking powder.

(4) That a further study be made of Chittick's method for the determination of lead in baking powder (page 218).

(5) That in view of the fact that Wichmann's modification of the Seeker-Clayton method gives difficulty as applied to modern baking powders, no further study be made of this method at the present time.

(6) That the Wagner-Ross method for total fluorids (page 131) be studied.

(7) That efforts be made to develop a chemical method for the determination of zinc in baking powders.

A NOTE ON THE HYDROGEN ION CONCENTRATION AT WHICH IRON IS PRECIPITATED FROM HYDROCHLORIC ACID SOLUTION BY AMMONIUM HYDROXID, SODIUM HYDROXID, AND HYDROGEN SULPHID.

By H. E. Patten[1] and G. H. Mains (Bureau of Chemistry, Washington, D. C.).

In working out the details of a method for determining lead in baking powders, tests were made as to the hydrogen ion concentrations at which ferric iron is precipitated from hydrochloric acid solution. A 1200 cc. solution containing 0.4 gram of iron in the form of ferric chlorid and 0.002 gram of lead in the form of lead nitrate was divided into 100 cc. portions. (The concentration of iron given corresponds to the maximum likely to be met with in phosphate baking powders.)

To one of these portions, dilute ammonium hydroxid was added from a burette, a few drops at a time. The hydrogen ion concentration of the solution expressed as P_H ($P_H = \log \frac{1}{C_H}$)[2] after each addition of ammonium hydroxid was determined by color comparison[3] using the following indicators, whose color changes had been checked by the hydrogen electrode.

The indicators used and their practical range of color changes were:

M-Benzolsulfosaurediphenylamin	P_H 1.2 to 2.3
Methyl orange	P_H 2.0 to 3.5
Tetrabromphenolsulphonephthalein	P_H 3.3 to 4.5
Methyl red	P_H 4.0 to 6.4
Phenolsulphonephthalein	P_H 6.5 to 8.5
Rosolic acid	P_H 7.0 to 7.5

The P_H values may be considered as correct to ±0.2. At a P_H of 3.3 the solution was perfectly clear. At $P_H = 3.5$ a very faint cloudiness, due to a colloidal precipitation of ferric hydroxid, could be seen. This became more pronounced as the P_H increased, giving a small amount of fine flocculent precipitate at $P_H = 5.5$ and becoming very heavy at $P_H = 6.0$. By the addition of hydrochloric acid the P_H was lowered, and the precipitate dissolved correspondingly, the solution becoming clear at $P_H = 3.3$. Duplicate results were secured on a second portion. The use of sodium hydroxid in place of ammonium hydroxid gave the same results as to the point at which the iron was held in solution.

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[1] Present address, Provident Chemical Works, St. Louis, Mo.

[2] *Cf.* Leonor Michaelis. Die Wasserstoffionenkonzentration. Berlin, 1914; S. P. L. Sorenson. *Compt. rend. trav. lab. Carlsberg*, 1909, **8**: 1.

[3] *Cf.* W. M. Clark and H. A. Lubs. *J. Bact.*, 1917, **2**: 1.

By the addition of various amounts of sodium hydroxid and hydrochloric acid, respectively, several portions of the solution were brought to certain different hydrogen ion concentrations. The P_H of each of these portions was then determined as before, and all of the portions were saturated with hydrogen sulphid gas. It was found that the highest P_H at which the iron was held in solution was approximately 3.3. At a P_H of 3.5 a small amount of iron sulphid was precipitated, increasing in amount as the P_H was increased. At P_H points lower than 3.3 the solution remained clear, the iron being held up. This was checked with several solutions containing the above concentration of iron (0.03 per cent).

It is customary to say that iron is precipitated by hydrogen sulphid from alkaline solutions, and not from acid solutions, without stating any definite limit as to the degree of acidity or alkalinity. We have found that iron in quantities up to 0.03 per cent can be held up in hydrochloric acid solutions at a P_H below 3.3, and that, in solutions of a higher P_H, it is precipitated as ferrous sulphid by hydrogen sulphid.

We have also found that when ammonium hydroxid or sodium hydroxid is added to this same concentration of ferric iron in hydrochloric acid solution, the first formation of colloidal ferric hydroxid is noted at a P_H value very slightly greater than 3.3.

This P_H value 3.3, at which iron is held up in hydrochloric acid solutions, may be conveniently obtained in practice as follows:

> Place a 3 cc. portion of the solution in a test tube, and add 4 drops of the indicator tetrabromphenolsulphonephthalein[1] (0.1 gram in 250 cc. of alcohol). This indicator turns from green to purple through a P_H range of 3.3 to 4.5, showing the first faint tinge of purple at 3.3. If the sample in the test tube shows no purplish tint, add dilute ammonium hydroxid to the main solution, a few drops at a time, with constant stirring, and remove 3 cc. portions to test after each addition, until the value $P_H =$ 3.3 is reached. If this value is exceeded and the test tube sample shows more than a tinge of purple color, dilute hydrochloric acid may be added a few drops at a time until the correct shade is obtained.

For the determination of other P_H values, indicators and comparison solutions of known P_H may be made as outlined by Clark and Lubs[2].

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[1] H. A. Lubs and W. M. Clark. *J. Wash. Acad. Sci.*, 1915, **5**: 609.

[2] W. M. Clark and H. A. Lubs. *J. Biol. Chem.*, 1916, **25**: 479.

NOTE ON THE BEHAVIOR OF NEUTRAL AMMONIUM CITRATE IN CERTAIN PHOSPHATE SOLUTIONS.

By H. E. PATTEN[1] and G. H. MAINS, (Bureau of Chemistry, Washington, D. C.).

In the methods for the determination of lead in phosphate baking powders the use of neutral ammonium citrate is specified, but there seems to be much uncertainty among analysts as to the function it performs, and the qualifying conditions for its use. Therefore, tests were made upon hydrochloric acid solutions of phosphate baking powder to determine the hydrogen ion concentration at which precipitates of calcium phosphate and calcium citrate are formed.

The starch in a 100 gram sample of baking powder containing 56 grams of monocalcium phosphate was hydrolyzed by the addition of 300 cc. of concentrated hydrochloric acid, and the solution made up to 1 liter with water. This was divided into 100 cc. portions. To one of these, ammonium hydroxid was added a few drops at a time, and the P_H ($P_H = \log \frac{1}{C_H}$) of the solution determined after each addition by color comparison, using indicators checked by the hydrogen electrode. P_H values given may be considered correct to ± 0.2. When the P_H had increased to 2.3, the first colloidal precipitate of calcium phosphate was formed, at a temperature of 26°C. Lowering the temperature to 10°C. brought no change in the amount of precipitate. Twenty cc. of neutral ammonium citrate solution ($P_H = 7$) were added, but did not cause an appreciable change in the volume of the precipitate. The P_H of the solution after the addition of the ammonium citrate was 5.5. Upon standing overnight, a heavy precipitate of calcium citrate settled out. By the addition of hydrochloric acid to the solution, the P_H was lowered gradually, the precipitate dissolving correspondingly, and the solution becoming clear at a P_H of 2.4. The P_H was again gradually increased by the addition of the ammonium hydroxid until a value of 6.0 was reached, with no formation of precipitate, the temperature of the solution being 25°C. The solution was gradually heated over a Bunsen flame. At 95°C., a heavy precipitate of calcium citrate began to form, and settled rapidly. The P_H of the hot liquid was 6.2.

To a second portion of the solution 20 cc. of neutral ammonium citrate solution were added, giving a P_H of 3.1. Ammonium hydroxid was very gradually added and the P_H determined at intervals as above. Small amounts of gelatinous calcium citrate, which dissolved upon stirring, were formed at each addition of the ammonium hydroxid. At P_H values of 5.0 and higher, the liquid appeared clear, but calcium

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[1] Present address, Provident Chemical Works, St. Louis, Mo.

citrate settled out on standing overnight. At $P_H = 7.2$, a permanent gelatinous precipitate of calcium citrate was formed. The temperature of the solution was 22°C. Another portion of solution gave the first permanent precipitate of calcium citrate at $P_H = 7.1$.

Tests with ammonium molybdate solution showed that, when neutral ammonium citrate had been added to the phosphate solution, the precipitates obtained by standing overnight or by raising the P_H to 7.1 were not calcium phosphate but calcium citrate.

These experiments show that neutral ammonium citrate, when added to hydrochloric acid solution of phosphate baking powder, prevents the formation of a precipitate of calcium phosphate upon the addition of ammonium hydroxid. Calcium phosphate is held up in hydrochloric acid solution at P_H values more acid than 2.3 but precipitates at higher P_H values. When neutral ammonium citrate is added, the P_H is shifted greatly toward the alkaline end, tending still further to precipitate the phosphate. However, the ammonium citrate (broken up into citric acid) acts on the calcium phosphate, forming calcium citrate. At P_H values below 5.0, the calcium citrate is held in solution. At P_H values between 5.0 and 7.0, the precipitation is extremely slow in the cold, becoming noticeable only after several hours, but the precipitate settles out upon standing overnight. Raising the temperature of the solution hastens the precipitation. At $P_H = 7.1$, an immediate permanent precipitate of gelatinous calcium citrate is formed. After the precipitate is once formed, the addition of neutral ammonium citrate except in large excess will not clear up the solution. A comparatively small amount of hydrochloric acid, because of the sharp increase in hydrogen ion concentration (decrease of P_H), rapidly dissolves the gelatinous calcium citrate.

A convenient method of ascertaining the hydrogen ion concentration (P_H) of any solution is given herewith, since this determination has not yet come into general practice. The following directions are applicable only when a considerable volume of the solution to be tested is available. When a very small volume of solution is in question, other methods must be used[1].

Take several test portions of 3 cc. each and place in test tubes, add 3 to 5 drops of the indicator solution, and observe the color-shade produced. Each indicator solution has its own color change corresponding to a more or less definite hydrogen ion concentration. The quantitative directions for synthesizing and making up these indicator solutions are given by Clark and Lubs[2], and the same authors[3] have outlined very exactly a system of comparison solutions (or buffer solutions, as they

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[1] G. S. Walpole. Biochem. J., 1913, **7**: 410; Leonor Michaelis. Die Wasserstoffionenkonzentration. Berlin, 1914; W. M. Clark and H. A. Lubs., *J. Bact.*, 1917, **2**: 1.

[2] *J. Wash. Acad. Sci.*, 1915, **5**: 609.

[3] *J. Biol. Chem.*, 1916, **25**: 479.

term them) whose hydrogen ion concentration is determined by their quantitative composition, and is reproducible. The hydrogen ion value for each comparison solution is given, and opposite each value is given the color-shade of the appropriate indicator for that particular hydrogen ion concentration.

Further quotation of the great number of references to the literature of this subject is not given here since this is well covered in the authorities cited.

The specific indicators that we have used in the above work on citrate and phosphate solutions, together with their practical ranges of color change, are given below:

M-Benzolsulfosaurediphenylamin	P_H 1.2 to 2.3.
Methyl orange	P_H 2.0 to 3.5.
Tetrabromphenolsulphonephthalein	P_H 3.3 to 4.5.
Methyl red	P_H 4.0 to 6.4.
Phenolsulphonephthalein	P_H 6.5 to 8.5.
Rosolic acid	P_H 7.0 to 7.5.

J. K. Haywood made the following recommendations in behalf of the executive committee:

It is recommended—

(1) That the Committee on Amendment to the Constitution and By-Laws be discharged. This committee completed its work last year and should have been discharged at that time.

Approved.

(2) That the action of the president in appointing an associate referee on gelatin be approved.

Approved.

(3) That the action of the president in appointing an associate referee on eggs and egg products be approved.

Approved.

(4) That the Committee on Editing Methods of Analysis be continued for another year and that A. J. Patten be appointed to fill the vacancy due to the resignation of J. P. Street.

Approved.

(5) That a referee on the toxicity of feed, suggested by W. A. Withers of North Carolina, be not appointed, as it appears to the executive committee that the work of a referee on this subject is somewhat outside the scope of the work of this association.

Approved.

(6) That no separate section be held on soils, since it does not appear to the executive committee that it would be to the best interests of the association to break up into sections. The program is so arranged now that soils are discussed in the main meeting while drugs are discussed in a separate room, which practically amounts to a separate session for soils.

Approved.

William Frear of Pennsylvania made the following motion, which was duly seconded and adopted, that the executive committee, in the preparation of the program next year, be requested to keep carefully in mind this request of the soil chemists. He called attention to the fact that there were a great many subjects that required more time than has been given to them during the past five years.

The meeting adjourned at 4.30 p. m. for the day.

THIRD DAY.

WEDNESDAY—MORNING SESSION.

REPORT OF COMMITTEE ON RECOMMENDATIONS OF REFEREES.

By B. B. Ross (Alabama Polytechnic Institute, Auburn, Ala.), *Chairman.*

Your committee has made a report and has discharged a certain part of the duty assigned to it. As a result of the amendment to the constitution that was adopted a year ago, the Committee on Recommendations of Referees is delegated or authorized to recommend to the President of the Association the names of referees and associate referees to work on each of the subjects to be considered by the association. At a joint meeting of the Executive Committee, the Committee on Recommendations of Referees, and the President of the Association, this committee made such recommendations.

The chairmen of the various subcommittees will report on the recommendations of the various referees that have been approved for the coming year. Probably owing to war time conditions, a number of recommendations made a year ago have not been carried out by the present referees. Wherever possible it is hoped that work along these lines will be conducted during the coming year.

REPORT OF COMMITTEE A ON RECOMMENDATIONS OF REFEREES.

By A. J. Patten (Agricultural Experiment Station, E. Lansing, Mich.), *Chairman.*

[Phosphoric acid (basic slag, to cooperate with committee on vegetation tests on the availability of phosphoric acid in basic slag), nitrogen (special study of Kjeldahl method), potash, soils (nitrogenous compounds, lime requirements), inorganic plant constituents, insecticides and fungicides, and water.]

PHOSPHORIC ACID.

It is recommended that the following recommendations of 1916 be referred to the referee for 1918:

(1) That the study of the preparation of neutral ammonium citrate solution, its use in determining reverted phosphoric acid and possible substitutes for it in this determination, be continued.

Approved.

(2) That in view of the conditions resulting from the European war, whereby the price of molybdic acid has been more than quadrupled and 100 per cent molybdic acid practically removed from the markets of the United States, the referee study the determination of phosphoric acid with a view to recommending an optional method not requiring the use of molybdic acid.

Approved.

(3) That the volumetric method, dissolving the slag in sulphuric and nitric acids[1], be adopted as an official method for total phosphoric acid in basic slag.

Approved.

(4) That this association instruct its referee on phosphoric acid to give prominent attention to the question of methods of determining available phosphoric acid in slags, the chemical ingredients influencing the same, and the bibliography on the subject.

Sufficient reports are already in the hands of your committee to be of service to the referee on phosphoric acid in his chemical investigations. It seems unnecessary to the committee to wait until all of the vegetation results are at hand before tentative methods of analysis are submitted to the association.

Approved.

(5) It is further recommended that the papers presented by H. D. Haskins[2] on "The Effect of Mass and Degree of Fineness on the Percentage of Available Phosphoric Acid in Precipitated Phosphate" and by E. O. Thomas[3] on "Insoluble Phosphoric Acid in Organic Base Goods", be referred to the referee for 1918.

Approved.

NITROGEN.

It is recommended—

(1) That work on the West Coast refraction method be discontinued.

Approved.

(2) That the referee for 1918 study the Lunge nitrometer method, which is invariably used by the manufacturers of explosives, for the analysis of nitrate of soda.

Approved.

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[1] *J. Assoc. Official Agr. Chemists*, 1917, **3**: 90.
[2] *Ibid.*, 1920, **4**: 64.
[3] *J. Ind. Eng. Chem.*, 1917, **9**: 865.

(3) That the use of 10 grams of anhydrous sodium sulphate as a substitute for potassium sulphate in the Gunning method and any modification thereof be made official.

Approved.

(4) That the use of potassium permanganate, wherever it appears in the Kjeldahl method, be eliminated.

Approved.

(5) That a further study be made of the effect of glass wool in the ferrous-sulphate-zinc-soda method for nitrates.

Approved.

POTASH.

It is recommended—

(1) That the work on the availability of potash be continued.

Approved.

(2) That the study of the perchlorate method be continued.

Approved.

(3) That the official method for the preparation of potash solution[1] be revised to read as follows:

> Place 2.5 grams of the sample upon a 12.5 cm. filter paper and wash with successive portions of boiling water into a 250 cc. graduated flask until the filtrate amounts to about 200 cc. Add to the hot solution a slight excess of ammonium hydroxid and sufficient ammonium oxalate to precipitate all of the lime present, cool, dilute to 250 cc., mix, and pass through a dry filter.

Approved.

(4) That the paper presented by H. D. Haskins[2], entitled "A Modified Method for the Determination of Water-Soluble Potash in Wood Ashes and Treater Dust", be referred to the referee on potash for 1918.

Approved.

SOILS.

It is recommended—

(1) That the recommendations held over from 1916[3] be rescinded and the referee on soils for 1918 be given authority to take up such work as seems in his judgment most necessary.

Approved.

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 12.
[2] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 82.
[3] *Ibid.*, **3**: 520.

(2) That the title, "Lime Requirement of Soils", be changed to "Lime Absorption Coefficient".

Approved.

(3) That the work of the ensuing year be directed along lines which will fully develop the optimum conditions for carrying out the Jones method.

Approved.

INORGANIC PLANT CONSTITUENTS.

It is recommended—

(1) That the report of J. F. Breazeale[1] on "The Determination of Calcium in the Presence of Phosphates" and the recommendations approved by the association in 1916[2] be referred to the referee for 1918.

Approved.

(2) That the methods as outlined for calcium, magnesium, iron and aluminium[3] be further studied on solutions approximating the composition of the ash from cereals.

Approved.

(3) That the colorimetric method for the determination of manganese[4] be further studied.

Approved.

INSECTICIDES AND FUNGICIDES.

It is recommended—

(1) That further cooperative work be done on the Gyory method for titrating arsenious oxid in hydrochloric acid solution with a solution of potassium bromate.

Approved.

(2) That further cooperative work be done on the determination of lead, copper and zinc in the analysis of such preparations as Bordeaux-lead arsenate, Bordeaux-zinc arsenite, etc.

Approved.

(3) That cooperative work be done on the determination of total arsenic in London purple by first destroying the color by heating the sample with a mixture of zinc oxid and sodium carbonate.

Approved.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 124.

[2] *Ibid.*, **3**: 521.

[3] *Ibid.*, 329.

[4] *Ibid.*, 330.

(4) That cooperative work be done on the removal of coloring matter from London purple by the use of an adsorbent.

Approved.

(5) That the methods given by Roark[1] for total sulphur and for total lime be made official.

Approved.

(6) That methods for determining the monosulphid equivalent, thiosulphate sulphur, sulphid sulphur, and sulphate sulphur under the iodin titration method[2], be made tentative.

Approved.

(7) That the paper on "The Determination of Arsenic in Insecticides by Potassium Iodate" by George S. Jamieson[3] be referred to the referee for 1918.

Approved.

WATER.

It is recommended—

(1) That the method for the determination of barium[4] be adopted as official.

Approved.

(2) That the method for the determination of manganese[5] be adopted as an additional official method.

Approved.

(3) That further study be given to the rapid method for the determination of calcium and magnesium in industrial water[6].

Approved.

(4) That further study be given to the determination of free and albuminoid ammonia in water containing sulphids, with a view to modifying the official method in this respect.

Approved.

(5) That the referee for 1918 be instructed to continue the work on water along the lines suggested in the Report of the Referee on Water for 1917[6], giving particular attention to the selection of methods for determining lead, copper, zinc and tin in waters, and to the calculation of the milligram equivalents of the radicals found in water with a view to their use in the interpretation of water analyses.

Approved.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **3**: 354–5.

[2] *Ibid.*, **4**: 146.

[3] *J. Ind. Eng. Chem.*, 1918, **10**: 290.

[4] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 86.

[5] *Ibid.*, 85.

[6] *Ibid.*, 84.

(6) That the methods listed below, recommended in 1916[1] for adoption as official, be adopted as official this year. (Second presentation of the methods for action.)

(a) Method for the determination of lithium, potassium and sodium[1].

(b) Method for turbidity, **1** and **2**[2].

(c) Method for color, **3** and **4**[2].

(d) Method for odor, **5**[2].

(e) The Schulze-Trommsdorf method for the determination of required oxygen, **22** and **23**[3].

(f) Method I and Method II for dissolved oxygen, **24**, **25**, **26**, **27**, **28** and **29**[4].

(g) Method for the determination of specific gravity, **30**[5].

(h) Method for the determination of hydrogen sulphid, **37**[5].

(i) Method for temporary hardness, **70**[6].

(j) Method for alkalinity, **71**, **72**, **73** and **74**[6].

(k) Method for total hardness, **75** andd **76**[7].

(l) Method for permanent or non-carbonate hardness, **77**[7].

Approved.

(7) That the method for free carbon dioxid[8] remain a tentative method. (Second presentation of the method for action.)

Approved.

(8) That consideration be given to the Gutzeit method for the determination of arsenic with a view to having it printed in the methods for the analysis of water (as an additional official method)[9]. (Second presentation of the method for action.)

Approved.

(9) That the official reduction method for the determination of nitrogen in the form of nitrate be revised to read as outlined in the 1917 Report of the Referee on Water[10].

Approved.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **3**: 522.
[2] *Assoc. Official Agr. Chemists, Methods*, 1916, 35.
[3] *Ibid.*, 39.
[4] *Ibid.*, 40–1.
[5] *Ibid.*, 41.
[6] *Ibid.*, 50.
[7] *Ibid.*, 51.
[8] *Ibid.*, 42, 38.
[9] *Ibid.*, 171.
[10] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 92.

REPORT OF COMMITTEE B ON RECOMMENDATIONS OF REFEREES.

By H. C. Lythgoe (State Department of Health, Boston, Mass.), *Acting Chairman.*

[Foods and feeding stuffs (sugar, crude fiber, stock feed adulteration, organic and inorganic phosphorus, water), dairy products (separation of nitrogenous substances in milk and cheese), saccharine products (maple products, honey, sugar house products), drugs (medicinal plants, alkaloids, synthetic products, medicated soft drinks, balsams and gum resins, enzyms), testing chemical reagents and microanalytical methods.]

FOODS AND FEEDING STUFFS.

It is recommended—

(1) That a further study be made of sulphur dioxid in bleached grain.

Approved.

(2) That the method for determining the acidity of corn, as described by Black and Alsberg[1], be considered by the referee next year with a view to its adoption as an official method, and that the method be studied to determine whether changes are necessary to make it applicable to grains other than corn.

Approved.

SUGAR.

No recommendations were made by the referee. The committee therefore recommends that the following 1916 recommendations be continued:

(1) That the modifications proposed in 1915 for determining sucrose by acid and invertase inversion be further studied.

Approved.

(2) That the work upon determining small amounts of reducing sugars in the presence of sucrose be continued.

Approved.

(3) That the methods of determining copper by reduction of the oxid in alcohol vapors be investigated.

Approved.

(4) That the optical methods for estimating raffinose in beet products be examined with special reference to hydrolysis by means of enzyms.

Approved.

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[1] U. S. Bur. Plant Ind. Bull. 199: (1910).

(5) That details of mixing raw sugars be studied with a view to reducing moisture changes.

Approved.

(6) That the influence of temperature upon polarization by sugars other than sucrose be studied.

Approved.

(7) That recommendations 2, 3 and 5 made by W. D. Horne[1] be referred to the Committee on Editing Methods of Analysis.

Approved.

(8) That the referees continue in collaboration with the Bureau of Standards the preparation of a table of reduction factors for the more common reducing sugars.

Approved.

CRUDE FIBER.

(1) That the one filtration method[2] be further studied.

Approved.

(2) That the matter of a uniform filtering medium be further studied.

Approved.

STOCK FEED ADULTERATION.

(1) That the work on scratch feed be continued with a view to securing an accurate and satisfactory method of sampling.

Approved.

(2) That the work of developing a method for the quantitative determination of cottonseed hulls in cottonseed meal be continued.

Approved.

ORGANIC AND INORGANIC PHOSPHORUS.

(1) That the magnesia mixture method[3] for the estimation of water-soluble inorganic phosphorus in flesh be adopted as an official method, with one minor change of detail, in the interest of economy of reagents, namely, that the amount of magnesia mixture used in extracts from 10 to 12 gram samples be reduced from 50 to 10 cc.

The committee feels that sufficient collaboration has not been reported and recommends that final action be deferred until further collaboration.

Final action postponed.

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[1] *J. Assoc. Official Agr. Chemists*, 1919, **3**: 263.
[2] *Ibid.*, 256.
[3] *Ibid.*, 1916, **1**: 562; 1919, **3**: 264.

(2) That further work be done with the magnesia mixture method[1] on brain; and that other glandular tissues be studied.

Approved.

(3) That the referee consider the report of J. B. Rather on "The Determination of Phytin Phosphorus in Plant Products"[2].

Approved.

WATER.

It is recommended—

(1) That the method for the determination of water in foods and feeding stuffs by drying in vacuum over sulphuric acid[3] be adopted as official. (This method has been twice before recommended as an official method by the previous referee.) (Final reading.)

Approved.

(2) That the following method for the determination of water by drying over lime in vacuum be adopted as a tentative method, and be recommended for further study for the ensuing year:

Lime-Vacuum Method for Moisture.

Weigh 2 grams of the material into a suitable dish or crucible with a tightly fitted cover. Place in a vacuum desiccator over about 400 grams of freshly ignited powdered lime, and exhaust with a vacuum pump. After 24 hours, open the desiccator, forcing the incoming air through concentrated sulphuric acid, and make the first weighing. After weighing, replace the dish in the desiccator and repeat the process until constant weight is obtained. The lime should be changed on the third or fourth day and, with very wet substances, once again near the end of the process.

Approved.

(3) That the following method for the determination of water by drying over carbide in vacuum be adopted as a tentative method, and be recommended for further study:

Carbide-Vacuum Method for Moisture.

Weigh 2 grams of the material into a suitable dish or crucible with a tightly fitted cover. Place in a vacuum desiccator over about 400 grams of clean lumps of calcium carbide, and exhaust with a vacuum pump. After 24 hours, open the desiccator, forcing the incoming air through concentrated sulphuric acid, and make the first weighing. After weighing, replace the dish in the desiccator and repeat the process until constant weight is obtained. The calcium carbide should be changed on the third or fourth day and, with very wet substances, once again near the end of the process.

Approved.

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[1] *J. Assoc. Official Agr. Chemists*, 1916, **1**: 562; 1919, **3**: 264.

[2] *J. Am. Chem. Soc.*, 1917, **39**: 2506.

[3] *Assoc. Official Agr. Chemists, Methods*, 1916, 79.

DAIRY PRODUCTS.

It is recommended—

(1) That the Roese-Gottlieb method[1] for fat, as applied to plain ice cream, be adopted as official. (First reading.)

Approved.

(2) That the Harding-Parkin method[2] for fat in ice cream be adopted as a tentative method. (First reading.)

Approved.

(3) That the Schmidt-Bondzynski method[3] modified for the determination of fat in cheese be adopted as official. (First reading.)

Approved.

(4) That a further study be made of the Roese-Gottlieb method[1] as applied to malted milk and dried milk products of various fat content.

Approved.

(5) That a study be made of methods for the determination of moisture in milk products, including evaporated and condensed milk, dried milk, and malted milk.

Approved.

(6) That the tentative method for the determination of moisture in cheese[4] be further studied with a view to its adoption as an official method.

Approved.

SEPARATION OF NITROGENOUS SUBSTANCES IN MILK AND CHEESE.

The committee recommends that the following recommendations, adopted at the 1916 meeting, be continued for further study:

(1) That the referee for next year attempt to determine the relative amounts of some of the dissociation products in water-soluble and water-insoluble meat proteins.

Approved.

(2) That study be continued leading to the adoption of methods for the determination of the non-casein proteins and the products of protein decomposition in milk.

Approved.

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 289.
[2] *J. Ind. Eng. Chem.*, 1913, **5**: 131.
[3] *Assoc. Official Agr. Chemists, Methods*, 1916, 297.
[4] *Ibid.*, 296.

SACCHARINE PRODUCTS.

No report or recommendations.

DRUGS.

It is recommended—

(1) That the method for the determination of atropin in tablets[1], with the following change relative to drying the alkaloidal residue, be made tentative: "Dry in vacuo to a constant weight, and weigh as atropin".

Approved.

(2) That further work be done on the methods for separating quinin and strychnin, and that a method be submitted to the collaborators, which has a reasonable certainty of yielding concordant results.

Approved.

(3) That work be continued to find new sources of supplies or proper substitutes for drugs not now obtainable.

Approved.

(4) That work be continued to determine the value of a more extended use of weights of unit volumes in the analysis of crude drugs and spices.

Approved.

(5) That comparative work be resumed on the ricin method for the assay of pepsin[2] and that the methods outlined for the identification and assay of pepsin be studied cooperatively.

Approved.

(6) That the appointment of the referee on balsam be continued, and that a study be made of the methods of demonstrating the difference between the natural and the artificial product.

Approved.

(7) That the work on mixtures containing synthetic products be continued.

Approved.

(8) That a further study be made of the methods for the determination of strychnin in tablet triturates[3].

Approved.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **3**: 379.

[2] *Assoc. Official Agr. Chemists, Methods*, 1916, 363.

[3] *J. Assoc. Official Agr. Chemists*, 1919, **3**: 189; 1920, **3**: 379.

(9) That a further study be made of the method for the determination of strychnin in liquids[1] where it occurs as the only alkaloid.

Approved.

TESTING CHEMICAL REAGENTS.

No recommendations were made by the referee. The committee therefore recommends that the following 1916 recommendations be continued:

(1) That the work on the determination of alcohol in pharmaceutical preparations be continued.

Approved.

(2) That the study of methods for the determination of the strength of acetic anhydrid be continued.

Approved.

(3) That work on the testing of purity of immiscible organic solvents be continued.

Approved.

MICROANALYTICAL METHODS.

It is recommended that studies on quantitative methods be continued.

Approved.

REPORT OF COMMITTEE C ON RECOMMENDATIONS OF REFEREES.

By R. E. DOOLITTLE (Transportation Building, Chicago, Ill.), *Acting Chairman.*

[Food preservatives, coloring matters in foods, metals in foods, fruits and fruit products, canned vegetables, cereal foods, wines, soft drinks (bottlers' products), distilled liquors, beers, vinegars, flavoring extracts, meat and meat products (separation of nitrogenous compounds in meat products, meat extracts), edible fats and oils, spices and other condiments, cacao products, coffee, tea, baking powder.]

FOOD PRESERVATIVES.

It is recommended—

(1) That further work be done on Method II[2], submitted for collaborative work last year, for the determination of saccharin in the presence of mustard oil.

Approved.

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[1] *J. Assoc. Official Agr. Chemists*, 1919, **3**: 189; 1920, **3**: 379.

[2] *Ibid.*, 1920, **3**: 505.

(2) That other methods not dependent upon the sulphur component of saccharin be investigated.

Approved.

(3) That further work be done upon the determination of saccharin in baked flour preparations.

Approved.

(4) That the following methods be made official, the paragraph numbers and titles being given as they appear in the *Association of Official Agricultural Chemists, Methods,* 1916, 141-54. (Final action):

SALICYLIC ACID.

1 PREPARATION OF SAMPLE.—OFFICIAL.
2 *Ferric Chlorid Test.—Qualitative.—Official.*
4, 5 *Colorimetric Method.—Quantitative.—Official.*

BENZOIC ACID.

6, 7 PREPARATION OF SAMPLE.—OFFICIAL.
9 *Ferric Chlorid Test.—Qualitative.—Official.*
10 *Modified Mohler Test.—Qualitative.—Official.*
11 *Quantitative Method.—Official.*

SACCHARIN.

12 *Qualitative Test.—Official.*

BORIC ACID AND BORATES.

14 *Qualitative Test.—Official.*
15 *Quantitative Method.—Official.*

FORMALDEHYDE.

16 PREPARATION OF SAMPLE.—OFFICIAL.
17 *Phenylhydrazin Hydrochlorid Method.—Official.*
18 *Hehner Method.—Official.*
19 *Leach Method.—Official.*
20 *Phenylhydrazin Hydrochlorid and Sodium Nitro-prussid Test.—Official.*
21 *Phenylhydrazin Hydrochlorid and Potassium Ferricyanid Test.—Official.*
22 *Phenylhydrazin Hydrochlorid and Ferric Chlorid Test.—Official.*
23 *Phloroglucinol Method.*

FLUORIDS.

24 *Method I.—Modified Method of Blarez.—Official.*
25 *Method II.—Official.*

SULPHUROUS ACID.

30 *Method I.—Distillation Method.—Official.*

31 *Method II.—Direct Titration Method.—Official.*

32 DETERMINATION OF FREE SULPHUROUS ACID.—OFFICIAL.

FORMIC ACID.

38, 39, 40 *Quantitative Method.—Official.*

Approved.

COLORING MATTERS IN FOODS.

It is recommended—

(1) That the study of the natural coloring matters be continued.

Approved.

(2) That the data secured during the past year relative to the behavior of natural coloring matters with certain specified reagents be added to the table for the "Behavior of certain natural coloring matters with common reagents"[1] when this table is revised.

Approved.

METALS IN FOODS.

It is recommended—

(1) That the Penniman method for tin[2] be the subject of collaborative work during 1918.

Approved.

(2) That the Gutzeit method as modified during 1916[3] be the subject of collaborative work on baking powder materials during 1918.

Approved.

(3) That a study be made of methods for the determination of arsenic in gelatin and similar products.

Approved.

(4) That a study be made of methods for the determination of zinc, copper, and aluminium in foods.

Approved.

FRUITS AND FRUIT PRODUCTS.

It is recommended that methods for the detection of pectin from apple pomace, used in the manufacture of jellies and jams, be studied.

Approved.

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 166–7.
[2] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 172.
[3] *Ibid.*, **3**: 512.

CANNED VEGETABLES.

It is recommended—

(1) That the referee be instructed to study methods peculiarly adapted to the examination of canned foods, especially methods for the detection of spoilage and conditions which are likely to lead to spoilage.

Approved.

(2) That the methods for the hydrolysis of linamarin and the subsequent determination of hydrocyanic acid[1] be adopted as tentative methods.

Approved.

CEREAL FOODS.

It is recommended—

(1) That the work on the determination of moisture, gluten, soluble carbohydrates, cold water extract, chlorin, and ash be continued.

Approved.

(2) That the referee for the coming year study methods for the determination of fat in baked cereal products.

Approved.

WINES.

No recommendation.

SOFT DRINKS.

It is recommended that the work of the present year, particularly that for the determination of ginger and of capsicum in ginger ale and other ginger drinks, be continued.

Approved.

DISTILLED LIQUORS.

No recommendation.

BEERS.

No recommendation.

VINEGARS.

No recommendation.

FLAVORING EXTRACTS.

It is recommended—

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 151.

(1) That the following method for the determination of benzoic acid in almond extracts be adopted as tentative:

Oxidation Method for Determining Benzaldehyde and Benzoic Acid Together.

Measure 10 cc. of the extract into a 100 cc. flask, add 10 cc. of a 10% sodium hydroxid solution, and 20 cc. of U. S. P. hydrogen peroxid solution; cover with a watch glass and place in a water oven. Oxidation of the aldehyde to benzoic acid begins almost immediately and should be continued 5–10 minutes after all odor of benzaldehyde has disappeared, which usually requires 20–30 minutes. Remove the flask from the water oven, transfer the contents to a separatory funnel, rinsing off the watch glass, add 10 cc. of dilute sulphuric acid solution (1 to 5) and cool the contents of the funnel to room temperature under the water tap. Extract the benzoic acid with 4 portions of 25, 25, 20, and 20 cc. of ether, respectively, and wash the combined extracts with two portions of 5–10 cc. of water, or until all sulphuric acid is removed. Filter into a tared dish, evaporate at room temperature, dry overnight in a desiccator, and weigh the benzoic acid. Multiply the result by 10.

Multiply the grams per 100 cc. of benzaldehyde obtained in the sample by 1.151 to obtain the equivalent of benzoic acid, and subtract this from the grams per 100 cc. of total benzaldehyde and benzoic acid obtained above. The difference will be grams of benzoic acid per 100 cc. of the extract.

Approved.

(2) That the *Association of Official Agricultural Chemists, Methods*, 1916, 265–9, be changed as follows:

35 (a), *Phenylhydrazin solution.*

Line 2.—Eliminate the word "article" and substitute therefor the words "product in vacuo", making the sentence read: "A sufficiently pure product can be obtained by distilling the commercial product in vacuo, rejecting the first portions coming over which contain ammonia."

36, DETERMINATION.

Line 1.—After the word "Weigh" insert the words "accurately about", making the clause read: "Weigh accurately about 15 grams of the sample into a small, glass-stoppered flask;".

55, *Hortvet and West Method Modified.—Tentative.*

Change the last sentence to read as follows: "Multiply the weight of salicylic acid so found by 9.33 to obtain the per cent by volume of methyl salicylate."

Approved.

(3) That Wichmann and Dean's qualitative method for coumarin in vanilla extract be studied with a view to its possible adoption as a preliminary test for the purpose of shortening the official method when coumarin is absent. The method is as follows:

Make 10 cc. of extract alkaline with sufficient 10% sodium hydroxid solution, dilute with 15 cc. water, to reduce the alcoholic strength, and extract with 20 cc. of ether in a separatory funnel. The ether solution will be slightly colored when the brown lower layer has been drawn off. Add a few cc. of strong alcoholic potassium hydroxid solution and wash the mixture with 10 cc. of water. The ether layer will be white. This procedure removes all organic acids, coloring matter or saccharin that may be present. Place 1 cc. of 50% potassium hydroxid solution in a test tube and pour the ether solution of coumarin over it. After thoroughly shaking, hastily evaporate the ether. Then place the tube over a free flame and evaporate the water and fuse the potassium hydroxid. If coumarin is present in any amount, a change of color will be noticed as the evaporation of the water proceeds and fusion begins. Even very small quantities of coumarin in strong hot potassium hydroxid solution will show a greenish color that suddenly disappears as the heating is continued. The disappearance of the color shows that the coumarin has been converted into the salicylate and heating should be discontinued.

Take up the melt with a few cc. of water, acidify the solution with sulphuric acid and extract in a small separatory with 5–10 cc. of benzol. Benzol is preferred to any other solvent because of its low density, low solvent power for mineral acids, and because it will not dissolve any protocatechuic acid formed from vanillin that might possibly have been carried over with the ether. Remove the acid solution from the separatory and wash the benzol with a few cc. of water. After washing, filter the benzol into a test tube and test for salicylic acid with 1–2 cc. of water, containing a few drops of ferric chlorid solution. If no color develops on shaking, add 1–2 drops of N/10 sodium hydroxid solution to neutralize any trace of mineral acid that may be present and prevent the development of the purple color. This test can be conducted easily in 15 minutes, takes only 10 cc. of extract, and does not require dealcoholization or any complicated apparatus. The only evaporation necessary, that of the ether, can be done on a steam bath without appreciable loss. The change of color on fusion indicates its own end point.

Approved.

(4) That methods of analysis for imitation vanilla preparations containing large quantities of coumarin and vanillin be studied.

Approved.

(5) That the applicability of Hortvet and West's method[1] for alcohol in orange and lemon extract, with F. M. Boyle's details for alcohol in ginger extract, be considered in connection with the official methods and other available methods. Boyle's method is as follows:

To 25 cc. of the ginger extract, add 50 cc. of water, saturate with salt and shake with 75 cc. of petroleum ether. Allow to stand for 10 minutes, draw off the lower layer into a 200 cc. flask. Wash the petroleum ether with 50 cc. of saturated salt solution. This washing must be done carefully with moderate shaking, to avoid the formation of an emulsion. A slight emulsion at this point may be broken up by pouring back and forth into two separators. Add this wash water to the 200 cc. flask and make up to the mark with salt solution. Filter through a rapidly acting folded filter and determine the alcohol in 100 cc. of the filtrate by distillation.

Approved.

(6) That Mitchell's polarization method[2] for lemon and orange extracts be studied for the purpose of determining the most accurate

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[1] *J. Ind. Eng. Chem.*, 1909, **1**: 84.

[2] *Assoc. Official Agr. Chemists, Methods*, 1916, 262.

factors to be used, especially with reference to the natural variations in the oils and the influence of dilution.

Approved.

(7) That Albright's details of the Kleber method for citral in lemon and orange oils[1] be studied.

Approved.

MEAT AND MEAT PRODUCTS.

No recommendations.

SEPARATION OF NITROGENOUS COMPOUNDS IN MEAT PRODUCTS.

No recommendations.

MEAT EXTRACTS.

No recommendations.

EDIBLE FATS AND OILS.

It is recommended that the method for the detection of the adulteration of lard with fats containing tristearin[2] be adopted as a tentative method.

Approved.

SPICES AND OTHER CONDIMENTS.

It is recommended—

(1) That the associate referee's modification of the distillation method for determining moisture in whole spices[3] be further studied with a view to its adoption by the association.

Approved.

(2) That the method for the determination of volatile oil in mustard seed and mustard substitutes[4] be adopted as tentative.

Approved.

CACAO PRODUCTS.

No recommendations were received from the referee. Your committee, however, has been informed that the referee has had under investigation methods for the detection of adulteration in cocoa butters. It is therefore recommended that these studies be continued.

Approved.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **3**: 417.
[2] *Ibid.*, **4**: 200.
[3] *Ibid.*, **3**: 428.
[4] *Ibid.*, **4**: 149.

COFFEE.

It is recommended—

(1) That the Gorter method for the determination of caffein in coffee[1] be dropped.

Approved.

(2) That the Stahlschmidt method for the determination of caffein in coffee[1] be not made official this year.

Approved.

(3) That the Fendler-Stüber method for the determination of caffein in coffee[2] be adopted tentatively.

Approved.

(4) That the Fendler-Stüber method for the determination of caffein in coffee[2] be tried on other coffees, including raw coffee, with a view to its adoption as official next year.

Approved.

TEA.

No recommendations.

BAKING POWDER.

It is recommended—

(1) That the modified Corper-Bryan method for the electrolytic determination of lead in baking powder[3] be adopted as a tentative method. (First reading.)

Approved.

(2) That a further study be made of the electrolytic method proposed by Holbrook and McCreary[4], which uses a rotating cathode, and operates directly in an aqueous phosphoric acid suspension of the powder without preliminary hydrolysis of the starch.

Approved.

(3) That a study be made of the electrolytic separation and determination of zinc in baking powder.

Approved.

(4) That a study be made of Chittick's method for the determination of lead in baking powders[5].

Approved.

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 332.
[2] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 213.
[3] *Ibid.*, 221.
[4] *Ibid.*, 230
[5] *Ibid.*, 218.

(5) That in view of the fact that Wichmann's modification of the Seeker-Clayton method gives difficulty as applied to modern baking powders, no further study be made of this method at the present time.

Approved.

(6) That the Ross-Wagner method for total fluorids[1] be studied.

Approved.

(7) That efforts be made to develop a commercial method for the determination of zinc in baking powders.

Approved.

REPORT OF COMMITTEE ON EDITING METHODS OF ANALYSIS[2].

Your committee on Editing Methods of Analysis begs leave to report that the instructions given by the association at the 1916 meeting[3] have been carried out as follows:

(1) The following general reference tables have been removed from the text of the several chapters and inserted in a separate chapter designated as Chapter **XXX**:

1 Munson and Walker's Table[4]. For calculating dextrose, invert sugar alone, invert sugar in the presence of sucrose, etc.

2 Kröber's Table[5]. For determining pentoses and pentosans.

3 Table for densities of solutions of cane sugar at 20°C.[6].

4 Table of temperature corrections for changing percentages of sugar by specific gravity to true values at 20°C.[7].

5 Geerlig's Table[8]. For dry substances in sugar-house products by the Abbé refractometer, at 28°C.

6 Table of corrections for temperature to be used in conjunction with Table No. 5[9].

7 Alcohol Table[10]. For calculating the percentages of alcohol in mixtures of ethyl alcohol and water from their specific gravities.

8 Alcohol Table[11]. For calculating the percentages of alcohol in mixtures of ethyl alcohol and water from their Zeiss immersion refractometer readings at 17.5°–25°C.

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[1] *J. Ind. Eng. Chem.*, 1917, **9**: 116.
[2] Presented by R. E. Doolittle.
[3] *J. Assoc. Official Agr. Chemists*, 1920, **3**: 537.
[4] *Assoc. Official Agr. Chemists, Methods*, 1916, 88–96.
[5] *Ibid.*, 112–7.
[6] *Ibid.*, 125–6.
[7] U. S. Bur. Standards, Circ. **19**: (1916), 25.
[8] *Assoc. Official Agr. Chemists, Methods*, 1916, 127.
[9] *Ibid.*, 128.
[10] *Ibid.*, 194–207.
[11] *Ibid.*, 208–35.

(2) There has been included in Chapter XXX a table of the International Atomic Weights for 1916.

(3) The word "chlorids" has been substituted for the word "chlorin" in the phrase "wash free from chlorin" and similar phrases.

(4) The gravimetric factors have been restored to the body of the text, these factors being based upon the atomic weights given in the atomic weight table included in Chapter XXX.

(5) Specific directions for the preparation of solutions by weight or volume have been substituted in those methods in which the strength of solutions was expressed in terms of per cent.

(6) The methods and changes in methods reported by the referees and adopted by the association in 1916 have been included in the revised methods.

(7) The deletions, additions, and changes recommended by the Committee on Editing Methods of Analysis at the 1916 meeting and approved by the association, together with those recommendations made from the floor of the meeting, and adopted by the association, have been made with the following exception:

Under Chapter III, Inorganic Plant Constituents, cross references to methods under "Soils", have not been changed to references to methods under "Waters".

Your committee found that this change of the cross references from "Soils" to "Waters" would necessitate a complete rewriting of the methods under Inorganic Plant Constituents, including changes in procedure in the methods. The methods under Chapter IV, Waters, are differently grouped from those under Inorganic Plant Constituents and provide for the determination of elements not included under Inorganic Plant Constituents. In order to comply with this motion, it would have been necessary to rewrite entirely the methods under Inorganic Plant Constituents, in most cases giving the methods in detail without cross references. Often these details were different from the official methods for Inorganic Plant Constituents adopted by the association. It is therefore recommended that the methods as now submitted, which disregard the motion, be approved. It is further recommended that the 1916 motion be referred to the referee on Inorganic Plant Constituents.

(8) The use of proper names in the titles of methods has been omitted in so far as possible. Only such names have been retained as serve to identify properly certain procedures.

(9) Careful consideration has been given to the methods designated as "Provisional" in Bureau of Chemistry Bulletin 107, Revised, and "Tentative" in the revised methods printed in supplements to the

Journal of the Association of Official Agricultural Chemists in 1916 for criticism, and your committee recommends that the rules be suspended and the following methods be made official (final action):

I. FERTILIZERS[1].

1, MECHANICAL ANALYSIS OF BONE AND TANKAGE.

THOMAS OR BASIC SLAG.

46, MECHANICAL ANALYSIS.

47, PREPARATION OF SAMPLE.

48, 49, *Gravimetric Method.*

III. INORGANIC PLANT CONSTITUENTS.

18, CHLORIN IN PLANTS.

VII. INSECTICIDES AND FUNGICIDES.

57, *Modified Method of Benedikt and Lewkowitsch.*

58, POTASSIUM AND SODIUM.

VIII. FOODS AND FEEDING STUFFS.

3, *Drying in Vacuo Without Heat.*

12, GENERAL DIRECTIONS FOR RAW SUGARS.

13, PREPARATION AND USE OF CLARIFYING REAGENTS.

15, 16, *By Polarization Before and After Inversion with Invertase.*

18, DETERMINATION OF SUCROSE FROM REDUCING SUGARS BEFORE AND AFTER INVERSION.

24, 25, *Munson and Walker General Method.*

26, *I. Direct Weighing of Cuprous Oxid.*

28, 29, *II. A. H. Low Volumetric Method, Modified.*

30, *III. Volumetric Permanganate Method.*

31, *IV. Electrolytic Deposition from Sulphuric Acid Solution.*

32, *V. Electrolytic Deposition from Sulphuric and Nitric Acid Solution.*

33, *VI. Electrolytic Deposition from Nitric Acid Solution.*

34, *VII. Reduction in Hydrogen.*

35, 36, 38, 39, *Herzfeld Gravimetric Method.*

42, *General Gravimetric Method.*

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[1] The references given are to the *Assoc. Official Agr. Chemists, Methods*, 1916.

43, 44, 45, *Wein Method.*

46, *General Gravimetric Method.*

52, *General Gravimetric Method.*

53, 54, 55, *Allihn Gravimetric Method.*

56, REDUCING SUGARS OTHER THAN DEXTROSE.

61, 62, *Diastase Method with Subsequent Acid Hydrolysis.*

63, 64, PENTOSANS.

IX. SACCHARINE PRODUCTS.

1, PREPARATION OF SAMPLE.

3, **Drying upon Pumice Stone.**

4, **Drying upon Quartz Sand.**

10, *REFRACTOMETER METHOD.*

17, SOLUBLE AND INSOLUBLE ASH.

18, ALKALINITY OF THE SOLUBLE ASH.

19, ALKALINITY OF THE INSOLUBLE ASH.

21, NITROGEN.

22, *Method I.*

23, *Method II. (Double Dilution Method.)*

24, *Method I.*

25, *Method II.*

31, ALCOHOL IN SIRUPS USED IN CONFECTIONERY ("BRANDY DROPS").

HONEY.

34, PREPARATION OF SAMPLE.

35, MOISTURE.

37, SOLUBLE ASH.

38, ALKALINITY OF THE SOLUBLE ASH.

41, REDUCING SUGARS.

42, SUCROSE.

46, FREE ACID.

MAPLE PRODUCTS.

53, PREPARATION OF SAMPLE.

54, MOISTURE.

55, POLARIZATION.

56, REDUCING SUGARS AS INVERT SUGAR.

57, *By Polarization.*

58, *By Reducing Sugars Before and After Inversion.*

59, TOTAL ASH.

60, SOLUBLE AND INSOLUBLE ASH.

61, ALKALINITY OF THE SOLUBLE ASH.

62, ALKALINITY OF THE INSOLUBLE ASH.

X. FOOD PRESERVATIVES.

3, *Jorissen's Test.—Qualitative.*

13, *Quantitative Method.*

XIII. FRUITS AND FRUIT PRODUCTS.

1, PREPARATION OF SAMPLE.

2, ALCOHOL.

3, TOTAL SOLIDS.

4, *Direct Method.*

5, *Indirect Method.*

7, ALKALINITY OF THE ASH.

8, SULPHATE AND CHLORID.

9, TOTAL ACIDITY.

10, VOLATILE ACIDS.

14, *By Reducing Sugars Before and After Inversion.*

15, REDUCING SUGARS.

16, COMMERCIAL GLUCOSE.

18, ALCOHOL PRECIPITATE.

19, *Qualitative Test.*

XIV. CANNED VEGETABLES.

2, PREPARATION OF SAMPLE.

3, MOISTURE.

7, TOTAL ACIDS.

8, VOLATILE ACIDS.

XVI. WINES.

2, PREPARATION OF SAMPLE.

3, SPECIFIC GRAVITY.

4, ALCOHOL.

7, *Method I. (By Direct Weighing.)*

8, *Method II. (By Oxidation with Dichromate.)*

9, GLYCEROL IN SWEET WINES.

10, GLYCEROL-ALCOHOL RATIO.

11, From the Specific Gravity of the Dealcoholized Wine.

12, By Evaporation.

13, NON-SUGAR SOLIDS.

14, REDUCING SUGARS.

15, *By Reducing Sugars Before and After Inversion.*

16, *By Polarization.*

17, COMMERCIAL GLUCOSE.

18, ASH.

19, ASH-EXTRACT RATIO.

20, ALKALINITY OF THE WATER-SOLUBLE ASH.

21, ALKALINITY OF THE WATER-INSOLUBLE ASH.

22, PHOSPHORIC ACID.

23, SULPHURIC ACID.

24, CHLORIN.

25, TOTAL ACIDS.

26, *Method I.*

27, *Method II. (Hortvet Method.)*

28, FIXED ACIDS.

29, TOTAL TARTARIC ACID.

30, FREE TARTARIC ACID AND CREAM OF TARTAR.

33, CRUDE PROTEIN.

34, PENTOSANS.

XVII. DISTILLED LIQUORS.

1, SPECIFIC GRAVITY.

4, *Method II.*

7, ACIDITY.

8, ESTERS.

9, 10, ALDEHYDES.

11, 12, FURFURAL.

13, 14, FUSEL OIL.

15, SUGARS.

16, *Trillat Method.*

17, *Riche and Bardy Method.*

18, *Immersion Refractometer Method.* *(Leach and Lythgoe.)*

XVIII. BEERS.

1, PREPARATION OF SAMPLE.

3, SPECIFIC GRAVITY.

4, ALCOHOL.

7, *Method III.*

8, EXTRACT OF ORIGINAL WORT (APPROXIMATE).

9, DEGREE OF FERMENTATION.

10, TOTAL ACIDS.

11, VOLATILE ACIDS.

12, REDUCING SUGARS.

15, GLYCEROL.

17, PHOSPHORIC ACID.

XIX. VINEGARS.

2, PREPARATION OF SAMPLE.

3, SPECIFIC GRAVITY.

5, 6, GLYCEROL.

7, SOLIDS.

8, TOTAL REDUCING SUBSTANCES BEFORE INVERSION.

9, REDUCING SUGARS BEFORE INVERSION AFTER EVAPORATION.

10, REDUCING SUGARS AFTER INVERSION.

13, ASH.

14, SOLUBLE AND INSOLUBLE ASH.

15, ALKALINITY OF THE SOLUBLE ASH.

16, SOLUBLE AND INSOLUBLE PHOSPHORIC ACID.

17, TOTAL ACIDS.

18, FIXED ACIDS.

19, VOLATILE ACIDS.

20, COLOR.

21, *Fincke Method.*

23, PENTOSANS.

24, *Qualitative Test.*

25, TOTAL TARTARIC ACID.

XX. FLAVORING EXTRACTS.

VANILLA EXTRACT AND ITS SUBSTITUTES.

1, SPECIFIC GRAVITY.

4, 5, *Modified Hess and Prescott Method.*

6, NORMAL LEAD NUMBER.

7, TOTAL SOLIDS.

10, SUCROSE.

12, METHYL ALCOHOL.

LEMON AND ORANGE EXTRACTS.

17, SPECIFIC GRAVITY.

18, ALCOHOL.

20, *By Polarization.* (*Mitchell Method.*)

21, *By Precipitation.* (*Mitchell Method.*)

22, 23, *Chace Method.*

24, 25, *Hiltner Method.*

28, SUCROSE.

29, METHYL ALCOHOL.

LEMON AND ORANGE OILS.

32, SPECIFIC GRAVITY.

33, INDEX OF REFRACTION.

34, OPTICAL ROTATION.

35, 36, *Kleber Method.*

37, *Hiltner Method.*

38, *Chace Method.*

39, PHYSICAL CONSTANTS OF THE 10 PER CENT DISTILLATE.

49, *Chace Method.*

XXI. MEAT AND MEAT PRODUCTS.

MEAT.

1, PREPARATION OF SAMPLE.

2, MOISTURE.

5, TOTAL PHOSPHORUS.

MEAT EXTRACTS AND SIMILAR PRODUCTS.

32, PREPARATION OF SAMPLE.

33, MOISTURE.

35, TOTAL PHOSPHORUS.

36, CHLORIN.

XXII. DAIRY PRODUCTS.

MILK.

6, *Method I.*

7, *Method II.*

16, ACETIC SERUM.

CHEESE.

56, ACIDITY.

XXIII. FATS AND OILS.

2, At $\frac{20°C.}{4°}$

5, General Directions.

11, *Capillary Tube Method.*

12, 13, *Alcoholic or Aqueous Sodium Hydroxid Method.*

14, *Glycerol-Potassium Hydroxid Method.*

28, *Polenske Method.*

31, *Benedikt-Lewkowitsch Method.*

34, UNSAPONIFIABLE RESIDUE.

37, *Modified Renard Test.*

XXIV. SPICES AND OTHER CONDIMENTS.

SPICES.

1, PREPARATION OF SAMPLE.

4, SOLUBLE AND INSOLUBLE ASH.

5, ASH INSOLUBLE IN ACID.

8, *Winton, Ogden and Mitchell Method.*

9, VOLATILE AND NON-VOLATILE ETHER EXTRACT.

10, ALCOHOL EXTRACT.

12, COPPER-REDUCING SUBSTANCES BY DIRECT INVERSION.

13, STARCH.

14, CRUDE FIBER.

15, TANNIN.

PREPARED MUSTARD.

23, PREPARATION OF SAMPLE.

24, SOLIDS.

26, SALT.

29, ACIDITY.

30, COPPER-REDUCING SUBSTANCES.

TOMATO PRODUCTS.

34, PREPARATION OF SAMPLE.

40, ALKALINITY OF THE ASH.

42, REDUCING SUGARS BEFORE INVERSION.

43, REDUCING SUGARS AFTER INVERSION.

44, SUCROSE.

45, TOTAL ACIDS.

46, VOLATILE ACIDS.

48, FIXED ACIDS.

XXV. CACAO PRODUCTS.

1, PREPARATION OF SAMPLE.

4, ASH INSOLUBLE IN ACID.

5, SOLUBLE AND INSOLUBLE ASH.

6, ALKALINITY OF THE SOLUBLE ASH.

7, ALKALINITY OF THE INSOLUBLE ASH.

12, FAT.

XXVI. COFFEES.

ROASTED COFFEE.

4, PREPARATION OF SAMPLE.

8, ASH INSOLUBLE IN ACID.

9, SOLUBLE AND INSOLUBLE ASH.

10, ALKALINITY OF THE SOLUBLE ASH.

11, SOLUBLE PHOSPHORIC ACID IN THE ASH.

12, INSOLUBLE PHOSPHORIC ACID IN THE ASH.

16, CRUDE FIBER.

19, PETROLEUM ETHER EXTRACT.

XXVII. TEA.

2, PREPARATION OF SAMPLE.

3, MOISTURE.

6, SOLUBLE AND INSOLUBLE ASH.

7, ASH INSOLUBLE IN ACID.

8, ALKALINITY OF THE ASH.

9, PHOSPHORIC ACID IN THE ASH.

10, PETROLEUM ETHER EXTRACT.

12, CRUDE FIBER.

XXVIII. BAKING POWDERS AND THEIR INGREDIENTS.

1, PREPARATION OF SAMPLE.

3, 4, 5, *Method Using Knorr's Apparatus.*

6, 7, 8, *Method Using Heidenhain's Apparatus.*

9, RESIDUAL CARBON DIOXID.

10, AVAILABLE CARBON DIOXID.

11, ACIDITY.

13, *Goldenberg-Geromont-Heidenhain Method.*

14, *Qualitative Test.*

15, *Quantitative Method.*

16, POTASSIUM BITARTRATE.

17, *Direct Inversion Method.*

18, *Indirect Method.*

20, *Qualitative Test.*

21, INSOLUBLE ASH AND PREPARATION OF SOLUTION.

22, IRON AND ALUMINIUM.

23, CALCIUM.

24, POTASSIUM AND SODIUM.

26, SULPHURIC ACID.

27, AMMONIA.

Respectfully submitted,
R. E. Doolittle, W. A. Withers,
A. F. Seeker, J. P. Street,
G. W. Hoover, B. L. Hartwell,
Committee on Editing Methods of Analysis.

Adopted.

REPORT OF SECRETARY-TREASURER FOR

By C. L. ALSBERG (Bureau of Chemistry,

RECEIPTS.

1916		
Nov. 14	Bank balance	$221.07
Nov. 22	1915–16 dues from 4 organizations (Oakland, Calif., Canada, Wisconsin and Pennsylvania) received after the Secretary-Treasurer's report for 1916 had been made out	8.00
1917		
Nov. 12	Dues for the year 1916–17 from 76 Federal, State, Municipal and Canadian organizations (includes 1 subscription to *Journal* deposited under assumption it was for dues)	380.00
		$609.07

THE YEAR ENDING NOVEMBER 21, 1917.

Washington, D. C.), *Secretary-Treasurer.*

DISBURSEMENTS.

				Check No.
1916				
Nov. 25	Tips, New Willard Hotel, 1916 meeting		$5.00	62
Nov. 25	Telephone calls, New Willard Hotel, 1916 meeting		2.20	63
Nov. 25	1000 special request 2-cent envelopes		22.00	64
Dec. 4	Postage		3.00	65
Dec. 11	Stenographic report Tuesday afternoon meeting, November 21, 1916		8.50	66
Dec. 16	Partial payment for editing Vol. II, No. 4 of the *Journal* (Journal)		50.00	67
1917				
Jan. 10	Post office box rent for quarter ending March 31, 1917		2.00	68
Jan. 10	Postage (Journal)		3.00	69
Jan. 30	Expressage (Journal)		0.37	70
Feb. 12	Printing circulars		10.25	71
Feb. 12	Printing 1000 letterheads (Journal)		5.25	72
Feb. 16	Postage		5.00	73
Feb. 21	Certified public accountant (Journal)		150.00	74
Feb. 21	Transferring post office box, increased rent due	$0.36		
	Less refund on key	0.20		
			0.16	75
Apr. 16	Post office box rent for quarter ending June 30, 1917		2.00	76
May 19	Printing 1000 letterheads		5.25	77
May 22	Refund to Williams & Wilkins Co. which was deposited under assumption it was dues (Journal)		5.00	78
June 11	Editing remainder of Vol. II, No. 4 and part of Vol. III, No. 1 of the *Journal* (Journal)		50.00	79
June 21	1000 special request 2-cent envelopes	$22.56		
	Less return on mutilated stamped envelopes	0.56		
			22.00	80
June 22	Post office box rent for quarter ending September 30, 1917		2.00	81
July 6	On account reporting Wednesday meeting, November 22, 1916		50.00	82
July 24	To complete payment reporting Wednesday meeting, November 22, 1916		28.65	83
Aug. 6	250 1-cent stamped envelopes		2.86	84
Aug. 20	Postage		5.00	85
Sept. 11	Postage		5.00	86
Sept. 24	Post office box rent for quarter ending December 30, 1917		2.00	87
Nov. 1	Ribbon for 1917 badges		3.24	88
Nov. 1	Printing 1000 announcements of 1917 meeting		25.75	89
Nov. 1	400 tags for 1917 meeting		1.80	90
Nov. 12	Bank balance	$139.81		
	Less checks out	8.02		
			131.79	
			$609.07	

The Auditing Committee[1] has examined the above report and finds the same correct, all payments being substantiated by vouchers. The balance to credit is on deposit in the National Metropolitan Bank in the name of the association.

Respectfully submitted,
W. A. Withers,
B. H. Silberberg,
Auditing Committee.

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[1] W. D. Lynch was appointed a member of the Auditing Committee, but was called out of the city and was unable to meet with the committee to examine the report.

REPORT ON THE JOURNAL.

By C. L. ALSBERG (Bureau of Chemistry, Washington, D. C.), *Chairman, Board of Editors.*

As you know, certain differences have arisen between the executive committee, the board of editors, and the publishers of *The Journal.* The only serious differences are differences as to the rights and authority of the board of editors, as compared with the authority of the publishers. For reasons which seemed satisfactory to the editors, the matter of arbitration was not very greatly hastened in the beginning of the year. The publishers insisted that the question of authority, which arose originally, be arbitrated in connection with the publishing of the methods as a supplement to *The Journal* and the refusal of the publishers to publish that supplement in exactly the shape in which the board of editors wished it to be published. The publishers conceded the point finally, and then, having conceded, they insisted on having the matter arbitrated. There has been considerable correspondence and it looks now as if an adjustment soon can be made. I believe this difference can be adjusted and then I think *The Journal* will make up the time that has been lost.

As I reported last year[1], the publisher's statement showed a deficit of something around two hundred and seventy-five dollars, which was a very good showing for the first year of *The Journal.* As a matter of fact, I think there is no such deficit. That point is now being settled. The second volume has been subscribed to very generally. There are about nine hundred subscribers, and the indications are that there will be no deficit for the second volume and perhaps a slight surplus, even on the basis of figuring employed by the publishers, which will, of course, on my basis, if I am right, be a considerably larger surplus. The prospects are good that *The Journal* will be self-supporting.

I think I can assure the association that the delay in publishing the third volume, of which one number has appeared, will soon be remedied. The delay is only due to the fact that we have had this disagreement with the publishers. I have no doubt as to how it will be settled.

Before this report is acted upon, I wish to mention the matter of printing the methods in book form. The methods have been waiting to be printed in book form until the adoption of this report. We will at once put forward the printing of the methods in book form. I think we can anticipate that the methods will be ready in separate book form in the course of the winter. I think that is pretty definite. We can not

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[1] *J. Assoc. Official Agr. Chemists,* 1920, **3**: 578.

tell the exact price at which the book will be sold until the revised manuscript is received and we see how extensively it will have to be changed. The methods have been kept set up in type and that should make the printing less expensive than it would be if all the material had to be reset. All that is necessary is to make the changes. We shall make an effort to sell them to the members as cheaply as possible.

PRESENTATION OF GAVEL.

By R. N. Brackett (Clemson Agricultural College, Clemson College, S. C.).

I have noticed that the presiding officer of this association has had no symbol of authority which is the property of the association. It therefore occurred to me, when acting as your President last year, to present a gavel to the association.

It seems fitting that this donation should come from the South because our association had its beginning in the State of Georgia. It also seems fitting that this donation should come from South Carolina, since the discovery of phosphate rock deposits in my State and their subsequent use for the manufacture of acid phosphate and the use of this material as a fertilizer led to a study of methods of analysis of fertilizers which culminated in the organization of this association. It further seems fitting that this donation should come from the Clemson Agricultural College of South Carolina, since this gavel is made from a cedar which grew on the Fort Hill Plantation, the estate of John C. Calhoun, and upon this estate is located the Clemson Agricultural College of South Carolina. This property was bought in by Thomas G. Clemson, the son-in-law of John C. Calhoun, and when Mr. Clemson died he left the property to the State of South Carolina on condition that the State would establish and maintain on this property an agricultural and mechanical college.

There is a still further tie or point of contact between the estate of John C. Calhoun, the Clemson Agricultural College, and the Association of Official Agricultural Chemists in that John C. Calhoun received his education at Yale University, which furnished the first president of this association. I refer to Samuel W. Johnson.

I take pleasure in presenting to the association this little token of the esteem of the South and a reminder of the connection of the South with the association. I shall leave it in Washington to be marked as follows and turned over to the secretary of the association:

"Cedar from Fort Hill, John C. Calhoun Estate,
The Clemson Agricultural College, South Carliona.
A. O. A. C., 1917.
Presented by R. N. Brackett, President, 1916."

J. K. HAYWOOD: In the name of the association I wish to thank Dr. Brackett for this gavel. It will certainly be treasured in the archives of the association. We are very glad to have this gavel and will certainly keep a record of it that it may go down to posterity.

The meeting adjourned at 12.45 p. m. to reconvene at 2 p. m.

THIRD DAY.

WEDNESDAY—AFTERNOON SESSION.

REPORT OF COMMITTEE TO COOPERATE WITH OTHER COMMITTEES ON FOOD DEFINITIONS[1].

Your comittee submits for your information and action the following report of the activities of the Joint Committee on Food Definitions and Standards.

The definitions and standards formulated and recommended by the joint committee are, you will recall, submitted for approval first to your association and to the Association of American Dairy, Food and Drug Officials, according to the order of their times of meeting, and after approval by these bodies, or in cases of emergency calling for prompt action in the interim between their meetings, after the approval of their executive committees, are submitted to the Secretary of Agriculture. After approval by all three of the collaborating authorities, the definitions and standards are officially published by the Secretary of Agriculture for the information and guidance of those concerned.

Since the organization of the joint committee, definitions and standards for the following food products have been so published in Food Inspection Decisions of the United States Department of Agriculture Nos. 158, 160, 161, 162, 165, 169, 170 and 171:

Milk products:
- Condensed milk
- Evaporated milk
- Concentrated milk
- Sweetened condensed milk
- Condensed skimmed milk
- Sweetened condensed skimmed milk
- Dried milk
- Dried skimmed milk
- Malted milk

Gluten products:
- Ground gluten
- Gluten flour
- Gluten flour, self-raising
- "Diabetic" food

Maple products:
- Maple sugar
- Maple concrete
- Maple syrup

Noodles:
- Noodles
- Egg noodles
- Plain noodles
- Water noodles

Cacao products:
- Cacao beans, cocoa beans
- Cacao nibs, cocoa nibs, cracked cocoa
- Chocolate, plain chocolate, bitter chocolate, chocolate liquor, chocolate paste, bitter chocolate coatings
- Sweet chocolate, sweet chocolate coatings
- Cocoa, powdered cocoa
- Milk chocolate, milk cocoa, sweet milk chocolate or sweet milk cocoa

Edible vegetable fats and oils:
- Edible fats and edible oils
- Cacao butter, cocoa butter

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[1] Presented by William Frear.

Edible vegetable fats and oils—Continued:

- Coconut oil, copra oil
- Cochin oil
- Ceylon oil
- Corn oil, maize oil
- Cottonseed oil
- Olive oil, sweet oil
- Palm kernel oil
- Peanut oil, arachis oil, earthnut oil
- Poppy seed oil
- Rape seed oil, rape oil, colza oil

Edible vegetable fats and oils—Concluded:

- Soy bean oil, soy oil, soja oil
- Sesame oil, gingili oil, teel oil, benne oil
- Sunflower oil

Grades for commercial corn.

Macaroni, etc.:

- Macaroni, spaghetti, vermicelli
- Flour macaroni, flour spaghetti, flour vermicelli.

In addition to the foregoing, there are two definitions and standards that have been approved by both of the associations, but not yet published by the Secretary of Agriculture. These relate to baking powder and evaporated apples[1], respectively.

A third group of schedules, recommended by the joint committee after preliminary consideration by the trade and official interests concerned, and after public hearings, has been approved by the association of American Dairy, Food and Drug Officials, and is now submitted for your consideration and action.

The first of these schedules comprises a modification of definition 3, of the subdivision, Vegetables and Vegetable Products, of the standards proclaimed by the Secretary of Agriculture, June 30, 1906[2], a specific definition for canned peas and subordinate definitions and standards for canned pea grades. These grade definitions involve a revision of trade nomenclature and classification. This revision was made by the pea canners' section of the National Canners Association acting in consultation with a subcommittee of the Joint Committee on Food Definitions and Standards, was approved in February, 1916, by a conference of delegates from the above-named association and from the National Wholesale Grocers' Association, was thereafter carefully considered by the joint committee, and after a public hearing in February, 1917, was adopted by that committee for recommendation, on April 17, 1917.

CANNED VEGETABLES, CANNED PEAS AND CANNED PEA GRADES.

Definitions and standards adopted by the Joint Committee on Definitions and Standards, April 25, 1917:

1. *Canned vegetables* are properly matured and prepared fresh vegetables, with or without the addition of potable water, salt and sugar, as specified in the separate definitions for the several kinds of canned vegetables, sterilized by heat, with or without previous cooking in vessels from which they take up no injurious substance, and kept in suitable, clean, hermetically sealed containers.

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[1] Since published. U. S. Dept. Agr., Office of the Secretary, Circ. **136:** (1919), 22, 8.

[2] U. S. Dept. Agr., Office of the Secretary, Circ. **19:** (1906), 9.

2. *Canned peas* are the canned vegetables prepared from the well developed but still tender seeds of the common or garden pea (*Pisum sativum*) by shelling, winnowing and thorough washing, with or without grading and with or without precooking (blanching), and by the addition, before sterilization, of the necessary amount of potable water, with or without sugar and salt.

CANNED PEA VARIETIES.

3. *Early peas* are peas of early maturing sorts having a smooth skin.

4. *Sugar peas, sweet peas* are peas of later maturing varieties having a wrinkled skin and sweet flavor.

CANNED PEA GRADES.

5. *Fancy peas* are young, succulent peas of fairly uniform size and color, unless declared to be ungraded for size, with reasonably clear liquor, and free from flavor defects due to imperfect processing.

6. *Standard peas* are less succulent peas than the "fancy" grade, but green and of mellow consistency, of uniform size and color, unless declared to be ungraded for size, with reasonably clear liquor, though not necessarily free from sediment, and reasonably free from flavor defects due to imperfect processing.

7. *Substandard* peas are peas that are overmature, though not fully ripened, or that lack in other respects the qualifications for the standard grade.

CANNED PEA SIZES.

No. 1 peas are peas which were, before precooking (blanching), small enough to pass through a screen of $\frac{9}{32}$ inch (7 mm.) mesh.

No. 2 peas are peas which were, before precooking (blanching), small enough to pass through a screen of $\frac{10}{32}$ inch (8 mm.) mesh.

No. 3 peas are peas which were, before precooking (blanching), small enough to pass through a screen of $\frac{11}{32}$ inch (8.7 mm.) mesh.

No. 4 peas are peas which were, before precooking (blanching), small enough to pass through a screen of $\frac{12}{32}$ inch (9.5 mm.) mesh.

No. 5 peas are peas which were, before precooking (blanching), small enough to pass through a screen of $\frac{13}{32}$ inch (10.3 mm.) mesh.

No. 6 peas are peas not all of which were, before precooking (blanching), small enough to pass through a screen of $\frac{13}{32}$ inch (10.3 mm.) mesh.

There follows a partial schedule for soda water flavors and soda waters. What is here presented, is a mere beginning upon this numerous and complex group of products. The committee has believed that the items here presented have sufficient value to justify their being considered and acted upon at this time.

SODA WATER FLAVORS AND SODA, SODA WATER.

1. *Ginger ale flavor* is the water-soluble product obtained from ginger, with or without flavoring substances which do not simulate the flavor or pungent effect of ginger. The predominating flavor of the product is that of ginger.

2. *Ginger ale with capsicum flavor* is the water-soluble product obtained from ginger and capsicum, with or without other flavoring substances. The predominating flavor of the product is that of ginger.

3. *Sarsaparilla flavor* is the water-soluble product prepared with oil of sassafras and methyl salicylate or oil of wintergreen or oil of sweet birch and with or without other essential oils or extract of sarsaparilla.

4. *Ginger ale* is the carbonated or artificially carbonated beverage prepared with potable water, acidulated[1] sugar (sucrose) sirup and ginger ale flavor.

5. *Ginger ale with capsicum* is the carbonated or artificially carbonated beverage prepared with potable water, acidulated[1] sugar (sucrose) sirup and ginger ale with capsicum flavor.

6. *Sarsaparilla* is the carbonated or artificially carbonated beverage prepared with potable water, sugar (sucrose) sirup and sarsaparilla flavor. It may or may not be acidulated[1].

(Additional definitions and standards for soda water flavors, soda, soda water, under consideration.)

NOTE.—It is the opinion of the committee that the use of sugar color in ginger ale, ginger ale with capsicum or sarsaparilla, soda water flavor or the corresponding soda, soda water, does not require that they be labeled as imitation products.

The next schedule is a revision and expansion of the spice schedule[2]. The revision is the outcome of a great accumulation of additional data gathered by the Bureau of Chemistry in its work, by the committee for its revisional work, and furnished by the spice grinders of America in an exceptionally well-prepared and able presentation of their experience at a public hearing, and of experience gained by the Federal and State governments in enforcing the food laws. The revision includes the perfecting of eight of the definitions; the slight modification of limits in eight cases, usually so as to narrow the range of variation, though in two or three instances so as to widen the latitude as need therefor has been shown by additional analyses of authentic goods; the modification of botanical nomenclature in a few cases to meet the requirements of present usage; and the dropping of the definitions for cassia buds and for Bombay mace, the former because of inutility and the latter because of the committee's conclusion that the status of a spice should not be accorded to the product.

The expansion includes the formulation of standards to accompany the previously given definitions for twelve spices; the insertion of definitions, at present without corresponding standards, for eight spices; and of both definitions and standards for ten other spices not included in the schedule of 1906. The modifications of the paprika and mustard groups of spices are possibly those of major importance.

This schedule revision represents several years of study, at least two main hearings, and numerous consultations with various specialists, officials, the trade and others; to all of whom the committee is indebted for valued collaboration.

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[1] It is the opinion of the committee that citric acid when of the purity required by the U. S. Pharmacopoeia is permissible for the acidulation without a statement on the label.

[2] U. S. Dept. Agr., Office of the Secretary, Circ. **19**: (1906), 11.

SPICES.

Definitions and standards adopted by the Joint Committee on Definitions and Standards, July 29, 1917:

The term "dried" as used in this schedule refers to the air-dried product. The term "starch" as used in this schedule refers to starch as determined by the official diastase method. In the examination of the products listed in this schedule the methods of analysis of the Association of Official Agricultural Chemists should be followed, except where otherwise specified.

1. *Spices* are aromatic vegetable substances used for the seasoning of food. They are clean, sound, and true to name, and from them no portion of any volatile oil or other flavoring principle has been removed.

2. *Allspice, pimento,* is the dried, nearly ripe fruit of *Pimenta officinalis* (L.) Karst. It contains not less than eight per cent (8%) of quercitannic acid (calculated from the total oxygen absorbed by the aqueous extract), not more than twenty-five per cent (25%) of crude fiber, not more than six per cent (6%) of total ash, nor more than four-tenths per cent (0.4%) of ash insoluble in hydrochloric acid.

3. *Anise, aniseed,* is the dried fruit of *Pimpinella anisum* L. It contains not more than nine per cent (9%) of total ash, nor more than one and five-tenths per cent (1.5%) of ash insoluble in hydrochloric acid.

4. *Bay leaves* are the dried leaves of *Laurus nobilis* L.

5. *Capers* are the flower buds of *Capparis spinosa* L.

6. *Caraway, caraway seed,* is the dried fruit of *Carum carvi* L. It contains not more than eight per cent (8%) of total ash, nor more than one and five-tenths per cent (1.5%) of ash insoluble in hydrochloric acid.

7. *Cardamom* is the dried, nearly ripe fruit of *Elettaria cardamomum* White & Maton.

8. *Cardamom seed* is the dried seed of cardamom. It contains not more than eight per cent (8%) of total ash, nor more than three per cent (3%) of ash insoluble in hydrochloric acid.

9. *Red pepper* is the red, dried, ripe fruit of any species of *Capsicum.* It contains not more than eight per cent (8%) of total ash, nor more than one per cent (1%) of ash insoluble in hydrochloric acid.

10. *Cayenne pepper, cayenne,* is the dried, ripe fruit of *Capsicum frutescens* L., *Capsicum baccatum* L., or some other small-fruited species of *Capsicum.* It contains not less than fifteen per cent (15%) of nonvolatile ether extract, not more than one and five-tenths per cent (1.5%) of starch, not more than twenty-eight per cent (28%) of crude fiber, not more than seven per cent (7%) of total ash, nor more than one per cent (1%) of ash insoluble in hydrochloric acid.

11. *Paprika* is the dried, ripe fruit of *Capsicum annuum* L. It contains not more than eight and five-tenths per cent (8.5%) of total ash, nor more than one per cent (1%) of ash insoluble in hydrochloric acid. The iodin number of its extracted oil is not less than 125 nor more than 136.

12. *Hungarian paprika* is paprika having the pungency and flavor characteristic of that grown in Hungary.

(a) *Rosenpaprika, rozsapaprika, rose paprika,* is Hungarian paprika prepared by grinding specially selected pods of paprika, from which the placentæ, stalks, and stems have been removed. It contains no more seeds than the normal pods, not more than eighteen per cent (18%) of nonvolatile ether extract, not more than twenty-three per cent (23%) of crude fiber, not more than six per cent (6%) of total ash, nor more than four-tenths per cent (0.4%) of ash insoluble in hydrochloric acid.

(b) *Koenigspaprika, king's paprika,* is Hungarian paprika prepared by grinding whole pods of paprika without selection, and includes the seeds and stems naturally occurring with the pods. It contains not more than eighteen per cent (18%) of nonvolatile ether extract, not more than twenty-three per cent (23%) of crude fiber, not more than six and five-tenths per cent (6.5%) of total ash, nor more than five-tenths per cent (0.5%) of ash insoluble in hydrochloric acid.

13. *Pimenton, pimiento, Spanish paprika,* is paprika having the characteristics of that grown in Spain. It contains not more than eighteen per cent (18%) of nonvolatile ether extract, not more than twenty-one per cent (21%) of crude fiber, not more than eight and five-tenths per cent (8.5%) of total ash, nor more than one per cent (1%) of ash insoluble in hydrochloric acid.

14. *Celery seed* is the dried fruit of *Apium graveolens* L. It contains not more than ten per cent (10%) of total ash, nor more than two per cent (2%) of ash insoluble in hydrochloric acid.

15. *Cinnamon* is the dried bark of cultivated varieties of *Cinnamomum zeylanicum* Breyne or of *Cinnamomum cassia* (Nees) Blume, from which the outer layers may or may not have been removed.

16. *Ceylon cinnamon* is the dried inner bark of cultivated varieties of *Cinnamomum zeylanicum* Breyne.

17. *Saigon cinnamon, cassia,* is the dried bark of cultivated varieties of *Cinnamomum cassia* (Nees) Blume.

18. *Ground cinnamon*[1], *ground cassia,* is the powder made from cinnamon. It contains not more than five per cent (5%) of total ash, nor more than two per cent (2%) of ash insoluble in hydrochloric acid.

19. *Cloves* are the dried flower buds of *Caryophyllus aromaticus* L. They contain not more than five per cent (5%) of clove stems, not less than fifteen per cent (15%) of volatile ether extract, not less than twelve per cent (12%) of quercitannic acid (calculated from the total oxygen absorbed by the aqueous extract), not more than ten per cent (10%) of crude fiber, not more than seven per cent (7%) of total ash, nor more than five-tenths per cent (0.5%) of ash insoluble in hydrochloric acid.

20. *Coriander seed* is the dried fruit of *Coriandrum sativum* L. It contains not more than seven per cent (7%) of total ash, nor more than one and five-tenths per cent (1.5%) of ash insoluble in hydrochloric acid.

21. *Cumin seed* is the dried fruit of *Cuminum cyminum* L. It contains not more than eight and five-tenths per cent (8.5%) of total ash, nor more than one and five-tenths per cent (1.5%) of ash insoluble in hydrochloric acid.

22. *Curcuma, turmeric,* is the dried rhizome or bulbous roots of *Curcuma longa* L.

23. *Dill seed* is the dried fruit of *Anethum graveolens* L. It contains not more than ten per cent (10%) of total ash, nor more than three per cent (3%) of ash insoluble in hydrochloric acid.

24. *Fennel seed* is the dried fruit of cultivated varieties of *Foeniculum vulgare* Hill. It contains not more than nine per cent (9%) of total ash, nor more than two per cent (2%) of ash insoluble in hydrochloric acid.

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[1] The question of the use of cassia buds in ground cinnamon is under consideration.

25. *Ginger* is the washed and dried, or decorticated and dried, rhizome of *Zinziber officinale* Roscoe. It contains not less than forty-two per cent (42%) of starch, not more than eight per cent (8%) of crude fiber, not more than one per cent (1%) of lime (CaO), not less than twelve per cent (12%) of cold water extract, not more than seven per cent (7%) of total ash, not more than two per cent (2%) of ash insoluble in hydrochloric acid, nor less than two per cent (2%) of ash soluble in cold water.

26. *Jamaica ginger* is ginger grown in Jamaica. It contains not less than fifteen per cent (15%) of cold water extract, and conforms in other respects to the standards for ginger.

27. *Limed ginger, bleached ginger*, is whole ginger coated with carbonate of calcium. It contains not more than four per cent (4%) of carbonate of calcium, nor more than ten per cent (10%) of total ash, and conforms in other respects to the standards for ginger.

28. *Horse-radish* is the root of *Radicula armoracia* (L.) Robinson.

29. *Prepared horse-radish* is comminuted horse-radish, with or without a vinegar.

30. *Mace* is the dried arillus of *Myristica fragrans* Houtt. It contains not less than twenty per cent (20%) nor more than thirty per cent (30%) of nonvolatile ether extract, not more than ten per cent (10%) of crude fiber, not more than three per cent (3%) of total ash, nor more than five-tenths per cent (0.5%) of ash insoluble in hydrochloric acid.

31. *Macassar mace, papua mace*, is the dried arillus of *Myristica argentea* Warb.

32. *Marjoram* is the dried leaves, with or without a small proportion of the flowering tops, of *Majorana hortensis* Moench.

33. *Mustard seed* is the seed of *Sinapis alba* L. (white mustard), *Brassica nigra* (L.) Koch (black mustard), *Brassica juncea* Hook f. et Th., or varieties or closely related species of the types of *Brassica nigra* and *Brassica juncea.*

Sinapis alba (white mustard) contains no appreciable amount of volatile oil. It contains not more than five per cent (5%) of total ash, nor more than one and five-tenths per cent (1.5%) of ash insoluble in hydrochloric acid.

Brassica nigra (black mustard) and *Brassica juncea* yield six-tenths per cent (0.6%) of volatile mustard oil (calculated as allylisothiocyanate and determined by the method given in Service and Regulatory Announcements[1]). The varieties and species closely related to the types of *Brassica nigra* and *Brassica juncea* yield not less than six-tenths per cent (0.6%) of volatile mustard oil, similar in character and composition to the volatile oils yielded by *Brassica nigra* and *Brassica juncea.* These mustard seeds contain not more than five per cent (5%) of total ash, nor more than one and five-tenths per cent (1.5%) of ash insoluble in hydrochloric acid.

34. *Ground mustard* is the powder made from mustard seed, and conforms to the standards for mustard seed.

35. *Mustard flour* is the powder made from mustard seed with the hulls largely removed and with or without the removal of a portion of the fixed oil. It contains not more than one and five-tenths per cent (1.5%) of starch, nor more than six per cent (6%) of total ash.

36. *Prepared mustard, German mustard, French mustard, mustard paste*, is a paste composed of a mixture of ground mustard or mustard flour, with salt, a vinegar, and with or without spices or other condiments which do not simulate the color of yellow ground mustard. Calculated free from water, fat, and salt, it contains not more than

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[1] U. S. Dept. Agr., S. R. A., Chemistry, **20:** (1917), 59.

twenty-four per cent (24%) of carbohydrates (calculated as starch), not more than twelve per cent (12%) of crude fiber, nor less than five and six-tenths per cent (5.6%) of nitrogen derived solely from the materials herein named.

37. *Nutmeg* is the dried seed of *Myristica fragrans* Houtt., deprived of its testa, with or without a thin coating of lime (CaO). It contains not less than twenty-five per cent (25%) of nonvolatile ether extract, not more than ten per cent (10%) of crude fiber, not more than five per cent (5%) of total ash, nor more than five-tenths per cent (0.5%) of ash insoluble in hydrochloric acid.

38. *Macassar nutmeg, papua nutmeg, male nutmeg, long nutmeg,* is the dried seed of *Myristica argentea* Warb., deprived of its testa.

39. *Paradise seed, grains of paradise, Guinea grains, melegueta pepper,* is the seed of *Amomum melegueta* Roscoe.

40. *Parsley leaves* are the leaves of *Petroselinum sativum* Hoffm.

41. *Black pepper* is the dried immature berry of *Piper nigrum* L. It contains not less than six and seventy-five hundredths per cent (6.75%) of nonvolatile ether extract, not less than thirty per cent (30%) of starch, not more than seven per cent (7%) of total ash, nor more than one and five-tenths per cent (1.5%) of ash insoluble in hydrochloric acid.

42. *Ground black pepper* is the product made by grinding the entire berry of *Piper nigrum* L. It contains the several parts of the berry in their normal proportions.

43. *Long pepper* is the dried fruit of *Piper longum* L.

44. *White pepper* is the dried mature berry of *Piper nigrum* L., from which the outer coating, or the outer and inner coatings have been removed. It contains not less than seven per cent (7%) of nonvolatile ether extract, not less than fifty-two per cent (52%) of starch, not more than five per cent (5%) of crude fiber, not more than three and five-tenths per cent (3.5%) of total ash, nor more than three-tenths per cent (0.3%) of ash insoluble in hydrochloric acid.

45. *Saffron* is the dried stigma of *Crocus sativus* L. It contains not more than ten per cent (10%) of yellow styles and other foreign matter, not more than fourteen per cent (14%) of volatile matter when dried at 100°C., not more than six per cent (6%) of total ash, nor more than one per cent (1%) of ash insoluble in hydrochloric acid.

46. *Sage* is the dried leaf of *Salvia officinalis* L. It contains not less than one per cent (1%) of volatile ether extract, not more than twenty-five percent (25%) of crude fiber, not more than ten per cent (10%) of total ash, nor more than one per cent (1%) of ash insoluble in hydrochloric acid.

47. *Savory, summer savory,* is the dried leaf and flowering tops of *Satureja hortensis* L.

48. *Star aniseed* is the dried fruit of *Illicium verum* Hook. It contains not more than five per cent (5%) of total ash.

49. *Tarragon* is the dried leaves and flowering tops of *Artemisia dracunculus* L.

50. *Thyme* is the dried leaves and flowering tops of *Thymus vulgaris* L. It contains not more than fourteen per cent (14%) of total ash, nor more than four per cent (4%) of ash insoluble in hydrochloric acid.

The next schedule deals with milk and cream. This schedule also is in the nature of a revision and an expansion of the schedule of 1906[1]. The fundamental definition for milk is modified with respect to the mode of stating the limit of the colostral period, so that by the adoption of a

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[1] U. S. Dept. Agr., Office of the Secretary, Circ. 19: (1906), 6.

rule based chiefly upon edibility rather than upon an inelastic period, no edible milk may, in these days of milk shortage and high price, be made unsalable. There is a change with respect to the matter of a Federal milk standard the reason for which is stated in the schedule, and a corresponding change for skimmed milk. A lactic acid limit has been added for cream. A definition and standard for standardized or adjusted milk has been substituted for the former definition for blended milk. The definition for pasteurized milk and its primary products has been expanded and made more specific; also definitions for heavy cream, whipping cream, buttermilk and homogenized milk and cream, have been added, with specific standards for the first three of these items.

Doubtless the adoption of grade standards, both sanitary and chemical, for milks of various degrees of excellence may eventually solve some of the perplexities this product presents, but, in the committee's judgment, the country as a whole is not ready for the adoption of that method, though many of the centers of population doubtless are.

This schedule embodies the results of much collaborative work, extensive correspondence and a number of hearings.

MILK AND CREAM.

Definitions and standards adopted by the Joint Committee on Definitions and Standards, July 30, 1917:

1. *Milk* is the whole, fresh, clean, lacteal secretion obtained by the complete milking of one or more healthy cows, properly fed and kept, excluding that obtained within fifteen days before and five days after calving, or such longer period as may be necessary to render the milk practically colostrum-free.

The composition of milk varies so greatly that it is not practicable to fix limits which are applicable in all localities. It is, therefore, left to the State and Municipal authorities to adopt as high standards as their local production conditions may warrant.

2. *Standardized milk*, adjusted milk, is milk of which the original fat content has been changed by partial skimming, or by the addition of skimmed milk, cream, or milk rich in fat to maintain a declared percentage of milk fat. It contains not less than three and seventy-five hundredths per cent (3.75%) of milk fat.

3. *Skimmed milk* is milk from which substantially all of the milk fat has been removed.

4. *Cream, sweet cream*, is that portion of milk, rich in milk fat, which rises to the surface of milk on standing, or is separated from it by centrifugal force. It is fresh and clean. It contains not less than eighteen per cent (18%) of milk fat and not more than two-tenths per cent (0.2%) of lactic acid.

5. *Heavy cream* is cream that contains not less than forty per cent (40%) of milk fat.

6. *Whipping cream* is unpasteurized cream that contains not less than thirty per cent (30%) of milk fat.

7. *Pasteurized milk, pasteurized cream, pasteurized skimmed milk*, are milk, cream, and skimmed milk, respectively, that have been subjected to a temperature not lower

than 145 degrees Fahrenheit for not less than thirty minutes. Unless they are bottled hot, they are promptly cooled to 50 degrees Fahrenheit, or lower.

8. *Buttermilk* is the product that remains when fat is removed from milk or cream, sweet or sour, in the process of churning. It contains not less than eight and five-tenths per cent (8.5%) of milk solids not fat.

9. *Homogenized milk or homogenized cream* is milk or cream that has been mechanically treated in such a manner as to alter its physical properties with particular reference to the condition and appearance of the fat globules.

The last schedule now ready for your consideration pertains to cheese, for which the list of 1906 provided only a single, necessarily very broad and generic definition. The result of our work includes several modifications of the original generic definition. One of these modifications requires a declaration of artificial coloration except for such cheeses as are always so colored and in such manner as not to be deceptive as to quality. The schedule has presented numerous difficulties as to means of distinguishing the various types and as to nomenclature. Your committee trusts you may approve the choice of "American" instead of "domestic" or of no mark of origin at all, as the qualifying designation of place of manufacture for American-made cheeses. The alternative urged was to use the prefix "imported" for foreign-made cheese. In the judgment of the committee, the decision it has reached is best both for the American consumer and for the American exporter, and yet entirely just to the importer of foreign cheese and to the seller of home-produced goods.

The schedule represents only the more important cheese varieties, and is, in fact, a report of progress, since work upon other varieties is continuing.

The recommendations are based upon an extensive study, upon numerous consultations with dairy authorities, and upon several hearings largely attended by cheese experts, manufacturing and commercial.

CHEESE.

Definitions and standards adopted by the Joint Committee on Definitions and Standards, July 28, 1917:

1. *Cheese*[1] is the sound product made from curd obtained from whole milk, part skimmed or skimmed milk, goat's milk, or the milk of other animals, with or without added cream, by coagulating the casein with rennet, lactic acid, or other suitable enzym or acid, and with or without the further treatment of the separated curd by heat or pressure or by means of ripening ferments, special moulds, or seasoning.

A cheese bearing a varietal name indicating a special process and foreign origin, when made in America by the same process, is designated as American Camembert, American Emmenthaler, American Swiss, American Edam, or American Roquefort Cheese, as the

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[1] The name "cheese" as used without qualification in America refers to American cheese, American Cheddar cheese.

case may be, and, except for the place of manufacture, conforms to the definition and standard of the foreign cheese.

American cheeses made from cow's milk to resemble foreign varieties made from the milk of other animals, are designated in such a manner as to indicate that they are made from cow's milk.

2. *Whole milk cheese* is cheese made from whole milk.

3. *Skimmed milk cheese* is cheese made from skimmed milk.

In the case of cheese normally made from whole milk, when milk is used from which any of the fat has been removed, the approximate amount of this fat removal is stated in connection with the varietal name of the cheese; e.g., "Edam one-quarter skimmed milk", "one-half skimmed milk Edam", "three-quarter skimmed milk Edam", etc., as the case may be.

CHEESES MADE FROM WHOLE MILK.

4. *American cheese*, *American Cheddar cheese*, is the cheese made in America by the Cheddar process, from pressed curd obtained by the action of rennet on whole milk. It contains not more than thirty-nine per cent (39%) of water, and, in the water-free substance, not less than fifty per cent (50%) of milk fat.

5. *Stirred curd cheese*, *sweet curd cheese*, is the cheese made in America by a modified Cheddar process, from curd obtained by the action of rennet on whole milk, in which treatment of the curd after removal of whey yields a product of more open granular texture than American Cheddar cheese. It conforms in respect to moisture and fat content to the standard for American Cheddar cheese.

6. *American Limburger cheese* is the cheese made in America by the Limburger process, from unpressed curd obtained by the action of rennet on whole milk. The curd is ripened in damp atmosphere by special forced fermentation. It contains, in the water-free substance, not less than fifty per cent (50%) of milk fat.

7. *Brick cheese* is the quick-ripened cheese made in America by the Brick cheese process, from pressed curd obtained by the action of rennet on whole milk. It contains in the water-free substance, not less than fifty per cent (50%) of milk fat.

8. *Stilton cheese* is the cheese made in England by the Stilton process, from unpressed curd obtained by the action of rennet on whole milk, with or without added cream. The curd is ripened by special moulds which give it a peculiar blue or green color.

9. *Edam cheese* is the cheese made in Holland by the Edam process, from pressed curd obtained by the action of rennet on whole milk, and ripened by special slimy fermentation (*Bacillus viscosus*). It is commonly made in spherical form and coated with harmless red color and harmless drying oil.

CHEESES MADE FROM WHOLE MILK OR PARTLY SKIMMED MILK.

10. *Emmenthaler cheese*, *Swiss cheese*, is the cheese made in Switzerland by the Emmenthaler process, from pressed curd obtained by the action of rennet on whole milk or partly skimmed milk, and ripened by special gas producing bacteria, causing characteristic "eyes" or holes. It contains, in the water-free substance, not less than forty-five per cent (45.0%) of milk fat.

OTHER SCHEDULES IN PREPARATION.

The committee now has in course of preparation definitions and standards for the following products:

Fruit juices
Vinegars
Ice cream
Butter
Oleomargarine
Cereal products (flours, meals, etc.)
Bread
Canned vegetables
Jams, jellies, marmalades
Dried and frozen eggs
Meat extracts
Tomato pastes

Respectfully submitted,

WILLIAM FREAR,
JOHN PHILLIPS STREET,
JULIUS HORTVET,
Committee to Cooperate with Other Committees on Food Definitions.

Adopted.

REPORT OF COMMITTEE ON VEGETATION TESTS ON THE AVAILABILITY OF PHOSPHORIC ACID IN BASIC SLAG[1].

Your committee desires to submit another report of progress. It is hoped that the unfinished field experiments being carried on by those cooperating with your committee may be sufficiently complete by another year for the committee to be in position to make its final report on both the field and pot culture work. It would seem to your committee that in the meantime the referee on phosphoric acid, following last year's recommendations[2], should carry on sufficient analytical work to be in position, if possible, to recommend some method or methods which will be suitable for discriminating between high grade slags and those slags which may be adulterated or are products of processes possibly yielding inferior grades of slag.

As was stated in our report of last year[3], the results from the vegetation experiments so far obtained are available to the referee upon request. It is felt by your committee that these results will be of considerable value to the referee on phosphoric acid in forming an estimate of the availability of the phosphoric acid contained in true Thomas phosphate slags.

During the year, three series of pot culture experiments have been reported from the Massachusetts Agricultural Experiment Station. The final results from their field experiments have been withheld until a later date.

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[1] Presented by B. L. Hartwell.
[2] *J. Assoc. Official Agr. Chemists*, 1920, **3**: 519.
[3] *Ibid.*, 585.

The Illinois Agricultural Experiment Station has reported on four series of pot experiments in which rape, soy beans, wheat and clover were used.

The North Carolina Agricultural Experiment Station has also reported the results from its pot culture work but the results from the field experiments were withheld until those from the growing of one or two more crops on the plats had been secured.

C. B. Williams, H. D. Haskins,
B. L. Hartwell, C. G. Hopkins,
J. A. Bizzell,
Committee on Vegetation Tests on the Availability of Phosphoric Acid in Basic Slag.

Adopted.

REPORT OF COMMITTEE ON METHODS OF SAMPLING FERTILIZERS TO COOPERATE WITH A SIMILAR COMMITTEE OF THE AMERICAN CHEMICAL SOCIETY[1].

The committee appointed at the last meeting of the association to cooperate with a similar committee representing the American Chemical Society, to formulate methods for the accurate sampling of fertilizers, desires at this time simply to report progress and to suggest the features that seem essential in governing sampling methods in general. The personnel of both committees has been seriously depleted by the regretted death of one of our members, W. J. Jones, jr., who was a recognized authority on the subject and to whom we looked for much authentic data.

Under the supervision of F. S. Lodge, Chairman of the American Chemical Society Committee, two samples each comprising a ton of fertilizer, were prepared and carefully sampled by properly mixing, quartering, etc. The mixtures were then bagged and officially sampled by O. S. Roberts of Indiana, using the Indiana, the lard tryer, and the Massachusetts types of samplers. The bagged goods were then shipped from Chicago to Jeffersonville, Indiana, and to Atlanta, Georgia, respectively. As arranged by the late W. J. Jones, jr., his chief inspector, O. S. Roberts, has again taken samples from these goods, after arrival at their destinations, using the previously mentioned types of samplers. These samples will be submitted to the members of the two committees

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[1] Presented by C. H. Jones.

for analysis. It is hoped that a full report, based on the analytical data obtained, can be prepared for our next meeting.

The need of some approach to uniformity in the methods used in sampling fertilizers has long been recognized. During the year, the entire subject has been carefully reviewed by J. C. Brunnich representing the Department of Agriculture and Stock of Brisbane, Queensland. His report and the action taken thereon, April 20, 1917, has had our careful attention.

Your committee wishes to direct the attention of those interested to three features connected with the sampling of fertilizers:

1. *Types of samplers.*—Use a sampler that removes a core from the bag from top to bottom. The so-called Indiana type has proved very satisfactory.

2. *Amount of sample secured.*—Most State laws require a certain percentage of the stock to be sampled but do not specify necessarily the amount to be sent to headquarters by the sampling agent. It is suggested that at least a pound should constitute each official sample and that it be secured by thoroughly mixing, and then halving or quartering the entire amount taken from the bags.

3. *Care in subsampling in the laboratory.*—It is suggested that the entire sample submitted to the chemist be passed through a 10 mesh sieve, thoroughly mixed and then subdivided by halving or quartering. The portion thus secured is then reduced to a suitable fineness for analysis. It has been found desirable to grind such samples as carry 4 per cent or more of ammonia sufficiently fine to pass through a sieve having circular perforations $\frac{1}{2}$ mm. in diameter.

RECOMMENDATIONS.

It is recommended—

(1) That the work begun this year be continued.

  (a) That further study be made of the type of sampler to be used in securing samples.

  (b) That further study be made of the desirability of using a sieve having circular perforations $\frac{1}{2}$ mm. in diameter in place of the 1 mm. sieve now employed in the final preparations of the sample.

(2) That a third member be appointed on our committee.

(3) That at least a pound of the material should constitute each official sample sent to headquarters.

(4) That the entire sample submitted to the chemist be passed through a 10 mesh sieve previous to its subdivision for analysis.

Respectfully submitted,

C. H. Jones,
B. F. Robertson,
Committee on Methods of Sampling Fertilizers to Cooperate with a Similar Committee of the American Chemical Society.

Adopted.

REPORT OF COMMITTEE ON THE REVISION OF METHODS OF SOIL ANALYSIS[1].

The gigantic strides which have been taken by soil chemists in recent years in their progress on soil studies and on those bearing on the relation of soils to plants, have rendered necessary the revision, very materially, not only of the actual methods of analysis which have been in vogue in the past, but also our points of view and conceptions with reference to the value and validity of the procedure of soil analysis itself. This committee in its work has, therefore, tried to bear in mind the important advances referred to, and to revise the methods of soil analysis as much as possible in accordance with them. It must be obvious to all chemists, and particularly to all soil chemists, that the difficulties in the path of making a perfect revision are very numerous, and in some cases almost insuperable. Your committee has, however, done the best that it could in the light of the knowledge at hand, and submits the following methods for your consideration with the recommendation that they be adopted as the tentative methods of this association for the analysis of soils.

SOILS.—TENTATIVE.

1 DIRECTIONS FOR SAMPLING.

Remove from the surface all vegetable material not incorporated with the soil. Take a sufficient number of samples to insure securing a composite sample representative of the tract samples to a depth which will include the average depth of the plowed soil, usually about 7 inches, and a composite sample from each important and distinctly different soil stratum to the depth of 40 inches, using a soil tube or auger, whichever may be best adapted to the soil conditions. If a soil auger is used, before boring deeper the hole should be enlarged and carefully cleaned out with the soil auger to prevent contamination of the several substrata samples while being withdrawn. The sampling should be done when the soil is reasonably dry. Mix the samples of each depth thoroughly and dry in a well-aired, cool place.

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[1] Presented by C. B. Lipman.

It is recommended that the weight of a given volume of the soil as it lies in the field be taken for calculating the percentage results obtained by analysis to pounds per given area of the soil.

NOTE.—In view of the variability characteristic of field soils within small distances, it seems impossible at the present time to devise a perfect method for sampling. The method given above comes as near as any, that may now be adopted, to filling the requirements.

2

PREPARATION OF SAMPLE.

(**a**) Pulverize the air-dried soil, using porcelain pebble mill or other equally effective method which will not reduce the rock fragments, to pass through a sieve having circular openings 1 mm. in diameter. Thoroughly mix the sifted material and preserve in a suitable stoppered container. Weigh and discard the detritus.

(**b**) If necessary for the determination of total constituents, pulverize more finely a subsample of (**a**).

If, for any reason, deviations from this procedure are deemed necessary, they should be reported with the results.

3

MOISTURE.

Dry 2 grams of the sample in a wide-mouthed weighing bottle at 100°C. to constant weight. Report the loss of weight as percentage of the moisture of the water-free soil.

4

VOLATILE MATTER.

Ignite the soil from **3** in a platinum dish or suitable substitute to full redness, stirring occasionally until the organic matter is destroyed. If the soil contains appreciable quantities of carbonates, moisten it after cooling with a few drops of a saturated solution of ammonium carbonate; dry and heat to dull redness to expel the ammonium salts; cool in a desiccator and weigh.

Chemists are cautioned that this method gives only a crude approximation of the organic matter and is less accurate in soils containing much colloidal material.

TOTAL ORGANIC CARBON.

5

Sodium Peroxid Combustion Method.

Thoroughly mix 2 grams of soil (1 gram of soil high in organic matter), 0.75 gram of magnesium powder, and 10 grams of sodium peroxid, in a closed dry calorimeter bomb, by shaking the bomb back and forth. Explode the charge by means of electricity or by dropping a red hot plug into the bomb through a valve which closes automatically as soon as the plug enters. Remove the fused charge from the bomb, using as little hot water as possible, heat to boiling, and transfer to a receiving funnel of Parr's apparatus for total carbon[1]. From the acid funnel run 50 cc. of sulphuric acid (1 to 2) into a 150 cc. Erlenmeyer flask. Connect the apparatus and slowly add the contents from the receiving funnel. (The carbon dioxid generated passes through the capillary tube into the graduated burette). Heat the contents of the flask to boiling, then fill the flask with water from the receiving funnel to force the gases into the graduated burette, noting the temperature and pressure and the reading on the burette. Pass the gas into an ordinary absorption pipette containing a 30% potassium hydroxid solution. Shake the gas with the solution until carbon dioxid ceases to be absorbed. Return the residual gas to the graduated burette and again read the burette, noting the temperature and

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[1] *J. Am. Chem. Soc.* 1904; **26**: 294.

pressure. The difference in readings, calculated to uniform conditions of temperature and pressure, gives the number of cc. of carbon dioxid equivalent to the carbon in the sample.

Determine the carbonates as directed under **7** and subtract the carbon in the same from the total to obtain the organic carbon.

6 WET COMBUSTION METHOD.

Introduce 1–5 gram charges of soil, depending upon the organic matter content, into three 100 cc. Pyrex Erlenmeyer flasks[1]. Free the apparatus of atmospheric carbon dioxid, then introduce into each absorption tower 25 or 50 cc. of N/2 sodium hydroxid. Apply suction of 5 inches and run into each Erlenmeyer flask 10 cc. of the oxidizing mixture made as follows: Chromic anhydrid, 85 grams, dissolved in 100 cc. of water and diluted to 250 cc. with 85% phosphoric acid. Then add 25–40 cc. of a mixture consisting of equal parts of 85% phosphoric acid and concentrated sulphuric acid. Gently agitate the flasks and place a low flame under each. Continue the gentle agitation and heating for 30 minutes subsequent to attaining the boiling point. Between the Erlenmeyer flasks and the absorption towers, insert a glass bulb of about 1½ inches diameter, and bend the tube leading from this bulb into the Erlenmeyer flask so as to permit the return of the condensed water along the side of the Erlenmeyer flask. At the end of the agitation and aspiration, release the suction and wash out the absorbent into a 500 cc. flask. Then precipitate the sodium carbonate by means of an excess of a concentrated barium chlorid solution. Filter the supernatant liquid through a Büchner funnel by suction and wash the mass of the carbonate by a couple of decantations upon the filter as directed under **7.** Titration of the residual hydrate may then be made with N/10 acid, using phenolphthalein. Subtract the amount of carbon dioxid as determined under **7,** from the total. The difference represents the carbon dioxid derived from the oxidation of the organic carbon.

7 CARBONATE CARBON.

Pulverize the sample to pass a 100 mesh sieve, so as to expose to the action of the liberating acid as much as possible of any calcite which may be included in the quartz crystals. For soils low in carbonates, use 10, 25 or 50 gram charges in the quadruplicate shaking device[1]. For soils sufficiently high in carbonate to justify 2 or 5 gram charges, the single agitating device[2] may be used.

Introduce the charge into a 300 cc. Erlenmeyer flask; aspirate 5 minutes in order to free the apparatus of atmospheric carbon dioxid, release the suction and introduce 25 cc. of N/10 barium hydroxid into an elongated Camp absorption tower containing at its base an inverted test tube 2½ inches long. Place upon this tube alternating columns of ⅛ inch glass rods 2½ inches long and medium size glass beads in pockets 1½ inches deep. The absorption towers should be at least 25 inches long. The Erlenmeyer suction flask and tower[2] may also be used. Apply suction of 5 inches and then introduce 60 cc. of hydrochloric acid (1 to 10) upon the soil contained in the Erlenmeyer flask, regulating the intake of air by means of a screw cock placed just beyond the absorption tower. Aspirate for 30 minutes. Then release the suction and draw off the absorbent into a 500 cc. flask, washing the tower with a succession of fillings of carbon dioxid-free water, using a minimum volume of 250 cc. If the barium carbonate precipitate be light, the titration of residual hydrate may be made in the presence of the precipitate. If the barium carbonate precipitate be heavy, filter by suction through a 10 cm. Büchner

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[1] *J. Ind. Eng. Chem.*, 1915, **7**: 227.
[2] *Ibid.*, 1916, **8**: 341.

funnel into a large beaker placed under a bell jar. Add phenolphthalein to the filtrate and titrate the residual hydrate with N/20 acid. Sodium or potassium hydrate may be utilized in lieu of the barium hydrate, in which case the carbonate should be precipitated by means of a neutral solution of barium chlorid.

If the soil is known to be derived from the limited magnesite area, or if a soil or subsoil be from the glaciated region, where transported dolomite occurs in considerable amounts, the agitation and aspiration should be repeated or continued until the disintegration is completed, if necessary using hydrochloric acid (1 to 5).

TOTAL NITROGEN.

8

Gunning-Hibbard Method.

Digest 10 grams of soil in a 500 cc. Kjeldahl flask with 30–40 cc. of sulphuric acid and approximately 10 grams of salt mixture composed as follows: 10 parts of potassium sulphate or anhydrous sodium sulphate, 1 part of ferrous sulphate, $\frac{1}{2}$ part of copper sulphate. Continue the digestion until the mixture is colorless or nearly so. After cooling the mixture, dilute the contents of the flask with water as needed; add sufficient sodium hydroxid to neutralize the acid and distil into standard acid. Distil from the digestion flask, or, if preferred, transfer to copper flasks. Collect 150 cc. of the distillate and titrate the excess of acid with standard alkali, an indicator being used whose reactions are best gauged by the operator. It is suggested that N/10 or N/14 standard solutions be used for convenience.

9

Kjeldahl Method.

Proceed as directed under **8** with regard to the amount of soil and amount of acid, but instead of the salt mixture, add 0.7 gram of mercuric oxid or 0.65 gram of mercury. Mix immediately, heat over a slow flame, gradually increasing the heat. Continue the digestion until the mixture is colorless or nearly so. Oxidize the residue with potassium permanganate, adding a crystal or two at a time directly to the hot liquid, and shake until the liquid assumes a permanent green or purple color. After cooling the solution, dilute the contents of the flask as directed under **6**; then add 25 cc. of a potassium sulphid solution (40 grams of potassium sulphid in 1 liter of water) and an excess of alkali, after which proceed with the distillation and titration as directed under **8.**

10

SODIUM CARBONATE FUSION OF THE SOIL.

Thoroughly mix on glazed paper 1 gram of soil, ground to an impalpable powder, with 5 grams of sodium carbonate. Transfer carefully to a 40 cc. platinum crucible. Cover, heat at low redness until fusion begins, then increase the heat until a clear, quiet fusion results. Finally, give full heat of a Méker burner for 10 minutes, having the flame oblique to insure good oxidation. Pour into a large platinum dish set in water. Place the crucible and cover in a wide 200 cc. beaker. Cover with water, drop the infused lump in a platinum dish, and wash the contents of the dish into the beaker with dilute hydrochloric acid.

Add 15 cc. of concentrated hydrochloric acid to the contents of the beaker, cover, and place upon a steam bath until the fused mass has disintegrated (1–3 hours). Transfer the mixture to the platinum dish and evaporate to dryness on steam bath.

11

DETERMINATION OF SILICA.

Filter the mixture obtained as directed under **10** (a 9 cm. Büchner funnel with suction is very effective). Wash with hot water containing 1–2 cc. of hydrochloric acid per liter. Return the filtrate to a dish, preferably a casserole, and dehydrate on the steam bath until the silica assumes a crystalline appearance. Moisten with hydrochloric acid and repeat the dehydration. Add 5 cc. of concentrated hydrochloric acid and 100 cc. of hot water. Mix thoroughly and filter. Add the residue to the main portion of silica obtained from the first filtration. Make up the filtrate to 500 cc. and save for subsequent determinations. Place the two silica residues with filters in a porcelain crucible. Ignite slowly at first to burn off the filter paper and then with a strong flame, preferably a blast lamp, to constant weight.

12 FERRIC, ALUMINIUM AND TITANIUM OXIDS AND PHOSPHORUS PENTOXID.

To an aliquot of the solution from **10** (50 or 100 cc., according to the probable amount of iron present) add ammonium hydroxid, drop by drop, until the precipitate formed requires several seconds to dissolve, thus leaving the solution faintly acid. Heat nearly to the boiling point and add sufficient ammonium hydroxid to precipitate all of the iron, alumina, etc. Allow to boil in a covered beaker for about 1 minute, remove, and if no ammonia is given off (as detected by smelling) add more, drop by drop, until it can be detected. Do not allow the precipitate to settle, but stir and pour on the filter. Wash immediately with hot water containing ammonium nitrate, using a fine jet which is played around the edge of the precipitate, thus cutting it free from the paper in order to produce rapid filtration. Wash the precipitate several times, return it to the original beaker, dissolve with a few drops of hydrochloric acid and warm. Reprecipitate the iron, alumina and phosphoric acid with ammonium hydroxid as above, and wash until free from chlorids.

Dry the precipitate, remove it from the filter, and ignite over a Bunsen flame, the filter being incinerated separately and the residue added to the precipitate. Then ignite to bright redness, cool in a desiccator and weigh as ferric oxid (Fe_2O_3), aluminium oxid (Al_2O_3), titanium oxid (TiO_2), and phosphorus pentoxid (P_2O_5). Transfer this residue to a flask and digest with several cc. of sulphuric acid (1 to 4), heating to accelerate solution. When solution is complete, reduce with zinc and estimate ferric oxid with a standard solution of permanganate, as directed under "Inorganic Plant Constituents[1]."

Or, in lieu of the above, evaporate 50 or 100 cc. of the solution from **10** with the addition of 10 cc. of sulphuric acid until all hydrochloric acid is expelled, dilute with water, reduce with zinc and determine ferric oxid with a standard solution of permanganate as directed under "Inorganic Plant Constituents[1]."

Subtracting the ferric oxid, together with the phosphorus pentoxid (to be determined later) from the collective weights of ferric oxid and phosphorus pentoxid, gives aluminium and titanium oxids. For extreme accuracy consult "The Analysis of Silicate and Carbonate Rocks" by W. F. Hillebrand[2].

MANGANESE.

13 ELIMINATION.

Concentrate the solution from **12** to about 50 cc., cool, add ammonium sulphid, filter and wash with hot water. Use the filtrate in **15**.

14 DETERMINATION.

If it is desired to determine manganese quantitatively proceed as follows: Volatilize 1 gram of the original soil with hydrofluoric and sulphuric acids, ignite, fuse with potassium pyrosulphate, dissolve in water and nitric acid. Evaporate to dryness, again dis-

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 30.

[2] U. S. Geol. Surv. Bull. 422: (1910).

solve in water, add 25 cc. of nitric acid (1 to 3), add sodium bismuthate and complete as in the regular bismuthate method for manganese as given under "Waters[1]."

15 CALCIUM.

Evaporate the solution from **13** to about 50 cc., make slightly alkaline with ammonium hydroxid and add, while still hot, ammonium oxalate solution, drop by drop, so long as any precipitate is produced, adding a few cc. in excess to convert the magnesium also into oxalate. Heat to boiling, allow to stand for 3 hours or longer, decant the clear solution on the filter, pour 15–20 cc. of hot water on the precipitate and again decant the clear solution on the filter. Dissolve the precipitate in the beaker with a few drops of hydrochloric acid, add a little water, and reprecipitate, boiling hot, by adding ammonium hydroxid and a little ammonium oxalate solution. Allow to stand as before and filter through the same filter. Wash free from chlorids with hot water. From this point one of three methods may be used for determining calcium:

(1) Ignite the precipitate and determine the calcium as calcium oxid.

(2) Mix the oxalate precipitate, after incineration of the filter, with finely pulverized and dried ammonium sulphate and drive off the excess of the sulphate by carefully heating the upper portion of the crucible. Determine the calcium as calcium sulphate.

(3) Dissolve the calcium oxalate precipitate in dilute sulphuric acid, wash directly into the beaker and titrate while hot with a standard solution of potassium permanganate.

16 MAGNESIUM.

Evaporate the solution from **15** on the water bath to about 100 cc., add cautiously 20 cc. of concentrated nitric acid. Cover the beaker and evaporate to dryness on the hot plate to remove the ammonium salts. Add 5 cc. of hydrochloric acid and evaporate nearly to dryness. Dissolve the residue in hot water and a small amount of hydrochloric acid. If necessary, filter the solution and wash the filter paper with about 100 cc. of hot water. Precipitate the magnesium as magnesium ammonium phosphate by the addition of 3 cc. of a 10% ammonium phosphate solution and sufficient ammonium hydroxid to make the solution slightly alkaline. Stir the solution vigorously. Allow to stand 15 minutes; add 15 cc. of ammonium hydroxid and allow the precipitation to proceed overnight. Filter, wash the precipitate with 2% ammonium hydroxid solution, place in a porcelain crucible or filter in a platinum Gooch. Ignite and determine as magnesium pyrophosphate ($Mg_2P_2O_7$).

17 SULPHUR.

Evaporate 100 or 200 cc. of the solution from the original fusion nearly to dryness on a water bath to expel the excess of acid; then add 400 cc. of water; heat to boiling and add, drop by drop, a 10% hot barium chlorid solution until no further precipitation occurs. Continue near the boiling point for about 5 minutes; allow to stand for 5 hours or longer in a warm place, pour the liquid on a tared Gooch or on an ashless filter, treat the precipitate with 15–20 cc. of boiling water, transfer to the filter, and wash free from chlorids with boiling water. Dry the filter, ignite over a Bunsen burner, and weigh as barium sulphate.

For the determination of sulphur as sulphates, agitate the soil with water for 7 hours, proceeding otherwise as directed under **24.**

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[1] *Assoc. Official Agr. Chemists, Methods,* 1916, 47.

PHOSPHORUS.

18

Sodium Peroxid Method.

Place 10 grams of sodium peroxid in an iron or porcelain crucible and thoroughly mix with it 5 grams of the soil. If the soil has very little organic matter, add a little starch to hasten the action. Heat the mixture carefully by applying the flame of a Bunsen burner directly to the surface of the charge until the action starts. Cover the crucible until reaction is over and keep at a low red heat for 15 minutes. Do not allow fusion to take place. By means of a large funnel and a stream of hot water, transfer the charge to a 500 cc. volumetric flask, acidify with hydrochloric acid, and boil. Cool and make up to the mark. If the action has taken place properly, there should be no particles of undecomposed soil in the bottom of the flask. Allow the silica to settle and draw off 200 cc. of the clear solution.

Precipitate the iron, aluminium, and phosphorus with ammonium hydroxid; filter, wash several times with hot water, return the precipitate to the beaker, and dissolve the precipitate in hot hydrochloric acid, pouring the acid upon the filter to dissolve any precipitate remaining. Evaporate the solution and washings to complete dryness on a water bath. Take up with dilute hydrochloric acid, heating if necessary, and remove the silica by filtration. Evaporate the filtrate and washings to about 10 cc., add 2 cc. of concentrated nitric acid, and neutralize with ammonium hydroxid. Add nitric, acid until the solution is clear, avoiding an excess. Heat at 40–50°C. on a water bath add, 15 cc. of molybdate solution, keeping at this temperature for 1–2 hours. Let stand overnight, filter, and wash free of acid with 0.1% ammonium nitrate solution, and finally, once or twice with cold water. Transfer the filter to a beaker, and dissolve in standard potassium hydroxid (1 cc. = 0.2 mg. of phosphorus), titrate the excess of potassium hydroxid with standard nitric acid, using phenolphthalein as an indicator.

19

Volumetric Method.

Proceed as in the sodium peroxid method and then complete the determination as follows:

Allow to stand for 3 hours at a temperature not above 60°C., filter on a small filter paper or on a Gooch crucible and wash with cold water until two fillings of the filter do not greatly diminish the color produced with phenolphthalein by 1 drop of standard alkali. Return the filter and precipitate to the same beaker used for precipitating the phosphomolybdate, dissolve the yellow precipitate in standard sodium or potassium hydroxid, add a few drops of phenolphthalein solution and titrate the excess of alkali with standard acid; 1 cc. of the standard alkali should be made to equal 0.0005 gram of phosphorous pentoxid (P_2O_5).

20

Magnesium Nitrate Method.

Place 5 grams of soil in a porcelain dish. Moisten with 5–7 cc. of magnesium nitrate solution[1]. Dry on the water bath and burn off the organic matter at low redness. Cool, moisten slightly with water, add 10 cc. of concentrated hydrochloric acid, and digest for 2 hours on the water bath, keeping the dish covered with a watch glass and stirring 2 or 3 times during digestion. Make up to 250 cc., mix well, and transfer to a dry folded filter, pouring back on the filter until the solution runs through clear. Make the determination on aliquots corresponding to 2 or 4 grams of the soil, depending upon the amount of phosphorus present. Dry, take up with hydrochloric acid and water and filter, the filtrate and washings not exceeding 30–40 cc. Make alkaline with ammonium

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 2.

hydroxid, and dissolve the precipitate with concentrated nitric acid, using a slight excess. Add gradually, while shaking, 5–15 cc. of molybdate solution[1], shaking thoroughly. Keep the solution at 40–50°C. for 1 hour, let stand overnight at room temperature, filter, and wash well with cold water. Return the filter and the precipitate to the same flask and determine phosphorus volumetrically, using standard potassium hydroxid and nitric acid

21

POTASSIUM AND SODIUM.

Fuse 0.5 or 1 gram of soil (ground to impalpable powder) according to the J. Lawrence Smith method for total alkalies[2]. Transfer the fused mass to a porcelain dish, slake thoroughly with hot water, and grind finely with an agate pestle. After washing five times by decantation with hot water, transfer to a filter and wash well, 300 cc. of wash water being sufficient. To the filtrate add 10 cc. of concentrated hydrochloric acid, and evaporate as nearly as possible to dryness. Take up with hot water and 2 cc. of hydrochloric acid, filter with suction through a small filtering medium into a 150 cc. Jena beaker, to remove the material which would render final filtration extremely slow. If the determination of sodium is desired, evaporate to dryness in platinum and determine sodium by difference from the mixed chlorids. Concentrate the solution to 30 cc., add 1.5 cc. of a platinic chlorid solution (10 cc. containing 1 gram of platinum), evaporate to a sirupy consistency, and add 15 cc. of acidulated alcohol (to 2 liters of 95% alcohol add 152 cc. of hydrochloric acid, sp. gr. 1.20, and pass hydrochloric acid gas into solution to make it 2.25 N). Filter through a Gooch crucible, using suction, wash with 80% alcohol, then with ammonium chlorid solution, saturated with potassium platinic chlorid, and finally with 80% alcohol. Dry for 1 hour at 120°C. to remove alcohol, and weigh. Dissolve the precipitate with hot water, using suction, dry and weigh again.

QUALITATIVE TEST FOR SOIL REACTION.

22

Litmus Paper.

Place approximately 50 grams of air-dry soil in a small porcelain evaporating dish freshly rinsed with water. Add water and mix to a thick paste. By means of forceps, lay upon this a strip of the best grade of litmus paper. Press firmly against the soil by means of a clean spatula or glass rod, keeping the upper surface of the paper free from soil. Protect and permit to stand for 30 minutes, then note the color of the litmus paper.

23

DETERMINATION OF NITRATE NITROGEN.

Place 100 grams of air-dry soil in a mortar or porcelain dish, add, with constant stirring, 1 gram of lime, and 200 cc. of water. Allow to settle for 10–20 minutes. Filter directly through an ordinary filter paper and obtain a clear solution. Evaporate 25 cc. of the filtrate on the water bath. Cool, and add 2 cc. of phenoldisulphonic acid. Triturate thoroughly with a clean glass rod, add 25 cc. of water and strong ammonium hydroxid or potassium hydroxid, drop by drop, with constant stirring, until a permanent yellow color is obtained. Compare the solution in a colorimeter with a standard solution prepared in a similar manner to the unknown[3].

In the cases of solutions containing more than 6 parts per million of chlorin, those containing sulphates, or those which are deeply colored by organic matter, one of the reduction methods should be employed for making the nitrate determination[4].

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 1.
[2] *Ibid.*, 27.
[3] *Ibid.*, 37.
[4] *Ibid.*, 38.

24 DETERMINATION OF ALKALI SALTS.

To 100 grams of soil in a 500 cc. bottle, add 250 cc. of water. Stopper, shake thoroughly and allow to stand overnight. Filter through a Pasteur-Chamberland filter. Evaporate 50 cc. of the filtrate in a platinum dish on the steam bath to dryness. Ignite at a low red heat to drive off organic matter. Cool in a desiccator and weigh for total salts. Then dissolve the residue in a platinum dish in 10–15 cc. of hot water. Make up to 40 cc. in a graduated cylinder. Take 10 cc. and titrate with N/10 silver nitrate for chlorids. Take 10 cc. and titrate with N/10 hydrochloric acid for sodium carbonate. Determine sulphates by difference. When much gypsum is present, the solution of the salts in hot water must be filtered through a small filter, the gypsum weighed separately, and subtracted from the total amount of sulphates.

Respectfully submitted,

C. B. Lipman, E. C. Shorey,
W. H. McIntire, A. W. Blair,
R. Stewart,
Committee on the Revision of Methods of Soil Analysis.

Adopted. Methods adopted as the tentative methods of the association for the analysis of soils.

It was moved, seconded, and adopted that the N/5 acid digestion of soils be deleted.

REPORT OF COMMITTEE ON RESOLUTIONS[1].

Resolved, That this association record its deep regret at the loss of its esteemed fellow member, R. E. Stallings, who died July 11, 1917. Mr. Stallings has held the office of State Chemist of Georgia for the past ten years and has been a regular attendant at the meetings of this association since 1909. He was active and effective in regulatory work and has made his influence felt in his chosen field.

Resolved, That this testimonial be spread upon the records of the association and that the secretary be instructed to furnish a copy to his immediate family.

It is with great regret that we record the loss of one of our active members, W. J. Jones, jr., of La Fayette, Indiana, whose death occurred at his home August 31, 1917. As State Chemist of Indiana, Mr. Jones was distinguished by his creative work, and devoted years to the organization of efficient fertilizer and feeding stuff control. He was also an

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[1] Presented by A. S. Mitchell.

investigator in analytical research and made valuable contributions to our knowledge of methods relating to plant foods. His cordial manner endeared him to all with whom he came in contact.

Resolved, That it is the sense of the Association of Official Agricultural chemists that the death of Mr. Jones is a distinct loss to agricultural science and especially to workers concerned with legislation for the public welfare.

Resolved, further, That the secretary be instructed to send a copy of this resolution to Mrs. Jones.

Resolved, That the thanks of the association be tendered to the management of the New Willard Hotel for the use of the Assembly Room and for the many courtesies extended to the association and its members.

Resolved, That it is the sense of the association that much of the success of the meeting is due to the painstaking care and efficient work of Miss Nellie A. Parkinson, not only at the time of the meeting but throughout the year.

Resolved, That a vote of thanks be extended to the retiring president, J. K. Haywood, for the able and impartial manner in which he has presided over this session.

Respectfully submitted,

A. S. MITCHELL,
C. H. JONES,
W. D. BIGELOW,
Committee on Resolutions.

Adopted.

It was moved, seconded and adopted that the place and time of the next meeting be referred to the executive committee with power to act.

The convention adjourned.

PROCEEDINGS OF THE THIRTY-FIFTH[1] ANNUAL CONVENTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, 1919.

OFFICERS, COMMITTEES, REFEREES, AND ASSOCIATE REFEREES OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, FOR THE YEAR ENDING NOVEMBER, 1920.

Honorary President.

H. W. **Wiley**, Woodward Building, Washington, D. C.

President.

H. C. **Lythgoe**, State Department of Health, Boston, Mass.

Vice-President.

W. F. **Hand**, Agricultural College, Agricultural College, Miss.

Secretary-Treasurer.

C. L. **Alsberg**, Box 744, Eleventh Street Station, Washington, D. C.

Additional Members of the Executive Committee.

C. H. **Jones**, Agricultural Experiment Station, Burlington, Vt.
W. W. **Skinner**, Bureau of Chemistry, Washington, D. C.

PERMANENT COMMITTEES.

Cooperation with Other Committees on Food Definitions.

William Frear (State College, Pa.), *Chairman.*
Julius Hortvet, St. Paul, Minn.
C. D. Howard, Concord, N. H.

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[1] No regular meeting was held in 1918 because of war conditions. The Executive Committee, however, held a meeting in January, 1919, and planned the work for the year ending November, 1919.

Recommendations of Referees.

(Figures in parentheses refer to year in which appointment expires.)

B. B. Ross (Auburn, Ala.), *Chairman.*

SUBCOMMITTEE A: B. B. Ross (1920), (Polytechnic Institute, Auburn, Ala.), *Chairman*, C. C. McDonnell (1922), W. H. McIntire (1924). [Fertilizers (borax in fertilizers, preparation of ammonium citrate, nitrogen, potash), potash availability, inorganic plant constituents (sulphur and phosphorus in the seeds of plants, calcium and magnesium in the ash of seed), water, tanning materials and leather, insecticides and fungicides, and soils (sulphur in soils).]

SUBCOMMITTEE B: H. C. Lythgoe (1920), (State Department of Health, Boston, Mass.), *Chairman*, C. A. Browne (1922), E. M. Bailey (1924). [Foods and feeding stuffs (crude fiber, stock feed adulteration), saccharine products (honey, maple products, maltose products, sugar house products), dairy products (moisture in cheese), fats and oils, baking powder, drugs (alkaloids, arsenicals, synthetic drugs, alkaloids of opium, medicinal plants, enzyms), testing chemical reagents, soft drinks, and eggs and egg products.]

SUBCOMMITTEE C: R. E. Doolittle (1920), (Transportation Building, Chicago, Ill.), *Chairman*, W. W. Randall (1922), W. D. Collins (1924). [Food preservatives (saccharin), coloring matters (oil soluble colors), metals in foods, pectin in fruits and fruit products, canned foods (physical methods of examination, tomato products), cereal foods, wines, distilled liquors, limit of accuracy in the determination of small amounts of alcohol in beers, methods of analysis of near beers, vinegars, flavoring extracts, meats and meat products (separation of meat proteins, decomposition of meat products, gelatin), spices, determination of shells in cacao products, method for the examination of cacao butter, coffee, and tea.]

Board of Editors.

C. L. Alsberg (Box 744, Eleventh Street Station, Washington, D. C.), *Chairman.*

L. L. Van Slyke (1920).	C. B. Lipman (1922).
E. F. Ladd (1921).	R. E. Doolittle (1923).

Editing Methods of Analysis.

R. E. Doolittle (Transportation Building, Chicago, Ill.), *Chairman.*

B. B. Ross.	J. W. Sale.
A. J. Patten.	G. W. Hoover.

W. H. McIntire.

SPECIAL COMMITTEES.

Vegetation Tests on the Availability of Phosphoric Acid in Basic Slag.

C. B. Williams (College of Agriculture and Mechanic Arts,
West Raleigh, N. C.), *Chairman.*

W. B. Ellett.	H. D. Haskins.
B. L. Hartwell.	J. A. Bizzell.

Committee on Methods of Sampling Fertilizers to Cooperate with a Similar Committee of the American Chemical Society.

C. H. Jones (Agricultural Experiment Station, Burlington, Vt.), *Chairman.*
E. G. Proulx. B. F. Robertson.

Committee on Revision of Methods of Soil Analysis.

C. B. Lipman (University of California, Berkeley, Calif.), *Chairman.*
W. H. McIntire. A. W. Blair.
R. Stewart.

Committee on Quartz Plate Standardization and Normal Weight.

Frederick Bates (Bureau of Standards, Washington, D. C.), *Chairman.*
C. A. Browne. F. W. Zerban.

Referees and Associate Referees.

Fertilizers:

Referee: R. N. Brackett, Clemson College, Clemson College, S. C.

Borax in Fertilizers:

Associate referees: W. H. Ross, Bureau of Plant Industry, Washington, D.C.
R. B. Deemer, Bureau of Plant Industry, Washington, D. C.
G. F. Lipscomb, University of South Carolina, Columbia, S. C.

Preparation of ammonium citrate:

Associate referee: C. S. Robinson, Agricultural Experiment Station, E. Lansing, Mich.

Nitrogen:

Associate referee: I. K. Phelps, Bureau of Chemistry, Washington, D. C.

Potash:

Associate referee: T. E. Keitt, Agricultural Experiment Station, Experiment, Ga.

Potash availability:

Referee: A. G. McCall, Agricultural Experiment Station, College Park, Md.

Inorganic plant constituents:

Referee: J. H. Mitchell, Polytechnic Institute, Auburn, Ala.

Sulphur and phosphorus in the seeds of plants:

Associate referee: W. L. Latshaw, Agricultural Experiment Station, Manhattan, Kans.

Calcium and magnesium in the ash of seed:

Associate referee: A. J. Patten, Agricultural Experiment Station, E. Lansing, Mich.

Water:

Referee: J. W. Sale, Bureau of Chemistry, Washington, D. C.

Associate referee: J. C. Diggs, State Board of Health, Indianapolis, Ind.

Tanning materials and leather:

Referee: F. P. Veitch, Bureau of Chemistry, Washington, D. C.

Insecticides and fungicides:

Referee: J. J. T. Graham, Bureau of Chemistry, Washington, D. C.

Soils:

Referee: W. H. McIntire, Agricultural Experiment Station, Knoxville, Tenn.

Sulphur in soils:

Associate referee: (Not appointed.)

Foods and feeding stuffs:

Referee: G. L. Bidwell, Bureau of Chemistry, Washington, D. C.

Crude fiber:

Associate referee: G. L. Bidwell, Bureau of Chemistry, Washington, D. C.

Stock feed adulteration:

Associate referee: Miss B. H. Silberberg, Bureau of Chemistry, Washington, D. C.

Saccharine products:

Referee: H. S. Paine, Bureau of Chemistry, Washington, D. C.

Honey:

Associate referee: Sidney F. Sherwood, Bureau of Plant Industry, Washington, D. C.

Maple products:

Associate referee: (Not appointed.)

Maltose products:

Associate referee: O. S. Keener, Bureau of Chemistry, Washington, D. C.

Sugar house products:

Associate referee: F. W. Zerban, Penick & Ford, Ltd., New Orleans, La.

Drugs:

Referee: G. W. Hoover, U. S. Food and Drug Inspection Station, Transportation Building, Chicago, Ill.

Alkaloids:

Associate referee: A. R. Bliss, Emory University, Atlanta, Ga.

Arsenicals:

Associate referee: W. O. Emery, Bureau of Chemistry, Washington, D. C.

Synthetic drugs:

Associate referee: C. D. Wright, Bureau of Chemistry, Washington, D. C.

Alkaloids of opium:

Associate referee: C. K. Glycart, U. S. Food and Drug Inspection Station, Transportation Building, Chicago, Ill.

Medicinal plants:

Associate referee: A. Viehoever, Bureau of Chemistry, Washington, D. C.

Enzyms:

Associate referee: J. F. Brewster, Bureau of Chemistry, Washington, D. C.

Food preservatives (saccharin):

Referee: A. G. Lowenstein, U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, New York, N. Y.

Coloring matters (oil soluble colors):

Referee: W. E. Mathewson, Bureau of Chemistry, Washington, D. C.

Metals in foods:

Referee: W. F. Clarke, Bureau of Chemistry, Washington, D. C.

Pectin in fruits and fruit products:

Referee: D. B. Bisbee, U. S. Food and Drug Inspection Station, U. S. Custom House, St. Louis, Mo.

Canned foods:

Referee: W. D. Bigelow, National Canners Association, 1739 H Street, N. W., Washington, D. C.

Physical methods of examination:

Associate referee: J. H. Shrader, Bureau of Plant Industry, Washington, D. C.

Tomato products:

Associate referee: F. C. Blanck, National Canners Association, Easton, Md.

Cereal foods:

Referee: C. H. Bailey, University Farm, St. Paul, Minn.

Distilled liquors:

Referee: (Not appointed.)

Wines:

Referee: (Not appointed.)

Limit of accuracy in the determination of small amounts of alcohol in beers:

Referee: J. R. Eoff, Garrett & Co., St. Louis, Mo.

Methods of analysis of near beers:

Referee: (Not appointed.)

Vinegars:

Referee: R. W. Balcom, Bureau of Chemistry, Washington, D. C.

Flavoring extracts:

Referee: H. J. Wichmann, U. S. Food and Drug Inspection Station, Tabor Opera House Building, Denver, Colo.

Meats and meat products:

Referee: C. R. Moulton, University of Missouri, Columbia, Mo.

Separation of meat proteins:

Associate referee: C. R. Moulton, University of Missouri, Columbia, Mo.

Decomposition of meat products:

Associate referee: (Not appointed.)

Gelatin:

Associate referee: C. R. Smith, Bureau of Chemistry, Washington, D. C.

Dairy products:

Referee: Julius Hortvet, Old Capitol, St. Paul, Minn.

Moisture in cheese:

Associate referee: W. A. Egge, U. S. Food and Drug Inspection Station, Federal Office Building, Minneapolis, Minn.

Fats and oils:

Referee: R. H. Kerr, Bureau of Animal Industry, Washington, D. C.

Spices:

Referee: H. E. Sindall, Austin, Nichols & Co., Brooklyn, N. Y.

Determination of shells in cacao products:

Referee: W. C. Taber, U. S. Food and Drug Inspection Station, Park Avenue Building, Baltimore, Md.

Method for the examination of cacao butter:

Referee: W. F. Baughman, Bureau of Chemistry, Washington, D. C.

Coffee:

Referee: H. A. Lepper, Bureau of Chemistry, Washington, D. C.

Tea:

Referee: E. M. Bailey, Agricultural Experiment Station, New Haven, Conn.

Baking powder:

Referee: G. H. Mains, Bureau of Chemistry, Washington, D. C.

Testing chemical reagents:

Referee: W. D. Collins, Geological Survey, Washington, D. C.

Soft drinks:

Referee: W. W. Skinner, Bureau of Chemistry, Washington, D. C.

Eggs and egg products:

Referee: H. L. Lourie, U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, New York, N. Y.

MEMBERS AND VISITORS PRESENT, 1919 MEETING.

Abbott, J. S., 1212 Munsey Building, Washington, D. C.
Alexander, Miss L. M., Bureau of Markets, Washington, D. C.
Alsberg, C. L., Bureau of Chemistry, Washington, D. C.
Anderson, M. S., Bureau of Soils, Washington, D. C.
Appleman, C. O., Agricultural Experiment Station, College Park, Md.

Badger, C. H., Bureau of Chemistry, Washington, D. C.
Bailey, C. H., Agricultural Experiment Station, University Farm, St. Paul, Minn.
Bailey, E. M., Agricultural Experiment Station, New Haven, Conn.
Bailey, H. S., Southern Cotton Oil Company, Savannah, Ga.
Bailey, L. H., Bureau of Chemistry, Washington, D. C.
Balch, R. T., Bureau of Chemistry, Washington, D. C.
Balcom, R. W., Bureau of Chemistry, Washington, D. C.
Barnard, H. E., American Institute of Baking, Minneapolis, Minn.
Bartlett, G. M., Joseph Campbell Co., Camden, N. J.
Bartlett, J. M., Agricultural Experiment Station, Orono, Me.

Baston, G. H., Room 1132 Webster Building, Chicago, Ill.
Bates, Carleton, U. S. Glue Company, Milwaukee, Wis.
Bates, Frederick, Bureau of Standards, Washington, D. C.
Baughman, W. F., Bureau of Chemistry, Washington, D. C.
Beal, W. H., States Relations Service, Washington, D. C.
Bidwell, G. L., Bureau of Chemistry, Washington, D. C.
Bigelow, W. D., National Canners Association, 1739 H Street, N. W., Washington, D.C.
Birckner, V., Bureau of Chemistry, Washington, D. C.
Blair, A. W., Agricultural Experiment Station, New Brunswick, N. J.
Bletsch, C. F., State College, College Park, Md.
Bloomberg, Eugene, General Chemical Company, Baltimore Works, Baltimore, Md.
Bopst, L. E., Bureau of Chemistry, Washington, D. C.
Boyle, Martin, Bureau of Chemistry, Washington, D. C.
Boyles, F. M., McCormick and Company, Baltimore, Md.
Brackett, R. N., Clemson Agricultural College, Clemson College, S. C.
Bradbury, C. M., Department of Agriculture and Immigration, Richmond, Va.
Bradley, L. W., 258 Lawton Street, Atlanta, Ga.
Bradley, Mrs. L. W., 258 Lawton Street, Atlanta, Ga.
Brauns, D. H., Bureau of Chemistry, Washington, D. C.
Breckenridge, J. E., American Agricultural Chemical Co., New York, N. Y.
Brewster, J. F., Bureau of Chemistry, Washington, D. C.
Brown, B. E., Bureau of Plant Industry, Washington, D. C.
Browne, B. F., 424 Book Building, Detroit, Mich.
Browne, Mrs. Grace, 820 South University Avenue, Ann Arbor, Mich.
Bryan, A. H., Arbuckle Bros., New York, N. Y. (Since deceased.)
Buchanan, Miss Ruth, Bureau of Chemistry, Washington, D. C.
Burroughs, Miss L. C., State Department of Health, 16 West Saratoga Street, Baltimore, Md.

Carpenter, F. B., Virginia-Carolina Chemical Co., Richmond, Va.
Cathcart, P. H., National Canners Association, 1739 H Street, N. W., Washington, D. C.
Charron, A. T., St. Hyacinthe, Province of Quebec, Canada.
Chesnut, V. K., Bureau of Chemistry, Washington, D. C.
Christie, Alfred, jr., 1957 Fourth Street, N. E., Washington, D. C.
Clarke, W. F., Bureau of Chemistry, Washington, D. C.
Clevenger, J. F., Bureau of Chemistry, Washington, D. C.
Coe, M. R., Bureau of Chemistry, Washington, D. C.
Coleman, D. O., Bureau of Markets, Washington, D. C.
Collins, W. D., Geological Survey, Washington, D. C.
Cook, F. C., Bureau of Chemistry, Washington, D. C.
Crawford, C. W., Bureau of Chemistry, Washington, D. C.
Custis, H. H., Bureau of Animal Industry, Washington, D. C.
Cutler, W. V., Bureau of Soils, Washington, D. C.

Davidson, J., Bureau of Chemistry, Washington, D. C.
Deemer, R. B., Bureau of Plant Industry, Washington, D. C.
Donnet, John, State Department of Health, 16 West Saratoga Street, Baltimore, Md.
Doolittle, R. E., Transportation Building, Chicago, Ill.
Doran, J. M., Bureau of Internal Revenue, Washington, D. C.
Dunbar, P. B., Bureau of Chemistry, Washington, D. C.

Edmondson, Miss R. B., Bureau of Chemistry, Washington, D. C.
Ellett, W. B., Agricultural Experiment Station, Blacksburg, Va.
Elvove, Elias, Hygienic Laboratory, Washington, D. C
Emery, W. O., Bureau of Chemistry, Washington, D. C.
Emmons, F. W., Washburn-Crosby Co., Minneapolis, Minn.
Esty, J. R., 1318 Fifteenth Street, N. W., Washington, D. C.
Evenson, O. L., Bureau of Chemistry, Washington, D. C.

Fellers, C. R., U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, San Francisco, Calif.
Finks, A. J., Bureau of Chemistry, Washington, D. C.
Fitzgerald, F. F., National Canners Association, 1739 H Street, N. W., Washington, D. C.
Fletcher, G. A., 702 Ninth Street, N. W., Washington, D. C.
Fraps, G. S., Agricultural Experiment Station, College Station, Texas.
Frear, William, Agricultural Experiment Station, State College, Pa.
French, D. M., Alexandria Fertilizer and Chemical Co., Alexandria, Va.
Fry, W. H., Bureau of Soils, Washington, D. C.
Fuller, A. V., American Sugar Refining Co., New York, N. Y.
Fuller, F. D., Agricultural Experiment Station, College Station, Texas.
Fuller, H. C., Institute of Industrial Research, Washington, D. C.
Fulton, H. R., Bureau of Plant Industry, Washington, D. C.

Garby, C. D., Bureau of Chemistry, Washington, D. C.
Gardiner, R. F., Bureau of Soils, Washington, D. C.
Geagley, W. C., State Food and Drug Department, Lansing, Mich.
Gersdorff, C. E. F., Bureau of Chemistry, Washington, D. C.
Gersdorff, W. A., Bureau of Chemistry, Washington, D. C.
Gordon, N. E., Agricultural Experiment Station, College Park, Md.
Gordon, W. O., Mead, Johnson & Co., Evansville, Ind.
Gore, H. C., Bureau of Chemistry, Washington, D. C.
Graham, J. J. T., Bureau of Chemistry, Washington, D. C.
Grant, D. H., 5652 Maryland Avenue, Chicago, Ill.
Gray, M. A., Pillsbury Flour Mills Company, Minneapolis, Minn.

Halverson, H. A., State Dairy and Food Commission, St. Paul, Minn.
Hand, W. F., Agricultural College, Agricultural College, Miss.
Hanson, H. H., State Board of Health, Dover, Del.
Harris, J. R., Tennessee Coal, Iron and R. R. Co., Birmingham, Ala.
Hartwell, B. L., Agricultural Experiment Station, Kingston, R. I.
Haskell, S. B., Soil Improvement Committee, Baltimore, Md.
Haskins, H. D., Agricultural Experiment Station, Amherst, Mass.
Haywood, W. G., State Department of Agriculture, Raleigh, N. C.
Hazard, I. W., Gibbs Preserving Company, Baltimore, Md.
Hazen, William, Bureau of Soils, Washington, D. C.
Heath, W. H., Letchfield, Ill.
Henry, A. M., State Department of Agriculture, Tallahassee, Fla.
Higgins, C. H., Sears Roebuck Company, Chicago, Ill.
Hinds, Miss M. E., State Food and Drug Department, Nashville, Tenn.
Hite, B. H., Agricultural Experiment Station, Morgantown, W. Va.
Holmes, R. S., Bureau of Soils, Washington, D. C.

Hoover, G. W., U. S. Food and Drug Inspection Station, Transportation Building, Chicago, Ill.
Horne, W. D., National Sugar Refining Company, Yonkers, N. Y.
Hortvet, Julius, State Dairy and Food Commission, St. Paul, Minn.
Houghton, H. W., Hygienic Laboratory, Washington, D. C.
Howard, C. D., State Board of Health, Concord, N. H.
Huddleson, M., Office of Horticulture, Washington, D. C.
Hudson, Miss Ruby, Atlanta, Ga.
Humphreys, A. F., Room 120, House Office Building, Washington, D. C.
Hunter, A. C., Bureau of Chemistry, Washington, D. C.
Hurst, L. A., Bureau of Plant Industry, Washington, D. C.
Huttlinger, C. F., American Sugar Refining Co., New York, N. Y.

Ingle, M. J., Albion, N. Y.
Irwin, W. H., Swift and Company, Chicago, Ill.

Jackson, R. F., Bureau of Standards, Washington, D. C.
Jacobs, B. R., 1731 H Street, N. W., Washington, D. C.
Jamieson, G. S., Bureau of Chemistry, Washington, D. C.
Jarrell, T. D., Bureau of Chemistry, Washington, D. C.
Jodidi, S. L., 23 West Irving Street, Chevy Chase, Md.
Johnson, J. M., Hygienic Laboratory, Washington, D. C.
Jones, C. H., Agricultural Experiment Station, Burlington, Vt.
Jones, D. B., Bureau of Chemistry, Washington, D. C.
Jones, W. P., Union Trust Building, Washington, D. C.

Kebler, L. F., Bureau of Chemistry, Washington, D. C.
Keenan, G. L., Bureau of Chemistry, Washington, D. C.
Keener, O. S., Bureau of Chemistry, Washington, D. C.
Keister, J. T., Bureau of Chemistry, Washington, D. C.
Kellems, T. O., U. S. Food and Drug Inspection Station, U. S. Custom House, New Orleans, La.
Kellogg, J. W., State Department of Agriculture, Harrisburg, Pa.
Kerr, A. P., Agricultural Experiment Station, Baton Rouge, La.
Kerr, R. H., Bureau of Animal Industry, Washington, D. C.
Klein, David, Hollister-Wilson Laboratories, Chicago, Ill.
Knight, H. L., States Relations Service, Washington, D. C.
Kohman, E. F., National Canners Association, 1739 H Street, N. W., Washington, D. C.
Kunke, W. F., Bureau of Chemistry, Washington, D. C.

Langenbeck, Karl, 1625 Hobart Street, N. W., Washington, D. C.
Lathrop, E. C., 8096 DuPont Building, Wilmington, Del.
Latshaw, W. L., Agricultural College, Manhattan, Kans.
LeClerc, J. A., Miner-Hillard Milling Company, Wilkes-Barre, Pa.
LeFevre, Edwin, Bureau of Chemistry, Washington, D. C.
Leith, T. B., State College, College Park, Md.
Lepper, H. A., Bureau of Chemistry, Washington, D. C.
Lodge, F. S., Armour Fertilizer Works, Chicago, Ill.
Loomis, H. M., National Canners Association, Mills Building, Washington, D. C.
Love, R. F., Bureau of Internal Revenue, Washington, D. C.

Luckett, J. D., States Relations Service, Washington, D. C.
Lythgoe, H. C., State Department of Health, Boston, Mass.

Magnuson, H. P., Bureau of Soils, Washington, D. C.
Magruder, E. W., F. S. Royster Guano Company, Norfolk, Va.
Mangels, C. E., Bureau of Chemistry, Washington, D. C.
Manross, Miss L. M., Bureau of Chemistry, Washington, D. C.
Markovitz, L. N., Bureau of Chemistry, Washington, D. C.
Mason, G. F., H. J. Heinz Company, San Francisco, Calif.
Mathewson, W. E., Bureau of Chemistry, Washington, D. C.
McCall, A. G., Agricultural Experiment Station, College Park, Md.
McCallums, W. R., Washington Star, Washington, D. C.
McDonnell, C. C., Bureau of Chemistry, Washington, D. C.
McDonnell, H. B., Agricultural Experiment Station, College Park, Md.
McGill, A., Department of Health, Ottawa, Canada.
McHargue, J. S., Agricultural Experiment Station, Lexington, Ky.
McIntire, W. H., Agricultural Experiment Station, Knoxville, Tenn.
McLaughlin, J. A., Chemical Warfare Service, Edgewood Arsenal, Edgewood, Md.
McMillin, H. R., Bureau of Animal Industry, Washington, D. C.
Mehurin, R. M., Bureau of Animal Industry, Washington, D. C.
Meigs, E. B., Bureau of Animal Industry, Washington, D. C.
Mitchell, A. S., Bureau of Chemistry, Washington, D. C.
Mitchell, L. C., U. S. Food and Drug Inspection Station, Federal Office Building, Minneapolis, Minn.
Mix, Miss A. E., Bureau of Chemistry, Washington, D. C.
Montgomery, C. P., 117 Wall Street, New York, N. Y.
Moore, Charles, Bureau of Chemistry, Washington, D. C.
Morton, J. K., Bureau of Chemistry, Washington, D. C.
Motter, M. G., Hygienic Laboratory, Washington, D. C.
Moulton, R. C., University of Missouri, Columbia, Mo.
Moulton, S. C., Health Department, Washington, D. C.
Munch, J. C., Bureau of Chemistry, Washington, D. C.

Nelson, E. K., Bureau of Chemistry, Washington, D. C.
Newkirk, W. B., Bureau of Standards, Washington, D. C.

Offutt, Miss M. L., Bureau of Chemistry, Washington, D. C.
Osterhout, K. J., Bureau of Internal Revenue, Washington, D. C.

Palkin, S., Bureau of Chemistry, Washington, D. C.
Palmer, H. E., Bureau of Chemistry, Washington, D. C. (Since deceased.)
Palmore, J. I., Bureau of Chemistry, Washington, D. C.
Parkins, J. H., F. S. Royster Guano Co., Norfolk, Va.
Parkinson, Miss N. A., Bureau of Chemistry, Washington, D. C.
Patten, A. J., Agricultural Experiment Station, E. Lansing, Mich.
Patterson, H. J., Agricultural Experiment Station, College Park, Md.
Pettyjohn, O. A., Woodward, Okla.
Phelps, I. K., Bureau of Chemistry, Washington, D. C.
Pingree, M. H. American Agricultural Chemical Co., 2343 South Clinton Street, Baltimore, Md.
Plummer, J. K., State Department of Agriculture, Raleigh, N. C.
Pope, W. B., 1416 Newton Street, N. W., Washington, D. C.

Porch, M. B., H. J. Heinz Company, Pittsburgh, Pa.
Porter, A. H., New Prague Flouring Mill Company, New Prague, Minn.
Power, F. B., Bureau of Chemistry, Washington, D. C.
Powick, W. C., Bureau of Animal Industry, Washington, D. C.
Price, T. M., Department of Health, Washington, D.C.
Proulx, E. G., Agricultural Experiment Station, La Fayette, Ind.

Rabak, Frank, Bureau of Plant Industry, Washington, D. C.
Rabak, William, U. S. Food and Drug Inspection Station, Federal Office Building, Minneapolis, Minn.
Rains, Miss Opal, Bureau of Chemistry, Washington, D. C.
Read, Miss E. A., Bureau of Chemistry, Washington, D. C.
Read, J. W., Agricultural Experiment Station, Fayetteville, Ark.
Redfield, H. W., U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, New York, N. Y.
Reed, J. B., Bureau of Chemistry, Washington, D. C.
Reh, Miss Emma, National Canners Association, 1739 H Street, N. W., Washington, D. C.
Reid, W. D., Swift and Company, Baltimore, Md.
Reindollar, W. F., State Department of Health, 16 West Saratoga Street, Baltimore, Md.
Riley, J. G., Bureau of Internal Revenue, Washington, D. C.
Ripperton, J. C., Bureau of Soils, Washington, D. C.
Roark, R. C., General Chemical Company, Baltimore Works, Baltimore, Md.
Roberts, O. S., Agricultural Experiment Station, La Fayette, Ind.
Robinson, W. O., Bureau of Soils, Washington, D. C.
Robison, F. W., Detroit, Mich.
Ross, B. B., Polytechnic Institute, Auburn, Ala.
Ross, W. H., Bureau of Soils, Washington, D. C.
Rudnick, Paul, Armour & Co., Chicago, Ill.
Runkel, H., Bureau of Chemistry, Washington, D. C.
Runyan, E. G., 419 Tenth Street, N. W., Washington, D. C.

Sample, J. W., State Department of Health, Nashville, Tenn.
Schreiner, Oswald, Bureau of Plant Industry, Washington, D. C.
Scott, Miss D. B., Bureau of Chemistry, Washington, D. C.
Shaefer, W. E., Bureau of Chemistry, Washington, D. C.
Shannon, F. L., P. J. Ritter Company, Philadelphia, Pa.
Shook, C. F., Booth Packing Company, Baltimore, Md.
Shuman, R. L., 3519 Fourteenth Street, N. W., Washington, D. C.
Sievers, A. F., Bureau of Plant Industry, Washington, D. C.
Silberberg, Miss B. H., Bureau of Chemistry, Washington, D. C.
Sindall, H. E., Austin, Nichols & Co., Brooklyn, N. Y.
Skinner, J. J., Bureau of Plant Industry, Washington, D. C.
Skinner, W. W., Bureau of Chemistry, Washington, D. C.
Smith, A. M., Agricultural Experiment Station, College Park, Md.
Smith, C. M., Bureau of Chemistry, Washington, D. C.
Smith, C. R., Bureau of Chemistry, Washington, D. C.
Smith, J. G., Bureau of Soils, Washington, D. C.
Smith, Miss K. A., Bureau of Chemistry, Washington, D. C.
Smith, P. H., Agricultural Experiment Station, Amherst, Mass.
Smith, Miss S. L., States Relations Service, Washington, D. C.

Snell, J. F., Macdonald College, Province of Quebec, Canada.
Snyder, E. F., Bureau of Plant Industry, Washington, D. C.
Spencer, G. C., Bureau of Chemistry, Washington, D. C.
Stephenson, C. H., Bureau of Chemistry, Washington, D. C.
Stevenson, A. E., National Canners Association, 1739 H Street, N. W., Washington, D. C.
Stockhom, W. L., Bureau of Markets, Washington, D. C.
Stone, C. B., Bureau of Chemistry, Washington, D. C.
Strevig, W. E., 50 E. Forty-first Street, New York, N. Y.
Strowd, W. H., State Department of Agriculture, Madison, Wis.

Taylor, J. N., Bureau of Animal Industry, Washington, D. C.
Thatcher, A. S., Loose-Wiles Biscuit Company, Washington, D. C.
Thom, Charles, Bureau of Chemistry, Washington, D. C.
Tice, W. G., State Department of Health, Trenton, N. J.
Toll, J. D., The American Fertilizer, 1010 Arch Street, Philadelphia, Pa.
Treuthardt, E. L. P., 1810 Munitions Building, Washington, D. C.
Trowbridge, P. F., Agricultural Experiment Station, Agricultural College, N. Dak.

Valaer, Peter, jr., Bureau of Internal Revenue, Washington, D. C.
Veitch, F. P., Bureau of Chemistry, Washington, D. C.
Viehoever, Arno, Bureau of Chemistry, Washington, D. C.
Vollertsen, J. J., Morris & Co., Chicago, Ill.

Walker, P. H., Bureau of Standards, Washington, D. C.
Walls, H. R., State College, College Park, Md.
Walton, G. P., Bureau of Chemistry, Washington, D. C.
Weber, F. C., Bureau of Chemistry, Washington, D. C.
Weems, J. B., State Department of Agriculture, Richmond, Va.
Wiley, H. W., Woodward Building, Washington, D. C.
Wiley, R. C., Agricultural Experiment Station, College Park, Md.
Wiley, S. W., Wiley & Co., Inc., Baltimore, Md.
Wilkansky, M., Jaffa, Palestine.
Wilkansky, T., Jaffa, Palestine.
Williams, C. C., National Canners Association, 1739 H Street, N. W., Washington, D. C.
Wright, C. D., Bureau of Chemistry, Washington, D. C.

PRESIDENT'S ADDRESS[1].

THE MEAT PROBLEM.

By P. F. Trowbridge (Agricultural Experiment Station, Agricultural College, N. Dak.), *President.*

Every year at the North Dakota Agricultural Experiment Station we grow a field of corn. We keep a careful record of all the items of cost of production. About the first of September, we take a number of spring pigs, weighing from 100 to 175 pounds each, and turn them loose in that corn to do the harvesting. The pigs are provided with cots for shelter and have access to plenty of water. Some time during the month of November the corn is all harvested and the pigs (now hogs) are weighed and sent to market. We then calculate the yield in pounds of pork produced by an acre of corn and if the market has held steady or improved we calculate the dollars of profit per acre. Last year each acre of corn produced 289 pounds of pork, as shown by the increased gain in weight. At the market price at that time, the gross returns were forty-seven dollars and fourteen cents per acre. The corn crop was well sold. Our farmers are urged to grow corn (even in North Dakota), to grow hogs, and to hog off a field of corn every year.

By comparative data we figure that corn crop as yielding about 35 bushels per acre of shelled corn, or 1960 pounds. Using the average figures as quoted by Henry and Morrison[2] for the composition of flint corn, we produced 204 pounds of protein, 98 pounds of fat, and 1360 pounds of carbohydrates per acre. Our 289 pounds of pork produced will have, we will assume, about the average composition of the entire hog. In fact, the per cent of protein will be lower and the per cent of fat will be higher. Fifteen per cent of the weight of the hog will be waste offal, leaving 246 pounds of hog carcass and edible offal, such as heart, tongue, liver, etc. Of this 246 pounds, about 13 per cent is bone and skin, non-edible portions. This leaves 214 pounds of edible pork produced by the 1960 pounds of corn.

This 214 pounds of pork will be about 40 per cent lean meat and 60 per cent fat meat. An analysis of the 85.6 pounds of lean meat is about as follows:

	Per cent
Protein	18.00
Fat	20.00
Ash	1.00
Water	61.00

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[1] Presented Tuesday morning, November 18, 1919, as special order of business for 11.30 o'clock.
[2] W. A. Henry and F. B. Morrison. Feeds and Feeding. 15th ed., 1915, 633.

This will yield approximately as follows:

	Pounds
Protein	15.4
Fat	17.1

Analysis of the 128.4 pounds of fat meat will show about as follows:

	Per cent
Fat	77.00
Protein	3.5
Ash	0.2
Water	19.3

This, in turn, will give a yield approximately equivalent to the following:

	Pounds
Fat	98.9
Protein	4.5

From the 289 pounds of hog we have obtained for human consumption 19.9 pounds of protein and 116 pounds of fat. A large portion of the fat meat will be rendered into lard and the protein of the cracklings will be lost as food for man. The actual protein available will, therefore, be less than the above figure; also there will be an appreciable loss of fat in the cracklings.

We see then that, in changing our acre of corn, 1960 pounds, to pork, we have fed 204 pounds of protein which could have been used direct for human food, and have, in return, less than 20 pounds of protein in the meat. We have lost more than 90 per cent of the protein. We have fed 98 pounds of fat and have, in return, 116 pounds of fat. We have gained 18 pounds of fat, and to produce this we have used up 1360 pounds of carbohydrates and 184 pounds of protein for which we have no direct return, except in the somewhat greater digestibility of the meat protein and fat over these constituents in the corn and in the added energy value of the 18 pounds of fat.

In the acre of corn we have, on the equivalent energy basis, 1650 pounds of digestible nutrients and, if we assume the pork to have a digestibility coefficient of 95 and put the fat on a protein energy equivalent basis, we have recovered in the edible pork 267 pounds of digestible nutrients. This shows 83.8 per cent loss of digestible nutrients, 16.2 per cent of the corn being returned as available human food. The grain from the field of corn if used direct as human food will furnish six times as much digestible nutrients as when put into pork products.

Research studies at the University of Missouri[1] on the energy cost of beef fattening show that Steer No. 18, a three year old thin feeder, was

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[1] Mo. Agr. Expt. Sta. Research Bull. **30**: (1919).

carried on a maintenance cost experiment and slaughtered and analyzed as a check animal. Steer No. 121, weighing almost exactly the same as the check animal and being as near a duplicate as could be selected, was put on full feed for 153 days. This steer had gained 502 pounds in live weight when it was slaughtered and analyzed. Three hundred pounds of this gain in live weight were due to increased weight of the lean and fat flesh of the carcass. The lean and fat flesh of the check steer, No. 18, contained 64 pounds of protein and 65.5 pounds of fat, while the lean and fat flesh of Steer No. 121 contained 93.7 pounds of protein and 223.6 pounds of fat. There was, therefore, a gain in the 300 pounds of edible flesh of 29.7 pounds of protein, and 158.1 pounds of fat. The edible offal (heart, brains, tongue, liver) of Steer No. 121 weighed about 10¼ pounds more than that of the check steer. These organs were not analyzed separately, so we estimate added edible protein as 1 pound and the fat as 5 pounds. In order to make this gain in edible protein and fat, the steer consumed 2401.3 pounds of digestible nutrients calculated to an equivalent energy basis. The equivalent energy basis of the edible protein and fat, based on a digestion coefficient of 95, is 377.9 pounds. This shows that, on the ration used during the 153 days fattening period, about 15.73 per cent of the energy equivalent in the digestible nutrients consumed is recovered in the form of edible digestible nutrients available for human consumption. The ration fed the steers consisted of 8 parts of corn coarsely ground, 1 part of linseed meal, and 0.4 as much alfalfa hay as grain.

It would seem from the data given that only about one-sixth of the digestible nutrients consumed by the hog or the steer becomes available as food for man. Part of the feed of the steer (the corn) could have been used directly as human food. Had the steer been fed entirely on such feed stuffs as were not directly available for human food, the per cent made available for man in the form of edible meat would, perhaps, have been a little less, but that lower amount available would have been a net gain in the production of food for the human race.

As I study the corn field data, I find my story only partly told. The hogs have eaten that part of the crop already fit for human food and have scarcely touched the fodder which, according to our records, is just about one-half the weight of the entire plant on an air-dry basis. This fodder is broken down and trampled in the dirt. Part of it can be plowed under to rot for humus, but much of it must be raked and burned to get the land in shape for the next crop. Parenthetically, I may note that this same field was put into corn again in 1919 for the hogging off experiment. Complete data are not available, as the hogs have not been sold. The yield is fully twice as great as last year, but because of the slump in the market price of hogs it looks as if, perhaps, we would give away the corn crop so far as the finances are concerned.

The acre of corn fodder contained, according to my calculations, 1023 pounds of digestible nutrients which, if consumed by a steer, would have produced about 161 pounds of nutrients in the form of meat for human consumption, as compared with 267 pounds of nutrients produced from the grain of the same field. By not organizing our feeding so as to utilize the entire digestible nutrients of the corn crop we have wasted about 38 per cent of the crop.

While hogging off the corn is profitable from the financial standpoint, see what extravagance it is from the point of view of producing food to feed a hungry world! The grain of the corn crop contained 1650 pounds of digestible nutrients available for human food. The 1023 pounds of digestible nutrients of fodder were capable of being transformed into 161 pounds of digestible units available for human consumption, making a total of 1811 pounds of digestible nutrients available for human consumption, from which we did obtain 267 pounds for human consumption, a utilization of only 14.7 per cent.

An average of 18 plots of wheat gives the grain as 29.1 per cent of the weight of the crop. On this basis, 20 bushels of wheat, or 1200 pounds per acre, would represent a yield of straw of ~~1024~~ 2924 pounds. Wheat straw in North Dakota is not highly regarded as feed and thousands of tons are burned annually, especially in the eastern part of the State. Unquestionably, the wheat crop can be handled so as to increase very greatly the quality of the straw for feed. This will come when more live stock is produced. The ~~1024~~ 2924 pounds of straw from a 20 bushel wheat crop contain ~~710~~ 1279 pounds of digestible nutrients capable of doing their part in a balanced ration in the production of meat equivalent to ~~112~~ 170 pounds of digestible nutrients for human food. Similarly, the oat straw from an acre of 40 bushel oat crop (data from 15 plats) is 55 per cent of the crop, weighs about 1564 pounds and contains 713 pounds of digestible nutrients.

At present market prices but very little wheat is being fed to animals (some to poultry). Approximately 30 per cent of the wheat milled is in the form of bran and shorts and is generally used for animal food. Much of this coarser portion of the wheat grain could be used direct as human food, but it would be of doubtful economy, as the human digestive tract is not equipped for the efficient handling of feed stuffs. Let us not let a food fad rob the cow of her bran mash.

The minimum price of hogs was fixed on the basis of the cost or value of 13 bushels of corn because of the prevalent custom of fattening hogs on corn and because, when so fattened, 13 bushels of corn are used for every 100 pounds increase in weight of the hogs. If corn is worth one dollar and a half per bushel for human food in competition with other cereals, the farmer can not afford to feed it to hogs unless the market price of the hogs is nineteen dollars and a half, or more, per hundred. When hogs are the above price, experimental data show that if pastured on alfalfa with a small amount of middlings, the returns from the alfalfa in pounds of pork are nearly one hundred dollars per acre. With a reasonable return on investment in land, pork on alfalfa has been produced for about six cents per pound. With beef or mutton the problem of direct competition with man in the feed consumed is not so great as with the pork production.

I have already shown that we can not produce the grain crops for man's cereal foods without producing at the same time about an equal tonnage of roughage. This roughage contains from 45 to 65 per cent as many pounds of digestible nutrients as the cereals, but these nutrients can not nourish man except as they are first utilized in the production of animal food.

The proper tillage of our soil requires the growing of a certain amount of the grass or leguminous crops which must be used for animal feed. A considerable per cent of the land of the country is either too wet, too dry, or too rough to be used for growing cereal crops and must always be in grass for pasturage or hay. The United States Government Soil Survey of North Dakota estimates that 17 per cent of the lands of the State west of the one hundredth meridian is too rough for farm cropping and should be used permanently for grazing.

Because of the opening of this vast country to the homesteader, the ranges have been broken up. Live stock to a large extent disappeared with the advent of the bonanza grain farm. It is no use to rotate crops and grow roughage without animals to feed. Animals can not be kept without fences, and in a good year a fortune is made on wheat or flax, so why bother with animals which mean labor during the winter months? Gradually, the western farmers are seeing that a one-crop system of agriculture can not permanently endure, and live stock is coming back.

At the Mandan Dry Land Station just west of the Missouri River the State Agricultural Experiment Station cooperates with the United States Department of Agriculture in the study of the problem of the native pastures. Primarily, the problem is to study the carrying capacity of the range pasture and the effect of close grazing upon the carrying capacity. As a secondary problem, we are studying the effect of the degrees of the grazing upon the condition of the steers and the quality of the meat.

We have just completed the fourth consecutive year of this grazing trial. The experiment is planned for ten years, and present results must not be considered as final until further confirmed. For purposes of comparison, the section of land selected was much more level than the usual range land. Fields of 100, 70, 50, and ~~40~~ 30 acres are fenced, also a 70 acre field divided into three parts, called the rotation pasture, and there is also a large reserve pasture. All fields are supplied with good water and are accessible to shelter sheds for hot and stormy weather.

The same weight of cattle are put on each pasture each spring—ten, two year old steers or equivalent. A few head are put into the reserve pasture to maintain the number on experiment in case of a loss. Cattle are weighed every 30 days. Those on the 30 acre pasture are weighed every 10 days after July first. They are kept on this pasture until the consecutive weighings show a loss in weight, when the pasture is reported as 100 per cent grazed and the animals are turned into the reserve pasture to observe the rapidity of recuperation. A few points seem to stand out strikingly.

The grazing season for 1919 was 140 days, the shortest in four years, ending October twentieth. The increase in the weight of the cattle was as follows:

100 acre pasture produced 2860 pounds of beef.
70 acre pasture produced 2990 pounds of beef.
70 acre rotation pasture produced 2570 pounds of beef.
50 acre pasture produced 2230 pounds of beef.
30 acre pasture produced 1790 pounds of beef.

The 30 acre pasture was 100 per cent grazed on September first, and the animals were transferred to the reserve pasture, making the season for that pasture 90 days.

Each acre of the 100 acre pasture produced 28.6 pounds of beef.
Each acre of the 70 acre pasture produced 42.7 pounds of beef.
Each acre of the 70 acre rotation pasture produced 36.7 pounds of beef.
Each acre of the 50 acre pasture produced 44.6 pounds of beef.
Each acre of the 30 acre pasture produced 59.7 pounds of beef.

Each year the 30 acre closely grazed pasture has produced more pounds of beef than the year previous.

The per acre returns on the 30 acre pasture were more than double those from the 100 acre pasture, but the per acre investment in cattle was three and one-third times as great on the 30 acre pasture. The cattle went on to the range at a calculated value of ninety dollars per head. If the gain is figured at ten cents per pound and the land valued at thirty dollars per acre, the 100 acre pasture yields a gross return of 7.3 per cent; the 30 and 70 acre pastures, 9.9 per cent; and the 50 acre pasture, 9.7 per cent.

During the grazing season of 140 days, the gain per steer per day was 2 pounds on the 100 acre pasture; 2.1 pounds on the 70 acre pasture;

1.99 pounds on the 70 acre rotation pasture; 1.59 pounds on the 50 acre pasture; and 1.99 pounds on the 30 acre pasture for the 90 days this pasture was grazed.

Before the first of June the cattle were all on the general range for a week or two, so that the first fill on grass was made before the weigh-in for the grazing period on June first. The gains during June were remarkable, averaging 4.356 pounds per day for all the steers. The gains were quite uniform, being the highest on the 70 acre pasture. After the first of July the daily gains dropped to 2.6 pounds on the 100 and 70 acre pastures, to 1.9 pounds on the 50 acre pasture, and to 1.23 pounds on the 30 acre pasture. During the last ten days in August, the steers on the 30 acre pasture lost in weight, so that this loss affects the daily average for the 90 days. From September first to October twentieth, 5 of the 30 acre pasture steers were on the reserve pasture with an abundance of feed and gained 1.3 pounds per day. During this same time, the steers that had been continuously on the reserve pasture gained only 0.2 pound per day. The small gains during the latter part of the season suggest that perhaps we overrate the nutritive value of the dry range grass, and we have already outlined an experiment to study the digestibility of the native grasses cut at different seasons.

September first two steers each from the 30 acre exhausted pasture and from the reserve pasture were shipped to the North Dakota Agricultural College and slaughtered in a test. They were sold to the local packing company, all bringing the same price, nine dollars and a half per hundred. The two 30 acre pasture steers dressed more than 1 per cent better than the reserve. The carcasses all graded medium, sold at fourteen dollars per hundred, and were rated as good quality range grass cattle.

The flavor of the meat was not noticeably affected by the close grazing, although all the sage on the pasture was eaten. From a carcass of a 30 acre pasture steer and also from one from the reserve pasture, (~~sixtieth~~ both Angus breed) a cross section of the fifth lumbar vertebra was separated into lean, fat, and bone, and showed 28.16 per cent of fat for the 30 acre pasture carcass and 22.03 per cent of fat for the reserve pasture carcass.

The other steers from the 30 acre pasture were put into the reserve pasture until October twentieth, when the trial was closed for the season and the cattle were shipped to the North Dakota Agricultural Experiment Station for a steer winter feeding experiment. One steer from each pasture was slaughtered. All were Angus except that one from the 70 acre pasture had enough Hereford blood to give him a white face.

Dressed weight and fat in cross section of the fifth lumbar vertebra of steers.

MATERIAL	30 ACRE	70 ACRE	70 ACRE ROTATION	100 ACRE	50 ACRE
	per cent	*per cent*	*per cent*	*per cent*	*per cent*
Dressed weight	56.49	55.36	53.68	52.39	51.77
Fat in cross section of the fifth lumbar vertebra	32.72	30.57	22.87	25.38	22.40

The carcasses from the 70 acre rotation pasture and the 50 acre pasture graded fair to medium, while the other three all graded good. The three carcasses were really as good beef as the average market would care to handle. These steers had had no grain whatsoever. They were typically range Angus steers. The quality of the meat was excellent. The close grazing of the range does not appear to affect the flavor of the meat.

It is worth noting that the 30 acre pasture steer lost weight during the last ten days of August and on the reserve pasture from September first to October twentieth gained only 1.3 pounds per day, yet this steer dressed the highest per cent and showed the greatest per cent of fat in the cross section. This steer also had the greatest amount of offal fat, 23.5 pounds. The 50 acre pasture steer had only 10.5 pounds of offal fat. All the data on this steer indicate that the 50 acre pasture was exhausted and the steers were losing rapidly in condition while barely holding their own in weight.

These steers were strictly grass steers. They did not know the taste of grain. The meat was produced entirely from products which man could not use as food and the meat thus produced was a definite increase in the nutrients available for man.

I have tried to use these illustrations to show that in the economical production of man's food the production and use of meat plays a very important part, because of the areas of our country that are only fit for the growing of grasses and because the production of cereal foods entails the production of nearly an equal weight of roughage in addition to the 30 per cent or more of milling by-products that are best suited for animal foods. To these should be added the protein concentrates from the seeds of flax, cotton, peanuts, soy beans, etc.

Our experience of the past years has called to our mind the seriousness of a world shortage in food. We have increased our consumption of fish, but have scarcely begun to realize the possibilities of our waters to augment the meat supply. Our attention has been called to the possibilities of the utilization of animal flesh other than beef, pork, mutton, and poultry for food. Probably some here have tried the generous whale steaks. In the United States quite a number of markets have been established for the sale of horse meat for human food. They have met with very indifferent success, but there appears to be a growing demand for the cured horse meat for export. The horse does better than the steer on the open ranges in making a living under adverse conditions. He is able to graze closer and will paw away the snow in winter to get at the grass when a steer will starve.

Horse meat is more like beef than it is like pork or mutton. In fact, most of us could not tell a roast of horse from one of beef. Our prejudice against eating an animal which has been of such service to man is hard to overcome. I hope the time may come when the meat of the horse will find its place on our tables without prejudice, yet I would not wish the stress of necessity to force us to it as was the case with Europe.

There is not time to discuss the question of the economy of milk production or its importance as animal food. A steer may gain from 300 to 800 pounds in a year, not over 40 per cent of which is solids available for human food. In the same length of time the dairy cow may add 100 pounds to her weight and produce more than her body weight in milk solids. She exceeds the steer in economy of production, but she should be made to manufacture her products from those feed stuffs not already available for human consumption.

Modern agriculture must not seek to make of man a vegetarian. Production and consumption of meat must be encouraged. Permanent agriculture demands crop rotation and the maintenance of soil fertility with the production of sufficient animals to manufacture into human food and into energy for man's use those products not directly available to him. This should be done with a minimum use of that food for animals which is in its form adapted to man's immediate use.

As I try to figure the possibilities of meat production from the hay, grass, and roughage of the cereal crops, I am almost incredulous at the magnitude. I will not attempt to give you the figures. I have used the Government reports of acreage and yields. I have used Henry and Morrison's[1] figures on the composition of the various roughages and on the digestible nutrients contained in them. I have estimated that one-half of all the cereal straw and corn fodder could be wasted and used for other than feed purposes. I have computed the number of cattle, sheep, horses, and mules and estimated that one-fourth of all the roughage should be reserved for the horses and mules. I have not counted in the feed value

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[1] W. A. Henry and F. B. Morrison. Feeds and Feeding. 15th ed., 1915, 633–4.

of any of the concentrates nor the cereals. I have used our experimental data which show that only about 15 per cent of the digestible nutrients can be transformed by the animals into digestible meat product nutrients for man's consumption. I have subtracted the over two and one-half billion pounds of meat and meat products we exported during 1918, and yet if we would only utilize what we already produce every man, woman, and child in our land might eat pork and poultry as of old and, in addition, have more than one and one-half pounds of beef or mutton every working day in the year.

FIRST DAY.

MONDAY—MORNING SESSION.

The thirty-fifth annual convention of the Association of Official Agricultural Chemists was called to order by the President, P. F. Trowbridge, of Agricultural College, North Dakota, on the morning of November 17, 1919, at 10:00 at the New Willard, Washington, D. C.

REPORT ON FOODS AND FEEDING STUFFS.

By G. L. Bidwell (Bureau of Chemistry, Washington, D. C.), *Referee.*

The referee has very little to report. He has consulted with his associate referees on several occasions, written letters regarding the work of the association, and given advice to a small extent. The investigational work, however, has been conducted by the associate referees and their reports will speak for themselves.

R. F. Jackson and C. L. Gillis (Bureau of Standards, Washington, D. C.) presented a paper on "The Double-Polarization Method for Estimation of Sucrose and the Evaluation of the Clerget Divisor"[1].

REPORT ON SUGAR.

By A. H. Bryan[2] (Arbuckle Bros., New York, N. Y.), *Associate Referee.*

DISCUSSION OF FORMER ASSOCIATE REFEREE'S RECOMMENDATIONS.

During the past two years it has been impossible for the associate referee to carry on any extended work upon the recommendations left over from the 1916 meeting, although many of these are of the greatest importance to chemists called upon for sugar determinations.

The recommendations of C. A. Browne (New York Sugar Trade Laboratory, 80 South Street, New York, N. Y.), former associate referee, called for further study along the following lines[3]:

(1) Upon the modifications proposed in 1916 for determining sucrose by acid and invertase inversion.

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[1] U. S. Bur. Standards Scientific Paper **375**: (1920).

[2] Since deceased.

[3] *J. Assoc. Official Agr. Chemists*, 1919, **3**: 263.

(2) Upon methods for the determination of small amounts of reducing sugars in the presence of sucrose.

(3) Upon the methods of determining copper by reduction of the oxid in alcoholic vapors.

(4) Upon the optical methods for estimating raffinose in beet products, using enzyms for the hydrolysis.

It is suggested that the first, second, and fourth recommendations be referred to the Carbohydrate Laboratory of the Bureau of Chemistry for study, as there is need in these recommendations for the continuous work of one man. The third recommendation could be studied by any chemist doing reducing sugar determinations, as it is a rapid method of determining the copper in the cuprous oxid precipitate from reducing sugar determinations. In the case of pure sugars, the weighing of the cuprous oxid gives good results, but with impure sugar products this method does not give true results, because of the contamination of the cuprous oxid with organic matter, and also with mineral salts. The changing of the cuprous oxid to cupric oxid by heating, or of the cuprous oxid to copper by alcoholic vapors, removes the organic matter, but does not remove the contamination by mineral salts. The most accurate method is to determine the copper in the precipitate volumetrically.

At the 1916 meeting, W. D. Horne (National Sugar Refining Company, Yonkers, N. Y.) asked for an investigation and report upon five points[1]. Upon some of these a little work has been conducted, but on others there seems to be no particular need for experimentation. The following suggestions on the points submitted by Horne are made:

(1) *That raw sugar samples be mixed in a mortar, instead of on a plate, to diminish moisture changes.*—Under normal conditions there is little moisture change by mixing samples of raw sugar on a glass plate, and a more thorough mixing in a shorter time is accomplished on the glass plate than in the mortar. It was impossible for the associate referee to mix as many samples in a given length of time in a mortar as upon a glass plate. This, however, may have been due to awkwardness on his part, as he has always used the glass-plate method. Comparative experiments showed a greater tendency for wet sugars to cling to the mortar than to the glass plate, and more difficulty was noted in cleaning the mortar than the glass plate. With certain raw sugars, as Philippine mats and hard, lumpy, dry sugar, the mortar gave a sample more even in appearance than the same sample mixed upon a glass plate, using a rolling pin or spatula to mash the lumps. Polarization, however, revealed no decided difference in favor of the one or the other method of mixing. It

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[1] *J. Assoc. Official Agr. Chemists*, 1919, 3: 263.

is believed, therefore, that raw sugar samples may be mixed on a glass plate or in a mortar, and yield as correct results one way as the other.

(2) *That the defecation be made with the minimum amount of lead subacetate requisite to cause flocculation and that an excessive quantity for producing a lighter-colored filtrate than is necessary to obtain a reliable reading be avoided.*—All printed regulations for the use of lead subacetate require that a minimum amount be used, which means just enough lead to cause a clear defecation. All sugar chemists know that increasing the quantity of lead subacetate changes the polarization, especially in low-grade sugars, and it is to be regretted that an absolute amount of lead subacetate to be used with raw sugars of a definite polarization can not be prescribed. This point is covered in the methods of analysis. The term "minimum quantity" is really a personal equation.

(3) *That polarizations be checked by readings above and below rather than by averaging.*—The usual method of polarizing sugars is to average a series of three to five successive readings. In each case the reading is the one in which the analyst believes he has an exact match of color in the field. The writer has been unable to obtain as concordant results by the method suggested as by the usual method.

(4) *That polarizations be made at 20°C., or that temperature corrections for levulose be included with those for sucrose.*—The methods of the International Commission for Uniform Methods of Sugar Analysis[1] which were adopted by this association require that polarizations be made at 20°C., but they do not offer any corrections for polarizations made at other than 20°C. Corrections for temperature in polarizations should be founded upon the substance that is being polarized, that is, the temperature correction for a raw beet sugar is different from that for a raw cane sugar; hence they are not interchangeable. The application of a correction for sucrose alone where other sugars are present is also wrong. The question of temperature corrections for raw cane sugars is discussed on page 326.)

(5) *That the Bureau of Standards be asked to certify to the most advisable Baumé scale and that it be adopted.*—The Baumé scale still clings in the sugar industry. Yet Baumé spindles are not graduated as finely as those standardized for the Brix scale. The ordinary Baumé spindle is generally divided into degrees and seldom finer than half a degree, while the ordinary Brix spindle is divided to a tenth of a degree. With 1° Baumé equalling 1.8° Brix, it is easily seen that by using a Brix spindle a more correct reading of the density can be obtained than by using a Baumé scale. The Baumé scale should be relegated to the shelf, and only the Brix scale used. If there is real need for a Baumé scale, however, the one in general use should be that advocated by this association.

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[1] Proceedings, Paris Convention, 1900.

AN AMERICAN-MADE POLARISCOPE.

During the past few years few, if any, polariscopes have been imported into this country; and of the few many showed big discrepancies in the scale. As the time seemed ripe for the production of an American polariscope, and makers were eager to produce it, it was decided to formulate specifications for a polariscope which would be accurate, simple, and easy of manipulation, and would embrace the best ideas of the users of these instruments. With this in view, C. A. Browne, after consultation with many sugar chemists, submitted to a committee of the American Chemical Society specifications for a polariscope. These were gone over very carefully and in the main approved. A sample instrument was built from these specifications and criticised by members of this committee.

Two points of difference arose: Optical system and normal weight.

Optical system.—The preference as to a polarizer was divided between the Lippich and Jellet-Cornu systems. The former consists of a large Nicol prism with a smaller Nicol prism (half-prism) covering just half of the field and placed in front of the large one. The two must be in exact alignment to obtain good results. In the Jellet-Cornu system there is only one prism. It is generally admitted that the Lippich system gives greater accuracy, and some held out for this, but it has the disadvantage of being a little more complicated and more easily put out of order than the Jellet-Cornu. For factory work and the ordinary run of polariscopic work, the Jellet-Cornu system can not be said by any to give unreliable readings due to the lower degree of sensitiveness. This question of optical system has been left to the opinion of the buyers of the new polariscope. With a Lippich system, however, the price will probably be some fifty to one hundred dollars more than with the Jellet-Cornu system.

Normal weight.—Upon this point decided opposition developed. With the start of a new polariscope many members of this committee thought it was time to adopt a rational normal weight. The German standard is 26 grams to 100 cc., and the French standard, 16.29 grams to 100 cc. Both of these figures are not easily divisible without a fraction, one of the reasons that a 20-gram standard should be the one adopted for the polariscope made in America, 20 grams being divisible by 2, 4, 5, 10, etc. In fact, any standard pipette will deliver an even number of grams of material when 20 grams are dissolved and made up to 100 cc. This proposed standard is also between the two present standards. It is also superior to the 26-gram, in that it will allow of a normal weight of dextrose and lactose. Obviously, this proposed 20-gram standard is of great advantage to the food chemist who takes aliquot parts of his solution used for polarization to make other determinations. It is also of great use in control work around sugar houses, both cane and beet, as well as in refineries. With the French standard of 16.29 there has been no objection to the smallness of the charge used for analysis, or inaccuracy in reading due to this small charge, nor have objections arisen to the use of the 26-gram standard because of inaccuracies of reading. This objection, however, has been raised in connection with the adoption of the proposed 20-gram standard.

Another reason for adopting this new standard of 20 grams is that it is proposed to have it absolutely accurate. At present there are in use in this country polariscopes standardized for 26.048 grams in true cc., in Mohr cc., and for temperatures of 17.5° and 15°C., in addition to the late standard of 26 grams to 100 cc. at 20°C. Even this last standard for the polariscope has been found by Bates (Bureau of Standards, Washington, D. C.) to be incorrect. The 100° point on the scale reads only 99.895 when using 26.00 grams of pure sugar in 100 cc. and polarizing at 20°C. The United States Treasury Department has adopted this new value in the valuation of raw sugar for duty purposes, and the Bureau of Standards uses it for standardizing quartz plates. This procedure then adds another standard for the 26-gram instrument to the already numerous ones. E. Saillard of France has recently shown that the normal weight for the French instrument is not 16.29 but 16.26[1]. In view of these inaccuracies and the many standards for 26-gram instruments, the American polariscope should be made with a standard which is correct, and not open to error, and which is rational. The 20-gram standard will answer this purpose.

The 20-gram standard is not new, for Sidersky and Pellet advocated the adoption of the normal weight of 20 grams as an international sugar scale at the Second International Congress of Applied Chemistry, held in Paris in 1896. This standard of 20 grams has been voted upon favorably by the Louisiana Sugar Chemists and the Hawaiian Sugar Chemists. Many English chemists have spoken favorably of it; and the French chemists are willing to accept it if it becomes international. It is the desire of all advocating this standard that the value for 20 grams be checked by the physical laboratories of all countries.

A committee, composed of C. A. Browne, C. E. Coates (State University, Baton Rouge, La.), and G. W. Rolfe (3 Dana street, Cambridge, Mass.), has been appointed by the American Chemical Society to get in touch with the various governments abroad with a view to arranging an international standard for the polariscope. It is the purpose of this committee to take up this matter with the various chemists in this country. It is suggested that a committee be appointed from this association to look into the question of the adoption of this new 20-gram standard, if it can be made international.

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[1] *J. fabr. sucre*, 1919, **60**: No. 13

STANDARDIZATION OF QUARTZ PLATES BY THE BUREAU OF STANDARDS.

For the past year or so the Bureau of Standards has been standardizing the quartz plates sent them on the value found by Frederick Bates and R. F. Jackson of that Bureau, that the 100° point is low by 0.105°, and not on their former value which has had practical universal usage.

A quartz plate with a German Reichsanstalt value of 99.895°V. would be certified by the Bureau of Standards as reading 100°V. The application of quartz plates with the new Bureau of Standards value to saccharimeters equipped with Reichsanstalt scales may cause the introduction of serious errors.

In making a reading upon a polariscope it is customary first to see that the zero point of the scale is in perfect adjustment. The standard quartz plate is then introduced and any deviation from the certified value employed as a correction in the readings. If the vernier of the polariscope be changed so that the scale reading agrees with the new plate, readings made in the vicinity of zero will be in error to the extent of 0.105 and invert readings in the vicinity of −30 will be in error to the extent of 0.14. These errors at various points of the scale may be considerably multiplied in case of diluted solutions. To avoid such discrepancies, polariscopes equipped with the Reichsanstalt scale should be controlled only by quartz plates standardized according to the Reichsanstalt value, for only in this way is the scale correct at all its points. In other words, scales and control plates should be measured according to one and the same standard.

Most sugar chemists who have gone into the subject are free to admit that the 100° point is low. There is grave doubt, however, whether it is as low as the Bureau of Standards makes it. The work upon which this value was founded has been severely criticised by sugar chemists abroad, and until it has been checked it is not advisable to make changes in the old international value. It is much less advisable to apply this correction for the 100° point in the way the Bureau of Standards is doing. If there is an error in the 100° point, the only correct way is to increase the normal weight and use the old scale, or to change the graduation of the scale so that the error will be distributed equally from 0 to 100.

In view of these facts, it is most strongly recommended that when quartz plates are sent to the Bureau of Standards for certification they be requested to certify to the old value of the 100° point, and not their new one—at least until this new value has been agreed to internationally.

TEMPERATURE CORRECTIONS FOR RAW CANE SUGARS.

At the twenty-second annual meeting of this association in 1905[1], and again at greater length at the twenty-fifth meeting[2], C. A. Browne called

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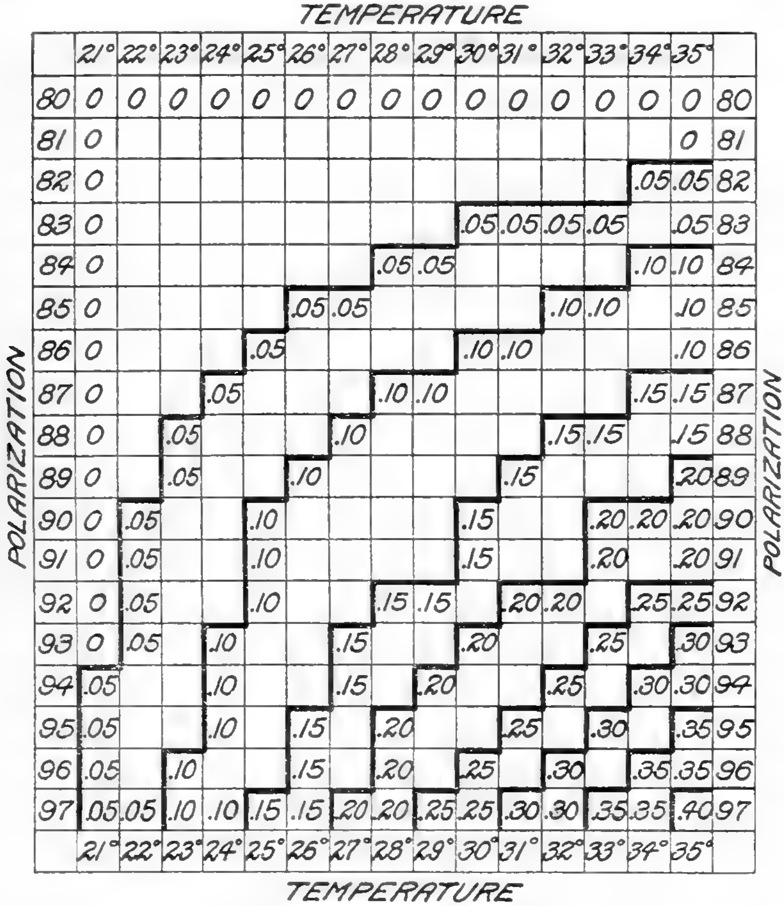
[1] U. S. Bur. Chem. Bull. **99**: (1906), 20.

[2] *Ibid.*, **122**: (1909), 221.

attention to the temperature factor in polarizations of raw cane sugars. At the twenty-eighth meeting in 1911[1], W. D. Horne showed the effect of temperature correction on polarizations of raw cane sugar. Comparative results on the polarizations of raw cane sugars when polarized at 20°C., and when polarized at varying temperatures, but corrected by Browne's temperature correction formula[2], are here recorded. The general formula for correcting the polarizations (P) at t° of any raw cane sugar to $P^{20°}$ is as follows:

$$P^{20°} = P^{t} + 0.0015\,(P^{t} - 80)\,(t° - 20).$$

The chart shown in Fig. 1 gives corrections for sugars polarizing from 80 to 97, and for temperatures from 21° to 35°C., based on this formula.



	21°	22°	23°	24°	25°	26°	27°	28°	29°	30°	31°	32°	33°	34°	35°	
80	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	80
81	0														0	81
82	0													.05	.05	82
83	0									.05	.05	.05	.05		.05	83
84	0							.05	.05					.10	.10	84
85	0					.05	.05					.10	.10		.10	85
86	0				.05					.10	.10				.10	86
87	0			.05				.10	.10					.15	.15	87
88	0		.05				.10					.15	.15		.15	88
89	0		.05			.10					.15				.20	89
90	0	.05			.10					.15			.20	.20	.20	90
91	0	.05			.10					.15			.20		.20	91
92	0	.05			.10			.15	.15		.20	.20		.25	.25	92
93	0	.05		.10			.15			.20			.25		.30	93
94	.05			.10			.15		.20			.25		.30	.30	94
95	.05			.10		.15		.20			.25		.30		.35	95
96	.05		.10			.15		.20		.25		.30		.35	.35	96
97	.05	.05	.10	.10	.15	.15	.20	.20	.25	.25	.30	.30	.35	.35	.40	97
	21°	22°	23°	24°	25°	26°	27°	28°	29°	30°	31°	32°	33°	34°	35°	

FIG. 1. CORRECTIONS FOR POLARIZATIONS MADE AT OTHER THAN 20°C.

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[1] U. S. Bur. Chem. Bull. **152**: (1912), 207.
[2] *J. Ind. Eng. Chem.*, 1909, **1**: 567.

In this chart the corrections vary by 0.05°, as this is the lowest difference most sugar chemists use for their polarizations. This chart has been in use by the writer for some five years in the polarizations of raw cane sugars. Table 1 gives the comparative results of 4,117 polarizations made in 1915, 1916, and 1917 at 20°, together with the polarizations made at ordinary room temperature and corrected by this chart. The polarizations at 20°C. are those of the New York Sugar Trade Laboratory; the others are the associate referee's at room temperature, with the corrections.

TABLE 1.

Results of polarizations at 20°C., and at room temperature, with corrections.

RESULTS WITH 20°C. POLARIZATION.	PER CENT
Same or within 0.05°	48.9
0.05–0.15° higher	16.4
0.05–0.15° lower	17.4
0.15–0.25° higher	6.2
0.15–0.25° lower	5.9
0.25–0.35° higher	2.1
0.25–0.35° lower	1.7
0.35–0.45° higher	0.5
0.35–0.45° lower	0.5
0.45 up higher	0.3
0.45 up lower	0.1

During the year 1915, the average temperature-corrected polarization was 0.010° higher than that made at 20°, in 1916, 0.010° lower, and in 1917, 0.001° lower.

These results indicate that the temperature correction formula prepared by C. A. Browne will give average results closely approximating the true polarization at 20°C. This formula can not be expected, however, to give exact results in each case, due to the varying percentages of fructose and glucose in samples of raw sugar of the same polarizations.

The adoption of this formula is recommended for use in correcting the polarizations of raw cane sugars when made at any temperature other than 20°C. It is recommended also that this formula should be substituted for the quite frequently used formula of $P^{20°} = P^{t}[1 + 0.0003(t - 20)]$ which corrects only for sucrose, and gives too large corrections when applied to raw sugars containing sugars other than sucrose.

RECOMMENDATIONS.

It is recommended—

(1) That the following recommendations left over by the former associate referee be referred to the Carbohydrate Laboratory of the Bureau of Chemistry for study:

(*a*) Further study upon the modification proposed in 1916 for determining sucrose by acid and invertase inversion.

(*b*) Further study for the determination of small amounts of reducing sugars in the presence of sucrose.

(*c*) Further study upon the optical methods of estimating raffinose in beet products, using enzyms for the hydrolysis.

(2) That the methods of determining copper by reduction of the oxid in alcoholic vapors be further studied.

(3) That under the heading in the official methods, "Preparation of Sample.—Tentative." **IX**, Saccharine Products[1], the following be added:

(**d**) *Raw sugars.*—Mix thoroughly on a glass plate in the shortest possible time with spatula and glass or iron rolling pin in case of lumps, or in a large clean dry mortar.

(4) That Browne's temperature formula for correcting the polarization of raw cane sugars to that of 20° be adopted:

$$P^{20^\circ} = P^t + 0.0015\ (P^t - 80)\ (t^\circ - 20)$$

but where the percentage of levulose is actually determined, use the formula

$$P^{20^\circ} = P^t + 0.0003^\circ S\ (t - 20) - 0.00812^\circ L\ (t - 20).$$

(5) That the question of the adoption of the Baumé scale of the Bureau of Standards in place of the one now in use be further considered, possibly by a committee.

(6) That where standard quartz plates for the German or Ventzke scale are sent for certification, the Bureau of Standards be requested to certify on the old value of 100°V. = 34.657° (circular degrees) for sodium light at 20°C. in place of their new value of 34.620° until this has been adopted internationally.

(7) That a committee be appointed to get in touch with the committee of three appointed by the American Chemical Society, upon an international normal sugar weight, and also to ascertain the views of all members of this association upon the adoption of an international standard normal weight of 20 grams.

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[1] *Assoc. Official Agr. Chemists. Methods*, 1916, 121.

DENSIMETRIC AND POLARISCOPIC STANDARDIZATION IN REFERENCE TO THE ASSOCIATE REFEREE'S REPORT ON SUGAR.

By FREDERICK BATES and R. F. JACKSON (Bureau of Standards, Washington, D. C.).

In view of the current situation relative to certain important scientific procedures in the sugar industry which are treated in the report of the associate referee on sugar, page 321, the writers desire to present the following comments:

BAUMÉ SCALE.

W. D. Horne (National Sugar Refining Company, Yonkers, N. Y.), at the thirty-third annual convention of the association, asked for an investigation and report upon a number of points. Among them was the following[1]: "That the Bureau of Standards be asked to certify to the most advisable Baumé scale and that it be adopted". Relative thereto, the associate referee advises as follows: "The Baumé scale should be relegated to the shelf and only the Brix scale used. If there is real need for a Baumé scale, however, the one in general use should be that advocated by this association."

It so happened, that, simultaneously with Horne's recommendation, the Bureau of Standards, in response to frequent requests, had been preparing a Baumé scale to meet the very situation which was responsible for this recommendation[2].

It is unnecessary to enter into a discussion of the chaotic condition relative to the Baumé scale which existed prior to the advent of the Bureau of Standards scale. About twenty scales were in existence in various parts of the world, among which were two different scales published in the methods of this association[3]. None of these scales was based on accurate scientific data. The writers believe that it would be a step forward could the Baumé scale be abandoned. It is, however, still firmly entrenched, so far as practical use is concerned, and the only feasible plan which presented itself for its elimination was to educate the users to the advantages to be obtained by using the so-called Brix scale. With that end in view, the Bureau of Standards scale gives in parallel columns the value of each degree Baumé in terms of Brix. It is probable that the use of this scale will gradually result in the elimination of the term Baumé. The new Baumé scale has three features which should render its adoption a matter of routine:

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[1] *J. Assoc. Official Agr. Chemists*, 1919, **3**: 264.
[2] U. S. Bur. Standards Technologic Paper **115**: (1918).
[3] *Assoc. Official Agr. Chemists, Methods*, 1916, 124; U. S. Bur. Chem. Bull. **107**, rev.: 1910, 221.

(1) It is based upon the specific gravity values of Plato[1] which are considered the most reliable.

(2) It is based on 20°C., the most convenient and widely accepted temperature for sugar work.

(3) It is based on the modulus 145, which has already been adopted by the Manufacturing Chemists Association of the United States, by the Bureau of Standards, and by all American manufacturers of hydrometers.

Since the issuance of the new scale, it has come into general use and all spindles submitted to the Bureau of Standards for general scientific and sugar work are standardized upon it.

The writers would therefore recommend for your consideration the importance of the adoption of the new Bureau of Standards Baumé scale in accordance with the recommendation of Horne. It is felt that action upon this matter is of importance to this association and that the present unsatisfactory condition will continue indefinitely should the indefinite advice of the associate referee be adopted, namely, "If there is real need for a Baumé scale, however, the one in general use should be that advocated by this association".

NORMAL WEIGHT.

The associate referee's recommendations under this heading, page 324, constitute an argument in favor of changing the present international normal sugar weight of 26 grams to 20 grams. The suggested change emanated from conditions incidental to the war. It is perhaps unnecessary to repeat the well-known fact that for many years the best efforts of the leading sugar chemists of the world, especially at the meetings of the International Sugar Commission, have been directed towards securing an international normal sugar weight. The importance of international agreement upon this weight is obviously of the utmost importance. In order to obtain the present normal weight of 26 grams, the old so-called German weight of 26.048 was abandoned by the International Sugar Commission in favor of 26 grams, and at the same time the standard temperature of 20°C. was adopted. The advantages which have accrued to the sugar industry throughout the world because of the international scale have been tremendous. The 26-gram weight was adopted by practically the entire civilized world, the one exception being France, the latter nation refusing to change its normal weight of 16.29 grams. It is now stated, upon whose authority it is not known, that the French chemists are willing to accept 20 grams, providing it is made international, but recently E. Saillard[2], a French sugar chemist of international

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[1] *Wiss. Abh. der Kaiserlichen Normal-Eichungs-Kommission*, 1900, **2**: 153.

[2] *J. fabr. sucre*, 1919, **60**: No. 13.

reputation, has published an article in which he opposes both the 20- and 26-gram normal weight in favor of 16.29 grams. The inconvenience to the sugar industry of practically the entire world would be very great, indeed, should such a radical change be adopted in the normal weight as that advocated by the associate referee. It is believed that the sugar companies would continue to use the present international normal weight.

The associate referee states that a committee has been appointed by the American Chemical Society to get in touch with the various governments abroad with a view to arranging an international standard for the polariscope. The writers believe that the associate referee is somewhat in error in this statement in that this committee was appointed merely to make inquiries as to whether certain European nations, especially England and France, would be willing to adopt a 20-gram normal weight. The writers do not believe that the American Chemical Society has delegated authority to any committee to get in touch with the various governments abroad with a view to arranging an international standard for the polariscope. The associate referee's suggestion that a committee be appointed from this association to get in touch with the committee from the American Chemical Society and with the members of this association on the question of the adoption of the new 20-gram standard, providing it can be made international, is practically equivalent to committing this association to the adoption of the 20-gram weight, and the writers wish to protest against committing this association in any such manner.

STANDARDIZATION OF QUARTZ PLATES BY THE BUREAU OF STANDARDS.

The associate referee asserts, page 326, that the application of the new Bureau of Standards value of quartz plates may cause the introduction of scale errors. This conclusion results from a misconception of the proper method of applying corrections. He suggests two methods of correcting for scale errors. In the first, he adjusts the zero at zero and then determines the deviation of the scale from the certified value of the plate to obtain the correction. If this correction is applied proportionately to the observed rotation, no discrepancy can result. There is therefore no error to be multiplied.

In the second method, he adjusts the instrument at the 100° point to accord with the certified value of the plate. He then concludes that readings in the vicinity of zero will be in error by 0.105 and at −30 by 0.14. In other words, he advocates the curious procedure of determining the scale errors by means of a certified plate and then deliberately neglecting to apply the corrections thus ascertained. He further concludes that polariscopes equipped with a Reichsanstalt scale should be controlled by the incorrect Reichsanstalt standard in order that the plates shall agree with the instrument scales. In answer to this, we submit the obvious fact that the control plate should be used to standardize the scale and not the reverse.

The associate referee further remarks that most sugar chemists who have gone into the subject are free to admit that the 100° point is low, but there is grave doubt whether it is as low as the Bureau of Standards makes it. The latter part of this assertion is evidently based upon his further statement that "The work upon which this value was founded has been severely criticised by sugar chemists abroad". The writers wish to enter a solemn protest against any such misstatement of facts. So far as our information goes, there has only been one statement which could be construed as a criticism of the work of the Bureau of Standards in determining the 100° point of the saccharimeter. The criticism referred to is a statement by A. Herzfeld[1] (Institut für Zuckerindustrie, Amrumstrasse, Berlin, N. 65, Germany), who was the joint collaborator of Otto Schönrock (Physikalisch-Technische Reichsanstalt, Berlin, N. W., Germany) in determining the previously accepted value of the 100° point. The so-called criticism is approximately 150 words in length and is confined to a short discussion of several minor points involved in the preparation of the sugar used by the Bureau of Standards. Herzfeld merely conveys the idea of a possibility of the existence of a slight error due to these causes. He does not furnish the slightest proof that any error was made. On the contrary, all measurements and all contributory data developed since the determination of the constant by the Bureau of Standards have merely served to verify the correctness of the new value. Fortunately, or unfortunately, depending upon the point of view taken, the correction of the error in the old value of the 100° point operates in favor of the producers of sugar and against the buyers of sugar. For many years the buyers of raw sugars have had the advantage of a lowering of the test of over 0.°1S.

In view of these facts, the writers desire to advise against the adoption by this association of the associate referee's recommendation that members sending quartz plates to the Bureau of Standards for standardization request that the Bureau of Standards certify on the old value of the 100° point in place of the present corrected value until this has been adopted internationally.

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[1] *Z. Ver. Zuckerind.*, 1917, **67**: 407.

THE ATTITUDE OF THE NEW YORK SUGAR TRADE UPON THE NEW BUREAU OF STANDARDS VALUE FOR STANDARDIZING SACCHARIMETERS[1].

By C. A. Browne (New York Sugar Trade Laboratory, New York, N. Y.).

The question of the standardization value of normal quartz plates for saccharimeters, which A. H. Bryan discusses in his report, page 326, is a matter of extreme importance, as the difference of 0.1 sugar degree between the official standards of Berlin and Washington will mean a difference of over one million dollars in the valuation of sugars handled by the New York Sugar Trade. This conflict of opposing standards has already caused some uneasiness in the trade, and the matter has been carefully considered by the Directors of the New York Sugar Trade Laboratory, who represent equally the buyers and sellers of raw sugar.

The standard of graduation for the German or Ventzke scale which has been employed by the New York Sugar Trade up to the present time is the one which is being used by practically all members of the International Commission for Uniform Methods of Sugar Analysis, the methods of which are the official methods of the New York Sugar Trade. The Directors of the New York Sugar Trade Laboratory, after careful consideration of the question, voted unanimously against the adoption of the new Bureau of Standards value for standardizing saccharimeters for the following reasons:

(1) That the new Bureau of Standards value has been criticised by European investigators and until it has been confirmed by testing bureaus in other countries and has been agreed to internationally it would be exceedingly unwise to adopt a value which might have to be changed again to something else. The Directors of the New York Sugar Trade Laboratory do not wish to make any departure in methods or standards unless the proposed changes have a fair prospect of permanency, especially in international transactions, otherwise the sugar trade will be subjected continually to disturbances of this kind.

(2) Granting that there may be a slight error in the present German standard, and even granting that the new Bureau of Standards value may be correct, the Directors of the New York Sugar Trade Laboratory are of the opinion that no injustice is being done at present to the sellers of raw sugar for the reason that the minus error due to scale graduation is offset by an equal or greater error due to the volume of the lead precipitate in clarification.

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[1] Presented by G. L. Bidwell.

If a scale error exists it should by all means be corrected, and the true standardization value fixed by international agreement. When this scale error is corrected, the counter-balancing lead precipitate error should also be corrected either by dry lead defecation, as proposed by W. D. Horne, or by other accurate means.

I might say in conclusion that this decision of the Directors of the New York Sugar Trade Laboratory is in complete agreement with the opinion of Prinsen Geerligs of Holland and other European authorities.

COMMENTS ON RECOMMENDATIONS PROPOSED BY A. H. BRYAN.

By W. D. Horne (National Sugar Refining Company, Yonkers, N. Y.).

The following comments are presented relative to recommendations 3, 4, 5, and 6 of the associate referee on sugar, page 329:

(3) In mixing sugar samples, particular care should be taken to avoid excessive exposure to the air. Speed is of first importance. Avoidance of exposure of thin layers of sugar to the air may be helped by mixing in a mortar instead of on a plate.

(4) In cases where the invert sugar is known, more accurate results for individual samples may be obtained by using the formula

$$P^{20°} = P^{t} + 0.0003°S\,(t - 20) - 0.00812°L\,(t - 20)$$

where S = sucrose and L = levulose (one-half the invert sugar).

(5) The Baumé scale is so thoroughly entrenched in the industries that it will continue to be used and must be taken into consideration. The Bureau of Standards new scale and table is of particular value, having the modulus 145, which is close to the averages heretofore in use, is a round number and has already been accepted by the Manufacturing Chemists Association. The reference of sugar solutions at 20°C. to water at 20°C. is also a great advantage.

(6) The Bureau of Standards work on the 100° point of the polariscope was conducted with the utmost precaution and the one criticism of it is merely a suggestion that some inversion of the pure sucrose may have occurred. No evidence of such inversion is given, however, and it is not at all probable that it occurs, since the sugar solutions used never were heated above 35°C.

The government will doubtless continue to employ the corrective value and it is important that all remaining errors of polarization, especially elevation due to the volume of the lead precipitate, be eliminated as soon as possible.

A SUGGESTED MODIFICATION OF THE METHOD FOR CRUDE FIBER[1].

By L. D. HAIGH (University of Missouri, Columbia, Mo.), *Associate Referee on Crude Fiber in Foods and Feeding Stuffs.*

The study of the crude fiber determination this year has centered around the filtration operations. The treatment of feeds with dilute acid and alkali produces solutions which do not pass easily through ordinary filtering media. This has led to the use of muslin or linen as filtering media. The uncertainty that the cloth holds all the fine particles of fiber and the fact that no two chemists would be likely to use the same variety of cloth in filtering, renders this mode of filtering undesirable. Because of these reasons, the writer has preferred the one filtration method since it avoids one of these doubtful operations.

The associate referee has been unable to find a quick filtering medium which could be substituted for the muslin or linen cloth. The feeds most difficult to filter in the crude fiber determination are those of high protein content, such as linseed and cottonseed meal. If the protein in the feed could be dissolved without producing a colloidal solution and removed before the acid treatment, the filtration could be conducted without difficulty, using asbestos, thereby avoiding loss of fine material. To test this, experiments were conducted with various proteolytic enzyms. The best results were obtained with pepsin. It is, therefore, suggested that a preliminary treatment with pepsin be made in order to render the protein soluble. It may then be separated from the undissolved material by filtration through asbestos. Subsequent treatment of the residue with 1.25 per cent acid and alkali will not produce solutions which are difficult to filter, such as are obtained with the old procedure. The cloth filtration can then be abandoned and a filtering medium used, such as asbestos, which is more certain to retain all the insoluble material.

The time lost in trying to filter the colloidal solutions from the regular method is largely saved, even though another step, which adds about 40 minutes' work to the process, is introduced. The results should be entirely comparable with those obtained by the old method and have the additional advantage of greater accuracy.

INSTRUCTIONS TO COLLABORATORS.

Two samples were prepared—silage and cottonseed meal. Both samples had been washed with ether and brought to a stable, air-dry condition before they were distributed. The collaborators were asked to run

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[1] Presented by C. R. Moulton.

each of these samples as follows: (1) By the regular official method; (2) by the regular official method, preceded by a preliminary digestion at 40°C. for 30 minutes, with pepsin in the presence of dilute hydrochloric acid, after which the residue should be filtered, using an asbestos filter; (3) by the one filtration method, preceded by the above-mentioned digestion with pepsin; (4) in addition to the above, the acid filtrate from Method 2 was carried through the one filtration method to determine how much material precipitated from this acid solution by the alkali digestion remains to contribute to the result on crude fiber.

Some wide variations are observed in the results of the nine collaborators with the method using pepsin. The results, especially with the cottonseed meal, indicate to some extent a lack of uniformity in the samples. A uniform mixture of the ingredients of cottonseed meal is difficult to make and maintain. However, every reasonable effort was made to avoid this error.

A further experiment conducted in the writer's laboratory by W. S. Ritchie and T. H. Hopper is shown in the table as Method 5. It was thought possible that the hydrochloric acid washing of the residue could be omitted in the one filtration method when the preliminary digestion with pepsin was made.

Two of the collaborators reported that the solutions were difficult to filter where pepsin had been used. This variation may be due to some collaborators having used pepsin, which was weak or utterly useless.

The writer believes that in the removal of the protein from the feed before the digestion with acid, can be found the solution of the errors of the crude fiber method.

RECOMMENDATION.

It is recommended—

That Method 2 receive further study and that suggestions and criticism be solicited.

Loss on ignition (crude fiber) in sample of ether-washed silage.

ANALYST	METHOD 1	METHOD 2	METHOD 3	METHOD 4	METHOD 5
	per cent	*per cent*	*per cent*	*per cent*	*per cent*
L. D. Haigh, W. S. Ritchie and T. H. Hopper, University of Missouri, Columbia, Mo.	18.55	17.80	0.10	17.91	18.41
	18.35	17.95	0.13	18.32	18.47
Average	18.45	17.88	0.12	18.12	18.44
A. E. Smoll, Jacob Dold Packing Company, Wichita, Kans.	17.12	17.26	0.62	19.28	....
	17.23	17.27	0.46	19.55	....
Average	17.18	17.27	0.54	19.42	....
F. N. Smalley, Southern Cotton Oil Co., Savannah, Ga.	17.07	16.51	0.225	17.18	....
	18.04	16.25	0.235	17.18	....
	17.69	....	....	....	....
Average	17.60	16.38	0.23	17.18	....
J. M. Pickel, Department of Agriculture, Raleigh, N. C.	17.78	....	....	....	....
	18.01	....	....	....	....
Average	17.90	....	.....	....	....
S. H. Wilson and J. F. King, Department of Agriculture, Atlanta, Ga.	16.00	17.40	0.40	17.03	....
	16.15	17.30	0.37	....	....
Average	16.08	17.35	0.39	17.03	....
W. D. Richardson, Swift and Co., Chicago, Ill.	17.72	17.72	0.23	17.88	....
	17.50	17.85	0.21	17.87	....
	....	....	0.18	17.83	....
Average	17.61	17.79	0.21	17.86	....
W. F. Hand and L. B. Sandiford, Agricultural and Mechanical College, Agricultural College, Miss.	16.24	17.36	0.57	16.10	....
	17.06	16.55	0.06	17.76	....
	17.70	16.91	0.12	17.00	....
	17.01	17.35	....	....	....
Average	17.00	17.04	0.25	16.95	....
G. L. Bidwell and L. E. Bopst, Bureau of Chemistry, Washington, D. C. Average	17.67	17.34	0.15	17.01	....
H. B. McDonnell and L. H. Van Wormer, Agricultural Experiment Station, College Park, Md.	16.70	17.65	0.125	17.37	....
	17.05	17.55	0.15	17.20	....
Average	16.88	17.60	0.14	17.29	....

Loss on ignition (crude fiber) in sample of ether-washed cottonseed meal.

ANALYST	METHOD 1	METHOD 2	METHOD 3	METHOD 4	METHOD 5
	per cent	*per cent*	*per cent*	*per cent*	*per cent*
L. D. Haigh, W. S. Ritchie and T. H. Hopper	Rejected	16.62	0.19	16.43	16.10
	16.09	15.80	0.13	16.14	16.02
Average	16.09	16.21	0.16	16.29	16.06
A. E. Smoll	14.01	15.42	0.94	15.89	....
	14.13	15.40	0.97	15.62	....
Average	14.07	15.41	0.96	15.76	....
F. N. Smalley	14.00	15.58	0.12	15.23	....
	13.18	15.89	0.12	14.67	....
	13.22	....	....	....	....
Average	13.47	15.74	0.12	14.95	....
J. M. Pickel	15.63	....	....	....	....
	15.85	....	....	....	....
Average	15.74	....	....	....	....
S. H. Wilson and J. F. King	14.18	15.15	0.20	....	....
	14.40	14.83	0.40	....	....
Average	14.29	14.99	0.30	13.23	....
W. D. Richardson	15.39	14.85	0.24	15.32	....
	15.20	14.95	....	15.50	....
	15.15	14.93	....	15.43	....
Average	15.25	14.91	0.24	15.42	....
W. F. Hand and L. B. Sandiford	13.77	14.25	0.62	13.57	...
	13.30	14.38	0.07	13.40	....
	13.87	14.45	0.89	13.61	....
	13.64	14.70	0.86	14.06	....
	....	13.94	....	....	....
Average	13.65	14.34	0.61	13.66	....
G. L. Bidwell and L. E. Bopst. Average	15.47	15.77	0.10	14.92	....
H. B. McDonnell and L. H. Van Wormer	14.05	15.50	0.075	15.20	....
	14.35	15.15	0.10	15.40	....
Average	14.20	15.33	0.09	15.30	....

REPORT ON STOCK FEED ADULTERATION.

By B. H. SILBERBERG (Bureau of Chemistry, Washington, D. C.),
Associate Referee.

The phase of feed analysis which has been most prominent during the past year or two is that of the quantitative determination of percentage of ingredients, particularly of mixed feeds. It was therefore considered desirable to confine this year's work to investigations along this line. In this connection two samples were sent to the collaborators, one a cottonseed meal for determination of hulls, according to a method devised by the associate referee, and the other a molasses feed or horse feed.

PART I.—COTTONSEED MEAL.

This was in a sense a continuation of the work done two years ago on hull determination in cottonseed meal. The low results on hull content obtained by the collaborators at that time were thought to be largely due to the use of a rough mortar. The use of a glass mortar and pestle was therefore recommended and the directions were slightly revised. As stated in the last report[1], an ordinary commercial sample was used since it is impossible to simulate manufacturing processes under laboratory conditions. The exact hull content of the sample was therefore unknown. Of the sixteen collaborators some were using the method for almost the first time, while others had had quite a little experience with it. The results as shown by the table varied from 16.0 to 25.3 per cent of hulls. This, needless to say, indicates that the personal element introduces so large a factor of error as to make the method impracticable for general use. Probably the fact that the meal contained a great deal of lint made it more difficult to work, but it must be conceded that a method which is not adaptable to any type of sample is not worthy of general adoption.

One cause of the variation in results appears to be the method of drying the hulls after separation. Air drying to constant weight seems to be the most desirable method, but in some sections of the country where the humidity is high, this is too prolonged an operation. It is evident from the character of the results that if any method similar to this one is used, very definite instructions which will take into consideration the varying atmospheric conditions in different parts of the country must be given.

Two of the collaborators each suggested another method. One was based on the determination of the crude fiber figure. The other method

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **3**: 41.

is one which involves the actual separation of the hulls from the meal and sounds very promising. It is to be hoped that during the next year these methods and possibly others may be given a thorough trial.

PART II.—HORSE FEED.

This particular type of molasses feed was chosen for two reasons: (1) Because it is probably the commonest type of mixed feed; and (2) because it is the easiest type in which to identify the ingredients and possibly to determine their approximate percentages. At any rate, if the percentage of ingredients in such a sample could not be determined with a fair degree of accuracy it would be reasonable to assume that it would be an impossibility to do so in almost any other kind of mixed feed. In fact, one of the collaborators protested against the use of this kind of feed as a basis for conclusions on this subject, claiming that it was much too easily analyzed to be representative of the average type of mixed feed presented to the analyst. Nevertheless, this analyst failed to find two of the ingredients most easily identified microscopically, and reported twice as much of one of the others as was actually present. The principal ingredients were ordinary commercial products purchased from feed stores. The cracked corn was fairly clean and was a mixture of white and yellow corn; the oats were practically free from weed seeds but contained a slight trace of barley; the alfalfa was an ordinary sample of meal of good color; the cottonseed meal was of prime quality (39.8 per cent of protein); the cottonseed hulls were ground delinted hulls; the peanut shells were ground to pass through a 1 mm. (20 mesh) sieve; and the molasses was cane, commonly called blackstrap. The chemical analysis was worked out, according to the ingredients and amounts present, from Henry and Morrison's "Feeds and Feeding"[1] and the guaranty based thereon.

The following information was given the collaborators upon which to base their identification of ingredients and their approximate percentages:

This represents an adulterated molasses feed with the following chemical guaranty and declared list of ingredients:

CHEMICAL GUARANTY		INGREDIENTS
	per cent	
Protein	12	Corn, oats, barley, alfalfa, cottonseed meal, and molasses.
Fat	3	
Crude fiber	20	

This is very similar to samples which have come under the associate referee's notice.

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[1] W. A. Henry and F. B. Morrison. Feeds and Feeding. 16th ed., 1916.

The ingredients were mixed in the customary manner employed in mixed feed plants; that is, the warm molasses was mixed with the dusty, dry ingredients and the grains added afterwards. Each ingredient was carefully weighed and an exact percentage added. The feed was kept thoroughly mixed in order that each subsample should be uniform.

The reports of the collaborators showed an eager and earnest spirit of cooperation, and without question represented the best efforts of feed analysts from various parts of the country who are considered most active and competent in this field of work. The results are therefore of particular interest. The feed contained 20 per cent of corn; the results of the collaborators varied from 11 to 21.5 per cent. There was 15 per cent of oats present; reports varied from 10 to 19 per cent, although three reported the oats correctly. No barley was added, but a slight trace was present as a contamination of the oats. It is more than likely that some subsamples contained no barley. The highest amount reported was 0.9 per cent. Twenty-five per cent of alfalfa was present; the reports varied from 24.6 to 35 per cent, although all but the one high one were remarkably close. The amount of cottonseed meal added was 10 per cent; only two of the collaborators found any cottonseed meal, one reporting 4 per cent and the other 35.9 per cent. There was also 10 per cent of cottonseed hulls used in addition to the meal; reports on these ranged from none to 30.3 per cent, all but the one reporting 20 per cent or over. Five per cent of peanut shells were added, and not one of the collaborators discovered them. This is of special interest in view of the fact that one of the proposed laws relative to mixed feeds contained a clause providing that when 5 per cent or over of a low-grade ingredient was used, the percentage present should be stated on the label. The amount of molasses present was 15 per cent; reports ranged from 2 to 25 per cent.

These results speak for themselves, but it is impossible to refrain from a few comments. In the first place, one surprising and outstanding fact is that in practically every case the work, even the identification of the ingredients, was done by chemists instead of microscopists and apparently without the use of a microscope. Of course there is nothing to prevent any one with chemical training from also taking special training in plant histology, including microscopy. But the results show how utterly out of the question it is for one without such special training, no matter how good a chemist he may be, to do the microscopic work, that is, the identification of ingredients, on feeds. There is no one ingredient of feeds which is more easily identified microscopically than cottonseed meal. A few years ago the writer showed an analyst how to identify cottonseed meal in a mixed feed and he afterwards identified it positively in a feed which contained less than a hundredth of a per cent. This horse feed contained at least 7 or 8 per cent of hull-free cottonseed meal and yet only two analysts found any present, and two of the others specifically stated in their reports that they saw no evidence of the presence of cottonseed meal. While 5 per cent of some ingredients in certain mixtures would be difficult to identify, even for a microscopist, there are at least three distinctive types of tissue which would enable the plant histologist to identify peanut shells.

This whole question is neither original nor new, nor is it the first time it has been presented at a scientific meeting. A. L. Winton says[1], "Without a certain amount of botanical training, * * * * a chemist is no more fitted to take up microscopical analysis than a botanist without chemical training is fitted to work at quantitative analysis".

The inevitable conclusion is that unless a prodigy can be found who has been trained both as a chemist and a plant histologist or microscopist, the chemist and microscopist must work together for efficient feed analysis, for one can not do the work of the other.

With regard to the work for the ensuing year, it seems advisable that, in view of the suggestions made by collaborators, the work on hull determination in cottonseed meal be continued. A few years ago, work was begun on methods of sampling scratch feed containing grit or shell. The results so far obtained show that the methods of mixing commonly used do not insure a homogeneous sample. A method which will do this is most necessary, and it is suggested that work along this line be continued. Several years ago it was recommended that a key or outline

TABLE 1.

Amount of hulls found in cottonseed meal by different collaborators.

ANALYST	PERCENTAGE OF HULLS REPORTED SHOWING NUMBER OF DETERMINATIONS MADE BY EACH ANALYST					AVERAGE
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
A	22.0	21.8	....	....	....	21.9
B	24.4	24.8	21.0	22.4	....	23.15
C	16.2	15.9	16.4	15.5	....	16.0
D	24.6	24.0	....	....	....	24.3
E	18.3	18.2	18.3	....	....	18.3
F	20.4	21.3	....	....	....	20.85
G	19.3	19.2	....	....	....	19.25
H	20.7	19.4	20.5	....	....	20.2
I	16.1	17.8	16.3	....	....	16.7
K	19.2	18.6	19.3	18.9	19.4	19.1
L	21.7	23.5	23.9	....	....	23.0
M	23.2	20.8	....	....	....	22.0
N	20.0	....	....	....	....	20.0
O	23.3	....	....	....	....	23.3
P	20.2	....	....	....	....	20.2
Q	25.8	24.8	....	....	....	25.3

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[1] Proc. Eighth Intern. Cong. Appl. Chem., 1912, **18**: 361; *Am. J. Pharm.*, 1913, **85**: 132.

for the qualitative detection of various products used in feeds be presented. Such an outline has been prepared by the associate referee for the Information Sheets of the Bureau of Chemistry on the Microscopic Examination of Feeds and Feeding Stuffs and may be had on application to the associate referee.

TABLE 2.

Amount of different ingredients found in a mixed feed by different collaborators.

INGREDIENTS	PERCENTAGES PRESENT	PERCENTAGES REPORTED BY ANALYSTS						
		A	B	C	D	E	F	G
Corn................	20	15	11.4 10.4	14.8	19	21.5	20	21.3
Oats................	15	15	10.0 11.8	11.7	19	18.9	15	15.9
Barley..............	Trace; contamination of oats	Trace	0.9	0.3	..	....	..	0.0
Alfalfa..............	25	25	28.7 27.0	27.8	26	24.6	35	24.9
Cottonseed meal.....	10	..	0.0 0.0	0.0	0	..	4	35.9
Cottonseed hulls......	10	20	28.9 24.7	19.9	20	30.3	20	....
Peanut shells.........	5	..	....	....	..	....	..	....
Molasses............	15	25	21.5 24.1	24.0	16	4.7	6	2.0

No report on organic and inorganic phosphorus in foods and feeding stuffs was made by the associate referee.

REPORT ON WATER IN FOODS AND FEEDING STUFFS[1].

By J. O. CLARKE (U. S. Food and Drug Inspection Station, U. S. Custom House, Savannah, Ga.), *Associate Referee.*

In food control work a number of different procedures are used for the determination of moisture or water, the method employed depending on the nature of the material and the equipment of the laboratory. These methods can be classified as heating, desiccator, refractometer,

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[1] Presented by G. L. Bidwell.

densimetric, or other procedures. The temperature, pressure, type of apparatus, and other factors differ in each class. It is not surprising, therefore, that the determination of water is quite variable and that it is next to impossible for two laboratories to obtain good checks.

As ordinarily determined, the water or moisture in food products is derived from several sources, the principal ones being loosely bound moisture, water of constitution, and water derived from the decomposition of organic materials. If the moisture is determined by difference, low boiling and gaseous substances other than water will be lost. In the process of drying, other changes, more or less pronounced, always take place. Certain constituents may be oxidized, as in linseed meal, or other substances containing a drying oil. Many feed materials lose weight on heating until a certain minimum weight is obtained, when they begin to increase in weight.

As ordinarily used, the term "per cent of moisture", or "per cent of water" in a food material is intended to mean the loosely bound water in the material, or, in substances in which the water occurs as a solvent, sirups, etc., the actual uncombined water present. It is not possible in ordinary work to determine this water without including water from other sources or other volatile materials. For the purposes of this association, a few well-selected methods are needed which will give a consistent measure of the moisture in such substances as feed materials, and a measure of the actual water in such substances as sirup. Such selected methods should give comparative results when used with care with the equipment found in the ordinary laboratory.

The official methods of the association for this determination vary, depending on the nature of the product. Under Foods and Feeding Stuffs[1], two methods appear, one calling for drying at the temperature of boiling water in vacuo, or in a current of dry hydrogen, and the other for drying to constant weight in a vacuum over sulphuric acid. Under "Saccharine Products"[2] a number of other methods appear, which depend on various well-known principles. Ordinarily, throughout the published methods, the determination of water in any class of products is referred to one of these general chapters. On feed materials, both official methods give about the same results in the writer's hands. Some other laboratories get a little higher result with one method than with the other. Neither method in its present form can be depended upon to give concordant results in the hands of different operators.

It is quite possible that the treatment in vacuo at the temperature of boiling water is too strenuous to remove the moisture from some materials without other changes being too pronounced. It may be a better

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 79.

[2] *Ibid.*, 121.

procedure to dry such material at a lower temperature in vacuo. The statement "in a current of dry hydrogen or in vacuo" is extremely vague, and it is not probable that concordant results could be obtained if these instructions were followed. The simple specification "in vacuo" may mean less than 1 mm., or 70 mm. or more absolute pressure. It is suggested that the succeeding associate referee work out the best temperature, pressure, and other conditions to be used in this method, and so modify the wording of the method that the conditions will be exactly the same in any laboratory where the method is used. If it appears advisable to use dry hydrogen for some materials, a distinct method should be adopted for such materials to be carried out under rigidly specified conditions.

For several years the associate referees on water in foods and feeding stuffs have devoted most of their energies to a study of desiccator reagents. As a result of this work, one official method calling for the use of sulphuric acid in a vacuum desiccator has been adopted by the association. A large number of common desiccator reagents have been tried, but sulphuric acid is perhaps the best. The details of this method as published are fairly complete. Some attention should be given to the length of time before drying is considered complete. Weighing every 24 hours to constant weight is not satisfactory, and is very tedious and time consuming. It has been the writer's experience that a single fairly long drying period, two or three days, is sufficient to desiccate most substances. Specific instructions should be included regarding the preparation of the reagent.

Under "Fruits and Fruit Products", a method is given for "Total Solids"[1]. While this method can possibly be stretched to cover dried fruit products, it would simplify matters greatly to adopt a method especially designed for the determination of water in dried fruits.

RECOMMENDATIONS.

It is recommended—

(1) That further work on desiccator reagents, other than sulphuric acid, be discontinued.

(2) That the associate referee study the existing official general methods for water in foods and feeding stuffs with a view to rewording and fixing rigidly the conditions of temperature, pressure and other factors.

(3) That a definite method applicable to the determination of water in dried fruits be designed and submitted to the association.

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 177.

THE DETERMINATION OF WATER IN CEREAL AND MEAT PRODUCTS.

A Comparison of Results in a Vacuum Desiccator and in a Vacuum Oven at 65°C.

By F. C. Cook (Bureau of Chemistry, Washington, D. C.).

INTRODUCTION.

The determination of moisture or loss of weight in different materials on heating at various temperatures or treatment under various conditions is one of the simplest and most important determinations made by the chemist. It is a determination subject to great variation, depending on the method used and the material employed.

Many moisture determinations are made by heating the samples in a vacuum oven at 65°C. until no further loss of weight results. Another common procedure is to heat the sample at the temperature of boiling water until constant weight is obtained. With certain classes of products the method of Benedict and Manning[1], using a vacuum desiccator and no heat, is recommended. The fact that several days are required to insure complete loss of moisture by this method makes it inapplicable in many cases. Numerous other procedures for the determination of moisture, such as the calcium carbide method[2], and the method of Brown and Duvel[3], which is used for the determination of water in uncooked cereals, have been employed.

COMPARISON OF METHODS.

Two methods of determining moisture were employed in this study, viz, the vacuum desiccator method and heating in a vacuum oven at 65°C. The materials used were samples of fish, veal, meat extracts, finely divided malted cereal preparations, and malted milks. The cereal products contained large amounts of either starch or sugars. These samples were kept in Mason jars with rubbers and tops. Two grams of the finely divided samples, placed in aluminium dishes 2 inches in diameter with tightly fitting covers, were taken for the determinations by each method. The samples for the two sets of determinations were weighed at the same time in order to equalize conditions as far as practicable.

The vacuum oven was held at a temperature of 65°C. and a vacuum of 24 inches was employed. The samples were allowed to remain in the oven for 14 hours, when the covers were placed on the dishes and they

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[1] *Am. J. Physiol.*, 1905, **13**: 309.
[2] U. S. Bur. Chem. Circ. **97**: (1912).
[3] U. S. Bur. Plant Ind. Bull. **99**: (1907).

were transferred to a desiccator before weighing. It was found that in most cases no additional loss of weight resulted on leaving the samples in the oven more than 14 hours.

The desiccators employed in the vacuum desiccator method were of the Scheibler form, 6 inches in diameter and of strong glass with a rubber stopper carrying the stop-cock wired and sealed in place. About 150 cc. of sulphuric acid were placed in the bottom of each desiccator and an inverted glass stopper of the hollow type containing 10 cc. of absolute ether was placed in the acid below the rack which held the aluminium dishes. The samples, also a mercury manometer, were placed on the racks of the desiccators and 15 minutes after connecting the house vacuum system to the desiccators all the air was removed from the desiccators. The vacuum was made complete, as shown by the manometer in each desiccator, by carefully shaking the desiccators to insure the absorption of the remaining traces of the ether fumes by the sulphuric acid. In order to secure rapid absorption of moisture, the desiccators were shaken every few hours to break the film of moisture which settled on the surface of the sulphuric acid. The samples were allowed to remain in the desiccators for 5 days when they were weighed and replaced in the desiccators for an additional period of 48 hours. In practically all cases, the first 5-day period was sufficient to remove all moisture that could be removed by this method.

COMPARISON OF RESULTS.

In Table 1, results for moisture in powdered malted cereal preparations are given by the two methods. The samples which were commercial products consisted largely of sugars or starch and protein with a little fat.

TABLE 1.

Moisture in powdered malted cereal preparations.

SAMPLE NUMBER	DESCRIPTION OF MATERIAL	VACUUM DESICCATOR METHOD	VACUUM OVEN 65°C.
		per cent	*per cent*
1	High in sugars, low in starch	1.93	2.42
2	Low in sugars, high in starch	6.53	5.99
3	Low in sugars, high in starch	1.66	1.50
4	High in sugars, low in starch	3.18	3.37
5	High in sugars, low in starch	1.76	2.05
6	High in sugars, low in starch	2.15	2.50
7	High in sugars, low in starch	2.29	2.74
8	High in sugars, low in starch	2.50	2.60
9	Low in sugars, high in starch	9.01	8.20
10	Low in sugars, high in starch	7.83	6.81
11	Low in sugars, high in starch	6.66	4.80
12	Low in sugars, high in starch	5.89	5.15
13	Low in sugars, high in starch	7.70	6.91

Samples 1, 4, 5, 6, 7 and 8 were composed of a large percentage of sugars and contained but little starch. These samples gave a larger loss of weight (moisture) on heating in the vacuum oven at 65°C. than in the vacuum desiccator. The other samples which contained large amounts of starch and but little sugars gave a greater loss of weight (moisture) in the vacuum desiccator than in the vacuum oven at 65°C.

Samples 1, 2, 3 and 4 were heated an additional 4 hours in the vacuum oven at 100°C. with the following results:

TABLE 2.

Samples used for results in Table 1 heated 4 hours additional in vacuum oven at 100°C.

SAMPLE NUMBER	DESCRIPTION OF MATERIAL	MOISTURE
		per cent
1	High in sugars, low in starch	3.48
2	Low in sugars, high in starch	7.65
3	Low in sugars, high in starch	2.43
4	High in sugars, low in starch	4.85

These samples were then returned to the vacuum oven for 6 hours' additional heating at 100°C. Samples 2 and 3 showed an increase of weight while Samples 1 and 4, which were low in starch and high in sugars, did not show an increase of weight.

TABLE 3.

Moisture in malted milk products.

SAMPLE NUMBER	VACUUM DESICCATOR METHOD	VACUUM OVEN 65°C.
	per cent	*per cent*
1	1.76	2.05
2	2.15	2.50
3	2.29	2.74
4	2.50	2.60

In Table 3, similar results for moisture determined by the two methods on four samples of malted milks are given. These products contained a large amount of sugars, dextrins, etc., and practically no starch. In all four samples a greater loss of weight resulted on heating in the vacuum oven at 65°C. for 14 hours than in the vacuum desiccator method.

The samples which had been in the vacuum desiccator were placed in an oven at 100°C. for 4 hours and the samples which had been heated in the vacuum oven at 65°C. were returned to the same oven for 4 hours' additional heating. The results are as follows:

TABLE 4.

Moisture results of additional heating of samples.

SAMPLE NUMBER	OVEN 100°C.	VACUUM OVEN 65°C.
	per cent	*per cent*
1	2.58	2.75
2	3.18	3.24
3	3.36	3.50
4	2.89	3.72

While the samples heated at 65°C. all gave higher results, there was but little difference except in the case of Sample 4.

The samples, the analyses of which are reported in the foregoing tables, contained such a low percentage of moisture that the influence of the various conditions of the methods employed may be magnified in the results, and, therefore, appear to a greater extent than with samples higher in moisture. Samples of practically identical moisture content and containing both sugars and starch, and starch products in considerable amounts, do not show these differences in moisture results (loss of weight) by the two methods. There is no certainty that the higher figures are the correct moisture results in any of the samples tested.

The results obtained by determining the loss of weight or moisture in pure proteins, fat, starch and various sugars can hardly be applied to these products, since they are complicated mixtures of these substances, and are the result of various methods of manufacture. The carbohydrate composition of the cereal products tested showed a greater influence on the moisture results (loss of weight) than the moisture methods employed.

In Table 5 results are given by the two methods for moisture in veal, fish and meat extracts. With veal and fish, the results were practically

TABLE 5.

Moisture in meat, fish and meat extracts.

SAMPLE NUMBER	DESCRIPTION OF MATERIAL	VACUUM DESICCATOR METHOD	VACUUM OVEN 65°C.
		per cent	*per cent*
1	Veal	75.62	75.57
2	Veal	73.79	74.50
3	Mackerel	75.73	73.39
4	Butterfish	77.13	77.69
5	Meat extract	11.26	10.45
6	Meat extract	18.23	13.63*

* Not to constant weight.

identical by the two methods, while a greater loss of weight resulted with meat extracts by the vacuum desiccator method, than on heating at 65°C. in the vacuum oven.

Meat and fish are complex organic substances containing protein, fat and only a little carbohydrate. The fact that identical moisture results were obtained by the two methods may be explained on the basis of the low carbohydrate content of the veal and fish. The high percentage of moisture, viz. 75 per cent, masked any variations which were caused by the small percentage of carbohydrates present. In the case of meat extracts, Table 5, the physical character of the samples played an important role, as the large expansion of the samples which took place in the vacuum desiccator facilitated the removal of the moisture and, therefore, may explain the higher results obtained in the desiccator method.

SUMMARY.

A difference in the moisture content (loss of weight) of malted cereal preparations high in starch and similar preparations high in sugars by the vacuum desiccator method and by the method of heating at 65°C. for 14 hours in a vacuum oven was obtained.

Those samples high in starch gave lower moisture results and those high in sugars gave higher results on heating in a vacuum oven at 65°C. for 14 hours than by the vacuum desiccator method.

With meat and fish, which are practically carbohydrate free, identical results were obtained by the two methods. Meat extracts gave a higher moisture result (loss of weight) in the vacuum desiccator than in the vacuum oven at 65°C.

COMMERCIAL FEEDING STUFFS.

By A. McGill (Department of Health, Ottawa, Canada).

So far as Canada is concerned the Commercial Feeding Stuffs Act of 1909[1] has for its object such control of all feeds for cattle as shall assure, to the purchaser, a determinate minimum food value in the article which he buys. This, of course, assumes that he buys intelligently, *i. e.*, that he knows what he wants, and demands this under a definite name, understood by all the parties to the transaction. Feeds for farm stock are either subject to legal inspection, or are permitted to be sold without inspection.

Hay, straw, roots, wet brewers' grains, as well as oats, barley, corn, etc., in the unground state, are not subject to any official inspection.

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[1] Statutes of Canada, 1909, ch. 15.

The reason is clear. These articles are, from their nature, as readily judged for quality by the ordinary purchaser as they could be by a government official.

All other feeds are subject to inspection for the equally intelligible reason that their value is neither readily nor certainly appreciable to the purchaser. Indeed, the only way in which the superiority of one article of bran or chop feed over another article of like species can be found, without chemical analysis, is by actually feeding the material and observing the results. Feeds which are subject to inspection are again divided into two classes: Those which may be sold without registration; and those which must be registered before being legally offered for sale.

Bran, shorts (middlings) and chop feeds are not required to bear any registration number, or any guaranty of their nutritive value. This is because the terms bran, shorts and chop feeds are legally defined. The feeds in question are required to possess the subjoined percentage values:

Standards for bran, shorts and chop feeds.

SUBSTANCE	PROTEIN (minima)	FAT (minima)	FIBER (maxima)
	per cent	*per cent*	*per cent*
Bran	14	3	10
Shorts (middlings)	15	4	8
Chop feed	10	2	10

The purchaser of any one of these feeds is assured of obtaining a nutritive value at least equal to the value above given.

There is, however, nothing in the act which prevents a manufacturer of bran, shorts, or chop from registering his product, and from guaranteeing a higher value for it than the minimum value required by the act. Should a manufacturer of bran, etc., elect to register and to guarantee a value higher than the minimum which the act requires for such class of feed, it is incumbent upon such manufacturer to live up to the guaranty set by himself. If, for example, he chooses to warrant his bran as containing 16 per cent of protein, and 5 per cent of fat, but puts on the market an article containing only 14 per cent of protein and 3 per cent of fat, such article will be adulterated, under the act, although it meets the minimum requirements for legal bran.

All feeds other than those described above must be registered before being offered for sale. The registration number must be affixed to every package offered for sale, and constitutes identification of the brand. Every registration number corresponds to a definite brand of feed, whose minimum value in nutrient matter is guaranteed by the manufacturer and is on file in this department. The guaranteed value must also appear on every package offered for sale. The purchaser knows what value he is getting when he buys a registered feed.

It is the duty of this department to inspect all feeds on the market, except such as are specifically exempt from inspection. Experience has shown that there are on the market certain feeds which may not be classed under any of the headings given, without injustice to the manufacturer and misunderstanding by the purchaser. Thus, in reporting an inspection of 396 samples of feeds in 1913, I wrote as follows[1]:

> A considerable number of samples, which appear to answer a description of low grade flour, were found in the above collection; but these were supplied indiscriminately as shorts or middlings, as indicated by the maxima for fiber and for fat, though most of them were sold as middlings.
>
> In order to discover whether the present collection indicates any accepted distinction between the terms, I have collated the results for 72 out of 73 samples sold as shorts, and for 54 out of 68 samples sold as middlings, being, in each case, the number of samples, respectively, which meet the requirements of legal definition.
>
> Seventy-two samples of shorts give results as follows:
>
> *Shorts—average values.*
>
> | | FAT | PROTEIN | FIBER |
> |---|---|---|---|
> | | *per cent* | *per cent* | *per cent* |
> | Means | 4.65 | 16.20 | 6.3 |
> | Maxima | 6.48 | 18.46 | 8.0 |
> | Minima | 3.44 | 14.00 | 3.9 |
>
> Fifty-four samples of middlings give the following results:
>
> *Middlings—average values.*
>
> | | FAT | PROTEIN | FIBER |
> |---|---|---|---|
> | | *per cent* | *per cent* | *per cent* |
> | Means | 4.47 | 15.95 | 5.2 |
> | Maxima | 6.26 | 18.02 | 8.1 |
> | Minima | 3.02 | 14.13 | 1.4 |
>
> The results appear to justify the conclusion that the article sold as shorts is not substantially different from the article sold as middlings; and this conclusion is corroborated by the expressed opinion of many millers, in answer to questions by our inspectors. At the same time, it is to be noted that the samples characterized by very low fiber, low fat and a high starch content (indicating flour) are usually sold as middlings, rather than as shorts.
>
> Examples of this are found in the following:

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[1] Can. Lab. Inland Rev. Dept. Bull. 254: (1913), 6.

Feed flour—illustrations.

NUMBER	FAT	PROTEIN	FIBER	REMARKS
	per cent	*per cent*	*per cent*	
46084	2.16	10.37	1.5	Low in proteids and fiber.
36956	1.55	12.73	0.7	Low in proteids, fat and fiber.
43271	2.08	12.51	0.8	Low in fat, fiber and proteids.
43821	2.32	13.26	1.3	Low in proteids, fat and fiber.
47413	2.66	15.11	1.9	Low in fat and fiber.
47414	3.16	12.56	1.8	Low in proteids and fiber.
38185	2.66	11.42	0.9	Low in proteids, fat and fiber.
38189	2.02	12.03	0.5	Low in proteids, fat and fiber.
47611	2.37	14.61	1.1	Low in fat and fiber.
47618	1.98	13.04	0.8	Low in fat and fiber.
47621	2.28	12.86	1.0	Low in fat and fiber.

Feeds of this kind should be sold as feed flour, or by some name which would distinguish them from legal shorts or middlings. While they possess considerable value for feeding purposes, they must be regarded as adulterated under the act when offered as shorts or middlings. Their low fiber content is an advantage from the point of view of nutritive value, since this is replaced by starch in amount equivalent to the difference between the legal maximum for fiber (8 per cent) and the actual fiber present.

The protein value of this article is in many cases quite as high as that of shorts or middlings, but its fat value is usually distinctly lower, owing to more complete removal of the germ of the wheat.

It is matter for consideration whether this description of feed should not be sold under the name "Feed Flour".

It is desirable, in order to secure efficient administration of the act in question, that there should be no misunderstanding of the terms used in naming these feeds. The term "bran" (French, *son* or *son sec*) seems to be well understood. It is a product of the milling of a single grain, and is required to contain not less than 14 per cent of protein, nor less than 3 per cent of fat, and not more than 10 per cent of crude fiber.

Shorts and middlings are at present held to be synonymous, at least as far as feeding value is concerned. Some objection has been taken to this classification, but the recorded analyses of numerous market samples seem to indicate its justification. The French *recoupe*, *gru blanc*, *remoulage*, and *son gras* are believed to be equivalents for middlings, while *son fin*, *petit son*, and *gru rouge* are taken as equivalents for shorts. It is much to be desired, however, that explicit definition and identity of synonyms should be legally decreed.

In order to clearly show the difference between feed flour and shorts (or middlings) proper, I may cite the following examples:

Types of feed flours.

SOURCE OF INFORMATION	PROTEIN	FAT	FIBER
	per cent	*per cent*	*per cent*
From Bulletin 231*:			
46084	11.20	2.16	1.5
36956	12.73	1.55	0.7
43271	12.51	2.08	0.8
43281	13.26	2.32	1.3
28512	14.61	4.64	1.5
47413	15.11	2.66	1.9
47414	13.60	3.16	1.8
43866	15.20	3.40	1.8
48415	14.83	3.32	1.4
38185	11.42	1.99	0.9
38186	15.79	3.08	1.8
38188	14.13	2.82	2.6
38189	12.03	1.81	0.5
47611	14.61	2.37	1.1
47618	13.04	1.98	0.8
47621	13.71	2.28	1.0
Means	13.61	2.60	1.3
From Bulletin 254†:			
39160	15.71	3.50	1.3
50947	14.00	2.54	2.3
49676	13.78	2.04	1.4
Means	14.50	2.69	1.7
From Bulletin 311‡:			
63526	16.55	4.60	1.3
61593	16.75	3.34	0.8
52047	13.25	2.74	0.9
55126	16.85	3.06	0.7
55127	17.65	3.42	1.8
55130	18.20	2.99	0.7
55131	16.60	3.04	1.7
55135	15.60	3.96	0.9
62033	17.00	3.04	0.9
Means	16.49	3.35	1.1

* Can. Lab. Inland Rev. Dept. Bull. 231: (1912).
† *Ibid.*, 254: (1913).
‡ *Ibid.*, 311: (1915).

As already pointed out, the most noteworthy deviation from the standard for shorts (middlings) is the very low fiber, which, however, is accompanied by a lower percentage of fat, and in some cases by a lower protein value. It appears to me that this article should be placed on the market under a distinctive name, in which case it will be necessary to define it independently of shorts (middlings), under which term it apparently finds sale at present.

Chop feed is defined as "whole grain, of one or more kinds, more or less finely ground, and contains not less than 10 per cent of protein, not less than 2 per cent of fat, and not more than 10 per cent of crude fiber". The French terms *moulee* and *grains concasses* are usually accepted as equivalents.

It is apparent that the addition of any material which has been separated from any grain, or the abstraction of any portion of the grain which has been ground to constitute the feed, must be regarded as an adulteration of a chop feed. In every such case the article becomes a mixed feed and must be sold under a registered number, with attached guaranty of value. If, for example, hominy is partially degermed corn, it can not be sold as a chop feed, since the partial removal of the germ makes it no longer a ground whole grain. Such an article can be sold only when properly registered.

I think that the protein, fat and fiber requirements fixed for bran, shorts and chop will not be found to be unduly exigent. They are based on extensive work done upon commercial samples sold under the respective names. Yet it is desirable to consider the miller's point of view which has recently been very well put before me in the following words:

> At the back of the laudable object of obtaining regular values for the users of feed stuffs, particularly bran, shorts and middlings, there should be a desire to foster the milling industry upon sound principles and economic lines of production. Flour is the main product of wheat, and wheat is milled for the flour, not for the by-products, bran and middlings. While it is advisable for some standard to be set, it is certainly not wise that the standard should be so set as to encourage waste by the inclusion of flour in the offals which should be extracted and used for human food. It may be said that a good miller is, among other things, known by his ability to abstract a large proportion of flour from the wheat which he grinds, and this miller's bran would contain a higher rate of fiber than that of his less efficient competitor. A large number of such millers distributing the offals of defective milling systems will tend to the compilation of data of a misleading nature, and particularly in regard to the factor most influenced, namely, the fiber.

The same correspondent suggests that a minimum, as well as a maximum, content of fiber should be set for shorts (middlings), thus differentiating the variety which I have above referred to as feed flour, and placing it in a class by itself.

I have before me the definitions of feeding stuffs adopted by the Association of Feed Control Officials of the United States in January of this year. These definitions include the terms bran (recognizing corn, bran, rice bran, wheat bran), shorts or middlings (differentiating buckwheat, oat and wheat products), and chop, and the definitions furnished are essentially identical with those which obtain in Canada.

Minimum values, and limits of permissible variation, however, are not stated. This is apparently consequent upon the assumption that the producer in each case furnishes a guaranty of value with his bran, shorts and chop. I would respectfully suggest that in the case of bran, shorts and chop, it should be quite possible to legalize fixed minimum values, and to permit these classes of feed to be sold without specific, individual guaranty. I make this suggestion, having regard to the convenience of both buyer and seller. It is true that a certain variation in feed value occurs between the bran produced from one lot of wheat and that from another lot. The difference is very small, however, and when we bear in mind the way in which bran is usually produced and sold, it is easily recognized that a great, and, I believe, unnecessary addition is made to the cost of production, when the miller is required to store and evaluate separately the bran obtained from each lot of wheat, or other grain, milled by him. In the smaller mills especially it is usual to sell the greater part of the output of bran and shorts or middlings locally, and so long as the milling is honestly done, the continuous product of the mill has an average character not far removed from the standard limits above set, so far as Canada is concerned.

It is, of course, quite otherwise with mixed feeds and with feeds which are essentially by-products of other than straight milling processes. I have reference here, in particular, to such feeds as digester tankage, gluten meal, oil cake, etc. These can be sold fairly only with a guaranty of value, and specific identification by registration number or some equivalent.

During recent years a material known as screenings has been much in evidence as a component of feeds. Many of the State laws define screenings as follows: "Screenings are the smaller, imperfect grains, weed seeds, and other foreign material, having feeding value, separated in cleaning the grain".

I am given to understand that two distinct varieties of screenings, known respectively as elevator screenings and mill screenings, are recognized in the milling industry.

Elevator screenings is the offal separated by a process of screening from the wheat (or other grain) at the elevator, prior to the grading of the grain.

Mill screenings is the offal remaining in the wheat as furnished to the miller after treatment at the elevator, and separated by a screening process at the mill, prior to grinding the grain for production of flour.

I take it that, essentially, these two varieties of screenings do not greatly differ, the mill screenings being simply that residuum which the elevator treatment failed to remove from the grain. There is, however, this important distinction to be made. Elevator screenings are not sold with the graded wheat. Mill screenings, on the contrary, are present in the wheat as sold to the miller, who claims the right to include them with the products of milling operations. He separates them from the wheat before putting it through the rollers, because their presence in the flour would be objectionable, but at a later stage of the continuous milling process he claims a right to introduce them and they finally appear among the bran and shorts. The miller does not consider that he violates requirements for legal bran and shorts by such procedure, and describes his product as "mill-run".

Elevator screenings of necessity vary greatly in character from year to year and according to locality. As our grain fields become more and more infested with weeds, the seeds of such weeds are more and more in evidence. As as rule, elevator screenings possess decided nutrient value. The following results are quoted from a report by F. T. Shutt of the Dominion Experimental Farm, Ottawa, Canada:

Screenings from elevator of Fort William (1915).

	per cent
Moisture	9.04
Protein	13.00
Fat	7.64
Carbohydrates	51.68
Fiber	13.48
Ash	5.16

If otherwise unobjectionable, they may constitute a component of mixed feeds, sold under registration and with a guaranty. Of course, the addition of elevator screenings to bran or shorts would constitute adulteration so long as bran and shorts are defined as straight products of milling grain.

Unfortunately, these elevator screenings contain a large proportion of the seeds of various mustards and other weeds possessing bitter and even toxic properties. Shutt found that so-called black seeds constituted 38 per cent of the screenings at Fort William elevators in 1915. The removal of these black seeds left a material having the following composition:

	per cent
Moisture	10.32
Protein	14.25
Fat	5.67
Carbohydrates	61.81
Fiber	5.22
Ash	2.73

Shutt suggests the grading of screenings under government supervision. The desirability of some control over the character of elevator screenings is apparent from the reports of injury resulting from their use. Not only does the unpalatability of these weed seeds cause pigs and other stock to refuse them, but if starved into eating the feed death frequently results. Recent reports from the officials in charge of feeding stuffs in twelve States of the United States inform me that cases of poisoning probably caused by weed seeds are recorded. The pathologist of the Department of Agriculture, Ottawa, Canada, favors me with the subjoined account of experiments conducted by him in January, 1919.

> Our first experiment was with three pigs, to which we fed a liberal quantity of weed seed. The immediate result was that the pigs refused to eat any more of the material and vomited a great deal. The experiment was started on December 31. From that date until January 9 we contrived by various means to induce the pigs to eat small quantities of the material. We sometimes used sugar and at other times mixed the weed seeds with shorts or other material. The invariable result was that whenever the pigs swallowed any quantity of the seeds they vomited and became ill. On January 9 one of the pigs died and on opening its stomach we discovered a large red patch, as large as the palm of the hand, on the most dependent portion of the stomach where the poisonous seeds had been lying. There were other lesions which need not be described here.
>
> The other pigs were kept for a considerable period after this and were fed on ordinary food until February 10, at which time they had recovered and had improved in condition. On this date, they were forcibly fed after being starved for one meal and were given approximately 1½ ounces of ground weed seeds. One of the pigs vomited immediately the material he had been given; the other retained it for about half an hour. The result was that the pig that retained the material was found dead the following morning. The other pig was still alive, though it was not looking at all well. We made post mortems on both these pigs and in the first one found that the stomach was much congested and showed several ulcerations. The other pig showed them also, but they were not so distinct.
>
> To sum up: Our experiments are of a preliminary nature but there is no doubt in my mind that as these weed seeds belong to the mustard family they are dangerous and harmful to animals. This view is in accord with several authors such as Pammel[1], Lander[2] and Long[3].

The poisonous characters indicated as above for weed seeds which may, for our purpose, be grouped under the general heading of wild mustards (ball mustard, *Neslia paniculata*, wild mustard, *Brassica arvensis* and other species, hare's ear mustard, *Coringia orientalis*, tumbling mustard, *Sisymbrium altissimum*, etc.) are exactly such as might be expected to result from the use of the condimental mustards (*Sinapis alba*, *Brassica nigra* and *Brassica juncea*) in virtue of their vesicant properties, if these were used as feeds.

Whether these wild mustards may be made available for condimentary purposes or not, I am unable to say. It is asserted, however, that the so-called black seeds separated from elevator screenings find a market for use in the manufacture of the lower grades of this spice, particularly for so-called prepared or French mustard, and for pickles. What is of immediate importance to the officials charged with the inspection of feeds is to decide whether the material known as elevator screenings

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[1] M. H. Pammel. Manual of Poisonous Plants. 1911.
[2] G. D. Lander. Veterinary Toxicology. 1912.
[3] H. C. Long. Plants Poisonous to Live Stock. 1917.

should be permitted to find a market without some guaranty of its unobjectionable character for feeding purposes. It seems to me that separation of these poisonous seeds should be required. Whether or not a profitable use for them may be found, or their destruction insisted upon, does not immediately concern us. But that such a dangerous material as elevator screenings should be allowed to enter commerce uninspected and undefined is surely a serious lapse in feed control.

METHODS FOR ESTIMATING LIMITS OF CHLORIDS IN CHEMICALS.

By L. F. KEBLER, *Referee on Testing Chemical Reagents*, and W. H. HEATH (Bureau of Chemistry, Washington, D. C.).

During the past five years the quality of chemicals for use in medicine and for chemical analysis, available in this country, has either not improved or has really become inferior. The reason for this condition is, of course, attributed to the world war disturbances. No material improvements in methods of analyzing or detecting impurities or limit tests, in these chemicals, have been made during this time. In fact, most of the methods obtaining a decade ago are practically in use at present.

Several good text books contain instructions commonly used in testing reagent chemicals. Some of them, however, are prepared by or under the auspices of chemical manufacturers, so that the standards prescribed naturally are adapted to the chemicals prepared by the manufacturers compiling the publications. In case of disagreements on the part of chemists and manufacturers, other than the sponsors of these publications, however good they may be, such standards are not gracefully accepted by competing firms.

The United States Pharmacopœia, recognized by law, prescribes tests and standards for many chemicals used for medicinal purposes. The element of a competitor's standards is here eliminated.

The claim is made for most reagent chemicals now available that they contain no more than given maximum amounts of certain impurities, the nature of which, together with the amounts, are shown on the labels. Experience has shown that the statements on these labels are sometimes not so reliable as they might be. One of the essential difficulties is the fact that the methods of testing chemicals, either reagents or medicinal, leave entirely too wide a range for the personal equation. For example, in the case of limit tests, the methods prescribe such vague terms as opalescence, faint opalescence, slight opalescence, at most a slight opalescence, no turbidity, slight turbidity, no precipitate, faint precipitate, cloudiness, etc. No provision is made for enabling the analyst to determine by comparison or otherwise, what is, or is not, a turbidity, a slight precipitate, a faint precipitate, or an opalescence. Neither is he told whether or not a turbidity or an opalescence is permissible in case the limit prescribed is "no precipitate". It can readily be seen that such vague limiting adjectives are confusing. Furthermore, there are entirely too many limiting adjectives.

It would, of course, be very expensive, at times difficult, and generally unnecessary to use elaborate methods for determining the exact percentages of small amounts (often traces) of contaminating substances, such as chlorids, etc. A complicated method for the estimation of arsenic in reagent inorganic acids[1], worked out several years ago, is excellent and gives reliable results, but it is so tedious and involved that it is not adapted to routine work.

But little work of a definite character on the estimation of small amounts of impurities in chemicals has been published. The results of the referee's studies of the question in relation to the chlorid content of certain chemical reagents and the chemicals recognized by the United States Pharmacopœia, ninth revision, are as follows:

Determinations by the several nephelometers now on the market gave unsatisfactory results, because of the length of time consumed in making comparisons and the difficulty of thoroughly cleaning the apparatus after each determination. Long, selected test tubes also gave unsatisfactory results. Uniform 50 cc. Nessler tubes, with a graduation mark at the 25 cc. point, proved to be best adapted to this work.

N/100 hydrochloric acid was selected for the reason that it is always easily prepared from the N/10 standard stock solution, and because it does not introduce into the solution an unnecessary base.

The changes produced under given conditions, for convenience, are classified as follows:

N/100 Hydrochloric Acid.

	cc.
Slight opalescence	0.5
Opalescence	1.0
Slight turbidity	1.5
Turbidity	2.0
Precipitation	3.0

In order to prepare the standard in the case of opalescence, for example, introduce 22 cc. of water into a selected Nessler tube, add 1 cc. of N/100 hydrochloric acid, 1 cc. of dilute (10 per cent) nitric acid, and 1 cc. of N/10 silver nitrate solution, then mix well. In other words, the standard specified, in case of opalescence, is that opacity produced by an intimate mixture consisting of 1 cc. of N/100 hydrochloric acid, 1 cc. of dilute nitric acid, 1 cc. of N/10 silver nitrate solution, and enough water to make 25 cc.

With methods available for preparing standards, the procedure for testing individual chemicals can readily be worked out. Table 1 gives the approximate amounts of the several chemicals to be used. All chemicals, however, can not be tested by the same

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[1] *J. Am. Chem. Soc.*, 1906, 28: 178.

TABLE 1.

Chlorid in chemicals.

CHEMICAL	MAXIMUM AMOUNT OF N/100 HYDROCHLORIC ACID PERMITTED IN STANDARD COMPARISON TUBE	EQUIVALENT MAXIMUM WEIGHT OF IMPURITY PERMITTED	MAXIMUM IMPURITY PERMITTED, CALCULATED AS	WEIGHT OF SAMPLE	VOLUME OF SAMPLE	METHOD OF PREPARATION	TIME LIMIT
	cc.	*gram*	*per cent*	*grams*	*cc.*		*minutes*
		HCl:	HCl:				
Acetic anhydride	0.10	0.000036	0.0072	....	0.5	1	5
Acid, acetic	0.50	0.00018	0.0072	....	2.5	1	5
Dilute	0.50	0.00018	0.0012	....	15.0	1	5
Glacial	0.50	0.00018	0.0022	....	8.0	1	5
Acid, benzoic, natural	0.50	0.00018	0.036	0.5	....	2	5
Synthetic	1.00	0.00036	0.072	0.5	....	2	5
Acid, hydriodic, dilute	0.50	0.00018	0.036	....	0.5	3	1
Acid, hydrobromic, dilute	2.00	0.00036	0.072	....	0.5	3	1
Acid, lactic	0.10	0.000036	0.0144	....	0.25	1	5
Acid, nitric	3.00	0.00108	0.0434	....	2.5	1	5
Dilute	3.00	0.00108	0.0061	....	17.5	1	5
Acid, phosphoric	0.10	0.000036	0.0144	...	0.25	1	1
Dilute	0.10	0.000036	0.0020	....	1.75	1	1
Acid, salicylic	0.10	0.000036	0.0014	2.5	....	2	5
Acid, sulphuric	0.10	0.000036	0.0144	....	0.25	1	5
Dilute	0.10	0.000036	0.00288	....	1.25	1	5
Acid, trichloracetic	0.50	0.00018	0.0152	....	1.25	1	1
		NH_4Cl:	NH_4Cl:				
Ammonium carbonate	0.50	0.00026	0.0208	1.25	....	1	2
Ammonium nitrate	1.00	0.00052	0.0208	2.5	....	1	5
Ammonium oxalate	0.10	0.000052	0.0052	1.0	...	1	5
Ammonium sulphate	1.00	0.00052	0.0208	2.5	....	1	5
		$BaCl_2+2H_2O$:	$BaCl_2+2H_2O$:				
Barium hydroxid	0.50	0.00062	0.124	0.5	....	1	5
Barium nitrate	0.10	0.000122	0.01	1.25	....	1	5
		$CaCl_2+2H_2O$:	$CaCl_2+2H_2O$:				
Calcium bromid	2.00	0.00075	0.30	0.25	....	3	1
Calcium glycerophosphate	1.50	0.00118	0.47	0.25	....	1	1
		HCl:	HCl:				
Chloral hydrate	1.00	0.00036	0.0208	1.25	....	1*	1
			Cl:				
Chloroform	0.10	0.000035	0.00035	....	10	1†	5
			HCl:				
Codein phosphate	0.10	0.000036	0.0144	0.25	....	1	1
Codein sulphate	0.10	0.000036	0.0144	0.25	....	1	1
Ethyl chlorid	1.00	0.00036	0.0208	....	1.25	1*	1
Ethyl carbamate	0.50	0.00018	0.012	1.5	....	1	5
		$FeCl_3+6H_2O$:	$FeCl_3+6H_2O$:				
Ferric ammonium sulphate	2.00	0.0018	0.144	1.25	....	1	5
		HCl:	HCl:				
Hexamethylenamin	0.50	0.00016	0.036	0.5	...	1	5
		Cl:	Cl:				
Iodin	0.50	0.000175	0.070	0.25	...	3	1
		LiCl:	LiCl:				
Lithium bromid	1.25	0.000105	0.105	0.10	....	3‡	1

* Dissolve in U. S. P. alcohol instead of water.

† Shake with 22 cc. of water in a separatory funnel, allow to separate, withdraw the water, and proceed by Method 1 from acidulation.

‡ Pass gas through 22 cc. of N/10 nitric acid and proceed by Method 1 from dilution to 24 cc.

TABLE 1.—Concluded.

CHEMICAL	MAXIMUM AMOUNT OF N/100 HYDROCHLORIC ACID PERMITTED IN STANDARD COMPARISON TUBE	EQUIVALENT MAXIMUM WEIGHT OF IMPURITY PERMITTED	MAXIMUM IMPURITY PERMITTED, CALCULATED AS	WEIGHT OF SAMPLE	VOLUME OF SAMPLE	METHOD OF PREPARATION	TIME LIMIT
	cc.	*gram*	*per cent*	*grams*	*cc.*		*minutes*
Magnesium sulphate.......	1.00	$MgCl_2+6H_2O$: 0.00103	$MgCl_2+6H_2O$: 0.082	1.25	....	5	5
Nitrous oxid.............	0.50	Cl: 0.000175	Cl:	....	2000	1‡	5
Oxygen...................	0.50	0.000175	....	....	2000	1‡	5
Paraldehyde..............	0.25	HCl: 0.00009	HCl: 0.0036	....	2.5	1	5
Pilocarpin nitrate.........	1.00	Pilocarpin HCl: 0.00244	Pilocarpin HCl: 0.198	1.25	....	1	5
Potassium chromate.......	1.00	KCl: 0.00074	KCl: 0.0059	1.25	....	1§	5
Potassium dichromate......	1.00	0.00074	0.0089	0.835	....	1§	5
Potassium nitrate..........	0.50	0.00037	0.03	1.25	....	1	5
Quinin salicylate..........	0.50	Quinin HCl+ $2H_2O$: 0.00198	Quinin HCl+ $2H_2O$: 0.396	0.5	....	4 or 2	5
Quinin tannate............	1.50	0.00594	2.37	0.25	....	4 or 2	5
Salol.....................	1.00	HCl: 0.00036	HCl: 0.072	0.50	....	4	5
Sodium bicarbonate........	0.50	NaCl: 0.000216	NaCl: 0.0216	1.0	....	1	15
Sodium cacodylate.........	2.00	0.00116	0.232	0.5	....	1	5
Sodium oxalate............	0.20	0.00016	0.0232	0.5	....	1	5
Strontium iodid...........	2.00	$SrCl_2+2H_2O$: 0.00266	$SrCl_2+2H_2O$: 0.53	0.5	....	3	1
Sulphonal................	1.00	HCl: 0.00036	HCl: 0.07	0.5	....	4	5
Sugar....................	0.25	0.00009	0.0036	2.5	....	1	5
Trional..................	1.00	0.00036	0.07	5	....	4	5
Water, distilled...........	0.10	Cl: 0.000035	Cl: 0.00015	....	23	1	5
Zinc oxid................	0.50	$ZnCl_2$: 0.00034	$ZnCl_2$: 0.034	1	....	1	5

‡ Pass gas through 22 cc. of N/10 nitric acid and proceed by Method 1 from dilution to 24 cc.
§ Heat to 50°C. just before adding the silver nitrate.

method, because of different physical properties, different reactions, and different characters of the precipitates produced. Methods have been worked out for a number of chemicals, in the case of chlorids, to meet these features.

The methods tested are as follows:

(1) Dissolve the prescribed weight of chemical in about 15 cc. of water, acidulate with dilute (10%) nitric acid, add enough water to make exactly 24 cc., then add 1 cc. of N/10 silver nitrate solution, and mix thoroughly.

(2) Mix the chemical to be tested with chlorid-free calcium carbonate in water, dry, and ignite. After cooling, add about 15 cc. of water and enough nitric acid to render slightly acid, dilute to 24 cc. with water, then add 1 cc. of N/10 silver nitrate solution, and mix well.

(3) Mix or dissolve the sample in a mixture of 25 cc. of water and 5 cc. of dilute nitric acid, and add enough silver nitrate solution to completely precipitate all of the halogens present. Filter, and wash the precipitate with water, rejecting the filtrate and washings. Digest the precipitate for 10 minutes with 5 cc. of ammonium carbonate, U. S. P. test solution, cool, filter, and wash with 15 cc. of water. Collect the filtrate and washings in a comparison tube, acidify with concentrated nitric acid, add water to make 25 cc., mix well, and compare with the standard.

(4) Add about 15 cc. of water to the chemical to be tested and 1 cc. of dilute (10%) nitric acid, warm, shake, filter, and wash with enough water to make 24 cc. Add 1 cc. of N/10 silver nitrate solution and mix well.

(5) Proceed as for Method 1, except that the standard must contain an equivalent amount of the chlorid-free salt to be tested.

Place the prepared standards and unknown solutions in Nessler tubes. When ready to make the observation, add the N/10 silver nitrate solution simultaneously to the standard and the unknown solution, with thorough mixing.

RECOMMENDATIONS.

It is recommended—

(1) That immediate steps be taken to study methods for testing chemical reagents in order to provide definite details that will minimize the personal equations now obtaining.

(2) That steps be taken at an early date looking towards the publication of a compendium of methods for testing chemical reagents.

(3) That, if this association is not in position to undertake this task, this recommendation be transmitted to the Council of the American Chemical Society for consideration and action.

No report on microanalytical methods was made by the referee.

The appointment of the following committees was announced by the president:

Committee on auditing: G. L. Bidwell of Washington, D. C.; W. F. Hand of Mississippi; and E. M. Bailey of Connecticut.

Committee on nominations: A. J. Patten of Michigan; J. M. Bartlett of Maine; and B. L. Hartwell of Rhode Island.

Committee on resolutions: W. D. Collins of Washington, D. C.; H. B. McDonnell of Maryland; and Julius Hortvet of Minnesota.

The meeting adjourned at 12 m. to reconvene at 2 p. m.

FIRST DAY.

MONDAY—AFTERNOON SESSION.

The president announced the appointment of the following committee to consider the Baumé scale and report not later than Wednesday morning: W. D. Horne of New York; Frederick Bates of Washington, D. C.; Paul Rudnick of Illinois; E. W. Magruder of Virginia; and R. E. Doolittle of Illinois.

No referee on the subject of phosphoric acid was appointed and no report on this subject was presented.

No associate referee on basic slag, to cooperate with the committee on vegetation tests on the availability of phosphoric acid in basic slag, was appointed and no report on this subject was presented.

No report was presented by the committee on vegetation tests on the availability of phosphoric acid in basic slag.

REPORT ON NITROGEN.

By I. K. **Phelps** (Bureau of Chemistry, Washington, D. C.), *Referee.*

In the investigation of the Dupont nitrometer for the determination of nitrate nitrogen, but two collaborators were obtained. This has been due to the exigencies of the war and the high pressure under which the laboratories which were supplied with Dupont nitrometers have been subjected and to the fact that most of the laboratories which are accustomed to collaborate with the referee are not supplied with Dupont nitrometers. It is believed, however, that with the return to normal conditions collaborators can be procured. It is, therefore, recommended that this subject be continued for further investigation.

REPORT ON SPECIAL STUDY OF THE KJELDAHL METHOD[1].

By H. W. DAUDT (Jackson Laboratory, E. I. du Pont de Nemours & Co., Wilmington, Del.), *Associate Referee.*

On account of war activities, no work on the collaborative study of the Kjeldahl method was accomplished last year. Although the collaborators for 1918 were only required to take up during 1919 the work previously requested and to report the results of their work in time for the 1919 meeting, only three complete reports on the work submitted in 1918 have been received.

The work herein reported is largely a continuation of the investigations which had been made by I. K. Phelps and the writer. In fact, it is this study of the effect of the proportions and variations of reagents, and of time on the hydrolysis of difficultly decomposable substances, that has been made the subject of collaborative work. In addition, the retention of ammonia in the presence of mercury and copper salts during the distillation has been studied. The retention of part of the ammonia as a mercuric amino compound, when it is attempted to quantitatively distil ammonia from an alkaline solution in the presence of mercuric salts, was early recognized by H. Wilfarth[2], who used alkali sulphids to remove the mercury as sulphid before making the distillation. This work has been checked repeatedly by various investigators, including the associate referee, and the necessity of adding a reagent to remove the mercury has been so emphasized that further work along this line was deemed unnecessary. Because of the high cost of potassium sulphid and the difficulty in obtaining suitable material on the market, it appeared desirable to attempt to substitute this reagent by the cheaper and more easily procurable sodium thiosulphate. The experiments outlined in the following directions were, therefore, planned:

THE EFFICACY OF SODIUM THIOSULPHATE IN THE PRECIPITATION OF MERCURY.

Dissolve exactly 5 grams of the ammonium sulphate in water and dilute the solution to 500 cc. (preserve the remainder of the solution for experiments with copper sulphate). After thoroughly mixing, pipette 20 cc. portions of this solution into 500 cc. Kjeldahl flasks. Add 300 cc. of water and an acid solution of mercuric sulphate obtained by dissolving 0.7 gram of mercuric oxid in a mixture of 15 cc. of water and 15 cc. of sulphuric acid. Then proceed as follows:

(*a*) Add 25 cc. of a 4% potassium sulphid solution, agitate, then add 70 cc. of a saturated solution of sodium hydroxid. After connecting the flask to the condensing

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[1] Presented by I. K. Phelps.

[2] *Chem. Zentr.*, 1885, **16**: 17, 113.

apparatus, agitate and distil into standard acid in the usual manner until 200 cc. of the distillate have been collected.

(*b*) Add 25 cc. of an 8% solution of sodium thiosulphate, agitate, then add 20 cc. of a saturated solution of sodium hydroxid. Proceed as in (*a*).

(*c*) Proceed as in (*b*) except that 12.5 cc. of 8% sodium thiosulphate solution are used.

(*d*) Add 20 cc. of sodium hydroxid solution so that it forms a separate layer in the bottom of the flask. Then add 2 grams of sodium thiosulphate crystals. After connecting the flask to the condensing apparatus mix thoroughly by agitation and distil in the usual manner.

It is essential that blank determinations be made on the sulphid and thiosulphate and corrections made therefor. Make all determinations in duplicate.

The addition of thiosulphate was made in the two ways mentioned above in order to determine whether or not any sulphur dioxid passed into the condenser before neutralization of all of the acid solution.

TABLE 1.

Efficacy of sodium thiosulphate in the precipitation of mercury.

ANALYST	PRECIPITANT*			
	Potassium sulphid (*a*)	2 grams of thiosulphate in solution (*b*)	1 gram of thiosulphate in solution (*c*)	2 grams of thiosulphate crystals (*d*)
	gram	*gram*	*gram*	*gram*
J. J. Vollertsen, Morris & Co., Chicago, Ill.	0.04252	0.04252	0.04244	0.04252
Paul Rudnick and N. A. Gray, Armour & Co., Chicago, Ill.	0.0420	0.0416	0.0423	0.0422
W. D. Richardson, Swift & Co., Chicago, Ill.	0.04205	0.04122	0.04194	0.04202

* Average of duplicate results reported.

The results of all of the collaborators indicate that potassium sulphid can be replaced by sodium thiosulphate, that the quantity necessary for complete precipitation of 0.7 gram of mercury (as sulphid) is 1 gram and that the manner of adding the reagent has no influence on the results.

THE RETENTION OF AMMONIA BY COPPER SULPHATE.

It seemed desirable to make a study of the effect of copper sulphate on the distillation of ammonia, both in the presence of a slight excess of sodium hydroxid and in the presence of a larger excess. The directions sent to the collaborators for this part of the work were as follows:

Pipette 20 cc. portions of the ammonium sulphate solution used in the experiments in which the efficacy of sodium thiosulphate was studied, into 500 cc. Kjeldahl flasks.

To each portion add the amounts of copper sulphate crystals and the amount of saturated sodium hydroxid solution indicated in Table 2, then add 300 cc. of water. Distil 200 cc. of the solution in the usual manner into standard acid solution.

The results are given in Table 2.

TABLE 2.

Retention of ammonia in the presence of copper sulphate.*

ANALYST	COPPER SULPHATE	SATURATED SODIUM HYDROXID	NITROGEN FOUND
	gram	*cc.*	*gram*
J. J. Vollertsen	0.0	2.0	0.04235
	0.0	30.0	0.04235
	0.5	2.0	0.04235
	0.5	30.0	0.04235
	1.0	2.0	0.04194
	1.0	30.0	0.04194
Paul Rudnick and N. A. Gray	0.0	2.0	0.0420
	0.0	30.0	0.0423
	0.5	2.0	0.0421
	0.5	30.0	0.0420
	1.0	2.0	0.0420
	1.0	30.0	0.0422
W. D. Richardson	0.0	2.0	0.04073
	0.0	30.0	0.04234
	0.5	2.0	0.04083
	0.5	30.0	0.04117
	1.0	2.0	0.04120
	1.0	30.0	0.04214

* Average of duplicate results reported.

There is apparently a slight discrepancy in some of the results, but the conclusion can be drawn from the results of all of the collaborators that copper sulphate, in amounts of 0.5 gram or less, does not cause the retention of ammonia.

For the study of the effect of the proportion of the reagents, sulphuric acid, alkali sulphate, and heavy metal catalyst, and of time of digestion, the following directions were sent out, together with the required samples:

STUDY OF THE HYDROLYSIS OF REFRACTORY COMPOUNDS AND SUBSTANCES.

Into 500 cc. Kjeldahl flasks introduce the weights of the compounds indicated in Tables 3 and 4. Add the reagents indicated and boil briskly. The flasks should rest in a perforation 2½ inches in diameter. In the experiments in Table 4, where 0.2 gram of mercuric oxid is used, it is essential to drive off the hydrochloric acid from the pyridin zinc chlorid and from the nicotin zinc chlorid by first heating the weighed flask containing all of the sulphuric acid and then replacing carefully the sulphuric acid lost by volatilization. When the products of hydrolysis have cooled to room temperature, add enough water so that, after the addition of 35 cc. of 4% potassium sulphid

TABLE 3.

Study of the hydrolysis of certain refractory substances.*

ANALYST	POTASSIUM SULPHATE	SODIUM SULPHATE	CATALYST		TIME	NITROGEN IN BEAN MEAL†	NITROGEN IN PEPPER†
			Mercuric oxid	Copper sulphate			
	grams	*grams*	*gram*	*gram*	*hours*	*per cent*	*per cent*
J. J. Vollertsen..........	10	...	0.2	...	2	7.40	2.46
	10	...	0.7	...	1½	7.40	2.54
	10	...	0.7	...	2	7.44	2.63
	..	8.2	0.7	...	1½	7.48	2.54
	..	8.2	0.7	...	2	7.56	2.63
	..	10.0	0.7	...	1½	7.56	2.71
	..	10.0	0.7	...	2	7.48	2.67
	..	10.0	...	0.5	1½	7.36	2.13
	..	10.0	...	0.5	2	7.44	2.17
W. D. Richardson.......	10	...	0.2	...	2	6.978	2.083
	10	...	0.7	...	2½	6.992	2.115
	10	...	0.7	...	2	7.002	2.168
	..	8.2	0.7	...	1½	7.083	2.098
	..	8.2	0.7	...	2	7.097	2.217
	..	10.0	0.7	...	1½	7.094	2.141
	..	10.0	0.7	...	2	7.080	2.195
	..	10.0	...	0.5	1½	6.999	1.996
	..	10.0	...	...	2	7.013	2.075
Paul Rudnick...........	10	...	0.19	...	2	7.07	2.19
	10	...	0.65	...	1½	7.08	2.09
	10	...	0.65	...	2	7.08	...
	..	8.2	0.65	...	1½	7.08	2.11
	..	8.2	0.65	...	2	7.17	2.18
	..	10.0	0.65	...	1½	7.12	2.22
	..	10.0	0.65	...	2	7.15	2.25
	..	10.0	...	0.5	1½	7.09	1.96
	..	10.0	...	0.5	2	7.00	2.02
	..	10.0	...	0.5	2	7.02	...

* Digestion was made with 25 cc. of sulphuric acid in all cases.
† One gram of material used.

solution and later the required amount of sodium hydroxid solution, the total volume becomes about 375 cc. Distil in the usual manner into standard acid solution. (It is suggested that sodium alizarin sulphonate be used as the indicator.)

The effect of varying reagents and time is more pronounced with compounds containing nitrogen in the ring, as for example with pyridin, nicotin and hyroxyquinolin, than with protein-containing substances such as soy bean meal. Pepper, being a piperidin derivative, falls into the first-mentioned class.

It is apparent that difficulty has been found on the part of the collaborators in hydrolyzing completely the most refractory compounds, pyridin, nicotin, and hydroxyquinolin. This is probably caused by a difference in the intensity of heat from that maintained by the associate referee and his collaborators. This seems apparent from information sup-

TABLE 4.

Study of the hydrolysis of refractory compounds.

ANALYST	SULPHURIC ACID	POTASSIUM SULPHATE	SODIUM SULPHATE	CATALYST		TIME	HYDROXYQUINOLIN*	PYRIDIN ZINC CHLORID*
				Mercuric oxid	Copper sulphate		Nitrogen found	Nitrogen found
	cc.	*grams*	*grams*	*gram*	*gram*	*hours*	*per cent*	*per cent*
J. J. Vollertsen..	25	10	...	0.2	...	2	7.1	6.7
	25	10	...	0.2	...	2½	6.6	5.8
	25	10	...	0.7	...	2½	7.9	9.3
	35	10	...	0.7	...	2½	7.9	7.5
	20	10	...	0.7	...	1½	7.7	9.3
	25	10	...	0.7	...	1½	6.8	5.4
	25	10	...	...	0.5	2½	5.9	5.6
	25	10	...	...	1.0	2½	5.8	5.8
	20	...	8.2	0.2	...	2½	6.3	4.8
	20	...	8.2	0.7	...	2½	7.8	9.6
	25	...	8.2	.7	...	2½	8.0	8.0
	25	...	10.0	0.7	...	2½	7.8	8.7
	20	...	8.2	0.7	...	2	7.7	6.9
	20	...	8.2	0.7	...	1½	7.2	6.7
	20	...	8.2	0.65	...	2	6.5	7.3
	20	...	8.2	0.65	...	1½	6.4	7.3
Paul Rudnick ...	20	10	...	0.19	...	2	5.9	6.5
	25	10	...	0.19	...	2½	4.4	6.2
	25	10	...	0.65	...	2½	6.3	7.3
	35	10	...	0.65	...	2½	5.2	5.7
	20	10	...	0.65	...	1½	6.7	6.9
	25	10	...	0.65	...	1½	4.9	6.2
	25	10	...	...	0.5	2½	4.8	6.5
	25	10	...	...	1.0	2	3.7	5.8
	20	..	8.2	0.19	...	2½	5.7	7.1
	20	..	8.2	0.65	...	2½	6.6	7.4
	25	..	8.2	0.65	...	2½	6.5	7.4
	25	..	10.0	0.65	...	2½	6.6	7.2
W. D. Richardson	20	10	...	0.2	...	2	6.4	6.3
	25	10	...	0.2	...	2½	6.4	5.5
	25	10	...	0.7	...	2½	7.1	6.7
	35	10	...	0.7	...	2½	6.6	5.9
	20	10	...	0.7	...	1½	7.3	6.6
	25	10	...	0.7	...	1½	6.9	6.6
	25	10	...	0.5	0.5	2½	7.0	5.6
	20	10	...	1.0	1.0	2	6.9	4.4
	20	..	8.2	0.2	...	2½	7.3	6.1
	20	..	8.2	0.7	...	2½	7.3	7.7
	25	..	8.2	0.7	...	2½	7.2	6.4
	25	..	10.0	0.7	...	2½	7.2	6.8
	20	..	8.2	0.7	...	2	7.4	6.6
	20	..	8.2	0.7	...	1½	6.9	6.6

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* Four-tenths gram of the compound was used in all cases.

plied by the collaborators as to the size of the perforation on which the flasks rested, and of the sizes of the flames used. Later experience of the associate referee with gasoline gas, showed that some of the conditions had to be changed when intense heating could not be obtained in order to secure good results; that is, the amount of sulphuric acid had to be lowered and the time of digestion lengthened. The results bear out certain facts, however. Copper sulphate is not so effective a catalyst as mercury or mercuric oxid. Two-tenths of a gram of mercury or mercuric oxid is not sufficient, even in the presence of alkali sulphate, to decompose refractory compounds completely into ammonium sulphate. Thirty-five cc. of sulphuric acid when used with 10 grams of potassium sulphate, or its equivalent 8.2 grams of sodium sulphate, give results entirely too low. The use of 20 cc. of acid instead of 25 cc. with 10 grams of potassium sulphate or with either 8.2 or 10 grams of sodium sulphate gives a more rapid hydrolysis. A period of digestion of less than 2 hours should not be employed unless it is known that a certain substance requires less than 2 hours for hydrolysis. For the very refractory nitrogen heterocyclic compounds, a digestion of 2½ hours should be employed.

It has been noted that when protein substances are heated too rapidly at the beginning of the digestion, and particularly when acid has not come in contact with all of the sample, low results are obtained because of the loss of nitrogen in volatile form. In order to overcome this, such samples should be digested very slowly and carefully at first. This period, or that required for the expulsion of water when aqueous solutions are under examination, should not be considered as part of the time required for digestion. In other words, only the time of actual boiling of the sulphuric acid should be counted. The time of clearing up, *i. e.*, the time required for decolorization of the mixture, is not a safe criterion of complete hydrolysis. In some cases, as with pyridin derivatives, the digestion mixture may be colorless, even though little or no hydrolysis to ammonium sulphate has taken place, whereas in other cases, as when an excess of carbohydrates is present, all the nitrogen may be in the form of ammonium sulphate and the mixture may yet be discolored.

In the present official Kjeldahl-Gunning-Arnold method, the original procedure of Arnold was copied verbatim. When the Kjeldahl-Gunning-Arnold, "K. G. A.", or Arnold method was adopted it was understood that the method to be adopted was the method using 0.7 gram of mercury or mercuric oxid, 20 to 25 cc. of sulphuric acid and 10 grams of potassium sulphate, and not the original method of Arnold, calling for 40 to 45 cc. of sulphuric acid, 20 grams of potassium sulphate, 0.7 gram of mercury or mercuric oxid and 1 gram of copper sulphate. The method commonly called the Kjeldahl-Gunning-Arnold method is essentially the method of Dyer.

SUMMARY.

Summarizing the report, it can be stated that:

(1) Potassium sulphid can be replaced by sodium thiosulphate in precipitating mercury as sulphid.

(2) Copper sulphate, if present in amounts not exceeding 0.5 gram, causes no retention of ammonia.

(3) Two-tenths of a gram of mercury or mercuric oxid is not sufficient for the hydrolysis of refractory substances.

(4) Copper sulphate is not so effective a catalyst as mercury.

(5) The proportion of acid to alkali sulphate is important. For instance, with 10 grams of potassium sulphate, or 8.2 of sodium sulphate 35 cc. of sulphuric acid will not give complete decomposition. Twenty cc. is the most effective volume of sulphuric acid, although where considerable carbonaceous matter is present 25 cc. can be used to advantage. With 20 cc. of sulphuric acid, 8.2 grams of sodium sulphate and 10 grams of potassium sulphate can be substituted for each other.

(6) The time during which the preliminary digestion is conducted should not be taken into account in timing the period of digestion, but only the period of brisk boiling should be counted. The length of the period of digestion depends on the proportion of reagents and, of course, on the nature of the sample under analysis. Unless this time is definitely known for the particular substance, a period of 2 hours or more should be employed for the digestion.

RECOMMENDATIONS.

It is recommended—

(1) That the Kjeldahl-Gunning-Arnold method be amended as follows:

(*a*) Change "45–50 cc. of sulphuric acid" to "20–25 cc. of sulphuric acid".

(*b*) Omit "1 gram of copper sulphate".

(*c*) Change "20–25 grams of potassium sulphate" to "10 grams of potassium or sodium sulphate".

(*d*) After "potassium sulphid" insert "sodium sulphid or sodium thiosulphate".

(*e*) Replace the statement that digestion be continued until the mixture is colorless by a statement to the effect that 2 hours of continuous boiling of the sulphuric acid alkali sulphate mixture are required. Less than 2 hours should be consumed only when it has been shown that the particular sample does not require the full 2 hours.

(2) That the Kjeldahl method be specifically limited as an official method to substances to which it is particularly applicable.

(3) That the Gunning method and its modifications be specifically limited as an official method to substances to which it is particularly applicable.

(4) That the next associate referee be instructed to collect data and, if necessary, conduct collaborative work to determine for what common substances the Kjeldahl and Gunning methods can be used.

(5) That some statement, indicating the necessity for the careful heating of protein-containing substances at the beginning of the digestion, be incorporated into the official method, such statement to advise mixing the sample and acid mixture and to warn against heating portions of the sample not in intimate contact with the sulphuric acid.

REPORT ON POTASH[1].

By T. E. Keitt[2] (Agricultural Experiment Station, Experiment, Ga.), *Referee.*

Samples were prepared to send out to those who had signified their willingness to cooperate, but as there was nothing in the way of improvement of method to add to the instructions sent out by the former referee, it was decided, on account of the wide range of results shown by the former cooperators, to work on the outline of the method and endeavor to prepare a very definite procedure, especially with reference to mixed fertilizers.

The referee has serious misgivings about a method requiring burning below redness and subsequent lixiviation ever becoming satisfactory in the hands of the average analyst. He, therefore, undertook a combination of the moist combustion method first proposed by de Roode[3] and subsequently reported by Keitt and Shiver[4]. The outline as finally formulated and used follows:

MOIST COMBUSTION PERCHLORIC ACID METHOD.

Place 2.5 grams of the sample upon a 12.5 cm. filter paper and wash successively with portions of boiling water into a 250 cc. flask, until the washings amount to 200 cc. Acidify the solution with 5 cc. of concentrated hydrochloric acid. While hot, precipitate the sulphates by adding drop by drop, in slight excess, normal barium chlorid

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[1] Presented by C. C. McDonnell.

[2] Present address, Keitt and Caldwell, Newberry, S. C.

[3] *J. Am. Chem. Soc.*, 1895, **17**: 85.

[4] *J. Ind. Eng. Chem.*, 1918, **10**: 219; 1919, **11**: 1049.

solution acidified with hydrochloric acid (10 cc. usually suffice). Cool, make to the mark and shake. Allow the precipitate to settle. Transfer a 25 cc. aliquot, corresponding to 0.25 gram, to a porcelain evaporating dish, add 30 cc. of aqua regia and evaporate to dryness on a hot plate; add a second 30 cc. of aqua regia and evaporate to dryness; then add about 10 cc. of concentrated hydrochloric acid and 20 cc. of water and evaporate to dryness. Dissolve in 20 cc. of hot water and add 5 cc. of perchloric acid (sp. gr. 1.12); evaporate on a hot plate or steam bath until copious fumes evolve. Remove and run the liquid around the bottom of the dish. If solidification does not occur upon cooling, continue the evaporation. It can be cooled rapidly by floating the dish on water. Take up the residue with 5 cc. of water and add a second portion of 5 cc. of perchloric acid; evaporate the solution until dense fumes of perchloric acid appear. Remove so that the residue does not appear baked. In case it does, repeat the last operation. After *cooling* add 20 cc. of 95% alcohol, stir and allow to stand 30 minutes. Decant the alcohol through a Gooch crucible, carrying an asbestos pad about $\frac{1}{8}$ inch thick, then wash the precipitate twice by decantation with 95% alcohol containing 0.2% perchloric acid; transfer the precipitate to a Gooch using the same wash, and wash until the filtrate and washings amount to about 75 cc. Finally wash twice with alcohol ether (1 to 1), using 3–5 cc. each time, to remove all of the perchloric acid. Dry for 30 minutes at 120°C. Weigh, dissolve the potassium perchlorate from the Gooch with about 200 cc. of hot water. Use another receiver and wash the pad with alcohol or ether to facilitate drying, dry 30 minutes and weigh. Calculate the loss as potassium perchlorate and potassium oxid, using the factor 0.34.

Comparative results with the Lindo-Gladding, de Roode, and de Roode-perchloric acid method for determining potash.

FORMULA	LINDO-GLADDING METHOD	DE ROODE METHOD	DE ROODE-PERCHLORIC ACID METHOD
	per cent	*per cent*	*per cent*
Cottonseed meal	1.70	1.78	1.65
Cottonseed meal mixture	4.68	4.70	4.66
Complete fertilizer	4.26	4.41	4.38
Complete fertilizer	4.45	4.74	4.59
Complete fertilizer	3.91	4.02	3.86
Complete fertilizer	4.05	4.10	4.14
Average	3.84	3.96	3.88

RECOMMENDATIONS.

It is recommended—

(1) That the work on the availability of potash be continued.

(2) That the study of the perchlorate method be continued to include a study of the moist combustion method outlined in this report.

THE EFFECT OF MANURE-SULPHUR COMPOSTS UPON THE SOLUBILITY OF THE POTASSIUM OF GREENSAND.

By A. G. McCall (Agricultural Experiment Station, College Park, Md.).

Recent work at several of the agricultural experiment stations upon the effect of manure-sulphur composts upon the availability of the phosphorus in floats has suggested the desirability of a similar study with respect to the effect of sulphur composts upon the solubility of the potassium of greensand. Accordingly, two series of composts were prepared, using as the basis for one series a greensand from New Jersey containing 5.9 per cent of potassium, and for the other series a Maryland greensand containing 1.4 per cent of total potassium. The materials added were the same for each series and were as follows:

Compost No. 1.—Nothing.
Compost No. 2.—Sulphur, 500 grams.
Compost No. 3.—Sulphur, 500 grams; manure, 500 grams.
Compost No. 4.—Sulphur, 500 grams; manure, 250 grams; soil, 250 grams.
Compost No. 5.—Sulphur, 500 grams; soil, 500 grams.
Compost No. 6.—Sulphur, 500 grams; soil, 500 grams; aluminium sulphate, 0.02 per cent; ferrous sulphate, 0.02 per cent.
Compost No. 7.—Sulphur, 500 grams; manure, 250 grams; soil, 250 grams; calcium carbonate, 10 grams.

Commercial flowers of sulphur, partially rotted yard manure, Collington sandy loam soil and precipitated calcium carbonate were the materials used. After being thoroughly mixed, each compost was placed in a glazed pot and water added to one-half of the water-holding capacity, after which each compost was inoculated with sulphofying organisms. The pots were kept in the greenhouse, covered with two thicknesses of muslin, and once each week the water lost by evaporation was replaced and the composts removed from the pots and mixed to provide aeration.

For the water extractions, a 75-gram sample from each compost was weighed, air-dried, and 50 grams of the air-dried material shaken every 30 minutes for 8 hours with 500 cc. of distilled water. After standing overnight, the contents of the flasks were again shaken and filtered rapidly through folded No. 3 Whatman filter papers. The acidity of the water extracts was determined by first boiling an aliquot, cooling and titrating with N/10 sodium hydroxid. Sulphur was determined by acidifying aliquots with 2 cc. of concentrated hydrochloric acid and precipitating at boiling temperature with barium chlorid. The potassium determinations were made gravimetrically by the platinic chlorid method from aliquots of the water extracts, first eliminating the soluble organic matter, silicates, iron, aluminium and phosphorus by evaporation with sulphuric acid, ignition and precipitation.

The duration of the experiment was 23 weeks, water extracts being made at the beginning, and at the end of each week for the first nine weeks and thereafter at the end of the twelfth, fifteenth, seventeenth, twentieth and twenty-third weeks.

In the composts consisting of greensand, sulphur and manure, there was a slight but gradual accumulation of water-soluble acidity up to the end of the fifth week, after which there was a very rapid increase for three weeks. For the remainder of the period, the acidity remained at a high and practically constant level. When half of the manure was replaced with an equal quantity of soil, the acidity was greatly reduced, the maximum in one case being reached in 12 weeks and in another only after 15 weeks. When the manure was entirely replaced by soil, the acidity increased gradually to the end of the twenty-third-week period, but the amount developed was only about one-third as much as when equal weights of manure and soil were used.

The accumulation of soluble sulphates closely paralleled the development of acidity, and in every case, with the increase in acidity and the accumulation of sulphates, there was a corresponding increase in the amount of potassium in the water extract. The potassium, however, continued to increase for some weeks after the acidity and sulphates had attained their maximum. The outstanding results of the experiment may be summarized as follows:

SUMMARY.

1. In composts consisting of greensand, manure and soil in different proportions, an appreciable amount of the potassium of the greensand was made water-soluble through sulphofication. The most effective compost contained sulphur and manure in equal amounts.

2. The composts in which a part of the manure was replaced by soil showed a marked decrease in the amount of acidity and sulphur oxidized, as well as in the amount of potassium made soluble. When all of the manure was replaced by soil the rate of sulphofication was so slow that only a very small amount of potassium was found in the water extract.

3. The addition of small amounts of ferrous and aluminium sulphates failed to stimulate sulphofication.

4. Calcium carbonate, added to the sulphur-manure-soil compost, produced a stimulating effect during the early part of the period, but failed to increase the acidity or the soluble potassium above the maximum reached by the corresponding composts in which no calcium carbonate was used.

5. A greater total *amount* of water-soluble potassium was recovered in the composts containing the high potassium greensand, but a larger

percentage of the total potassium was liberated from the low potassium greensand.

6. In the composts containing manure, the total amounts of potassium recovered in the water extracts varied from 9.1 per cent to a maximum of 41.3 per cent of the total initial amount present.

A COMPARISON OF RESULTS OBTAINED BY THE DE ROODE, OFFICIAL LINDO-GLADDING AND FORMER OFFICIAL LINDO-GLADDING METHODS FOR THE DETERMINATION OF POTASH IN MIXED FERTILIZERS[1].

By E. R. TOBEY (Agricultural Experiment Station, Orono, Me.).

For some time the official Lindo-Gladding method of determining potash has not proved satisfactory to many fertilizer manufacturers and chemists. They contend that this method does not account for all of the water-soluble potash, the source of error being due to occlusion. A second source of error of an opposite nature is caused by the diminished volume of the solute, due to the volume occupied by the precipitate formed on the addition of ammonium hydroxid and ammonium oxalate.

To overcome these difficulties, several different methods have been suggested by chemists but none have shown sufficient worth to be adopted as official. A method suggested by T. E. Keitt and H. E. Shiver, both of the Agricultural Experiment Station, Experiment, Ga., using the de Roode method as a skeleton, promised, from determinations made by them, to overcome the objections of the manufacturer and chemist. The method is claimed to be applicable to all commercial fertilizers, including concentrated salts, and is as follows:

> Place 10 grams of the sample in a 500 cc. flask and add 300 cc. of water. Keep the contents of the flask at the boiling temperature for approximately 30 minutes, cool and dilute to volume. After allowing to stand until the material has settled, filter and draw out 50 cc., an aliquot representing 1 gram. Place the aliquot in a porcelain dish and add 3–5 cc. of nitric acid to destroy any organic matter that may be present. Evaporate to dryness over a water bath, take up with hot water and an excess of hydrochloric acid. Evaporate again to dryness, take up with hot water, adding several drops of hydrochloric acid, and enough platinic chlorid to precipitate all of the potash present. (Thus all of the details through the precipitation are carried out on one bath, in almost one operation, and in a very short time.) Cover the precipitate with the acidulated alcohol. Allow to stand 15–20 minutes, in which time all iron, aluminium and magnesium will dissolve; filter, and wash with the acidulated alcohol solution until the washings are colorless, washing free of the excess of platinic chlorid. Next wash well with ammonium chlorid (saturated with potassium chlorplatinate). This washing should be thorough, for the accuracy of the method is largely dependent upon this

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[1] Presented by J. M. Bartlett.

operation; six or seven washings usually suffice. Wash thoroughly with 95% alcohol to remove the ammonium chlorid; then dry and weigh the precipitate and calculate the results as in the older method.

PREPARATION OF ACIDULATED ALCOHOL.

To each 1000 cc. of 95% alcohol, add 75 cc. of concentrated hydrochloric acid, then pass dry hydrochloric acid gas into the mixture until 1 cc. of the alcohol neutralizes 2.25 cc. of normal potassium hydroxid, using phenolphthalein as an indicator. The hydrochloric acid gas may be prepared by using C. P. sodium chlorid and concentrated sulphuric acid, or by heating concentrated hydrochloric acid and first passing the gas through sulphuric acid and then into the alcohol.

The writer made several analyses of mixed fertilizers using the de Roode, official Lindo-Gladding, and former official Lindo-Gladding methods. The former official Lindo-Gladding method differs from the official method only in the method of obtaining the solution of the potash.

The results obtained are far from satisfactory, as may be seen by consulting the following table. It will be noted that the majority of results obtained by the official Lindo-Gladding method are lower than the corresponding results by the former official method. Those obtained by the de Roode method are so unsatisfactory that they are presented for no other reason than to invite suggestions as to the cause of the writer's error or errors.

In the following table it will be noted that 5 cc., 10 cc. and 15 cc. of hydrochloric acid, together with hot water, were used, with varying results, in taking up the residue from the nitric acid evaporation. The method does not advise one in regard to evaporation to a "sirupy consistency" after the addition of the platinic chlorid but the writer in all cases, except the four given in the column labelled "evaporated to a thin sirupy consistency", made this evaporation. In the case of the four exceptions, only a partial evaporation was made, that is, to a very thin sirupy consistency. Although a thorough washing was made with the ammonium chlorid solution, the results obtained are so much higher than those obtained by the regular official method that it is evident the final precipitate contained some material in addition to the potash salt.

After the work was done, the results of which are reported in the table, it was thought that possibly the very high results obtained in some samples were due to ammonia salts not being completely removed before the platinum chlorid was added. Consequently several of the samples which contained ammonia were analyzed by the de Roode method, except that after evaporating with hydrochloric acid they were heated sufficiently to drive off any ammonia that might be present. The averages of the results obtained on eleven samples were: not ignited, 4.29 per cent of potassium oxid; ignited, 4.17 per cent. This would seem to show that the high results were not due to ammonia.

This method was successfully used on mixed fertilizers and potash salts by Keitt, Shiver and Padgett[1] with very satisfactory results. The chief advantages of the method are: Ease of manipulation; less time consumed; no loss by sputtering; and no chance for occlusion.

Potash, expressed as K_2O, obtained by different methods.

NUMBER	DE ROODE METHOD ACIDIFIED WITH 5 CC. OF HYDROCHLORIC ACID	DE ROODE METHOD ACIDIFIED WITH 10 CC. OF HYDROCHLORIC ACID	DE ROODE METHOD ACIDIFIED WITH 15 CC. OF HYDROCHLORIC ACID	DE ROODE METHOD ACIDIFIED WITH 5 CC. OF HYDROCHLORIC ACID EVAPORATED TO A THIN SIRUPY CONSISTENCY	FORMER METHOD	OFFICIAL METHOD
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
4923	5.35	...	4.50	4.90	3.87	3.60
4942	3.78	...	...	...	3.62	3.69
4956	4.59	...	4.69	4.09	3.83	3.87
4960	5.30	...	4.71	4.93	3.78	3.51
4961	5.17	...	4.29	...	3.75	3.41
4962	4.12	...	3.55	...	2.90	2.78
4979	4.67	...	3.63	...	2.90	2.76
4984	4.17	...	3.42	...	2.88	2.52
4996	3.55	...	...	...	3.55	3.50
5000	5.35	...	4.40	...	3.93	3.77
5001	5.17	...	4.32	...	3.94	3.76
5002	5.03	...	4.45	...	3.95	3.79
5004	...	4.50	4.52	5.13	3.87	3.68
5005	...	4.32	4.36	...	3.89	3.74
5007	...	3.96	...	...	3.84	3.44
5012	...	1.77	...	...	1.95	1.45
5013	...	2.06	...	...	1.93	1.55
5025	...	4.13	3.93	...	3.71	3.76
5032	...	2.22	...	...	1.96	1.94
5048	...	4.38	4.25	...	3.96	3.84
5054	...	4.35	3.58	...	3.39	3.46
5071	...	3.68	...	...	3.74	3.78
5072	...	4.40	3.94	...	3.95	3.96
5076	...	3.80	...	...	3.76	3.71
5080	...	...	4.11 3.85	...	3.36	3.28
5083	...	...	3.07	...	2.95	2.80
5090	...	...	1.58 1.55	...	0.85	0.87
5105	...	...	3.86	...	3.57	3.29
5133	...	...	1.98 1.89	...	1.63	1.56
5135	...	...	1.01 0.97	...	0.65	0.60
5142	...	...	4.32 4.36	...	3.58	3.64
5145	...	...	3.75 3.53	...	2.90	2.75

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[1] *J. Ind. Eng. Chem.*, 1918, **10**: 219.

REPORT ON WATER[1].

By J. W. Sale (Bureau of Chemistry, Washington, D. C.), *Referee.*

A method for the determination of iodin[2] and a method for the determination of bromin[3] were selected for cooperative work. These methods gave promise of greater accuracy than is obtainable by the colorimetric methods of this association[4]. The need for better methods for iodin and bromin has been felt for some time by analysts, especially those concerned with the examination of mineral waters and brines.

The method for iodin in the presence of chlorin and bromin follows:

REAGENTS.

(**a**) *5% solution of equal weights of sodium hydroxid and sodium carbonate.*
(**b**) *Sulphuric acid (1 to 10).*
(**c**) *4% sodium hydroxid solution.*
(**d**) *10% potassium permanganate solution.*
(**e**) *Alcohol about 95%.*
(**f**) *Potassium iodid crystals.*
(**g**) *Hydrochloric acid (concentrated).*
(**h**) *N/20 sodium thiosulphate.*

REACTIONS.

$$KI + 2\ KMnO_4 + H_2O = KIO_3 + 2\ KOH + 2\ MnO_2.$$
$$KIO_3 + 5\ KI + 6\ HCl = 6\ KCl + 3\ H_2O + 3\ I_2.$$
$$3\ I_2 + 6\ Na_2S_2O_3 = 6\ NaI + 3\ Na_2S_4O_6.$$

DETERMINATION.

Take such a quantity of the brine or water as will contain not more than 0.1 gram of iodin or more than 10.0 grams of total salts. Adjust the volume to 100–150 cc., add a sufficient quantity of the solution of sodium hydroxid and sodium carbonate, reagent (**a**), to precipitate the calcium and magnesium. Boil, filter off the precipitate of calcium and magnesium, and wash with hot water; introduce the filtrate into an Erlenmeyer flask, adjust the volume to about 100 cc., neutralize with dilute sulphuric acid, reagent (**b**), and add 1 cc. of the solution of sodium hydroxid, reagent (**c**). Heat to boiling, add an excess of potassium permanganate, reagent (**d**), about 0.5 cc. excess, continue heating until the precipitate begins to coagulate and then allow to cool. Add sufficient alcohol, reagent (**e**), or hydrogen peroxid to bleach the permanganate color and set the beaker on a steam bath. When the precipitate has settled, filter and wash the precipitate with hot water. After cooling, add 1–2 grams of potassium iodid, acidify with hydrochloric acid and titrate with N/20 thiosulphate. One-sixth of the iodin titrated represents the amount originally present (1 cc. of N/20 thiosulphate solution equals 1.058 mg. of iodin).

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[1] Presented by W. W. Skinner.
[2] *J. Ind. Eng. Chem.*, 1919, **11**: 563.
[3] *Ibid.*, 954.
[4] *Assoc. Official Agr. Chemists, Methods*, 1916, 47.

The method for bromin in the presence of chlorin, but not iodin follows:

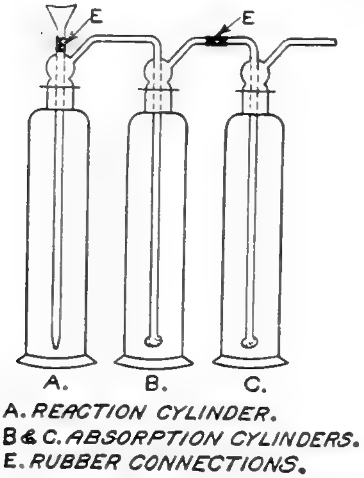

FIG. 1. GAS ABSORPTION APPARATUS.

APPARATUS.

Two high form Dreschel gas washing bottles.
An ordinary wash bottle.
Join the three wash bottles as in Fig. 1.

REAGENTS.

(a) *Sulphite reagent.*—Solution containing 4 grams of sodium sulphite and 0.8 gram of sodium carbonate per 100 cc.

(b) *Chromium trioxid crystals.*
(c) *3% solution of hydrogen peroxid.*
(d) *Potassium iodid crystals.*
(e) *N/20 thiosulphate.*

REACTIONS.

$$2\ CrO_3 + 6\ HBr = Cr_2O_3 + 3\ H_2O + 3\ Br_2.$$
$$2\ H_2CrO_4 + 3\ H_2O_2 = Cr_2O_3 + 2\ O_2 + 5\ H_2O.$$
$$Na_2SO_3 + 2\ Br + H_2O = 2\ HBr + Na_2SO_4.$$

DETERMINATION.

Evaporate the sample of water or brine, which should not be too acid, to dryness or nearly so. Charge the reaction cylinder A, Fig. 1, by introducing glass beads to a depth of about 1 inch, followed by 15 grams of solid chromium trioxid, and finally enough glass beads to fill the cylinder half full. Add 20 cc. of the solution of sodium sulphite and sodium carbonate, reagent (a), to the first absorption cylinder and 5 cc. to the second. Dilute each to about 200 cc. Connect the three cylinders and draw a current of air through slowly. Wash the sample into the reaction cylinder with water sufficient to make about 25 cc. of solution. Aspirate until the contents of the reaction cylinder are in solution and thoroughly mixed, then discontinue, close the inlet tube with a small piece of rubber tubing and clamp, and reduce the pressure in the apparatus slightly by sucking out some air in order to guard against any possible escape of bromin at the ground glass stopper. Allow to stand overnight, then aspirate with a rather strong current of air (about $\frac{1}{2}$ to $\frac{3}{4}$ liter per minute) for 3 hours, adding four 2 cc. portions of 3% hydrogen peroxid at 30-minute intervals. Stop the aspiration and evaporate the contents of the two absorption cylinders nearly to dryness. Clean out the reaction cylinder and freshly charge with glass beads and 15 grams of chromium trioxid. Into the first absorption cylinder put 10 grams of potassium iodid dissolved in 200 cc. of water and into the second 3 or 4 grams in a like amount of water. Connect the apparatus, draw through a slow current of air and transfer the contents of the evaporating dish to the reaction cylinder by means of the small funnel, using 25 cc. of water. Aspirate until all of the bromin is evolved (about 1 hour) and titrate the potassium iodid solution with thiosulphate (1 cc. of N/20 thiosulphate = 3.996 mg. of bromin).

It will be noted that the method for bromin is to be used only in the absence of iodin. The authors state that iodin may be removed from the sample in which bromin is to be determined subsequently, as follows:

Introduce the neutral or slightly acid sample, which should contain not more than 0.1 gram of bromin or 10 grams of total salts into a distillation flask and adjust to a volume of approximately 75 cc., add 1.5–2.0 grams of ferric sulphate and distil the liberated iodin with steam into 100 cc. of a potassium iodid solution (10 grams of potassium iodid per 100 cc.). The potassium iodid solution may be titrated with sodium thiosulphate solution and the result used to check the figure obtained by the permanganate method.

TABLE 1.

Composition of samples sent to collaborators.

(Expressed as milligrams per 10 cc.)

SOLUTIONS	CHLORIN	BROMIN	IODIN	SULPHATE	BICARBONATE	MAGNESIUM	CALCIUM	POTASSIUM	SODIUM
Iodin....	603.0	340.6	34.13	1.5	3.8	0.3	1.4	177.1	391.0
Bromin..	603.0	50.25	....	2.7	6.4	0.5	2.4	24.6	391.0

TABLE 2.

Collaborators' results on synthetic sample of brine.

ANALYST	IODIN FOUND IN IODIN SOLUTION		BROMIN FOUND IN BROMIN SOLUTION	
	Mg. per 10 cc.	Mg. per 2 cc.	Mg. per 10 cc.	Mg. per 2 cc.
J. H. Mitchell, Clemson Agricultural College, Clemson College, S. C.	32.7	6.8	45.00	....
	33.0	6.8	46.8	....
	33.0	6.9	38.4*	....
	33.5	6.9	....	....
	...	7.0	....	....
W. G. Koupal, State Water Survey Division, Urbana, Ill.	34.6	6.9	49.0	9.6
	34.5	7.0	49.2	9.6
	34.5	6.9	48.9	9.6
D. K. French, Dearborn Chemical Co., Chicago, Ill.	42.4*	8.4*	47.8	....
	42.6*	8.4*	46.7	....
	42.8*	8.6*	44.7	....
	47.8*	...	....	....
W. F. Baughman, Bureau of Chemistry, Washington, D. C.	...	...	49.7	....
	...	...	49.7	....
	...	...	....	....
W. E. Shaefer, Bureau of Chemistry, Washington, D. C.	34.5	7.0	49.7	9.9
	34.7	7.0	48.7	9.7
	34.6	7.0	49.3	10.2
L. H. Enslow, Filter Plant, Gatun, Canal Zone, Panama.	33.6	6.8	40.6*	....
	33.8	6.8	42.0*	....
	33.6	6.8	39.8*	....
	...	...	40.6*	....
J. W. Sale	34.2	6.9	48.7	9.6
	34.1	6.8	48.9	9.4
	...	...	49.0	9.0
Average....................	33.9	6.9	48.2	9.6
Theory.....................	34.13	6.83	50.25	10.05

* Omitted from average.

DISCUSSION OF IODIN METHOD.

Results obtained by using hydrogen peroxid in place of alcohol in the method for iodin, as suggested by Enslow, are contained in Table 3.

The results obtained are satisfactory. It is suggested that the use of hydrogen peroxid be optional in the method for iodin.

TABLE 3.

Results obtained by the use of hydrogen peroxid in place of alcohol to destroy excess permanganate in the method for iodin.

IODIN FOUND BY ENSLOW		IODIN FOUND BY SHAEFER	
Mg. in 10 cc.	Mg. in 2 cc.	Mg. in 10 cc.	Mg. in 2 cc.
34.6	6.9	34.1	6.9
34.6	6.9	34.0	lost
34.6	6.9	34.0	7.0
Theory......34.13	6.83	34.13	6.83

An average of 33.9 mg. of iodin was obtained by the collaborators, the theory being 34.13 mg. The difference is 0.23 mg. or 0.7 per cent. On the smaller sample of iodin solution taken for analysis an average of 6.9 mg. of iodin was obtained, the theory being 6.83 mg. The difference is 0.07 mg. or 1.0 per cent. These averages are based on fifteen determinations in the first instance and sixteen in the second. The results obtained by one of the analysts have been omitted from the average as they were excessively high, due, perhaps, to the incomplete removal of the excess permanganate.

After carefully considering the results submitted and the comments of the analysts, it is the opinion of the referee that the method for iodin is worthy of adoption as a tentative method by the association.

DISCUSSION OF BROMIN METHOD.

There appears to be no objection to the modification of the method for bromin suggested by Koupal and Shaefer, viz. that the chromic acid be added to the reaction chamber in solid form and that the sample after concentration be washed in, using as little wash water as possible. The last figures reported by the referee were obtained by using this modification of the method and they are satisfactory.

An average of 48.2 mg. of bromin was obtained by the collaborators, the theory being 50.25 mg. The difference is 2.05 mg. or 4.1 per cent. On the smaller sample of bromin solution taken for analysis, an average of 9.6 mg. of bromin was obtained, the theory being 10.05 mg. The difference is 0.45 mg. or 4.4 per cent. These averages are based on sixteen determinations in the first instance and nine in the second. Five determinations have been omitted from the average on the large sample, four of them because the analyst was so situated that adequate laboratory equipment for conducting the test was not available.

The method for bromin consistently gives slightly low results. The very best results obtained were 0.5 mg. low, and other results which may be regarded as fairly satisfactory were 1.0 to 1.5 mg. low. Under these circumstances, it may be well to state that the standard potassium bromid solution used in making up the synthetic sample was standardized by two analysts, both by weighing the potassium bromid as such and by precipitating the bromin as silver bromid, so that the theoretical figure for bromin content is undoubtedly correct.

In considering these results it must be borne in mind that the determination of bromin in mineral water and brines is one of the most tedious and unsatisfactory of any of the methods. The method proposed, while seemingly full of detail, requires, as a matter of fact, comparatively little attention after the analysis is started. It is freely conceded that it is not a method for an inexperienced worker, but there are no methods for bromin in the literature that are both accurate and rapid. It is believed that the method here proposed gives more dependable results than the colorimetric method of the association[1]. Under the circumstances set forth above, it is suggested that the method for bromin be adopted by the association as a tentative method. It is thought that the colorimetric methods for iodin and bromin should be retained by the association as tentative methods, as they will continue to give value from a qualitative standpoint.

DETERMINATION OF THE AMMONIAS IN WATER CONTAINING SULPHID.

Additional analytical work on the determination of free and albuminoid ammonia in water containing sulphid, particularly as to the effect of the reagent used on the quantity of albuminoid ammonia obtained has been done during the past year and some of the results of numerous tests are contained in Tables 4 and 5. The laboratory work was carried out by W. E. Shaefer under the direction of the referee.

Two methods of preventing sulphid from interfering with the determination of free ammonia had been shown previously to be satisfactory. In one of these methods, which for reference will be called "the acid method", the sample is acidified with a measured amount of sulphuric acid, the sulphid boiled off, the sample made alkaline with sodium carbonate and ammonias determined by distillation and nesslerization of the distillate in the usual manner. In the other method, which for refer-

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 47.

ence will be called "the cadmium chlorid method", the sample containing sulphid is made alkaline with sodium carbonate, the sulphid precipitated with cadmium chlorid and, without filtering, the ammonias are determined in the usual way.

TABLE 4.

*Free and albuminoid ammonia in water containing sulphid** (*sulphid removed by boiling in acid solution.*)

(Analyst, W. E. Shaefer.)

EXPERIMENT NUMBER	HYDROGEN SULPHID PRESENT	SULPHURIC ACID EXCESS N/2 ADDED	SODIUM CARBONATE EXCESS 5N ADDED	NITROGEN AS FREE AMMONIA		DIFFERENCE	POSSIBLE ERROR IN READING NESSLER TUBES	NITROGEN AS ALBUMINOID AMMONIA		DIFFERENCE	POSSIBLE ERROR IN READING NESSLER TUBES
				Present	Found			Present	Found		
	mg.	*cc.*	*cc.*	*mg.*	*mg.*			*mg.*	*mg.*		
1	0.5	30	5	0.060	0.050	−0.010	0.003	0.014	0.009	−0.005	0.001
2	0.5	30	5	0.060	0.058	−0.002	0.003	0.014	0.019	+0.005	0.002
3	0.5	30	5	0.160	0.160	0.000	0.012	0.014	0.017	+0.003	0.001
4	0.5	30	5	0.160	0.164	+0.004	0.012	0.014	0.014	0.000	0.001
5	5.0	30	5	0.085	0.094	+0.009	0.006	0.162	0.175	+0.013	0.010
6	5.0	30	5	0.085	0.104	+0.019	0.007	0.162	0.174	+0.012	0.010
7	5.0	30	5	0.114	0.142	+0.028	0.009	0.114	0.125	+0.011	0.008
8	5.0	30	5	0.166	0.171	+0.005	0.012	0.076	0.098	+0.022	0.006
9	5.0	30	5	0.166	0.182	+0.016	0.012	0.076	0.102	+0.026	0.006
10	0.5	30	5	0.517	0.544	+0.027	0.024	0.051	0.059	+0.008	0.004
11	5.0	30	5	0.517	0.560	+0.043	0.032	0.051	0.061	+0.010	0.003
12	5.0	30	5	0.517	0.624	+0.107	0.032	0.051	0.075	+0.024	0.006
13	5.0	30	5	0.558	0.552	−0.006	0.024	0.103	0.124	+0.021	0.008
14	125.0	30	5	0.517	0.576	+0.059	0.032	0.051	0.071	+0.020	0.006
15	125.0	30	5	0.517	0.504	−0.013	0.024	0.051	0.049	−0.002	0.004
16	125.0	30	5	0.558	0.576	+0.018	0.032	0.103	0.121	+0.018	0.008
..	....	..	..	4.757	5.061	....	0.276	1.107	1.293	....	0.084

* PROCEDURE: Measured amounts of sulphuric acid were added to samples containing varying, but known quantities of sulphid and free and albuminoid ammonias. The sample was boiled until free from sulphid (*i. e.*, about 20 minutes), made alkaline with sodium carbonate and the ammonias determined in the usual manner. The samples were prepared from boiled Washington City tap water, a standard solution of ammonium chlorid, a solution of hydrogen sulphid, and an infusion of leaves.

By reference to Table 4, it will be noted that a total of 4.757 grams of nitrogen in the form of free ammonia was contained in sixteen samples, whereas 5.061 grams of nitrogen were found, a plus error of 6.4 per cent. The possible error in the reading of the Nessler tubes in all sixteen tests, however, was 0.276 gram of nitrogen as free ammonia, or 5.8 per cent, which, subtracted from the total plus error, leaves 0.6 per cent error on the free ammonia, due to the modification of the method.

These data indicate that "the acid method" of preventing the sulphid from interfering is preferable to "the cadmium chlorid method". The

TABLE 5.

Free and albuminoid ammonia in water containing sulphid (sulphid removed by precipitation with cadmium chlorid).*

(Analyst, W. E. Shaefer.)

EXPERIMENT NUMBER	HYDROGEN SULPHID PRESENT	CADMIUM CHLORID 10% SOLUTION ADDED	SODIUM CARBONATE 5N ADDED	NITROGEN AS FREE AMMONIA		DIFFERENCE	POSSIBLE ERROR READING NESSLER TUBES	NITROGEN AS ALBUMINOID AMMONIA		DIFFERENCE	POSSIBLE ERROR READING NESSLER TUBES
				Present	Found			Present	Found		
		cc.	*cc.*	*mg.*	*mg.*			*mg.*	*mg.*		
1	0.5	5	5	0.023	0.041	+0.018	0.002	0.023	0.008	−0.015	0.000
2	0.5	5	5	0.023	0.036	+0.013	0.002	0.023	0.012	−0.011	0.001
3	0.5	5	5	0.173	0.160	−0.013	0.010	0.023	0.021	−0.002	0.002
4	0.5	5	5	0.173	0.175	+0.002	0.010	0.023	0.009	−0.014	0.001
5	0.5	5	5	0.523	0.530	+0.007	0.030	0.023	0.008	−0.015	0.000
6	0.5	5	5	0.523	0.500	−0.023	0.030	0.023	0.009	−0.014	0.001
7	5.0	5	5	0.073	0.059	−0.014	0.004	0.023	0.009	−0.014	0.001
8	5.0	5	5	0.073	0.058	−0.015	0.003	0.023	0.009	−0.014	0.001
9	5.0	5	5	0.173	0.112	−0.061	0.008	0.023	0.016	−0.007	0.001
10	5.0	5	5	0.173	0.096	−0.077	0.008	0.023	0.103	+0.080	0.006
11	5.0	5	5	0.523	0.290	−0.233	0.020	0.023	0.164	+0.141	0.009
12	5.0	5	5	0.523	0.350	−0.173	0.020	0.023	0.119	+0.096	0.006
13	125.0	25	5	0.073	0.023	−0.050	0.003	0.023	0.060	+0.037	0.004
14	125.0	25	5	0.073	0.021	−0.052	0.001	0.023	0.061	+0.038	0.004
15	125.0	25	5	0.173	0.092	−0.081	0.008	0.023	0.089	+0.066	0.005
16	125.0	25	5	0.173	0.076	−0.097	0.004	0.023	0.105	+0.082	0.005
..	....	..	..	3.468	2.619	....	0.163	0.368	0.802	....	0.047

* PROCEDURE: Measured amounts of sodium carbonate and of cadmium chlorid were added to samples containing varying, but known quantities of sulphid and free and albuminoid ammonia. The ammonias were then determined in the usual manner. The samples were prepared from boiled Washington City tap water, a standard solution of ammonium chlorid and a solution of hydrogen sulphid.

boiling off of the sulphid in acid solution has been practiced in the laboratory of the referee for several years and has been found satisfactory. It is believed that this modification can now be recommended as official.

It has occurred to the referee that the methods of water analysis should be broadened to include the examination of products closely allied to water, such as brine and salt. In some instances the methods for water could be used without modification. In others, however, considerable modification would be necessary. It is believed that the interest of the future referees and collaborators in the work would be increased, and a real need for proper methods for the analyses of these products would be filled by the extension of the methods. In order that the association may take some action, this suggestion will be put in the form of a recommendation.

RECOMMENDATIONS.

It is recommended—

(1) That the method for the determination of barium[1], be adopted as official. (Second and final presentation of the method for action.)

(2) That the method for the determination of manganese[2] be adopted as an additional official method. (Second and final presentation of the method for action.)

(3) That the method for the determination of iodin in the presence of chlorin and bromin, page 380, be adopted as a tentative method. (First presentation of the method for action.) The method has not been published in the Proceedings as provided by By-law No. 7.

(4) That the method for the determination of bromin in the presence of chlorin but not iodin, page 381, be adopted as a tentative method. (First presentation of the method for action.) The method has not been published in the Proceedings as provided by By-law No. 7.

(5) That the method given below for free and albuminoid ammonia in samples containing sulphid be adopted as official. (First presentation of the method for action.) The method has not been published in the Proceedings as provided by By-law No. 7.

FREE AND ALBUMINOID AMMONIA.

(In samples containing sulphid.)

REAGENTS.

(a) *N/2 solution of sulphuric acid.*

(b) *5 N solution of sodium carbonate.*

(c) *Ammonia-free water.*

(d) *Standard ammonium chlorid solution.*—One cc. is equivalent to 0.01 mg. of nitrogen in the form of ammonia (NH_3).

(e) *Nessler reagent.*—Dissolve 50 grams of potassium iodid in a minimum quantity of cold water. Add a saturated solution of mercuric chlorid until a slight permanent precipitate is formed. Add 400 cc. of a solution containing 200 grams of potassium hydroxid (or an equivalent quantity of sodium hydroxid), dilute to 1 liter, allow to settle, and decant.

(f) *Alkaline potassium permanganate solution.*—Dissolve 200 grams of potassium hydroxid and 8 grams of potassium permanganate in water and dilute to 1 liter.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, 4: 86.
[2] *Ibid.*, 85.

DETERMINATION.

Place 500 cc. of the sample in a beaker or casserole, add 30 cc. excess of N/2 sulphuric acid solution. Boil the solution carefully until free of sulphid (about 20 minutes). Add about 300 cc. of distilled water and 8 cc. of 5 N sodium carbonate solution to a distillation flask connected as described in the official method[1], and distil until free from ammonia. Cool and add the cooled sample which has been freed from sulphid. Proceed with the distillation, addition of alkaline permanganate solution, etc., as described in the official method.

(6) That the methods on water be extended to cover the examination of allied products, such as brine and salt.

(7) That continued study be given to the determination of iodin and bromin and the heavy metals and to the use of equivalents in studying the character of waters.

REPORT ON SOILS.

By C. B. Lipman[2] (University of California, Berkeley, Calif.),
Referee on Soils.

The report covers work undertaken two years ago on "The Total Phosphorus Determination". Of those who had promised to collaborate, only two sent in reports; namely, H. C. McLean (Agricultural Experiment Station, New Brunswick, N. J.) and L. A. Steinkoenig (Bureau of Soils, Washington, D. C.). The two former official methods[3] for total phosphorus determination, namely, the magnesium nitrate method and the sodium peroxid method which the association adopted two years ago as tentative[4], were studied. Two California soils, the Davis soil and the Oakley soil, were employed. The results obtained by the two collaborators are given in the following table:

Determination of total phosphorus—results as phosphorus of water-free soil.

DAVIS SOIL				OAKLEY SOIL			
MCLEAN		STEINKOENIG		MCLEAN		STEINKOENIG	
Magnesium nitrate method	Sodium peroxid method	Magnesium nitrate method	Sodium peroxid method	Magnesium nitrate method	Sodium peroxid method	Magnesium nitrate method	Sodium peroxid method
per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
0.066	0.061	0.056	0.066	0.045	0.045	0.043	0.036
0.066	0.063	0.055	0.067	0.043	0.046	0.043	0.035
0.065	0.061	....	....	0.045	0.045	....	....
0.064	0.061	....	....	0.045	0.044	....	....

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[1] *Assoc. Official Agr. Chemists, Methods,* 1916, 36.
[2] Presented by W. H. MacIntire.
[3] *Assoc. Official Agr. Chemists, Methods,* 1916, 25-6.
[4] *J. Assoc. Official Agr. Chemists,* 1920, **4**: 295.

In Steinkoenig's case, two additional methods to those for which directions were issued were tried on the same samples. On studying these results, your referee finds that determinations made in quadruplicate by McLean in every case with each method and with each soil agreed well among themselves. The duplicate results obtained by Steinkoenig also agree well among themselves. The agreement between the results of the two collaborators is not so good as could be desired. Mc Lean's results show that there is little choice between the two methods, though there is a possibility that the magnesium nitrate method is a little more thorough. This is just a possibility, however, and is offset by a certain difficulty in dehydration which was experienced by McLean with the method. With the Oakley soil even this slight difference between the two methods seems to have disappeared. In the case of McLean's results, the magnesium nitrate method is superior for the Oakley soil and the sodium peroxid method superior for the Davis soil. On the whole, your referee can not see sufficient difference between the figures submitted to justify drawing any other conclusion than that both methods so far adopted remain as they are for at least another year.

RECOMMENDATION.

It is recommended that the study of the determination of total phosphorus in soil be continued with a larger number of soils and that an attempt be made to secure more collaborators.

There was no associate referee on the nitrogenous compounds of soils and no report on this subject was presented.

REPORT ON THE LIME ABSORPTION COEFFICIENT OF SOILS.

By W. H. MacIntire (Agricultural Experiment Station, Knoxville, Tenn.), *Associate Referee.*

During the past two years abnormal conditions have seriously handicapped any extensive work along collaborative lines. The unusual demand placed upon station facilities has necessitated the suspension of all work not of primary importance. For this reason, those who offered collaborative assistance have been unable to fulfill the proffer made.

However, following the more exhaustive previous report[1] made upon the results secured through selective collaboration upon representative procedures, and the adoption of the recommendation that the Jones

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 108.

calcium acetate method be further studied, the writer has given, with some assistance, special attention to the fundamentals of the method and the technic of the procedure. Exchange of opinions as to the merits of the method has brought out the fact that the Jones method is well adapted to its intended use. Without in any sense being derogatory to other procedures, which may be well adapted to certain conditions, it seems to be the consensus of opinion that the Jones calcium acetate method fills the need of an absorption method to replace the abandoned sodium nitrate procedure.

Results from the Vermont and Virginia Agricultural Experiment Stations indicate a close parallel between results secured by the Jones procedure and those obtained by the method of Veitch. While it is true that the principles underlying the Veitch procedure are fundamentally correct from a physical chemical viewpoint, the method is not so well adapted to rapid laboratory manipulation as is the Jones procedure.

Results secured during three years, and in particular the slight variations reported in the factor, found applicable to different soils, together with the factor utilized in the Jones procedure, tend to justify the conclusion that the method, after certain slight editorial changes and modifications in technic are introduced, may well be adopted tentatively as a laboratory procedure for the purpose of determining the lime absorption coefficient of soils. These slight modifications have been presented to the originator of the procedure, C. H. Jones, Agricultural Experiment Station, Burlington, Vt., and have received his sanction.

JONES METHOD FOR DETERMINING LIME ABSORPTION COEFFICIENT.

Take 5.6 grams of soil, add 0.5 gram of calcium acetate (tested reagent), place in a 3-inch mortar and mix with a pestle. Add sufficient water (room temperature) to make a fairly stiff paste. Pestle for 20 seconds, add 30 cc. of water and continue the mixing for 30 seconds. Wash into a 200 cc. flask and keep the bulk down to about 160 cc. Let stand with occasional shaking for 15 minutes. Make up to a bulk of 200 cc., mix and filter through a dry filter. Discard the first 10–15 cc. which may be cloudy. For rapidity and efficiency the Büchner funnel is recommended. Titrate 100 cc. of the clear filtrate, using phenolphthalein as an indicator, with N/10 sodium hydroxid. This reading multiplied by 2 gives the number of cc. of N/10 alkali required to neutralize the acetic acid in 200 cc. of the solution. This figure times the factor 1.8 times 1000 indicates the pounds of lime (CaO) required per 2,000,000 pounds of soil.

RECOMMENDATION.

If further study of the tentative method be thought advisable, it is recommended that such a study be made upon the factor of soil type in its influence upon variation in the factor to be applied.

REPORT ON INORGANIC PLANT CONSTITUENTS.

By J. H. MITCHELL[1] (Clemson Agricultural College, Clemson College, S. C.), *Referee.*

I wish to review, briefly, the work that has been done on this subject during the past five years. In 1915 A. J. Patten[2] showed that the present molybdate method for the determination of iron, aluminium, phosphorus, calcium and magnesium was not accurate when used on material containing a large amount of phosphorus and low percentage of calcium and magnesium. This condition exists in such material as the ash from cereals, legumes and many seeds. No collaborative work was done in 1915, but the referee did some work on a method for calcium and magnesium. The following recommendation was made: "That suitable methods be devised for the determination of iron, aluminium, calcium and magnesium in the ash from seeds".

In 1916 Patten[3] presented a method for determining calcium and magnesium in the presence of phosphoric acid, iron and aluminium; also a method for determining manganese colorimetrically. No collaborative work was done, but it was recommended that the methods as outlined for calcium, magnesium and manganese be further studied on solutions approximating the composition of the ash of cereals, seeds, etc.

In 1917 and 1918 no recommendations were made, so the present referee decided to continue the study of the methods for the determination of calcium, magnesium and manganese in the presence of a large amount of phosphorus, as recommended and approved in 1916.

A synthetic ash solution was made of approximately the compostion of the ash of certain seeds, cereals and legumes. It contained iron, aluminium, phosphorus, calcium, magnesium and manganese in the following proportions:

	per cent
Ferric oxid (Fe_2O_3)	1.43
Aluminium oxid (Al_2O_3)	1.93
Phosphorus pentoxid (P_2O_5)	38.73
Calcium oxid (CaO)	5.46
Magnesium oxid (MgO)	4.30
Manganomanganic oxid (Mn_3O_4)	0.30

A sample of this solution was sent to eight chemists. The following table shows the results obtained:

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[1] Presented by W. L. Latshaw.

[2] *J. Assoc. Official Agr. Chemists*, 1917, **3**: 153.

[3] *Ibid.*, 1920, **3**: 329.

Results on synthetic solution.

CHEMIST	CALCIUM OXID	MAGNESIUM OXID	MANGANOMANGANIC OXID
	per cent	*per cent*	*per cent*
Benj. Freeman, Clemson Agricultural College, Clemson College, S. C.	5.32	4.82	....
A. P. Kerr, Department of Agriculture and Immigration, Baton Rouge, La.	5.60	4.34	...
J. F. King, State Department of Agriculture, Atlanta, Ga.	...	4.20	....
W. L. Latshaw, Agricultural Experiment Station, Manhattan, Kans.	5.45 5.54 5.48	4.47 4.53 4.50	....
J. S. McHargue, Agricultural Experiment Station, Lexington, Ky.	5.27 5.27 5.33	4.66 4.72 ...	.... 0.341 ....
J. H. Mitchell	5.44 5.44 5.50	4.24 4.25 4.29	0.332 0.326 ....
Percy O'Meara, Agricultural Experiment Station, E. Lansing, Mich.	5.57 5.54 5.60	4.80 4.76 4.76	0.322 0.325 0.334
Average	5.45	4.52	0.33
Theory	5.46	4.30	0.30
Difference	0.01	0.22	0.03

These results vary somewhat, yet taken as a whole they are very good, especially those for calcium. The results on magnesium show a little wider variation and are, on the average, higher than the theory. Owing to a difficulty in obtaining potassium periodate, only a few results were obtained for manganese. These, however, were very good.

The following is an outline of the methods sent to the different chemists:

CALCIUM.

Remove 25 cc. of the solution, representing 0.5 gram of ash, and dilute to 200 cc., add a few drops of alizarine or methyl orange and make slightly ammoniacal. Add very dilute hydrochloric acid (1 to 10) until the solution is just faintly acid, followed by 10 cc. of N/2 hydrochloric acid and 10 cc. of 2.5% oxalic acid. Boil the solution and add, with constant stirring, 15 cc. of a saturated solution of ammonium oxalate, and continue to heat until the precipitate becomes granular. Cool and add, with constant stirring, 8 cc. of 20% sodium acetate solution, and allow to stand 12 hours. Filter, and wash with hot water until free from chlorids. Dissolve the precipitate in hot, dilute sulphuric acid and titrate with N/10 potassium permanganate solution. In dissolving the precipitate it is best to first wash it off the paper into a beaker, and dissolve the portion remaining on the paper with hot dilute sulphuric acid (1 cc. N/10 $KMnO_4$ = 0.0028 gram CaO).

MAGNESIUM.

To the combined filtrate and washings from the calcium determination, add 25 cc. of strong nitric acid and evaporate to dryness. Take up with dilute hydrochloric acid and make to a volume of about 100 cc. Add 5 cc. of a 10% sodium citrate solution and 10 cc. of sodium hydrogen phosphate solution, or enough to precipitate all of the magnesium. Add dilute ammonium hydroxid, with constant stirring, until the solution is faintly alkaline; then add about 25 cc. of strong ammonium hydroxid and set aside in a cool place overnight. Filter and wash with 2.5% ammonium hydroxid solution. Dissolve the precipitate in dilute hydrochloric acid and reprecipitate as before. Allow to stand several hours, filter and wash free of chlorids with 2.5% ammonium hydroxid solution, ignite and weigh as magnesium pyrophosphate.

MANGANESE.

To 25 cc. of the solution, representing 0.5 gram of ash, add about 15 cc. of concentrated sulphuric acid and evaporate to expel hydrochloric acid. When the solution has reached a small volume, add 5–10 cc. of nitric acid and continue the evaporation. It is neither necessary nor desirable to evaporate until dense fumes appear, since the ferric sulphate then dissolves with difficulty. Nitric acid may be present, but not hydrochloric. Add water, a little at a time, heat until the iron salts have dissolved, and dilute to about 150 cc. Add about 0.3 gram of potassium periodate, heat just to boiling for a few minutes and allow to cool. The standard is prepared in the following manner: To a volume of water equal to the sample, add 15 cc. of sulphuric acid and sufficient pure ferric nitrate, free from manganese, so that this solution will contain about the same amount of iron (about 1 per cent) as the sample. Add standard permanganate solution, noting the amount, until the color is slightly darker than the sample, and then the same amount of periodate and boil as before. When cool, transfer the sample and standard to 250 cc. flasks and make to the mark. If the color is weak it may be necessary to make to smaller volume. Compare the colors in any standard colorimeter. Report as Mn_3O_4.

These methods seem to be quite promising, and with further study probably can be perfected so as to give excellent results.

The associate referee, W. L. Latshaw, has taken considerable interest in this work. In addition to making the determinations of calcium and magnesium, as outlined in these methods, he has made a comparative study of the present official method and these new methods on samples of cottonseed meal and alfalfa meal. He has also been working on a method for the determination of iron and aluminium in the filtrate from the magnesium. His results and comments are as follows:

I am highly pleased with the results obtained by the methods as outlined. They have a distinct advantage over the old procedure for the determination of calcium and magnesium in material high in phosphorus, as it seems impossible even with several reprecipitations of the phosphorus, iron and aluminium, to remove all of the calcium and magnesium. I tried this out very carefully, using the sample submitted, also the cottonseed meal and alfalfa meal. The results on calcium and magnesium from the collaborative sample and cottonseed meal sample were very low, as compared with the results obtained by the methods outlined for collaborative work, while the results obtained with alfalfa showed much better agreement with the two methods, although somewhat lower with the official method. I attributed these low results to the larger amount of phosphorus in the cottonseed meal and in the sample submitted for analysis. The amount was so much less in the case of alfalfa that it did not make it difficult to wash the precipitate free of calcium and magnesium, thus allowing for the fairly good comparison in the case of the alfalfa.

I have tried several procedures for the recovery of iron and aluminium after the removal of calcium and magnesium. While I have not obtained any definite results, the work performed has been sufficient to convince me that is is entirely feasible to find a good working method, and I think we should work towards this end, another year.

I have done a little work on phosphorus. The fusion method I used for determining phosphorus was originally worked out for the determination of iodin in thyroid glands of animals and is applicable to halogens. I think that some work should be done during the coming year on phosphorus and chlorin, especially in the seeds of plants.

The following table indicates the results obtained by these different methods. The material was ashed in a platinum dish by the aid of an electric muffle furnace, according to the official method for the preparation of the ash. Ten grams of the material were used in the case of the cottonseed meal, and 5 grams of alfalfa meal.

Determination of calcium and magnesium in cottonseed and alfalfa meal.

(Analyst, W. L. Latshaw.)

MATERIAL	PROPOSED METHOD		OFFICIAL METHOD	
	Calcium oxid	Magnesium oxid	Calcium oxid	Magnesium oxid
	per cent	*per cent*	*per cent*	*per cent*
Cottonseed meal	0.29	0.94	0.064	0.324
	0.30	0.93	0.105	0.360
	0.23	0.91	0.105	....
Alfalfa meal	2.20	0.37	1.96	0.335
	2.24	0.37	1.86	0.310
	2.05	0.40	....	....

The phosphoric acid content of the cottonseed meal was approximately 2 per cent, while that of the alfalfa meal was in the neighborhood of 0.5 per cent.

RECOMMENDATIONS.

It is recommended—

(1) That the methods as outlined in this report for calcium and magnesium be made tentative for the ash of such material as seeds, and that further work be done on them during the coming year.

(2) That the colorimetric method for manganese be further studied, possibly with the use of several oxidizing agents.

(3) That a method be devised for the determination of iron and aluminium in the filtrate from the magnesium.

(4) That a study of the present method for phosphorus and chlorin be made on material corresponding to the ash of seeds.

REPORT ON INSECTICIDES AND FUNGICIDES[1].

By O. B. WINTER (Agricultural Experiment Station, E. Lansing, Mich.), *Referee.*

The insecticide work for 1918 and 1919, in accordance with the recommendations of the association, has been a further cooperative study along the following lines:

1. The determination of lead, zinc and copper in a product which may contain arsenic, antimony, lead, copper, zinc, calcium, magnesium, etc., *e. g.*, Bordeaux-lead arsenate with Bordeaux-zinc arsenite.
2. The removal of the color in London purple before the determination of the total arsenic.
3. The comparison of the official iodin, the Gyory bromate, and the Jamieson iodate methods for the determination of arsenic.

The referee has also considered methods for the determination of the calcium, magnesium, zinc, and soluble arsenic in calcium and magnesium arsenates and zinc arsenite, the total arsenic in magnesium arsenate, and the arsenic trioxid in calcium and magnesium arsenates, since the association has no methods, either official or tentative, for making these determinations.

Early in 1918, five collaborators, including the referee, manifested a willingness to cooperate in the work, and samples were sent to them with directions for carrying on the work. Only two reports were received during 1918. The same work was continued in 1919 and four additional reports were received.

BORDEAUX-LEAD ARSENATE WITH BORDEAUX-ZINC ARSENITE.

The sample of Bordeaux-lead arsenate with Bordeaux-zinc arsenite was prepared by thoroughly mixing known quantities of lead arsenate, zinc arsenite, and dry Bordeaux mixture. Each of these materials was prepared and analyzed according to the directions used in preparing similar materials for the insecticide work in 1917[2]. By calculation, this sample should contain 17.38 per cent of lead oxid; 7.49 per cent of copper; and 11.31 per cent of zinc oxid.

The methods sent to the collaborators for the determination of lead, copper, and zinc were as follows:

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[1] Presented by A. J. Patten.

[2] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 134.

GENERAL PROCEDURE FOR THE ANALYSIS OF A PRODUCT CONTAINING ARSENIC, ANTIMONY, LEAD, COPPER, ZINC, IRON, CALCIUM, MAGNESIUM, ETC.

(Applicable to such preparations as Bordeaux-lead arsenate; Bordeaux-zinc arsenite; Bordeaux-Paris green; Bordeaux-calcium arsenate, etc.)

LEAD OXID (PbO)[1].

COPPER (CuO)[1].

ZINC OXID (ZnO)[2].

The results obtained on this sample are found in Table 1.

TABLE 1.

Bordeaux-lead arsenate with Bordeaux-zinc arsenite.

ANALYST	LEAD OXID (PbO)	COPPER (Cu)	ZINC OXID (ZnO)
	per cent	*per cent*	*per cent*
1. J. J. T. Graham, Bureau of Chemistry, Washington, D. C.	17.61	7.78	10.80
	17.35	7.74	10.88
	17.53	7.78	11.12
	17.31	...	....
Average....................	17.45	7.77	10.93
2. C. B. Stone, Bureau of Chemistry, Washington, D. C.	17.52	7.56	11.14
	17.58	7.60	11.24
	17.51	7.64	11.00
	17.43	7.52	....
	17.57	...	....
Average....................	17.52	7.58	11.13
3. Percy O'Meara, Agricultural Experiment Station, E. Lansing, Mich.	17.51	...	11.23
	17.38	7.24	11.30
Average....................	17.44	7.24	11.27
4. O. B. Winter, Agricultural Experiment Station, E. Lansing, Mich.	17.25	7.52	11.15
	17.41	7.50	11.30
Average....................	17.33	7.51	11.23
General average..............	17.44	7.53	11.14
Theory......................	17.38	7.49	11.31

DISCUSSION.

As stated in the 1917 Report on Insecticides[3], the problem involved in making the determinations found in Table 1 is a question of the

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 136.
[2] *Ibid.*, 137.
[3] *Ibid.*, 134.

separation of lead, copper and zinc in the presence of other compounds. Since in such a problem it is not easy to make a comparison with any standard method, especial care was taken to analyze the individual compounds before the composite sample was prepared. These results are given in the last line in the table. It must be borne in mind, however, that these results would vary somewhat if it had been possible for each analyst to make the individual determinations.

It is to be regretted that so few chemists have cooperated in this work, especially since the results do not agree so well as might be desired. The small amount of work done hardly justifies that any action be taken on the method but it should receive further cooperative study.

LONDON PURPLE.

The London purple used in this work was a commercial sample which had been received at the writer's laboratory for analysis.

TOTAL ARSENIC.

REAGENTS.

Standard solutions.—As given under Paris green[1].

Zinc oxid-sodium carbonate mixture.—Mix four parts of zinc oxid with one part of dry sodium carbonate.

Blood charcoal.

DETERMINATIONS.

Official iodin method.—Determine as directed under London purple[2].

Zinc oxid-sodium carbonate method.—Weigh an amount of sample equal to the arsenic trioxid equivalent of 250 cc. of the standard iodin solution. Mix the sample thoroughly with several times its weight of the zinc oxid-sodium carbonate mixture in a shallow porcelain crucible and cover with a layer of the same mixture. Place the crucible, uncovered, in a muffle, heat, gently at first and finally for about 15 minutes at full heat. (The mass will not sinter.) Cool, transfer to a distillation flask and proceed according to the official distillation method for total arsenic in Paris green[3].

Adsorption method.—Proceed as directed under the official iodin method for total arsenic in Paris green, except that 3–4 grams of blood charcoal are added to the distillation flask before beginning the distillation.

The results obtained on this sample are found in Table 2.

COMMENTS BY ANALYSTS.

One analyst found it impossible to get a colorless solution in the official method. A colorless distillate was obtained by all the collaborators in the zinc oxid-sodium carbonate and blood charcoal adsorption methods. Two analysts noted a tendency to foam in the blood charcoal

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 63.
[2] *Ibid.*, 66.
[3] *Ibid.*, 64.

TABLE 2.

Total arsenic expressed as arsenic trioxid in London purple.

ANALYST	OFFICIAL METHOD	ZINC OXID-SODIUM CARBONATE METHOD	ADSORPTION METHOD
	per cent	*per cent*	*per cent*
1. J. J. T. Graham	39.67 39.60 39.52 39.56	39.78 39.87 39.90 39.90	39.03 39.15 39.11 ....
Average	39.59	39.86	39.10
2. C. B. Stone	39.52 39.48 39.56	39.72 39.72 39.87 39.88	39.13 39.13 39.27 39.27
Average	39.52	39.80	39.20
3. Percy O'Meara	39.50 39.62	40.20 40.10	39.80 39.80 40.30 40.20
Average	39.56	40.15	40.03
4. O. B. Winter	39.57 39.63 39.57	39.70 39.74 39.76	39.56 39.69 ....
Average	39.59	39.73	39.63
General average	39.56	39.89	39.49

adsorption method and a third analyst stated that the charcoal collected on the sides of the flask during the distillation and required an occasional shaking. Two analysts expressed a preference for the zinc oxid-sodium carbonate method, while a third preferred the blood charcoal adsorption method.

DISCUSSION.

A study of Table 2 shows that the results by the different analysts for total arsenic in London purple by the official method agree very closely indeed. With one exception the results by the zinc oxid-sodium carbonate method also agree well, but run about 0.30 per cent higher than by the official method. The results by the adsorption method are very low by two analysts. They are high by one analyst, and agree fairly well with the official method by another analyst. Undoubtedly, these methods deserve further study.

PARIS GREEN.

The Paris green used in this work was a commercial sample received at the writer's laboratory for analysis.

TOTAL ARSENIC.

REAGENTS.

Starch indicator.—Prepare as directed under Paris green.

Standard arsenic trioxid solution.—Prepare as directed under Paris green[1].

Standard iodin solution[1].—Prepare as directed under Paris green.

Standard potassium bromate solution.—Dissolve 1.687 grams of pure potassium bromate (dried at 140°C.) in water and dilute to 1 liter. One cc. of this solution is equivalent to 0.00300 gram of arsenic trioxid.

Standard potassium iodate solution.—Dissolve 3.244 grams of potassium iodate (dried at 140°C.) in water and dilute to 1 liter. One cc. of this solution is equivalent to 0.00300 gram of arsenic trioxid.

DETERMINATIONS.

Official iodin method.—Determine as directed under Paris green[2].

Gyory bromate method.—Proceed as directed under the official iodin method, except that the distillate is made to 500 instead of 1000 cc[3].

(a) Transfer a 100 cc. aliquot to a 500 cc. Erlenmeyer flask, heat almost to boiling and titrate with the standard bromate solution, using methyl orange as indicator (the indicator should not be added until near the end of the reaction).

(b) Same as (a) except titrate without heating.

Jamieson iodate method.—Proceed as directed under the Gyory bromate method until the distillate is made to a volume of 500 cc. Transfer 100 cc. aliquots to 250–500 cc. glass stoppered bottles, add 6 cc. of chloroform and about 10 cc. of concentrated hydrochloric acid, and titrate with the standard iodate solution until the disappearance of the iodin from the chloroform, when the mixture is thoroughly shaken[4].

NOTE.—In both the bromate and iodate methods the number of cc. of standard solution used, multiplied by 2, represents the per cent of total arsenic in the sample expressed as arsenic trioxid.

ARSENIC TRIOXID.

C. C. Hedges modified method.—Determine as directed under Paris green[5].

Gyory bromate method.—Weigh an amount of the sample equal to the arsenic trioxid equivalent of 50 cc. of the standard bromate solution, transfer to a beaker, add about 50 cc. of water and 15 cc. of concentrated hydrochloric acid, heat almost to boiling, and titrate with the standard bromate solution as directed under total arsenic.

Jamieson iodate method.—Weigh an amount of sample equal to the arsenic trioxid equivalent of 50 cc. of the standard iodate solution, transfer to a 250–500 cc. glass stoppered bottle, add 20 cc. of water and 30 cc. of concentrated hydrochloric acid, and titrate with the standard iodate solution as directed under total arsenic.

NOTE.—In these two latter methods, the number of cc. of standard solution used, multiplied by 2, represents the per cent of arsenious oxid in the sample.

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 63.
[2] *Ibid.*, 64.
[3] *Z. anal. Chem.*, 1893, **32**: 415.
[4] *J. Ind. Eng. Chem.*, 1919, **10**: 291.
[5] *Assoc. Official Agr. Chemists, Methods*, 1916, 64.

Remarks on the directions for analysis:

1. The reaction of a bromate, and of an iodate on arsenic trioxid undoubtedly may be represented by the following two equations[1]:

$$3\ As_2O_3 + 2\ KBrO_3 + HCl = 3\ As_2O_5 + 2\ KBr + HCl$$
$$As_2O_3 + KIO_3 + 2\ HCl = As_2O_5 + ICl + KCl + H_2O.$$

2. Sodium bromate or sodium iodate may be used instead of the potassium salts.

3. Both the standard bromate and iodate solutions were found approximately accurate by the referee. However, it is not definitely known that these salts as found on the market are sufficiently pure for making up the standard solutions as directed. Therefore, it is desirable that each collaborator standardize his solution against arsenic trioxid, and report the results.

The results obtained on this sample are shown in Tables 3 and 4.

TABLE 3.

Total arsenic expressed as arsenic trioxid in Paris green.

ANALYST	OFFICIAL METHOD	BROMATE METHOD		IODATE METHOD
		(Hot)	(Cold)	
	per cent	*per cent*	*per cent*	*per cent*
1. J. J. T. Graham	56.40	56.26	57.09	56.47
	56.40	55.99	57.09	56.30
	56.51	56.09	57.09	56.56
	56.59	56.46	57.09	56.19
	....	56.91	....	....
	....	56.81	....	....
Average..................	56.48	56.42	57.09	56.38
2. C. B. Stone	56.57	56.60	56.97	56.54
	56.63	56.60	56.79	56.36
	56.57	56.79	56.97	56.54
	....	....	56.97	56.53
Average..................	56.59	56.66	56.93	56.49
3. Percy O'Meara	56.40	57.00	57.00	56.53
	56.60	57.00	57.10	56.63
	56.80	....	....	56.63
	56.80	....	....	....
Average..................	56.65	57.00	57.05	56.60
4. O. B. Winter	56.49	56.55	56.80	56.40
	56.33	56.45	....	56.50
Average..................	56.41	56.50	56.80	56.45
General average............	56.53	56.65	56.97	56.48

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[1] A. B. Prescott and O. C. Johnson. Qualitative Chemical Analysis. 6th ed., 1915, 349, 358; *J. Ind Eng. Chem.*, 1918, **10**: 291.

TABLE 4.

Arsenic trioxid in Paris green.

ANALYST	OFFICIAL METHOD	BROMATE METHOD		IODATE METHOD
		(Hot)	(Cold)	
	per cent	*per cent*	*per cent*	*per cent*
1. J. J. T. Graham	....	55.42	....	55.58
	....	55.42	....	55.58
	....	55.52	....	55.58
Average....................	....	55.45	....	55.58
2. C. B. Stone	....	55.49	....	55.75
	....	55.68	....	55.55
	....	55.49	....	55.55
Average....................	....	55.55	....	55.62
3. Percy O'Meara	55.5	56.00	55.10*	55.2 56.2
	55.6	55.90†	55.20‡	55.4 55.9
	....	55.80	55.60§	55.0 55.9
	....	....	55.20§	55.2 56.2
	....	....	55.20	... ...
Average.....................	55.55	55.90	55.26	55.63
4. O. B. Winter	55.60	55.60	55.20	55.60
	55.50	55.60	55.40	55.70
	....	....	....	55.50
Average.....................	55.55	55.60	55.30	55.60
General average..............	55.55	55.62	55.28	55.61

* Did not warm at all.
† Boiled about 1 minute.
‡ Heated to 80°C.
§ Heated to about 55°C.

DISCUSSION.

A study of Table 3 and the comments on this work by the analysts, shows that the results by the Jamieson iodate method for determining total arsenic are very good. However, this method appears to consume too much time for ordinary commercial work and the technique is somewhat disagreeable.

The hot Gyory bromate method gave good results in the hands of most of the analysts, and it is very convenient. However, since the results by one of the analysts when using this method are low and he has shown that when the solution is not heated quite so high, his results are higher and more uniform, it may be well to state a maximum temperature for making this determination.

The cold bromate method runs too high—apparently the indicator is not sufficiently sensitive in a cold solution.

When determining the arsenious acid it is necessary to heat the sample somewhat in order to insure complete solution.

CALCIUM AND MAGNESIUM ARSENATES AND ZINC ARSENITE.

As indicated earlier in this paper, the methods for the analysis of insecticides should include directions for analyzing calcium and magnesium arsenates and zinc arsenite. Official methods for the total arsenic in calcium arsenate and the total arsenic and arsenic trioxid in zinc arsenite have been adopted. Standard methods for the total arsenic in magnesium arsenate, for the arsenic trioxid in calcium and magnesium arsenates, and for the water-soluble arsenic in all three of the above-named materials are needed. Possibly methods for the complete analysis of these compounds should be adopted.

J. J. T. Graham, associate referee on this subject, will present a paper on some work which he has done on the water-soluble arsenic in calcium arsenate. He states that the official method for the determination of the water-soluble arsenic in lead arsenate is used for the determination of the water-soluble arsenic in calcium arsenate in his laboratory. This method is also in use in the writer's laboratory. The methods adopted for the total arsenic and the water-soluble arsenic in lead arsenate are also in use in the writer's laboratory for the determination of the total and water-soluble arsenic in magnesium arsenate and the water-soluble arsenic in zinc arsenite. Since the association has neither official nor tentative methods for making these determinations, the referee believes the above methods should be considered.

It should also be stated that considerable complaint has come to the attention of the Michigan Agricultural Experiment Station regarding the burning of foilage, due to spraying with calcium and magnesium arsenates. Some work along this line has recently been done in the referee's laboratory, and a paper giving the results will be presented.

SUGGESTIONS FOR FUTURE WORK.

1. No satisfactory method was found for removing the color in order to determine the arsenic trioxid in London purple. The end point can be read quite accurately by the present official method but it is possible that a method may be found which will take less time and prove just as accurate.

2. It may be unnecessary to use carbon dioxid-free water for determining the water-soluble arsenic in lead arsenate.

RECOMMENDATIONS.

It is recommended—

(1) That further cooperative work be done on the methods for the determination of lead, copper and zinc in a compound that may contain arsenic, antimony, lead, copper, zinc, iron, calcium, magnesium, etc.

(2) That further cooperative work be done on the comparison of the zinc oxid-sodium carbonate and the blood charcoal adsorption methods with the official iodin method for the determination of the total arsenic in London purple.

(3) That further cooperative work be done on the comparison of the Gyory bromate and the Jamieson iodate methods for the titration of arsenic trioxid in hydrochloric acid solution, with the official iodin method for the determination of arsenic trioxid; and that special emphasis be placed on the temperature of the solution when titrating with potassium bromate.

(4) That the official method for the determination of total arsenic in lead arsenate be made official for the determination of total arsenic in magnesium arsenate; and that the official method for arsenic trioxid in lead arsenate be made official for the arsenic trioxid in calcium and magnesium arsenates.

(5) That a study be made of methods for the determination of calcium, magnesium and zinc, respectively, in calcium and magnesium arsenates and zinc arsenite.

(6) That a study be made of methods for the determination of the soluble arsenic in calcium and magnesium arsenates and zinc arsenite; and that special attention be given that these methods be measures of the safety for the use of these compounds for spraying purposes.

THE SOLUBILITY OF CALCIUM AND MAGNESIUM ARSENATES IN CARBON DIOXID AND ITS RELATION TO FOLIAGE INJURY.

By A. J. PATTEN (Agricultural Experiment Station, E. Lansing, Mich.).

In 1915, Scott and Siegler[1] published some results of spraying tests with calcium arsenate that seemed to recommend this material as a promising substitute for lead arsenate. Since then it has been put upon the market in considerable quantities by several manufacturers, and during 1919 magnesium arsenate was first offered for sale by one manufacturer.

These new preparations contain from 5 to 10 per cent more total arsenic oxid (As_2O_5) than lead arsenate and can be produced at a somewhat lower cost. When first put upon the market, calcium arsenate showed rather high solubilities but of the samples analyzed in the writer's laboratory during 1919, only one sample out of six, representing five manufacturers, showed a water-soluble content in excess of 1.50 per cent of arsenic oxid. In this one case the excess was only 0.37 per cent. The one sample of magnesium arsenate analyzed gave 1.25 per cent of water-soluble arsenic oxid.

In spite of these facts, however, many reports of more or less severe foliage injury have come from fruit growers who have used these arsenicals. The greatest injury has been obtained on peaches, but apples and other tree fruits were also damaged. In a spraying experiment conducted by the Horticultural Department of the Michigan Agricultural Experiment Station during the summer of 1919, foliage injury was noted on apples and peaches from the use of calcium arsenate (II) and severe injury from the use of magnesium arsenate (I). From the following table it will be seen that calcium arsenate (II) and magnesium arsenate (I) contained only 0.34 per cent and 1.25 per cent of water-soluble arsenic oxid, respectively. It is evident, therefore, that some other explanation must be found for the cause of the injury than the solubility in water.

In considering this problem, it was thought that carbon dioxid might be a factor, especially since the trees give off large quantities of carbon dioxid during the night, when the greatest deposition of moisture occurs in the form of dew. Therefore, it would be reasonable to suppose that the dew would contain a rather high concentration of carbon dioxid.

Acting upon this theory, the solubility of a number of samples of lead, calcium and magnesium arsenates in carbonated water was determined.

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[1] U. S. Dept. Agr. Bull. 278: (1915).

The official method for the determination of water-soluble arsenic was followed except that water saturated with carbon dioxid was used instead of pure water. The results are shown in the following table:

Results of solubility tests.

SAMPLE	TOTAL ARSENIC OXID	WATER-SOLUBLE ARSENIC OXID	CARBON DIOXID-WATER-SOLUBLE ARSENIC OXID	TOTAL ARSENIC OXID SOLUBLE IN CARBON DIOXID-WATER
	per cent	*per cent*	*per cent*	*per cent*
Lead arsenate (I)	32.05	0.49	0.29	0.90
Lead arsenate (II)	30.70	0.14	0.17	0.55
Lead arsenate (III)	31.90	0.57	0.23	0.61
Lead arsenate (IV)	31.70	0.49	0.29	0.91
Lead arsenate (V)	32.15	0.34	0.17	0.53
Calcium arsenate	47.80	1.87	21.6	45.2
Calcium arsenate (I)	46.75	0.34	17.3	37.0
Calcium arsenate (II)	44.75	0.92	16.9	37.6
Calcium arsenate (III)	43.40	0.45	9.9	22.9
Calcium arsenate (IV)	41.00	0.43	12.6	30.7
Calcium arsenate (V)	38.30	0.24	33.0	86.2
Magnesium arsenate (I)	32.13	1.25	13.4	41.7
Magnesium arsenate (II)	38.05	0.23	3.0	7.9

In studying these results, it will be noted that the solubility of lead arsenate was lower in carbon dioxid-water than in pure water in all but one sample. The differences were small, however, in all cases. With calcium arsenate there is considerable variation in the amount of arsenic oxid dissolved in carbon dioxid-water. This amount, however, bears no relation to the total or water-soluble arsenic and to the amount of free lime in the samples. The solubility of magnesium arsenate (I) was similar to that of the calcium arsenates. It was further demonstrated that by suspending 1 gram of magnesium arsenate (I) in 1 liter of water and passing carbon dioxid through it for 2 hours, with frequent shaking, 96 per cent of the arsenic oxid was dissolved.

After this work was completed, the sample of magnesium arsenate (II) was received from the manufacturer, and the results are shown in the table for the sake of comparison. It was claimed that this sample would show a low solubility, both in pure water and carbonated water. It will be seen, from the table, that it does exhibit characteristics quite different from those of magnesium arsenate (I). Upon passing carbon dioxid through a suspension of the sample in water for 2½ hours only 4.29 per cent of arsenic oxid was dissolved, or 11.28 per cent of the total. Its solubility in dilute mineral acids was also much less. Further investigation showed this sample to be magnesium pyroarsenate instead of the ordinary ortho form.

From the results presented herewith, together with the many reports of injury from varied sources, the evidence seems quite conclusive that the determination of water-soluble arsenic oxid is not a satisfactory measure of the safety in using these arsenicals. Whether calcium or magnesium arsenate under any circumstances can be used safely on tender foliage and, if so, whether its solubility in carbon dioxid can be used as a measure of the safety, are problems for the future. Furthermore, the insecticidal efficiency and the action of magnesium pyroarsenate on foliage must be investigated. All of these problems must be solved by field experiments, but the members of this association can assist greatly by exercising a careful chemical control over such experiments.

THE DETERMINATION OF WATER-SOLUBLE ARSENIC OXID IN CALCIUM ARSENATE.

AN INVESTIGATION TO DETERMINE THE CORRECTNESS OF THE OFFICIAL METHOD FOR WATER-SOLUBLE ARSENIC IN LEAD ARSENATE, WHEN APPLIED TO CALCIUM ARSENATE.

By J. J. T. GRAHAM (Bureau of Chemistry, Washington, D. C.), *Associate Referee on Insecticides and Fungicides.*

In the absence of an official method for the determination of water-soluble arsenic in calcium arsenate, the official method for water-soluble arsenic in lead arsenate[1] is being used for calcium arsenate by most of the laboratories charged with the examination of this product. In order to clear up several points in connection with the accuracy of this method, as applied to calcium arsenate, certain investigations were made which may be divided into two parts:

(1) The determination of whether or not shaking during the first part of the 24-hour digestion period gave any difference in results from shaking during the latter part of the period; (2), a comparison of the iodin titration method for the arsenic in the water extract, with the distillation and the gravimetric methods.

For the first part of the investigation three commercial calcium arsenates, A, B and C were used. Two-gram charges were transferred to Florence flasks with 1 liter of carbon dioxid-free water and placed in the constant temperature bath at 32°C. One set of determinations was started at 9 A. M. and shaken at 1-hour intervals during the first 8-hour period of the digestion, and the second set was started at 4 P. M. and

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[1] *Assoc. Official Agr. Chemists, Methods,* 1916, 68.

shaken at 1-hour intervals during the last 8-hour period of the digestion. At the expiration of the 24-hour digestion period for each set, they were removed from the bath, filtered, and the arsenic determined by the official method for water-soluble arsenic in lead arsenate[1], with the following results:

TABLE 1.

Water-soluble arsenic oxid.

SOLUTIONS SHAKEN DURING FIRST PART OF DIGESTION PERIOD			SOLUTIONS SHAKEN DURING LAST PART OF DIGESTION PERIOD		
Sample A	Sample B	Sample C	Sample A	Sample B	Sample C
per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
1.32	5.03	3.66	1.37	5.09	3.60
1.26	5.15	3.66	1.32	5.15	3.63
Average...1.29	5.09	3.66	1.35	5.12	3.62

These results are practically identical and show that it is immaterial whether the flasks are given the eight shakings during the first or the last part of the digestion period.

For the second part of the investigation, solutions were prepared as in the preceding case using four samples of commercial calcium arsenate, A, B, F and D, the shakings being made during the first part of the 24-hour digestion period. Extractions were made in quadruplicate and, after filtering, the solutions for each sample were combined in order to have sufficient material to make all determinations on the same solution. The arsenic was determined by the following methods:

(a) *Official method for water-soluble arsenic in lead arsenate*[1].

(b) *Distillation method.*—An aliquot of 250 cc. of the solution representing 0.5 gram of the sample, was placed in an Erlenmeyer flask, 4 cc. of sulphuric acid were added and the solution concentrated by boiling to about 50 cc. This was transferred to a distillation flask and the arsenic determined by the official distillation method[2], titrating the entire distillate.

(c) *Gravimetric method.*—In this determination aliquots representing 1 gram were used on solutions A, B and F, and aliquots of 0.5 gram were used on solution D. The aliquots were placed in Erlenmeyer flasks and boiled with 5 cc. of nitric acid. In the case of solutions A, B and F the boiling was continued until the volume was reduced to about 100 cc.

The solutions were transferred to beakers, cooled, and made alkaline with sodium hydroxid after adding 1–2 drops of phenolphthalein. They were then made very faintly acid with acetic acid and the arsenic was precipitated by adding a neutral solution of

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 68.

[2] *Ibid.*, 63.

silver nitrate with vigorous stirring[1]. The precipitate was allowed to settle, when it was filtered and washed with cold water. It was then dissolved off the paper with nitric acid (1 to 4), the paper thoroughly washed and 10 cc. of hydrochloric acid added, followed by 10 cc. of magnesia mixture. The acid was neutralized with ammonium hydroxid and about one-third of the volume of ammonia (sp. gr. 0.95) additional was added. The solution was stirred vigorously and allowed to stand overnight.

It was then filtered through a tared Gooch crucible, dried at 110°C., ignited in an electric muffle furnace (in which the temperature was gradually raised from low heat to about 900°C.) and finally weighed as magnesium pyroarsenate[2].

Table 2.

Comparison of results of water-soluble arsenic oxid by different methods.

METHOD	SAMPLE A	SAMPLE B	SAMPLE D	SAMPLE F
	per cent	*per cent*	*per cent*	*per cent*
Official method for lead arsenate	1.37	5.06	27.87	9.28
	1.37	5.06	27.72	9.26
Average..................	1.37	5.06	27.80	9.27
Distillation method	1.43	4.98	27.69	9.05
	1.43	4.98	27.64	9.00
Average..................	1.43	4.98	27.67	9.03
Gravimetric method	1.13	4.77	27.75	8.88
	1.08	4.80	27.54	8.77
Average..................	1.11	4.79	27.65	8.83

These results show good agreement by the three methods and indicate that the official method for the determination of water-soluble arsenic in lead arsenate can be used for the same determination in calcium arsenate.

SUMMARY.

In the determination of water-soluble arsenic in calcium arsenate it is immaterial whether the eight hourly shakings are made during the first or the last part of the 24-hour digestion period.

The official method for water-soluble arsenic in lead arsenate is applicable for the same determination in calcium arsenate.

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[1] Francis Sutton. Volumetric Analysis. 10th ed., 1911, 161.

[2] F. P. Treadwell. Analytical Chemistry. 3rd ed., 1911, 2: 206.

DRUG SECTION.

No report on drugs was made by the referee.

REPORT ON MEDICINAL PLANTS.

By ARNO VIEHOEVER (Bureau of Chemistry, Washington, D. C.), *Associate Referee.*

The report is divided into four parts:

I. Work concerning new sources of supplies or proper substitutes for drugs not now obtainable.

II. Value of volume weight determinations in the analysis of crude drugs and spices.

III Value of micro-sublimation in the analysis of plant products.

IV. Condition of domestic and imported drugs.

PART I.

Concerning new sources of supplies, attention was given to the utilization of a part of the ipecac plant generally referred to as "stem".

It has been shown by A. R. L. Dohme[1], and subsequently by different collaborators of the Bureau of Chemistry (E. H. Grant, J. B. Luther, Samuel Ginsburg and J. F. Darling, all of the U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, New York, N. Y.), that appreciable amounts of ether-soluble alkaloids are present in the so-called "stems". The United States Pharmacopœia limits the amount of stems to 5 per cent. Collective evidence has recently been obtained that this part, referred to as "stem", consists largely of the underground part of the axis, more properly referred to as rhizome; the young and still smooth roots, resembling the rhizome, probably at times have also been mistaken for stems. Steps have been taken to separate the material into roots, thin and smooth, and thick and wrinkled, into underground parts of the axis (rhizomes) and overground parts of the axis (stems). Further collaborative work, however, has been delayed, since it has not been possible to obtain sufficient supplies of "stemy" ipecac.

Your associate referee has also given some attention to the work of his associates, J. F. Clevenger, C. O. Ewing and E. E. Stanford, all of the Bureau of Chemistry, Washington, D. C., on new supplies, such as

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[1] *Am. J. Pharm.*, 1895, 67: 533.

piptostegia root (*Piptostegia pisonis Mart*) and man-root (*Ipomœa pandurata* (L.) Meyer). Both roots, like jalap root, are obtained from plants belonging to the *Convolvulaceæ*, and yield a purging and more or less resinous-like alcoholic extract[1].

The piptostegia root, a South American product, which reached this country under the misleading name "Brazilian Jalap", may prove to be a valuable product and a parallel drug to jalap. Concerning the value of the domestic man-root, the opinion is more divided. Further work on both substitutes appears justified, especially if it is carried on with a view to obtaining the active principles, which are glucosidal and quite possibly of saponin character.

Upon the suggestion of G. W. Hoover, the referee on drugs, work was done on the following:

I. Spanish digitalis (*Digitalis thapsi* L.)
II. Egyptian henbane (*Hyoscyamus muticus* L.)
III. Rhaponticum root (*Rheum rhaponticum* L.)

The object was to find in the literature and in new investigations tests which permitted ready identifications and differentiation of those substitutes from the official drugs, in whole as well as in the powdered state.

SPANISH DIGITALIS (*Digitalis thapsi* L.)

The distinguishing characters of both the Spanish and official digitalis are as follows:

DIGITALIS[2].

MACROSCOPIC CHARACTERS.

Digitalis purpurea L.	*Digitalis thapsi* L.
Leaves.—Ovate to oval, rather bluntly pointed, abruptly contracted into winged petioles.	*Leaves.*—Oblong to oblong lanceolate, sharp pointed, gradually tapering to short winged petioles or sessile base.
Margin.—Crenate. Veins more or less prominent below. Pale green or gray and densely pubescent on lower surface.	*Margin.*—Yellowish green on both surfaces, which are inconspicuously pubescent. (This color, while noted in all samples, may be due to improper drying.)

MICROSCOPIC CHARACTERS.

Digitalis purpurea L.	*Digitalis thapsi* L.
Hairs.—Of two sorts; most common, long, simple, 2–8 celled, some cells frequently collapsed: glandular hairs few, with 1- or 2-celled stalk and 1- or 2-celled glandular head.	*Hairs.*—One sort; glandular, long, stalk several celled, with 1- or 2-celled glandular head.

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[1] *J. Am. Pharm. Assoc.*, 1918, **7**: 855; 1919, **8**: 789.

[2] *Am. J. Pharm.*, 1917, **89**: 147.
H. H. Rusby. National Standard Dispensatory. 3rd ed., 1916, 575.
H. Kraemer. Scientific and Applied Pharmacognosy. 2nd ed., 1920, 617.
Am. J. Pharm., 1912, **84**: 201.
Botan. Centr., 1906, **8**: 267.
J. pharm. chimie, 1905, **22**: 56.
H. Zörnig. Arzneidrogen. 1909 (I): 140.

The most striking difference, which has been pointed out before, and which is of special value in the estimation of powders, is the character of the hairs. Only glandular hairs were found in *Digitalis thapsi*, while *Digitalis purpurea* has both glandular and non-glandular hairs.

EGYPTIAN HENBANE (*Hyoscyamus muticus* L.)

The distinguishing characters of both the Egyptian and official henbane are given in another table, as follows:

HYOSCYAMUS[1].

MACROSCOPIC CHARACTERS.

Hyoscyamus niger L.	*Hyoscyamus muticus* L.
Leaves.—Large, shortly and broadly petioled, 15–25 mm. long and two-thirds as broad, the upper becoming smaller and sessile; blades angularly ovate, acute, coarsely and angularly toothed or lobed, grayish green or slightly yellowish green.	*Leaves.*—Much narrower than *H. niger* and obovate. They are coarsely toothed or lobed at the summit but lack the very large and sharp lateral lobes of *H. niger*.
Stems.—Hollow, cylindrical, flattened, longitudinally furrowed and wrinkled, 3–4 mm. in diameter.	*Stems.*—Light colored and often as thick as the finger.
Capsules.—Globose, oblong, about 10 mm. long.	*Capsules.*—Very light colored and much longer than *H. niger*.
Calyx.—Tubular, 5-toothed, about 10 mm. long.	*Calyx.*—Limb farther prolonged beyond the capsule than *H. niger*.

MICROSCOPIC CHARACTERS.

Hairs. — Branched and non-branched, glandular and non-glandular. Branched hairs thin walled, but very numerous.	*Hairs.* — Branched and non-branched, glandular and non-glandular. Branched hairs thick walled, more numerous, characteristic.

The character of the hairs, and especially the abundance of branching hairs, and the peculiar manner of branching, are considered a practical means of differentiation, especially in powders.

The statement in literature that *Hyoscyamus muticus* has no glandular hairs could not be confirmed.

RHAPONTICUM ROOT (*Rheum rhaponticum* L.)

Some work was also done to find means to differentiate rhaponticum root and rhubarb.

Rhaponticum root consists usually of the rhizome branches, smaller in diameter and more conical in shape than those of the rhizome of genuine

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[1] H. H. Rusby. National Standard Dispensatory. 3rd ed., 1916, 836.
H. Kraemer. Scientific and Applied Pharmacognosy. 2nd ed., 1920, 595.
Am. J. Pharm., 1914, **86**: 531; 1915, **87**: 1.
Trans. Am. Chem. Soc., 1899, **75**: 72.
Bull. sci. pharmacol., 1907, **14**: 569.
H. Zörnig. Arzneidrogen. 1909 (I): 284.

rhubarb. The roots and rhizomes show a normal structure, while within the normal, less conspicuous cambium ring the rhizome of genuine rhubarb shows several open and abnormally developed vascular bundles, characterized by prominent pith rays, giving the bundles a stellate appearance.

Since the microscopic differentiation is quite difficult, attempts were made to base the differentiation on the fact that rhaponticin is present in *Rhaponticum*, but absent in *Rheum*. Tschirch has already recommended a chemical test, suggesting the following procedure:

> Boil 10 grams of the powdered drug for 15 minutes with 50 mils of 70% alcohol, filter, evaporate the filtrate to about 10 mils, cool and add 15 mils of ether. If no turbidity or precipitation develops in the ethereal solution within 24 hours, the absence of rhaponticin is indicated.

The test is based on the insolubility of rhaponticin in ether, in which rhein and emodin, present in rhubarb, are soluble. The test, tried both with pure drugs and mixtures, has thus far not given satisfactory results. The work is, therefore, being continued on this subject.

PART II.

Further data have been collected on volume weights. It has previously been recommended that the weight of 100, 500 and 1000 cc., more or less, of crude drugs, as far as they are of fairly uniform size, such as seeds and fruits, or of powder, such as *Lycopodium* or *Kamala*, can often be used for an immediate indication of inferiority due to worm infection, moldiness, immaturity, partial extraction, substitution, presence of sand, or other foreign matter, etc. It appears that the maximum weights obtained are most useful for practical purposes. Ewing states:

> With regard to volume weight determination, I think too, as you suggest, that it might possibly be the case that neither marjoram nor pennyroyal is suitable for the purpose. I do not think that either of these drugs is dispensed in a powdered form. Seeds, fruits and other products, of rather uniform size, as you mention, appear also to be best fitted for this purpose. Clevenger has gone over the specimens in our collection and selected quite a number of these products which appear suitable for such determinations.
>
> In each instance in Table 1, except in the case of the fennels, that volume which appeared most advantageous for the character of the material was used. For products of a round or oval shape, and preferably of a fairly uniform size, not exceeding about 0.6 cm. in diameter, 100 cc. is a sufficient sample. For products between 0.6 cm. and 1.2 cm., a 200 cc. sample would be preferable. For products larger in diameter, a 500 cc. or even sometimes 1000 cc. sample would be necessary. In the case of materials of other shapes, it is hard to generalize upon the size of the sample to be taken. For instance, 200 cc. seems adequate for ergot, while for a light product, such as cardamom fruits, 500 cc. would appear to be preferable because the actual deviations in the specific weight of any shipment would be greater and consequently would give greater accuracy to the determination. Our criterion of the accuracy of any particular volume

TABLE 1.

Volume weight determinations obtained by J. F. Clevenger.

PRODUCT	VOLUME	WEIGHT	REMARKS
	cc.	*grams*	
Anise seed	100	42.9	
Beans, Burma white	500	405.0	
Beans, Tepary	500	427.5	
Cardamom fruits	500	86.5	
Cassia buds	200	93.0	Slightly moldy.
Celery seed	100	49.5	
Cloves	200	75.0	
Cochineal	100	44.0	
Coffee, Bolivia	200	117.0	
Coffee, Black Jack	200	139.0	
Coffee, skimmings	200	110.0	Polished skimmings.
Colchicum seed	100	80.0	
Conium	100	52.0	
Conium seed	100	54.0	
Coriander	100	32.0	
Coriander, oval	100	29.0	
Cumin seed	100	33.5	
Dill seed	100	29.0	
Ergot, American	200	90.0	(1918) G. W. Hoover's sample.
Ergot, laboratory sample	200	95.0	
Ergot	200	94.0	Hecker, Jones, Jewell Co. Kept 2 years in desiccator over sulphuric acid.
Fennel, bitter	100	45.5	200 cc. would appear better, but sample at hand too small.
Fennel, German	100	29.5	200 cc. would appear better, but sample at hand too small.
Fennel, Levant	100	32.5	200 cc. would appear better, but sample at hand too small.
Juniper berries	100	32.0	
Larkspur	100	48.8	
Lycopodium	100	37.0	10% spore cases laboratory sample.
Lycopodium	100	42.5	
Pimenta	100	38.5	
Poppy seed (white maru)	100	57.0	
Poppy seed (ordinary)	100	63.0	
Stavesacre seed	100	36.0	
Wormseed, Levant	100	23.0	
Ergot, American*	200	95.0	Good quality.
		95.0	
		96.0	
Ergot, imported*	200	95.0	Fair quality.
		97.0	
		90.0	Poor quality.
Bitter fennel	100	46.5	
Wormseed, Levant	100	33.5	
Wormseed, German	100	29.5	
Kamala	100	66.5	
Lupulin	100	54.5	

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* Determinations made by A. Viehoever.

is that its value shall be the same relative value that one obtains in a larger specimen; for instance, 100 cc. of conium seed weighed 52 grams; 500 cc. weighed 259 grams, which is only 1 gram less than 5 × 52. It may, therefore, be taken for granted that the 100 cc. volume is sufficient for conium. From the shape and size of the particular illustration, one would expect in advance that 100 cc. would be ample, but in some instances, such as cloves, experimentation is necessary. One hundred cc. of cloves weighed 35 grams, 200 cc. weighed 75 grams, which is 5 more than twice the weight of 100 cc. The conclusion in that instance is that a 100 cc. sample is not sufficient for the purpose. As another illustration: 100 cc. of juniper berries were found to weigh 32 grams and 200 cc. weighed 64 grams. The conclusion is that a 100 cc. sample is satisfactory.

Attempts to secure certain supplies, such as different samples of *Kamala* and *Lycopodium*, have been thus far unsuccessful. C. J. Zufall (U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, New York, N. Y.) has informed the associate referee that he has collected considerable data, which he will submit later. The work should be continued.

PART III.

Extended experiments were undertaken to establish the usefulness of micro-sublimation in food and drug analysis. The results were quite satisfactory. A critical study of the apparatus recommended in the literature resulted in the development of improved apparatus and methods. The sublimation of santonin from wormseed, and its subsequent identification may be of especial interest. Within recent years, importations of santonin-free wormseed necessitated careful examination of every shipment. No definite microscopical differences of the santonin-free drug from the one containing santonin have as yet been detected. In fact, it is not clear whether the santonin-free drug is collected from a different species, a physiological variety containing no santonin, or collected too late in the season. A simple test showing the presence or absence of santonin in a sample of wormseed is therefore of especial value. While Tunmann, Heyl and Molisch obtained no satisfactory results, the experiments of the associate referee in subliming this drug were quite successful. The details, however, have not been sufficiently tried out by collaborators to warrant the associate referee recommending the adoption of this procedure as a tentative method.

PART IV.

The subject of conditions existing in the interstate and import trade of crude drugs has been discussed to a more or less detailed extent in recent publications of the Bureau of Chemistry, and especially the Pharmacognosy Laboratory[1]. It may, however, be of interest to demonstrate here some of the drugs which were found to be adulterated in whole or in part by other plant products.

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[1] *J. Am. Pharm. Assoc.*, 1919, **8**: 725, 717, 459.

Such drugs as hydrastis, aletris, sassafras, as well as pennyroyal leaves are more often found dirty, due to improper collection, than adulterated with other drugs. The standards existing for the maximum allowance of ash are evidently not based on the actual ash content of these drugs, but are arbitrarily fixed and are often much in excess of the natural ash content. The high ash, as the great amount of acid insoluble ash indicates, is due largely to sand or other mineral matter adhering to parts of the drug. This adhering sand apparently can not be readily removed other than by washing the drug at the time of collection, especially for root drugs. The surprising fact that no ash standard exists in this country for hydrastis may again be given emphasis[1].

With regard to foreign drugs, substitution is far more frequent, and the drug desired may be substituted either wholly or in part. The substitution products are generally closely related to the drug, the name of which is usually adopted in invoicing and labeling, or the substitute may only have superficial similarity. As examples, may be mentioned the substitution of sarsaparilla root by non-official sarsaparilla species, such as *Smilax utilis* Hemsley, *Brassica juncea* (L.) Cosson, (Indian mustard), by *Brassica napus* var. *dichotoma* Prain, (Indian rape), *Arnica montana* L., by *Heterotheca inuloides* Cass., and castor beans by *Jatropha curcas* L. products derived from plants belonging to the same families. In the case of foreign drugs, identification is far more difficult, if not at times impossible. The greatest difficulty is experienced with tropical or oriental drugs where insufficient data are available in literature, and often no comparative material is available at the museum. The extension and centralization of collections of natural plant products found anywhere in the world, with botanical specimens, is urgently needed.

RECOMMENDATIONS.

It is recommended—

(1) That work be continued to determine the value of a more extended use of volume weight determination in the analysis of crude drugs and spices.

(2) That work be continued to find new sources of supplies or proper substitutes for drugs not now available.

(3) That the subject of sublimation for the analysis of plant products, etc., be further studied.

(4) That the methods for the macroscopic and microscopic identification of *Digitalis thapsi* (Spanish digitalis), a recent substitute for *Digitalis purpurea*, and *Hyoscyamus muticus* (Egyptian henbane), a substitute for *Hyoscyamus niger*, be adopted as tentative methods.

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[1] *J. Am. Pharm. Assoc.*, 1920, 9: 779.

REPORT ON ALKALOIDS.

By A. R. Bliss, jr. (Emory University, School of Medicine, Emory University, Ga.), *Associate Referee.*

RECOMMENDATIONS.

It is recommended—

(1) That the following be added to the method for the assay for strychnin in tablets[1], to follow, "Dry at 100°C. to a constant weight and weigh as strychnin":

Check the weight of the strychnin by dissolving the residue in neutral alcohol, adding an excess of N/10 sulphuric acid, and titrating back with N/50 potassium hydroxid, using methyl red as the indicator. One cc. of N/10 sulphuric acid is equivalent to 0.0334 gram of strychnin and 0.0428 gram of strychnin sulphate. The U. S. P. factor for strychnin to strychnin sulphate is 1.2815.

The entire method should be further studied.

(2) That the following be added to the assay for strychnin in liquids (applicable to elixirs of iron and strychnin in the absence of other alkaloids)[2], to follow, "Dry at 100°C. to a constant weight and weigh as strychnin":

Check the weight of the strychnin by dissolving the residue in neutral alcohol, adding an excess of N/10 sulphuric acid, and titrating back with N/50 potassium hydroxid, using methyl red as the indicator. One cc. of N/10 sulphuric acid is equivalent to 0.0334 gram of strychnin and 0.0428 gram of strychnin sulphate. The U. S. P. factor for strychnin to strychnin sulphate is 1.2815.

The entire method should be further studied.

Comments.—The experiences of practically all workers in the alkaloidal field have shown rather conclusively the advisability of the volumetric check on the assays for the majority of the alkaloids.

(3) That the following method for the separation of quinin and strychnin be adopted as a tentative method:

Treat 50 mils of the sample in the usual way with citric acid and ammonia water, remove the precipitated alkaloids by shaking with ether-chloroform, and recover the mixed alkaloids by careful evaporation in a tared dish. Note the weight of the mixed alkaloids.

Dissolve the mixed alkaloidal residue in chloroform, add an excess of 5% sulphuric acid, and then entirely boil off the chloroform on a steam bath. Transfer the solution to a separatory funnel, and wash the dish with sufficient water to make a volume of about 250 mils. (One gram of strychnin requires 6420 mils of water for solution[3].)

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[1] *J. Assoc. Official Agr. Chemists*, 1919, 3: 189; 1920, 3: 379.

[2] *Ibid.*, 1920, 3: 379.

[3] U. S. Pharmacopœia, IX, 1916, 349, 416.

Then extract it with two 15-mil portions of ether, rejecting the ethereal fractions. Add an excess of ammonia water, and shake out the mixture with 7 portions of ether, using 35, 20, 10, 10, 10, 10 and 5 mils, carefully washing the stem of the separatory funnel each time and running such ether used for washing into the combined ethereal fraction. Wash the combined ethereal fraction with 5 mils of water and allow it to stand for 15 minutes to completely separate. Introduce a pledget of absorbent cotton into the stem of the separatory funnel, and very carefully run the ethereal fraction into a tared dish. Pour 5 mils of ether into the separatory funnel and run into the tared dish; repeat with 5 additional mils of ether. Finally, carefully wash the outside of the stem of the separatory funnel with ether, and run this also into the tared dish. Then very carefully evaporate the ethereal solution on a bath at 100°C. for an hour and weigh as quinin.

Shake out the ammoniacal liquid left after the foregoing treatments with ether with 7 portions of chloroform, using 35, 20, 10, 10, 10, 10 and 5 mils, carefully washing the stem of the separatory funnel with chloroform and running this chloroform also into the combined chloroformic fraction. Wash the combined chloroformic fraction with 10 mils of distilled water, and allow it to stand for 15 minutes. Introduce a pledget of absorbent cotton into the stem of the separatory funnel, and carefully run the chloroformic solution into a tared dish. Add 10 mils of chloroform to the contents of the separatory funnel, thoroughly agitate the mixture, and, when completely separated, run the chloroformic layer into the tared dish. Wash the outer and inner surfaces of the separatory funnel with a little chloroform, and run this also into the tared dish. Lastly, evaporate the chloroformic solution very carefully on a bath, removing the dish from the bath as the last portions evaporate. Dry the residue at 100°C. for 1 hour, and weigh as strychnin.

This method should be further studied.

Comments.—Although this method has been submitted to the collaborators, sufficient time has not yet elapsed for reports of individual results, modifications, criticisms, etc. The associate referee's work with this method has been reported[1].

Notes.—(*a*) The solution in chloroform with the addition of an excess of sulphuric acid, followed by the complete removal of the chloroform by boiling, is carried out to insure complete solution of the alkaloids.

(*b*) The extraction with two portions of ether at this point, before the precipitation with ammonia water, is carried out to remove the traces of oily matter derived from the oils usually used in flavoring an elixir.

(*c*) The volumetric check on the alkaloids is omitted because, in the case of the alkaloid quinin, it is unsatisfactory and gives simply a rough check, and, in the case of the alkaloid strychnin, the amount of the alkaloid usually obtained by gravimetric process from a 50-mil sample of the usual elixir is too small for volumetric check.

(4) That the following methods for the assay of physostigma and its preparations be adopted as tentative methods:

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[1] *J. Am. Pharm. Assoc.*, 1919, 8: 804.

ASSAY OF PHYSOSTIGMA.

(For alkaloids.)

Introduce 20 grams of physostigma in No. 80 powder into an Erlenmeyer flask of about 250 mils capacity, add 180 mils of ether and a mixture of 10 mils of water and 3 grams of sodium bicarbonate. Stopper the flask, shake at intervals during 4 hours, and allow to stand overnight. Shake the flask well for 15 minutes, and, when the solid matter has settled, carefully decant off as much as possible and as rapidly as possible through a plaited filter, collecting the filtrate in a 200-mil graduated cylinder. Measure the aliquot, and pour into an Erlenmeyer flask of about 250 mils capacity. Recover the ether on a bath, removing the flask from the bath as the last portions of ether evaporate. To the residue in the flask add 15 mils of N/10 sulphuric acid, 15 mils of water, and 3 mils of chloroform. Remove the chloroform completely on a bath, and then titrate back with N/50 potassium hydroxid, using methyl red as the indicator. Run a blank determination, and make any necessary correction. One mil of N/10 sulphuric acid is equivalent to 0.02752 gram of physostigma alkaloids.

ASSAY OF FLUIDEXTRACT OF PHYSOSTIGMA.

(For alkaloids.)

Pour 20 mils of the sample on some oak sawdust contained in an evaporating dish, and allow the mixture to evaporate spontaneously in a darkened place. Then treat the impregnated sawdust like the drug, cleaning the evaporating dish with the mixture of 10 mils of water and 3 grams of sodium bicarbonate.

ASSAY FOR POWDERED AND SOLID EXTRACTS OF PHYSOSTIGMA.

(For alkaloids.)

Carefully weigh 3 grams of the sample, mix with 15 mils of alcohol in an evaporating dish, and add 10 grams of oak sawdust, mixing well. Allow the mixture to evaporate spontaneously in a darkened place. Then treat the impregnated sawdust like the drug, cleaning the evaporating dish with the mixture of 10 mils of water and 3 grams of sodium bicarbonate.

ASSAY FOR TINCTURE OF PHYSOSTIGMA.

(For alkaloids.)

Pour 200 mils of the sample on 10 grams of oak sawdust contained in an evaporating dish. Dry on a covered steam bath, mixing well occasionally. Treat the impregnated sawdust like the drug, cleaning the evaporating dish with the mixture of 10 mils of water and 3 grams of sodium bicarbonate.

STANDARDS.

Drug[1].—Not less than 0.15 per cent of alkaloids.

Fluidextract.—Alkaloids in 100 mils, 0.15 gram.

Extracts[2].—Alkaloids, 1.7 to 2.3 per cent.

Tincture[3].—Alkaloids in 100 mils, 0.013 to 0.017 gram.

Comments.—The method (proposed by G. W. Ewe, H. K. Mulford Co., Philadelphia, Pa.) has been carefully tried out by the associate

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[1] U. S. Pharmacopœia, IX, 1916, 320.
[2] *Ibid.*, 158.
[3] *Ibid.*, 464.

referee, whose results agree very closely with those obtained by Ewe. The method given in the Ninth Revision of the United States Pharmacopœia[1] has been given a conscientious trial and has shown unsatisfactory results that are much lower than those obtained with the method recommended. The exact cause of the low results has not been traced, but it is believed to be due partly to decomposition of the alkaloid and partly to incomplete extraction. The former belief is strengthened by the fact that the aqueous alkaloidal extractions, both acid and alkaline, develop a strong pink color. Table 1 shows comparative results obtained by Ewe in a series of 8 assays.

TABLE 1.

Physostigma: Comparative results by U. S. P. IX and direct evaporation methods.*

SAMPLE	U. S. P. IX METHOD*	DIRECT EVAPORATION OF ALIQUOT	INDICATOR	REMARKS
Drug........	0.02†	0.14†	Methyl red	End reaction fair
Drug........	None	0.13†	Methyl red	End reaction fair
Drug........	0.11†	0.37†	Methyl red	End reaction fair
Fluidextract..	0.07‡	0.133‡	Cochineal	End reaction not so sharp as methyl red
Fluidextract..	0.059‡	0.178‡	Cochineal	End reaction not so sharp as methyl red
Fluidextract..	0.0821‡	0.150‡	Methyl red	End reaction fair
Tincture.....	0.00218‡	0.00611‡	Methyl red	End reaction fair
Tincture.....	0.01990‡	0.01305‡	Methyl red	End reaction fair

* U. S. Pharmacopœia, IX, 1916, 320.
† Expressed as per cent.
‡ Expressed as gram.

Although many blanks have been run, it has never been found that any neutralization of the standard acid results. In several cases a slight acidity has been indicated. This has been ascribed to experimental error, and it is advised that a blank be run with each assay to correct for experimental error. In no case did the acidity run above 0.07 mil of N/10 acid.

The method has been submitted to the collaborators, but sufficient time has not elapsed for reports of results. It is hoped that they will be received in time for the next meeting.

(5) That the assay for fluidextract of hyoscyamus of the United States Pharmacopœia[2] be adopted with the following changes:

Proceed as directed under "Fluidextractum Belladonnæ Radicis"[3], first line of the assay, modifying the process there given by using 25 mils of the fluidextract of hyos-

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[1] U. S. Pharmacopœia, IX, 1916, 320.
[2] *Ibid.*, 187.
[3] *Ibid.*, 178.

cyamus in place of "10 mils of fluidextract of belladonna root", and adding 30 mils of water and 5 mils of ammonia water, in place of "10 mils of water and 2 mils of ammonia water"; and just before ninth line inserting, "treat the residue twice with 5 mils of ether and evaporate to dryness each time."

Comments.—The modification of the United States Pharmacopœia[1] assay recommended was suggested by H. C. Fuller (Institute of Industrial Research, Washington, D. C.). The increase in the amount of water and ammonia water to be added was made because the pharmacopœial quantities have been found to be decidedly insufficient for fluidextract of hyoscyamus. The recommended modification has given much more satisfactory results in the hands of both Fuller and the associate referee. The method has been submitted to the collaborators.

(6) That the comparative study of the volumetric and the gravimetric methods for the assay of ipecac be continued.

(7) That work be done on methods for assaying the following preparations, and that methods be submitted to the collaborators which have a reasonable certainty of yielding concordant results: (*a*) Liniment of belladonna; (*b*) ointment of belladonna; (*c*) powder of ipecac and opium; (*d*) ointment of stramonium. Since there are no pharmacopœial assays for the preparations named, it is strongly urged that this recommendation be adopted.

REPORT ON SYNTHETIC PRODUCTS.

By C. D. WRIGHT (Bureau of Chemistry, Washington, D. C.), *Associate Referee.*

No material was submitted to collaborators this year. Several methods have been under consideration and have been tried out more or less extensively, with results which are embodied in the following recommendations:

RECOMMENDATIONS.

It is recommended—

(1) That the method of W. O. Emery and C. D. Wright for the valuation of hexamethylenetetramin tablets[2] be adopted as tentative.

(2) That the method of W. O. Emery for the estimation of monobromated camphor in migraine tablets[3] be adopted as tentative.

(3) That methods for the determination of phenolphthalein be studied further preparatory to cooperative work.

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[1] U. S. Pharmacopœia, IX, 1916, 187.
[2] *J. Ind. Eng. Chem.*, 1918, **10**: 606.
[3] *Ibid.*, 1919, **11**: 756

REPORT ON BALSAMS AND GUMS.

By E. H. Grant[1] (U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, New York, N. Y.), *Associate Referee.*

The following method for the determination of crude fiber in gum karaya was submitted to the collaborators:

> To 2 grams of the gum in a 500 cc. flask add 50 cc. of cold 1.25% sulphuric acid, in small portions. Shake gently so as to form as uniform a mixture as possible, taking care that it does not splash so high on the sides of the flask that the acid will not attack it later. Allow to stand overnight, then mix again, if necessary. Add 150 cc. of boiling 1.25% sulphuric acid, and connect the flask with an inverted condenser or place a funnel in its neck. Heat rapidly to boiling and then turn the flame down so that the solution boils gently. Heat for 30 minutes, counting from the time the boiling acid was added. Proceed as in the official method for crude fiber, beginning with the word "Filter", line 8[2].

RESULTS.

The results shown in Table 1 were obtained.

TABLE 1.

Crude fiber in gum karaya.

ANALYST	SAMPLE A*	SAMPLE B†
	per cent	*per cent*
W. S. Friedman, Agricultural Experiment Station, Stillwater, Okla.	0.32 0.46	2.10 2.06
C. K. Glycart, U. S. Food and Drug Inspection Station, 1625 Transportation Building, Chicago, Ill.	0.48 0.57	2.05 2.33
E. O. Eaton, U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, San Francisco, Calif.	0.50 0.52	2.50 2.67
J. I. Millner, U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, New York, N. Y.	0.60	2.10
E. H. Grant..	....	2.02
H. C. Fuller, Institute of Industrial Research, Washington, D. C.	0.13	1.09

* A composite of several samples of cleaned gum, which were released by the U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, New York, N. Y., as being of satisfactory quality.

† A composite of low-grade gums, separated during the process of cleaning, which were to be used for technical purposes only.

RECOMMENDATION.

It is recommended that further work be done on this method.

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[1] Present address, Wm. S. Merrell Co., Cincinnati, Ohio.

[2] *Assoc. Official Agr. Chemists, Methods*, 1916, 118.

THE TURNER REACTION FOR GURJUN BALSAM.

By J. B. LUTHER[1] (U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, New York, N. Y.).

During the course of the examination of various shipments of copaiba at this laboratory considerable difficulty was experienced in determining whether or not the products entered at this port contained gurjun balsam as an adulterant.

The official standards established for this product under the Food and Drugs Act have been designated in the United States Pharmacopœia. Some of these tests are extremely indefinite and efforts were made to employ, if possible, a test which was reliable if made under definite conditions.

The test given in the Ninth Revision of the United States Pharmacopœia for the detection of gurjun balsam[2] (known as the Turner reaction) is as follows:

> Dissolve 3 or 4 drops of the volatile oil separated from copaiba by distillation with steam, in 3 mils of glacial acetic acid, mix the solution with 1 drop of freshly prepared aqueous solution of sodium nitrite (1 in 10), and carefully underlay this with 2 mils of sulphuric acid. The acetic layer is not colored pink.

The principal defects of this test were: (1) That the sulphuric acid always chars at the contact layer and thereby obscures the coloration; (2) if the acid is added without much disturbance of the liquid it settles to the bottom of the test tube, giving no color in the acetic acid layer; (3) after the acid is added with a slight disturbing of the liquid, invariably some degree of color is formed from the start and increases in intensity on standing. In the hands of analysts not specially experienced in this work these reactions would probably lead to erroneous conclusions.

It became evident that to remedy these faults some test should be devised that is performed under conditions which can be controlled. Happily, this situation was met after a number of experiments were made and a modification of the Turner reaction was used which overcame the main objections by using a mixture of 5 per cent of concentrated sulphuric acid in glacial acetic acid instead of the concentrated acid. The charring was thereby avoided and more reliable and uniform results were obtained.

It is the writer's experience that of the various copaibas entering this country among which are Para, Maracaibo, Barranquilla, Ciudad Bolivar, Carthagena, Trinidad (all so named after the port of export in

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[1] Since deceased.

[2] U. S. Pharmacopœia, IX, 1916, 123.

Comparative results of the tests on the different copaibas.

NEW YORK NUMBER	DESCRIPTION OF SAMPLE	U. S. P. VIII*	U. S. P. IX†	MODIFIED TURNER'S TEST
74134	Ciudad Bolivar	Colorless at first. Violet develops after 40 seconds.	Colorless. Gradually blue.	Colorless 10 seconds, then gradually blue.
74162	Para	Colorless. Pink develops after 30 seconds.	Colorless. Gradually pink after 1 minute.	Colorless 10 seconds, then red.
74173	Maracaibo....	Colorless at first. Slight pink after 20 seconds.	Colorless. Pink after 30 seconds.	Slight pink after 1 minute. Deepens to violet on standing.
74174	Ciudad Bolivar....	Colorless at first. Blue after 20 seconds.	Colorless. Blue after 10 seconds.	Blue immediately.
74175	Barranquilla..	Colorless at first. Blue after 20 seconds.	Blue after 20 seconds.	Light blue.
74176	Maracaibo....	Colorless at first. Pink within 20 seconds.	Pink after 30 seconds.	Light blue within 5 seconds.
74178	Ciudad Bolivar....	Colorless at first. Blue after 14 seconds.	Colorless at first. Blue after 30 seconds.	Light blue—darkening.
74179	Trinidad.....	Colorless at first. Blue within 4 seconds.	Light blue first, darkening.	Blue immediately.
74190	Maracaibo....	Colorless at first. Light pink within 20 seconds.	Colorless at first. Pink to violet after 30 seconds.	Colorless—slowly light pink—deepens.
74191	Para.........	No coloration within 1 minute.	Colorless for 30 seconds, then brown-pink.	Colorless for 1 minute, then light pink.
74197	Carthagena...	No coloration within 1 minute.	Light yellow for 30 seconds.	Colorless for 2 minutes.
74199	Maracaibo....	Slight pink after 50 seconds.	Slight pink first, then darker.	Colorless. Pink on standing.
74306	Ciudad Bolivar....	Blue within 10 seconds.	Blue after 30 seconds. Pink at part of contact. Colorless at first.	Blue after 30 seconds.

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* U. S. Pharmacopœia, VIII, 1905, 117 (before corrections were made June 1, 1907).
† *Ibid.*, IX, 1916, 123.

South America, the principal place of production) that of Para gave the least trouble in examination. The other varieties gave extremely confusing reactions with the known gurjun tests. In nearly all cases varying degrees of color, including red, violet and blue, were produced on standing for different lengths of time. The volatile oil of Para gave hardly any color and was used as a blank for comparison and in mixtures containing definite amounts of gurjun.

The modified test must be made as follows:

Dissolve 4 drops of the volatile oil in 1 mil of glacial acetic acid; add 1 drop of a freshly prepared aqueous solution of sodium nitrite (1 in 10) and then add at once, mixing quickly, 2 cc. of a 5% solution by volume of concentrated sulphuric acid in glacial acetic acid.

The presence of gurjun is indicated by the formation of a violet coloration. As small an amount as 5 per cent of gurjun was detected, the color being obtained instantly. This modified test was applied to a number of different varieties and compared: First, with the old United States Pharmacopœia, Eighth Revision[1]. (test before the corrections were made to the United States Pharmacopœia in 1907), adding 4 drops of the volatile oil to a mixture of 1 cc. of glacial acetic acid and 4 drops of nitric acid; second, with the Turner test of the Ninth Revision of the United States Pharmacopœia[2]. The results are given in the table.

All of the known tests gave some color after a time which was very misleading. These errors are avoided in the modified test, the reaction being sharp and instantaneous, giving a clear violet coloration in the presence of gurjun, but in using this modified test, unless a violet coloration is produced instantly, no appreciable amount of gurjun is present.

SUMMARY.

In using Turner's reaction for gurjun balsam in copaiba[2], a time limit of 10 seconds must be made on the appearance of a pink color, since all copaibas give more or less coloration after longer standing.

A modified method offering several advantages is described.

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[1] U. S. Pharmacopœia, VIII, 1905, 117.
[2] *Ibid.*, IX, 1916, 123.

RECOMMENDATIONS BY THE ASSOCIATE REFEREE ON ENZYMS.

By J. F. Brewster (Bureau of Chemistry, Washington, D. C.), *Associate Referee.*

(1) That the tentative method for the determination of pepsin[1] be subjected to further study, with a view to making it official.

(2) That the method developed by V. K. Chesnut for the estimation of papain[2] be studied.

THE PHARMACOPŒIAL ASSAY FOR ALCOHOLS IN SANTAL OIL EXTENDED TO INCLUDE THE TRUE ACETYL VALUE.

By C. W. Harrison (U. S. Food and Drug Inspection Station, Park Avenue Building, Baltimore, Md.).

The examination of a sample of santal oil (No. 19326), which was grossly adulterated with a vegetable oil, called attention to the fact that the United States Pharmacopœia assay method for alcohols is not applicable where the santal oil has been adulterated with any saponifiable oil.

The method as now given is, in fact, a determination of the saponification value of the acetylated oil expressed as santalol. It would therefore include any other saponifiable bodies present which also would be calculated as alcohols, thus giving erroneous results. Obviously, then, it would be possible to grossly adulterate sandalwood oil with saponifiable oils and still have it meet the requirements of the United States Pharmacopœia with respect to specific gravity, optical rotation, and percentage of alcohols, although it would not be completely soluble in 70 per cent alcohol. This is illustrated by Investigation No. 19327 (Table 1), which contains 20 per cent of foreign oils. When assayed according to the United States Pharmacopœia, apparently this product contains the required 90 per cent of alcohols expressed as santalol, but, as a matter of fact, it is quite deficient in these bodies.

The object of this work was to extend the Pharmacopœial assay to include a determination of the alcohols from the acetyl value and to make this determination simple and rapid enough to be useful in regulatory work, it being well known that the determination of acetyl value

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[1] *Assoc. Official Agr. Chemists, Methods,* 1916, 363.
[2] *J. Assoc. Official Agr. Chemists,* 1920, **3**: 391.

as generally given is a rather laborious process. It is believed that the results obtained by the present assay method, which are now expressed as per cent santalol, should be more correctly stated as "saponification number of the acetylated oil". The acetyl value obtained by this extension of the official method gives more nearly the amount of alcohols present and could, therefore, with more reason be reported as percentage of santalol. With these two results available, the adulteration of a given sample of santal oil with a saponifiable oil becomes evident at once.

Except for conducting a blank on the alcoholic potash solution used for the saponification, thus eliminating the necessity for using an alcoholic potash solution of exact normality, the present United States Pharmacopœia method[1] was found satisfactory as far as it went. The method was extended, however, from the point where the acetylated oil is saponified. The true acetyl value was determined on this residue, the determination being rendered simple and effective by adopting for this purpose the large-size, modified Hortvet volatile-acid apparatus.

In detail, the method was worked out as follows:

Acetylate the oil according to the present method[1]. Weigh accurately, and saponify with 50 cc. of alcoholic potash as directed, measuring at the same time with the same pipette, drained for the same length of time, another 50 cc. portion of the alcoholic potash solution to carry through as the blank. Titrate the blank and the sample with N/2 sulphuric acid and phenolphthalein, and subtract the number of cc. of acid required to neutralize the sample from the number required to neutralize the blank, thus obtaining the number of cc. of N/2 potassium hydroxid required to saponify the weight of oil taken:

$$\text{Saponification number of acetylated oil} = \frac{\text{cc. N/2 potassium hydroxid} \times 28.06}{\text{weight of oil taken}}.$$

After determining the saponification number of the acetylated oil, render the flask faintly alkaline with 1–2 drops of alcoholic potash, place on the steam bath, and dealcoholize. (This can be easily and quickly accomplished by using suction to remove alcohol vapors.) After the alcohol has been completely removed and the liquid in the flask reduced to a small volume, carefully transfer it to the modified Hortvet apparatus, and carefully rinse the flask with successive portions of dilute (approximately 10%) sulphuric acid, adding these to the volatile acid apparatus to which also a few drops of methyl orange test solution have been added. Continue the addition of dilute sulphuric acid until the contents of the tube are distinctly acid to methyl orange. This should not require more than 10–15 cc. of dilute acid. It is necessary to keep this volume as small as possible. Then steam distil the product. Most of the volatile acids pass over in the first 100 cc. of distillate, but about 300 cc. are required to carry over all traces of volatile acids. Next titrate the distillate with N/2 alkali and phenolphthalein, expressing the results as santalol according to the United States Pharmacopœia method[2].

The results obtained on mixtures of santal oil with other vegetable oils prepared in the laboratory, are given in Table 1. The figures show

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[1] U. S. Pharmacopœia, IX, 1916, 296.
[2] *Ibid.*, 1916, 300.

TABLE 1.

Comparison of results by the official method with the distillation method.

INVESTIGATION NUMBER	DESCRIPTION	REFRACTIVE INDEX	OPTICAL ROTATION, 100 MM. TUBE	ALCOHOLS AS SANTALOL, U. S. P. METHODS	SAPONIFICATION NUMBER OF ACETYLATED OIL	ALCOHOLS AS SANTALOL, CALCULATED FROM ACETYL VALUE	RATIO SAPONIFICATION NUMBER OF ACETYLATED OIL TO SANTALOL
				per cent		*per cent*	
16631	Santal oil, U. S. P.	1.50*	−18.2†	89.7	193.7	85.1	1 : 2.28
16837	Santal oil, 90% Castor oil, 5% Balsam copaiba, 5%	1.502‡	−17.1‡	93.7	201.	83.9	1 : 2.39
16838	Santal oil, 85% Copaiba oil, 10% Olive oil, 5%	1.501*	−17.9§	83.1	181.4	74.0	1 : 2.45
19328	Santal oil, 80% Copaiba oil, 10% Castor oil, 5% Olive oil, 5%	1.4995*	−16.9†	87.1	188.8	73.9	1 : 2.55
19329	Santal oil, 80% Copaiba oil, 5% Olive oil, 15%	1.498*	−15.8*	86.2	187.2	69.6	1 : 2.69
19327	Santal oil, 80% Copaiba oil, 5% Coconut oil, 15%	1.4963*	−15.9§	90.6	195.3	72.3	1 : 2.70
19326	Sandalwood oil, grossly adulterated (commercial sample)	1.4795*	−3.1*	107.6	225.8	34.4	1 : 6.56

* At 25°C.
† At 28°C.
‡ At 29°C.
§ At 26°C.

that it is possible to add appreciable amounts of foreign oils and still, when assayed by the United States Pharmacopœia method, have present apparently over 90 per cent of alcohols calculated as santalol, as required by the United States Pharmacopœia. When the alcohols are calculated from the true acetyl value, however, this figure is materially reduced. It would appear also that the ratio of the saponification number of the acetylated oil to the acetyl value, expressed as santalol, furnishes a significant figure, this ratio becoming progressively greater with the increase in the amount of foreign oils.

The meeting adjourned at 5.30 p. m. for the day.

SECOND DAY.

TUESDAY—MORNING SESSION.

No report on saccharine products was made by the referee.

REPORT ON MAPLE PRODUCTS.

By J. F. **Snell** (Macdonald College, Quebec, Canada), *Associate Referee.*

Seven samples of sirup were distributed to collaborators in 1918 and work upon the same samples was continued in 1919. The samples were as follows:

No. 1.—Pure, Quebec 1917, Grade I; Canadian lead number, 2.74.

No. 2.—Pure, Quebec 1918, Grade II; Canadian lead number, 3.60.

No. 3.—Adulterated, prepared from No. 5 and a "brown" cane sugar.

No. 4.—Adulterated, prepared from No. 5 and granulated sugar.

No. 5.—Pure, Quebec 1918, Grade III.

No. 6.—Adulterated, prepared from a Quebec 1918, Grade I, maple sirup and a sugar cane product; Canadian lead number of the pure sirup, 3.97.

No. 7.—Adulterated, prepared from the same maple sirup used in No. 6 and a "brown" cane sugar.

In 1918 collaborators were instructed to study: (1) Preparation of the sample; (2) Winton lead method; (3) Canadian lead method; (4) conductivity value method. In 1919 they were asked to determine: (5) Ash values; (6) malic acid value. In both years they were asked to report refractometer moisture results on the samples as received and to state their judgment as to the purity of the samples, making use of supplementary methods if necessary. Basing his judgment upon the Canadian lead numbers, A. Valin (Laboratory of the Inland Revenue Department, Ottawa, Canada), in 1918, expressed the opinion that Samples 2, 5 and 7 were genuine, the other four probably adulterated. N. C. McFarlane (Macdonald College, Quebec, Canada), in 1919, applying the volumetric lead method[1], adjudged Nos. 1, 2 and 5 genuine and No. 7 doubtful. The other collaborators did not venture an opinion.

MOISTURE RESULTS.

The results of the moisture determinations are given in Table 1. The wide variation in results is not to be emphasized, inasmuch as it may be due partly to actual differences in the portions of the individual

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[1] *J. Ind. Eng. Chem.*, 1916, **8**: 241; *J. Assoc. Official Agr. Chemists*, 1920, **4**: 169.

samples included in the various sets of seven. The samples, after they were subdivided and bottled, were sterilized by heating the filled bottles in a covered pan of boiling water. G. J. Van Zoeren (Macdonald College, Quebec, Canada), who prepared the samples, reports that in this process he found it necessary to remove the cork stoppers and cover the bottles with inverted beakers. Inasmuch as *sets* were sterilized together it is not impossible that different quantities of water distilled out of the various portions of the samples of identical *number*.

The results are arranged in Table 1 according to a suggestion made by Hugh Main, London, England, to whom the results were sent for criticism. Main, not being advised of the possibility of actual differences in the portions of the samples, suggests that probably the control of temperature is the chief source of error. His custom is to work in a room kept as near 20°C. as possible, pass water at 20°C. through the prism jacket of the Abbé refractometer, bring the sample to exactly 20°C. in a small aluminium dish, stirring with a thermometer graduated in fifths or tenths, use the thermometer bulb to transfer the sirup to the prism and then immediately take several readings, bringing the shadow region to the cross lines alternately from above and below. Operating in this way, he finds that his staff agrees in independent readings of the same sample within 0.20 per cent of water and, except for very dark solutions (molasses, etc.) or those that are turbid, he would take this as the maximum allowable amount of deviation from the middlemost result. In Table 1 the results which would be rejected on this basis are printed in italics. In view of the considerable variation between the results of C and F and of P, Q, R and S on identical portions of the sample, there can be little doubt that greater attention should be paid to precision in temperature control.

PREPARATION OF SAMPLE.

Collaborators were instructed to study the following proposed revised directions and to compare them with the present tentative method:

(**a**) *Maple sirup.*—Determine the moisture according to **10**[1]. If sugar has crystallized out, redissolve it by warming. Shake up any sediment that remains and pour a suitable quantity for analysis (about 100 cc. for all determinations) into a casserole or beaker. Add one-fourth the volume of distilled water. (The advisability of neutralizing with ammonia at this point might well be studied at some future time.) Boil to a temperature of about 104°C. (219°F.). Filter the hot sirup through a plug of cotton wool in the point of a funnel. After cooling, redetermine the moisture according to **10**[1].

(**b**) *Maple sugar and other solid or semi-solid products.*—(The present report refers to sirup alone. The moisture method for maple sugar may best be made identical with that for some of the other saccharine products, bearing in mind that maple sugar sometimes contains considerable invert sugar.) Determine moisture in the sample in

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 126.

its original condition after thoroughly mixing. For all other determinations, prepare a sirup from about 100 grams of the material by dissolving in about 150 cc. of hot water, boiling to a temperature of 104°C. and filtering through cotton wool as in (**a**). After cooling, determine the moisture in the sirup according to **10**.

A few experiments were made by Van Zoeren and the writer on the relative rate of filtration of the hot sirup through cotton wool and through filter paper. Eimer and Amend's "best white", Whatman No. 4 and Carl Schleicher and Schüll's No. 597 papers were used. In all cases, the filtration was very much slower than through the cotton wool. The other collaborators all chose to prepare their samples by the cotton-wool method. This seems to be the most satisfactory method in the field and it is, therefore, recommended that the above directions be adopted in place of the present tentative method.

Valin in 1918 reported results on the unprepared samples, as well as on the samples prepared according to the proposed revised directions.

WINTON LEAD NUMBER.

Collaborators were instructed to prepare and analyze basic lead acetate solutions of specific gravity 1.25 (*a*) from Horne's lead subacetate, (*b*) from litharge and lead acetate, following the directions given below, and to use these two solutions comparatively in the Winton method and also in the Canadian lead method.

PREPARATION OF BASIC LEAD ACETATE SOLUTIONS.

(**a**) *From Horne's lead subacetate.*—Boil 280 grams of Horne's lead subacetate with 500 cc. of water. When solution is complete except for a slight sediment, allow to cool in the dish or pour off into a Pyrex beaker. Take the sp. gr. at 20°C. and dilute with recently boiled water to a density of 1.25.

(**b**) *From litharge and lead acetate.*—Weigh 215 grams of normal lead acetate crystals and 65 grams of litharge into a 1-liter evaporating dish. Add about 500 cc. of water. Heat to boiling and boil exactly 30 minutes. Allow to cool in the dish or pour off into a Pyrex beaker. Dilute to a density of 1.25 at 20°C. as in (**a**).

DETERMINATION OF THE ALKALINITY OF LEAD SUBACETATE SOLUTIONS.

Pipette 10 cc. of the solution (sp. gr. 1.25) into a small beaker or Erlenmeyer. Add exactly 50 cc. of N/2 oxalic or sulphuric acid. Mix and allow to stand until the precipitate is well settled. Filter into a 250 cc. volumetric flask, washing the precipitate thoroughly with water. Make up to the mark and titrate 50 cc. aliquots with N/10 sodium hydroxid, using phenolphthalein.

Half the difference between 50 and the number of cc. of N/10 sodium hydroxid used represents the number of cc. of N/10 sulphuric acid neutralized by 1 cc. of the lead subacetate solution.

DETERMINATION OF TOTAL LEAD IN THE LEAD SUBACETATE SOLUTIONS.

Pipette 5 cc. of the solution into a 250 cc. flask. Add sufficient acetic acid (about 1 cc. of 30% or 5N acid) to prevent precipitation on diluting, and make up to the mark. Treat 25 cc. aliquots with 25 cc. of water and 1 cc. of dilute sulphuric acid (if 5N, or

2 cc. if 2N). Mix, add 100 cc. of 95% alcohol and allow to stand 3 hours or more. Filter on a tared Gooch, wash with alcohol, dry in an oven and ignite to bright redness in a muffle. Cool and weigh.

Multiply the weight of the precipitate by 1.3665 $\left(\frac{Pb \times 2}{Pb\,SO_4}\right)$ to obtain the weight of lead per cc. of solution.

CALCULATION OF THE RATIO OF NEUTRAL TO BASIC LEAD.

Multiply the alkalinity of the solution (expressed in cc. of N/10 acid) by 0.01036 $\left(\frac{Pb}{2 \times 10{,}000}\right)$. The result represents the basic lead per cc. The difference between the total and the basic lead represents the neutral lead per cc. Calculate the ratio of neutral to basic lead to the second decimal place.

The results of the analyses are shown at the foot of Tables 5 and 6 (Canadian lead numbers). It will be seen that Horne's salt yielded solutions a little more variable in lead content but much more uniform in basicity than the litharge and lead acetate. The extreme variations among the five collaborators were:

Range of variation in composition of basic lead acetate solution.

SOLUTION	TOTAL LEAD	RATIO OF NEUTRAL TO BASIC LEAD
	grams per cc.	
Horne's salt	0.214—0.237	1.50—1.83
Litharge and lead acetate	0.215—0.233	1.33—2.75

The results of the Winton lead determinations are given in Tables 2 and 3, the figures being averages of duplicate results. The acetic acid blank was used in calculating these results. The blanks in which a cane sugar sirup was used in place of the acetic acid did not differ materially from the acetic acid blanks.

It will be noted that, although the differences are small, the solution prepared from Horne's salt gives a higher average result on six of the seven sirups. With the following six exceptions, all of the thirty-five results obtained by the individual collaborators on individual sirups are higher with Horne's salt solution than with that prepared from litharge and lead acetate: Valin, Sample 4, both prepared and unprepared; Van Zoeren, Samples 4 and 6; and McFarlane, Samples 3 and 6. As will be seen by reference to Table 6, the solutions prepared from litharge and lead acetate by Van Zoeren and McFarlane were exceptionally basic, having (at the density 1.25) ratios of neutral to basic lead lower than Horne's salt solutions made and analyzed by the same collaborators. It may also be noted that all three sirups in question (Samples 3, 4 and 6) were adulterated sirups. Were it not that results in the opposite direction were obtained by Bryan[1] with a similarly pre-

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[1] U. S. Dept. Agr. Bull. 466: (1917), 10.

pared litharge-lead acetate solution, it might be concluded that in general the 1.25 solutions prepared from Horne's lead subacetate have ratios of about 1.50 to 1.75 and, when diluted, give slightly higher Winton numbers than those prepared from litharge and lead acetate, which normally (at a density of 1.25) have over twice as much neutral as basic lead. For Bryan's similarly prepared litharge-lead acetate solution (his No. 5) the writer calculates the ratio of neutral to basic lead as 1.11. In five out of six pure sirups this gave slightly higher results than a more basic Horne's salt solution (his No. 4; ratio 1.14).

In the course of the work on the Winton lead method it was observed by Van Zoeren and the writer that when the filtrate from the lead subacetate precipitate was allowed to stand for a few days it became turbid. This suggested that results might be affected by unduly extending the interval between precipitation and filtration. The results shown in Table 4 indicate that error may arise from this source but the effect of time intervals of more reasonable length has not been investigated.

CANADIAN LEAD NUMBER.

Weigh the quantity of sirup containing 25 grams of dry matter, transfer to a beaker, add 50–75 cc. of water, boil gently for 2–3 minutes, transfer to a 100 cc. flask, cool and make up to the mark.

Pipette 20 cc. of this solution into a large test tube, add 2 cc. of lead subacetate solution (sp. gr. 1.25) and mix. Allow to stand 2 hours, filter through a tared Gooch, having an asbestos mat at least 3 mm. thick, wash four or five times with boiling water, dry at 100°C. and weigh. Multiply the weight of the dry precipitate by 20.

In the collaborative work of 1917, it was noted that Valin obtained lower results on all sampels than did Van Zoeren and the writer. After the report was completed, an interchange of solutions was made between the two laboratories and the following results obtained:

Comparison of Canadian lead numbers obtained in 1917 with different solutions of basic lead acetate.

SOLUTION	ALKALINITY CC. N/10 ACID PER CC. OF SOLUTION	CANADIAN LEAD NUMBERS		
		A. O. A. C. 1917 Sirup No. 2		Another sirup
		Valin	Snell	Valin
Litharge-lead acetate, 1915*	7.40	3.19	3.42	1.94
Litharge-lead acetate, 1917*	8.60	....	....	2.21
Horne's salt‡	8.72	....	3.75† 3.84§	2.29

* Prepared in Food and Drugs Laboratory, Inland Revenue Department, Ottawa, Canada.
† Result obtained in October.
‡ Prepared in Macdonald College Laboratory, Macdonald College, Quebec, Canada.
§ Result obtained in December.

This indicated that higher lead numbers might be obtained the more alkaline the basic lead solution. This indication is strikingly confirmed in the results tabulated in Tables 5 and 6. All except two of the collaborators invariably obtained higher results on each sirup when they used Horne's salt solution than when the other solution was employed. The exceptions were Van Zoeren, whose very basic litharge-lead acetate solution gave higher results in five instances out of six than his Horne's salt solution, the results being equal in the sixth instance; and McFarlane, whose two subacetate solutions were nearly equal in ratio and whose results are divided, four to three.

A notable feature of Tables 5 and 6 is the wide divergence between the results of the different collaborators and even in different series of experiments by the same investigator. This is still more clearly brought out in Table 7. Results, (F), by A. G. Woodman (Massachusetts Institute of Technology, Boston, Mass.), are without exception the highest or second highest, and Valin's results on the prepared sirup in 1918, (A), the lowest or second lowest. With Horne's solution Valin's 1919 results, (B), are high throughout and McFarlane's, (D), low. Valin's 1918 results on the unprepared sample, (G), and the writer's, (E), are always near the middle or average. Van Zoeren's results, (C), with the litharge-lead acetate solution are among the highest, a result which might be attributed to the very high basicity of his solution had not Woodman, with the least basic solution of all, obtained results equally high. Woodman sometimes allowed his solutions to stand more than the prescribed two hours but Valin states that overstanding does not render his results high.

Evidently, there is some unrecognized condition affecting the Canadian lead number. Valin (Column H) made some experiments with a modification of the Canadian lead method in which 5 cc. of a solution, made by diluting one volume of the specific gravity 1.25 solution of basic lead acetate to five volumes, were used in place of the 2 cc. of the 1.25 solution and the time of standing was extended to 3 or 4 hours. In these experiments, not only were the precipitates weighed but the unprecipitated lead in the filtrate was also determined, as in the Winton method, the object being to ascertain whether the variations between duplicates in the Canadian lead method might not be due to variations in the washing of the precipitates. In spite of the fact that the quantity of lead used in these experiments per gram of dry matter was only half as much as in the regular Canadian method, the lead numbers found (Tables 5 and 6, Column H) were close to those obtained by the regular method and the differences between the results obtained with Horne's and litharge-lead acetate solutions were practically the same as in the regular Canadian method.

The quantity of lead contained in 5 cc. of the diluted Horne's solution was 0.234 and that in the litharge-lead acetate 0.230, being close to the amounts found in 1 cc. of the 1.25 solutions. The percentages of these quantities of lead remaining in the filtrate from the lead precipitates are shown in Table 8. The maximum difference in the quantities of lead found in the filtrates from duplicates was 1.30 per cent, corresponding to a difference of 3 mg. of lead. Valin infers that the differences between duplicates are not due to the washing but to the actual amount of precipitate produced by the reagent.

ASH VALUES.

The ash values as found by three of the collaborators are given in Tables 9 and 10. The figures of Valin and McFarlane are averages of satisfactory duplicates (except Valin's alkalinities, which appear to be single determinations); the averages of the writer are of three or four measurements, some made in 1918, the others in 1919. The agreement among collaborators is closer on the ash values than on any of the other determinations.

In the Macdonald College Laboratory it is the custom to ash over an Argand burner until charring is complete, then transfer to a muffle. When the carbon is nearly all burned out, the dish is removed from the muffle, allowed to cool, the ash moistened with 0.5 to 1.0 cc. of water, evaporated to dryness and returned to the muffle. The moistening appears to facilitate the ashing and also diminishes the danger of mechanical loss of the fluffy ash. When the carbon is all burned out, it is allowed to cool again, about 0.1 gram of solid ammonium carbonate added, moistened with water, evaporated to dryness and heated gently in the muffle for 1 to 2 minutes. In treating the insoluble ash, the cooled ash is moistened immediately after ignition over the flame, so as to provide against mechanical loss of the light filter ash in transferring to the muffle. In the alkalinity determinations, methyl red is used in place of methyl orange.

MALIC ACID VALUE.

Malic acid value was determined by the Cowles method. The results, as given in Table 11, show wide variations, the writer's results being generally high and Valin's low, while those of the two other collaborators are distributed. McFarlane and the writer both warmed the solutions during standing. McFarlane adhered to a definite volume of wash alcohol; the writer did not. A blank determination was used by the writer but not by McFarlane. With Sample 1, the writer experimented with four solutions of calcium acetate which were made from two samples of calcium acetate (Merck's "dry" and Baker and Adamson's, labelled Ca $(C_2H_3O_2)_2$ $2H_2O$, but approximating closely to Ca $(C_2H_3O_2)_2$ H_2O (theory 31.84 per cent of calcium oxid; found 31.97 and 32.04 per cent; average 32.01 per cent) and varied slightly in concentration. Although individual results varied from 0.74 to 0.94, the variations did not appear to be related to the make-up of the solutions. In some instances, the precipitate was ignited and weighed (sometimes both as calcium oxid and as calcium carbonate), as well as titrated. The gravimetric results were always higher than the volumetric, but it was evident from the color that the ignited precipitates were not pure calcium compounds. A pair of determinations made with calcium chlorid, instead of calcium acetate, gave 0.72 and 0.71 (uncorrected for blank), as compared with the average of 0.86 obtained with the acetate. The procedure followed was the same with both salts.

In the original malic acid value method of Leach and Lythgoe[1] the chlorid was the calcium salt used, and its use was retained by Hortvet[2] in adapting the method to maple products. In his extensive work on pure maple sirups, Bryan[3] used both the Cowles method and a modification of the Hortvet method in which blanks were run. The range of variation of the value was narrower with the Hortvet method than with the Cowles, 0.29 to 1.60 as against 0.21 to 1.82; in other words, a range of 452 per cent of the minimum value (156 per cent of the average, 0.84) for the Hortvet method as against 767 per cent of the minimum (159 per cent of the average 1.01) for the Cowles method. Calcium chlorid has also the advantage that if incompletely washed out it will not affect the alkalinity of the ash of the precipitate. In spite of the preference of Cowles and Bryan[4] for the acetate, it seems to the writer possible that a method might be worked out with the chlorid, which would give results more consistent than those so far obtained with the Cowles procedure. In its present form, the latter is certainly not a satisfactory method.

Pozzi-Escot's suggestion[5] to substitute an alcoholic solution of barium bromid for the calcium salts is worthy of consideration.

CONDUCTIVITY VALUE.

Determination of the cell constant.—Prepare 0.1, 0.02 and 0.01 molar potassium chlorid solutions by dissolving respectively, 7.4560, 1.4912 and 0.7456 grams of pure, ignited potassium chlorid in water and making up to 1000 cc. at 18°C. In a 100 cc. beaker place 60 cc. of the 0.01 molar solution, insert a Van Zoeren or other dipping electrode, bring to 25°C. and measure the electrical resistance. Multiply the number of ohms found by 141.2. Rinse the electrode and beaker with the 0.02 molar solution, put in 60 cc. of this solution, measure its resistance at 25°C. and multiply by 276.8. Rinse

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[1] *J. Am. Chem. Soc.*, 1904, **26**: 380.
[2] *Ibid.*, 1536; U. S. Bur. Chem. Bull. **107**, rev.: 1912, 74.
[3] U. S. Bur. Chem. Bull. **134**: 1910, 18.
[4] *J. Am. Chem. Soc.*, 1908, **30**: 1285; U. S. Dept. Agr. Bull. **466**: (1917), 11.
[5] *Bull. assoc. chim. dist.*, 1908, **26**: 266.

with the 0.1 molar solution, put in 60 cc., measure its resistance at 25°C. and multiply by 1289. Average the three results (which should agree within 1 per cent) and multiply by 10^{-5}.

Method.—Weigh out the quantity of sirup containing 22 grams of dry matter. Transfer to a 100 cc. volumetric flask with warm water, cool and make up to the mark. Measure 60 cc. of the solution into a 100 cc. beaker, insert a Van Zoeren or other dipping electrode, bring to 25°C. (±0.1°) and measure the electrical resistance. Divide the constant of the cell by the observed number of ohms and multiply the result by 10^5.

The test was applied to the samples, both in the above form and in the form previously published[1], in which 20 cc. of the prepared sirup were measured out with a graduate into a 100 cc. beaker and the graduate rinsed with two successive portions of 20 cc. of water. Except in Sample 1, closer agreement between collaborators was obtained by using the more exact method, *i. e.*, making solutions containing exactly 22 grams of dry matter to 100 cc. (See Table 12.)

This test is so simple, so rapid and so useful that it may well be recommended to the association for adoption.

RECOMMENDATIONS.

It is recommended—

(1) That the directions for "Preparation of Sample"[2] be amended to read as follows:

53 PREPARATION OF SAMPLE.—TENTATIVE.

(**a**) *Maple sirup.*—Determine the moisture by one of the methods given under **54**(**a**). If sugar has crystallized out, redissolve it by warming. Shake up any sediment remaining and pour off into a casserole or beaker a suitable quantity for analysis (100 cc.). Add one-fourth the volume of distilled water. Boil to a temperature of 104°C. Filter hot through a plug of cotton wool in a funnel. After cooling redetermine the moisture according to **54**(**a**) and use the result to reduce the other values determined to the dry substance basis.

(**b**) *Maple sugar and other solid or semi-solid products.*—Determine moisture by one of the methods given under **54** (**b**) after thoroughly mixing the sample. For all other determinations prepare a sirup from about 100 grams of the material by dissolving in about 150 cc. of hot water, boiling to a temperature of 104°C. and filtering through cotton wool as in (**a**). After cooling determine the moisture in the sirup according to one of the methods of **54** (**a**).

(2) That in the tentative method for the determination of moisture, 54 (a)[2], the refractometer method, **10**[3], be given the preference and that the other method to be recommended in **54** (a) and (b) be decided upon by the referee on saccharine products. (See Report of Associate Referee on Maple Products 1917[4].)

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[1] *J. Ind. Eng. Chem.*, 1916, **8**: 331.
[2] *Assoc. Official Agr. Chemists, Methods*, 1916, 136.
[3] *Ibid.*, 126.
[4] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 157

(3) That the tentative method for total ash[1] be amended to read as follows:

Heat 5 grams of the prepared sirup over a low flame (an Argand burner is recommended) until completely charred. Transfer to a muffle and heat at low redness (not over 550°) until a white ash is obtained. (If desired, the ashing may be interrupted when the carbon is nearly all burned, and after cooling, 0.5–1.0 cc. of water may be added and evaporated.) After cooling, add about 0.1 gram of ammonium carbonate, free from non-volatile matter, and add 0.5–1.0 cc. of water. Evaporate to dryness and reheat in the muffle for 1–2 minutes. Cool in a desiccator and weigh.

(4) That the Canadian lead method be adopted as a tentative method with directions as follows:

CANADIAN LEAD NUMBER.—TENTATIVE.

REAGENT.

Standard basic lead acetate solution.—Boil 280 grams of Horne's lead subacetate with 500 cc. of water. When solution is complete except for a slight sediment, pour off or allow to cool and dilute with recently boiled distilled water to a density of 1.25 at 20°C.

DETERMINATION.

Weigh the quantity of prepared sirup (**IX, 53**[2]) containing 25 grams of dry matter, transfer to a 100 cc. volumetric flask, cool to 20°C. and make up to the mark.

Pipette 20 cc. into a large test tube, add 2 cc. of the standard basic lead acetate solution and mix. Allow to stand 2 hours. Filter on a tared Gooch, having an asbestos mat at least 3 mm. thick, wash four or five times with boiling water, dry at 100°C. and weigh. Multiply the weight of the dry precipitate by 20.

(5) That the caption above **IX, 63**[1] be amended to read, "Winton Lead Number.—Tentative".

(6) That in **IX, 65**, line 3[1], the words "at least" before "3 hours" be omitted.

(7) That the conductivity value method, as given on page 435, be adopted as a tentative method.

KEY TO OBSERVERS.

I. Using Abbé refractometer:
  - A. Woodman, 1918.
  - B. Valin, 1918.
  - C. Snell, 1918.
  - O. Woodman, 1919.

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 137.
[2] *Ibid.*, 136.

II. Using Féry refractometer:
D. Van Zoeren, 1918
E. Miss Dorothy Moule, Macdonald College, Quebec, Canada, 1918.
F. Snell, 1918.
P. Snell, August, 1919.
Q. McFarlane, August, 1919.
R. Snell, September, 1919.
S. McFarlane, September, 1919.
Identical samples—C and F.
Identical samples—P, Q, R and S.

TABLE 1.

Moisture in original samples, determined refractometrically.*

	SAMPLE 1	SAMPLE 2	SAMPLE 3	SAMPLE 4	SAMPLE 5	SAMPLE 6	SAMPLE 7
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
Maximum.....	O *43.06*†	O *32.40*	O *33.80*	Q *34.27*	E *33.70*	E *32.70*	Q *35.62*
	D *43.04*	A *32.30*	F *33.64*	A 34.15	D *33.57*	A 32.36	F 35.39
	F *42.79*	P 32.23	D *33.47*	R 34.15	O *33.50*	C 32.31	D 35.37
	A *42.75*	R 32.23	E 33.35	S 34.10	R 33.34	R 32.28	C 35.32
	P 42.58	D 32.12	C 33.33	D 34.03	C 33.27	.....	.....
Middle.......	E 42.50	F 32.06	P 33.17	C 34.00	A 33.20	F 32.26	O 35.31
						O 32.25	R 35.16
	C 42.48	E 32.05	R 33.09	F 33.86	B 33.07	E 32.22	A 35.14
	S 42.34	S 32.03	A *32.91*	B 33.82	F 33.05	P 32.21	B 35.12
	R 42.33	Q 32.01	B *32.72*	P *33.74*	S 33.05	S *32.04*	S *34.97*
	B *42.17*	C 31.98	S *32.62*	O *33.65*	Q 33.00	.....	.....
Minimum.....	Q *42.15*	B 31.92	Q *32.51*	E *33.41*	P *32.97*	Q *31.87*	P *34.91*
Range........	0.91	0.48	1.29‡	0.86	0.73	0.83	0.71

* *Assoc. Official Agr. Chemists, Methods*, 1916, 126.

† The italicized readings differ from the middle readings by more than 0.20, the limit adopted by Main (see page 429).

‡ Sample 3 was difficult to read in the Féry refractometer.

TABLE 2.

Winton lead numbers, dry basis, using Horne's salt solution.

SIRUP NUMBER	PREPARED SAMPLES				UNPREPARED SAMPLES	AVERAGE
	Valin	Van Zoeren	McFarlane	Snell	Valin	
1....................	2.06	2.70	2.65	2.33	2.19	2.39
2....................	2.50	2.85	3.10	2.66	2.56	2.73
3....................	1.93	2.87	2.29	2.33	2.06	2.30
4....................	1.96	2.14	2.59	2.09	1.76	2.11
5....................	2.42	2.70	2.42	2.50	2.33	2.47
6....................	2.00	2.65	2.37	2.14	2.09	2.25
7....................	2.50	2.93	2.52	2.79	2.51	2.65
Acetic blank, grams lead sulphate........	0.170	0.1697	0.1652	0.1692	0.170	....
Sugar blank, grams lead sulphate........	0.171	0.1666	0.1667	0.1683	0.171	....

TABLE 3.

Winton lead numbers, dry basis, using litharge-lead acetate solution.

SIRUP NUMBER	PREPARED SAMPLES				UNPREPARED SAMPLES	AVERAGE
	Valin	Van Zoeren	McFarlane	Snell	Valin	
1	2.05	2.63	2.46	2.21	2.18	2.31
2	2.36	2.78	2.33	2.54	2.46	2.49
3	1.89	2.84	2.62	2.30	1.93	2.32
4	2.12	2.42	2.01	1.92	1.81	2.06
5	2.07	2.67	2.35	2.34	2.14	2.31
6	1.85	2.67	2.44	1.98	1.92	2.17
7	2.29	2.88	2.48	2.56	2.29	2.50
Acetic blank, grams lead sulphate	0.168	0.1693	0.1611	0.1708	0.168	....
Sugar blank, grams lead sulphate	0.167	0.1686	0.1613	0.1697	0.167	....

TABLE 4.

Effect of overstanding of lead precipitate in Winton determination.

SIRUP NUMBER	SUBACETATE SOLUTION	WINTON LEAD NUMBER WET BASIS	APPARENT WINTON NUMBER IN OVER-HELD SOLUTION	TIME OVERHELD
				weeks
3	Litharge	1.56	1.70	4*
3	Horne's	1.58	1.91	4
4	Litharge	1.23	1.41	4*
4	Horne's	1.34	1.76	4
5	Litharge	1.53	1.87	4
5	Horne's	1.63	2.02	4

* Days.

TABLE 5.

Canadian lead numbers, using Horne's salt solution.

SIRUP NUMBER	PREPARED SAMPLES						UNPREPARED SAMPLES	MODIFIED METHOD
	A Valin 1918	B Valin 1919	C Van Zoeren 1918	D McFarlane 1919	E Snell 1918	F Woodman 1919	G Valin 1918	H Valin 1919
1	2.62	3.41	3.31	3.01	3.35	4.04	3.16	3.46
2	2.80	3.75	3.62	3.23	3.62	4.37	3.64	3.93
3	2.56	3.44	3.66	2.85	3.23	4.13	3.39	3.31
4	2.30	2.87	2.77	2.31	2.69	3.38	2.79	3.05
5	3.15	3.92	3.51	2.57	3.48	4.11	3.63	3.72
6	2.36	3.11	....	2.75	2.81	3.30	2.89	3.20
7	3.17	4.03	3.16	3.31	3.69	4.77	3.85	3.62
Alkalinity	8.69	8.95	8.55	8.10	8.51	7.48	8.69	....
Lead per cc.	0.223	0.232	0.2329	0.237	0.2337	0.2144	0.223	....
Ratio of neutral lead to basic lead	1.58	1.50	1.63	1.83	1.65	1.76	1.58	....

TABLE 6.

Canadian lead numbers, using litharge-lead acetate solution.

SIRUP NUMBER	PREPARED SAMPLES						UNPREPARED SAMPLES	MODIFIED METHOD
	A Valin 1918	B Valin 1919	C Van Zoeren 1918	D McFarlane 1919	E Snell 1918	F Woodman 1919	G Valin 1918	H Valin 1919
1	2.39	2.87	3.44	2.78	2.96	3.38	2.97	2.99
2	2.67	3.43	3.76	3.14	3.51	3.78	3.29	3.53
3	2.32	2.90	3.91	2.96	3.01	3.45	3.12	2.90
4	2.10	2.55	2.77	2.36	2.56	2.79	2.56	2.70
5	2.80	3.48	3.64	3.16	3.25	3.77	3.28	3.33
6	2.13	2.72	2.80	3.63	2.78	3.11	2.67	2.86
7	2.72	3.65	3.88	2.99	3.52	3.82	3.41	3.35
Alkalinity	6.83	6.87	9.65*	7.45	7.03	5.72	6.53	....
Lead per cc.	0.230	0.229	0.233	0.215	0.2293	0.2225	0.230	....
Ratio of neutral lead to basic lead	2.24	2.20	1.33	1.78	2.01	2.75	2.24	....

* In making up this solution, Van Zoeren added an old solution to the water with which he boiled the litharge and lead acetate.

TABLE 7.

Canadian lead numbers, arranged in order of magnitude.

	SAMPLE 1	SAMPLE 2	SAMPLE 3	SAMPLE 4	SAMPLE 5	SAMPLE 6	SAMPLE 7
Using Horne's salt: Maximum	F 4.04	F 4.37	F 4.13	F 3.38	F 4.11	F 3.30	F 4.77
	B 3.41	B 3.75	C 3.66	B 2.87	B 3.92	B 3.11	B 4.03
	E 3.35	G 3.64	B 3.44	G 2.79	G 3.63	G 2.89	G 3.85
Middle	C 3.31	E 3.62	G 3.39	C 2.77	C 3.51	....	E 3.69
	G 3.16	C 3.62	E 3.23	E 2.69	E 3.48	E 2.81	D 3.31
	D 3.01	D 3.23	D 2.85	D 2.31	A 3.15	D 2.75	A 3.17
Minimum	A 2.62	A 2.80	A 2.56	A 2.30	D 2.57	A 2.36	C 3.16
Range	1.42	1.57	1.57	1.08	1.54	0.94	1.61
Using litharge-lead acetate: Maximum	C 3.44	F 3.78	C 3.91	F 2.79	F 3.77	D 3.63	C 3.88
	F 3.38	C 3.76	F 3.45	C 2.77	C 3.64	F 3.11	F 3.82
	G 2.97	E 3.51	G 3.12	G 2.56	B 3.46	C 2.80	B 3.65
Middle	E 2.96	B 3.43	E 3.01	E 2.56	G 3.28	E 2.78	E 3.52
	B 2.87	G 3.29	D 2.96	B 2.55	E 3.25	B 2.72	G 3.41
	D 2.78	D 3.14	B 2.90	D 2.36	D 3.16	G 2.67	D 2.99
Minimum	A 2.67	A 2.67	A 2.32	A 2.10	A 2.80	A 2.13	A 2.72
Range	0.77	1.11	1.59	0.69	0.97	1.50	1.16

TABLE 8.

Modified Canadian lead method—study of duplicates by A. Valin.

SAMPLE NUMBER	CANADIAN LEAD NUMBERS			TOTAL LEAD LEFT IN FILTRATE	
	Using Horne's salt	Using litharge-lead acetate		Using Horne's salt	Using litharge-lead acetate
				per cent	*per cent*
1............	3.50	2.96		50.85	56.95
	3.44	3.02		51.28	55.65
Average.....	3.47	2.99	Difference....	−0.43	1.30
2............	3.88	3.52		43.61	49.13
	3.98	3.54		44.01	49.13
Average.....	3.93	3.53	Difference....	−0.40	0.00
3............	3.28	2.88		55.59	60.00
	3.36	2.92		54.70	60.43
Average.....	3.32	2.90	Difference....	0.89	−0.43
4............	3.12	2.70		57.27	62.17
	2.98	2.70		56.83	61.30
Average.....	3.05	2.70	Difference....	0.44	0.87
5............	3.66	3.34		50.00	56.52
	3.78	3.32		51.28	56.52
Average.....	3.72	3.33	Difference....	−1.28	0.00
6............	3.20	2.90		52.57	56.52
	3.20	2.82		52.57	56.52
Average.....	3.20	2.86	Difference....	0.00	0.00
7............	3.68	3.40		46.15	53.91
	3.56	3.30		46.58	53.91
Average.....	3.62	3.35	Difference....	−0.43	0.00

Table 9.

Ash values.

ANALYST	SAMPLE 1	SAMPLE 2	SAMPLE 3	SAMPLE 4	SAMPLE 5	SAMPLE 6	SAMPLE 7
Total ash:	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
Valin.	0.95	1.02	0.99	0.79	1.00	0.93	1.08
McFarlane. .	1.05	1.10	1.00	0.74	0.98	0.83	1.08
Snell.	1.00	1.03	1.00	0.85	0.96	0.84	1.07
Soluble ash:							
Valin.	0.52	0.69	0.62	0.45	0.55	0.48	0.71
McFarlane. .	0.70	0.78	0.63	0.40	0.57	0.52	0.66
Snell.	0.65	0.70	0.54	0.51	0.53	0.54	0.74
Insoluble ash:							
Valin.	0.43	0.33	0.37	0.34	0.45	0.45	0.37
McFarlane. .	0.36	0.32	0.42	0.34	0.41	0.31	0.42
Snell.	0.36	0.33	0.43	0.35	0.42	0.31	0.37
Ratios of soluble ash to insoluble ash:							
Valin	1.21	2.09	1.67	1.02	1.22	1.06	1.91
McFarlane. .	1.94	2.44	1.50	1.18	1.39	1.68	1.57
Snell.	1.81	2.12	1.26	1.46	1.26	1.74	2.00

Table 10.

Ash values—alkalinities.

(Expressed as cc. of N/10 solution per gram of sirup.)

ANALYST	SAMPLE 1	SAMPLE 2	SAMPLE 3	SAMPLE 4	SAMPLE 5	SAMPLE 6	SAMPLE 7
Total ash*:							
Valin.	1.62	1.79	1.62	1.91	1.68	1.38	1.68
McFarlane. .	1.54	1.69	1.68	1.26	1.62	1.38	1.62
Snell.	1.45	1.83	1.71	1.57	1.66	1.42	1.66
Soluble ash:							
Valin.	0.81	0.98	0.69	1.06	0.67	0.69	0.84
McFarlane. .	0.75	0.95	0.73	0.51	0.70	0.64	0.63
Snell.	0.75	0.91	0.73	0.59	0.69	0.62	0.76
Insoluble ash:							
Valin.	0.81	0.81	0.93	0.85	1.01	0.69	0.84
McFarlane. .	0.79	0.74	0.95	0.75	0.92	0.74	0.99
Snell.	0.72	0.91	0.98	0.98	0.97	0.80	0.90
Ratio of soluble to insoluble ash:							
Valin.	1.00	1.21	0.74	1.24	0.66	1.00	1.00
McFarlane.	0.95	1.23	0.77	0.68	0.76	0.86	0.64
Snell.	1.04	1.00	0.74	0.60	0.71	0.77	0.84

* By the addition of soluble and insoluble ash alkalinities.

TABLE 11.

Malic acid values.

SAMPLE NUMBER	VALIN	WOODMAN	MCFARLANE	SNELL
1	0.57	0.80	0.78	0.86
2	0.63	0.87	0.73	0.82
3	0.48	0.67	0.62	0.76
4	0.57	0.58	0.53	0.76
5	0.59	0.62	0.81	0.85
6	0.54	0.61	0.80	0.85
7	0.42	0.86	0.84	0.84

TABLE 12.

Conductivity values.

SIRUP NUMBER	USING A SOLUTION OF 22 GRAMS OF DRY MATTER TO 100 CC.			USING 20 CC. OF SIRUP			
	Woodman	McFarlane	Snell	Woodman	McFarlane	Snell	Van Zoeren
1	166	154	167	165	166	173	171
2	173	172	173	171	177	179	180
3	171	166	172	171	168	177	178
4	128	128	131	123	128	132	...
5	149	148	152	148	154	153	154
6	139	138	141	137	138	144	145
7	191	192	195	192	195	202	203

No associate referee on the subject of honey was appointed and no report on this subject was presented.

Messrs. C. S. Hudson[1] and S. F. Sherwood[2] (Bureau of Chemistry, Washington, D. C.), presented a paper on "The Occurrence of Melezitose in Honey"[3].

E. T. Wherry (Bureau of Chemistry, Washington, D. C.), presented a paper on "Crystallography of Melezitose"[4].

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[1] Present address, 204 Academy Ave., Trenton, N. J.

[2] Present address, Bureau of Plant Industry, Washington, D. C.

[3] *J. Am. Chem. Soc.*, 1920, **42**: 116.

[4] *Ibid.*, 125.

THE DETERMINATION OF ASH IN CANE SIRUPS AND MOLASSES.

By F. W. ZERBAN[1] (Louisiana Sugar Experiment Station, New Orleans, La.), *Associate Referee on Sugar House Products*[2].

Since there is a possibility that the percentage of ash in sirups and molasses may, in some way, play a part in the official food standards to be established for these products it seemed advisable to carry out a comparative study of the methods for the determination of ash, as adopted by the association. There are at present three such methods[3], all of them official. The two principal questions to be settled are: First, which of the three methods yields the most concordant results in the hands of different analysts; and second, how well the results obtained by the three methods agree with one another. The first of these questions appears to be the more important from the standpoint of food control, although the second should also be settled as well as can be done in a matter involving the determination of a constituent as vaguely defined as "ash". For the present, we shall call "ash" that part of a substance which remains after heating it in the presence of air to a temperature high enough to destroy its organic constituents or convert them into carbonates, but not sufficiently high to volatilize the inorganic constituents to any extent.

In order to get an answer to the first question, and at the same time some information on the second, three samples of genuine cane products were sent out to a number of chemists who had expressed their willingness to cooperate. One of the samples was a cane sirup; another a first centrifugal molasses; and the third a final molasses.

Collaborators were asked to determine the ash in each of these samples by all of the following methods:

Method I.

Place 5 grams of the sample in a 50–100 cc. platinum dish (or a smaller quantity, if only small dishes are available), add a few drops of pure olive oil, and heat slowly over a flame until swelling ceases. Then place the dish in a muffle and heat at a very dull red heat, until all the carbon is completely burned off. Repeat the heating in the muffle, until constant weight is obtained. ·Record the result.

Take up the residue with a little ammonium carbonate solution, reevaporate, and heat again in the muffle at very dull red heat to constant weight. Record the result.

Method II.

Place 5 grams of the sample in a 50–100 cc. platinum dish (or a smaller quantity, if only small dishes are available), add a few drops of pure olive oil, and carbonize the mass over a free flame at the lowest possible temperature. Dissolve the soluble salts in hot

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[1] Present address, Penick & Ford Ltd., Inc., Marrero, La.

[2] For the year ending November, 1918.

[3] *Assoc. Official Agr. Chemists, Methods*, 1916, 128.

water, and ignite the carbonaceous residue as directed in Method I. Then add the solution of soluble salts, evaporate to dryness at 100°C., and heat in a muffle at very dull red heat, until constant weight is obtained. Record the result.

Take up the residue with a little ammonium carbonate solution, reevaporate, and heat again in the muffle at a very dull red heat to constant weight. Record the result.

Method III.

Place 5 grams of the sample in a 50–100 cc. platinum dish or silica dish (or a smaller quantity, if only small dishes are available), add 0.5 cc. of concentrated sulphuric acid per 5 grams of sample (if less was taken, reduce the amount of sulphuric acid accordingly), heat gently over a free flame until the sample is well carbonized. Then heat in the muffle at low red heat to constant weight. Record the result.

Method IV.

Same as Method III, except that 1 cc. of sulphuric acid per 5 grams of sample is used.

TABLE 1.

Determination of ash in sirup.

ANALYST	METHOD I*	METHOD I†	METHOD II*	METHOD II†	METHOD III	METHOD IV	METHOD V
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
E. C. Freeland, Sugar Experiment Station, Audubon Park, New Orleans, La.	2.51	2.56	2.50	2.52	3.08	3.17	3.18
C. A. Gamble, New York Sugar Trade Laboratory, 80 South Street, New York, N. Y.	2.41	2.41	2.45	2.49	3.06	3.09	3.12
Roberta Hafkesbring, Morse Laboratory Company, 213 North Peters Street, New Orleans, La.	....	....	....	....	2.82	2.92	3.00
G. H. Hardin, New York Sugar Trade Laboratory, 80 South Street, New York, N. Y.	2.42	2.46	2.42	2.46	2.94	3.07	3.11
S. D. Melancon, Penick & Ford, Ltd., New Orleans, La.	2.64	2.74	2.76	2.69	3.02	3.12	3.09
J. E. Mull, New York Sugar Trade Laboratory, 80 South Street, New York, N. Y.	2.42	2.43	2.45	2.47	3.03	3.08	3.09
H. Z. E. Perkins, American Sugar Refining Company, New Orleans, La.	2.34	2.52	2.60	2.65	3.02	3.10	3.12
D. D. Sullivant, Penick & Ford, Ltd., New Orleans, La.	2.60	2.49	2.58	2.85	3.08	3.02	3.16
F. W. Zerban..........	2.56	2.58	2.61	2.65	3.17	3.16	3.20
Average...........	2.49	2.52	2.55	2.60	3.02	3.08	3.12

* Without the use of ammonium carbonate.
† With ammonium carbonate.

Method V.

Same as Method III, except that 2 cc. of sulphuric acid per 5 grams of sample are used.

Collaborators were asked to make duplicate determinations in each case, and if the results differed by 0.2 per cent or more, to run a third determination.

Ten analysts, to whom the writer wishes to express his thanks, reported results on all or part of the samples, and the results are given in Tables

TABLE 2.

Determination of ash in first molasses.

ANALYST	METHOD I*	METHOD I†	METHOD II*	METHOD II†	METHOD III	METHOD IV	METHOD V
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
E. C. Freeland	5.12	5.11	5.11	4.98	6.13	6.28	6.33
C. A. Gamble	4.90	5.02	4.95	4.98	6.00	6.29	6.30
Roberta Hafkesbring	....	....	....	....	5.84	5.94	5.96
G. H. Hardin	4.94	4.95	4.97	5.00	5.97	6.12	6.18
S. D. Melancon	5.15	5.30	5.85‡	5.32	6.41	6.32	6.48
J. E. Mull	4.96	4.97	4.98	4.93	6.02	6.22	6.34
H. Z. E. Perkins	4.68	4.77	4.86	5.00	5.98	6.22	6.34
D. D. Sullivant	5.04	4.84	5.06	5.18	6.21	6.21	6.29
J. M. Webre, Best Clymer Manufacturing Co., South Fort Smith, Ark.	....	....	....	....	6.04	6.07	6.09
F. W. Zerban	5.08	5.11	5.15	5.03	6.19	6.32	6.29
Average	4.98	5.01	5.01	5.05	6.08	6.20	6.26

* Without the use of ammonium carbonate.
† With ammonium carbonate.
‡ Not considered in average.

TABLE 3.

Determination of ash in final molasses.

ANALYST	METHOD I*	METHOD I†	METHOD II*	METHOD II†	METHOD III	METHOD IV	METHOD V
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
E. C. Freeland	6.36	6.66	6.98	6.66	8.09	8.34	8.48
C. A. Gamble	6.57	6.60	6.52	6.52	8.14	8.24	8.45
Roberta Hafkesbring	....	....	....	....	7.99	7.91	8.17
G. H. Hardin	6.55	6.60	6.60	6.59	8.22	8.32	8.58
S. D. Melancon	6.70	6.74	7.10	6.73	8.61	8.69	8.82
J. E. Mull	6.51	6.57	6.58	6.62	8.16	8.34	8.46
H. Z. E. Perkins	6.09	6.72	6.06	6.80	8.11	8.07	8.64
D. D. Sullivant	6.54	6.62	6.79	6.47	8.38	8.76	8.68
J. M. Webre	....	....	....	....	8.00	8.13	7.98
Average	6.47	6.64	6.66	6.63	8.19	8.31	8.47

* Without the use of ammonium carbonate.
† With ammonium carbonate.

1 to 3. Only the averages of duplicate determinations are shown, and where triplicates were run, the average of the figures nearest each other. On the whole, duplicates made by the same analyst showed very good agreement. Differences of 0.1 per cent or more occurred in 12 cases out of 59 in the sirup analyses, 14 out of 62 in the analyses of the first molasses, and 16 out of 55 in the analyses of the final molasses. Duplicates differed by 0.2 per cent or more in only one case in the sirup analyses, and in three in the analyses of the final molasses. In other words, the agreement is not so good in the more impure products.

The tables show clearly that the agreement between different analysts is not nearly so good as that between duplicates of the same analyst.

TABLE 4.

Maximum variations between the results obtained by the various analysts.

PRODUCT	METHOD I*	METHOD I†	METHOD II*	METHOD II†	METHOD III	METHOD IV	METHOD V
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
Sirup	0.30	0.33	0.34	0.39	0.35	0.25	0.20
First molasses	0.47	0.53	0.29	0.39	0.57	0.38	0.52
Final molasses	0.61	0.17	1.04	0.33	0.62	0.85	0.84

* Without the use of ammonium carbonate.
† With ammonium carbonate.

These maximum variations are, of course, more or less accidental, but they show, nevertheless, that the deviations between results are smaller in the higher grade products, as had been found to be the case for the individual analyst.

It is further noted that the results of one and the same analyst are usually in one direction from the average, either generally high or low, or near the average. This would seem to indicate that each analyst did all of his work under practically identical conditions, but that these conditions differed with different analysts. In one case, that of Roberta Hafkesbring, this was probably due to the fact that silica dishes were used instead of platinum, and this may be responsible for her results being quite below the average. Contact with silica at high temperatures may drive out sulphuric acid under formation of silicates. This point should be further investigated. But in the other cases, where platinum was used, the differences were most probably due to different temperatures being used for the incineration. It will therefore be most important to continue this cooperative work under conditions where the temperature is kept constant and at the same time measured, and to make determinations at different temperatures, in both platinum and silica dishes.

From the results obtained, some important conclusions on the accuracy of each method, in the hands of different analysts may be drawn.

To this end, the average probable error for each method and each product, as well as for the three combined, has been calculated by the expression $\frac{2}{3}\sqrt{\frac{S}{N-1}}$.

The averages included in Table 5 were calculated from all the individual determinations made by each particular method, using the formula $\frac{2}{3}\sqrt{\frac{S}{N-1}}$.

TABLE 5.

Average probable errors of the determinations.

PRODUCT	METHOD I*	METHOD I†	METHOD II*	METHOD II†	METHOD III	METHOD IV	METHOD V
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
Sirup..................	0.076	0.071	0.076	0.085	0.065	0.051	0.039
First molasses...........	0.101	0.111	0.073	0.087	0.107	0.079	0.097
Final molasses...........	0.116	0.043	0.228	0.077	0.131	0.183	0.173
Average............	0.091	0.078	0.135	0.081	0.101	0.113	0.112

* Without the use of ammonium carbonate.
† With ammonium carbonate.

It is readily seen that from the standpoint of chance of error, the sulphated ash method is superior to the others in the case of sirup, but this is not generally true in the case of first molasses, and less so with the final molasses. Even if consideration is given to the fact that the errors shown in the table for the sulphate method are in reality proportionately smaller, on account of the correction factor used in reducing sulphated ash to carbonated ash, the sulphated ash method does not seem to offer any advantages over Methods I and II with ammonium carbonate in point of chance of error. Apparently, these are the two methods by which different analysts show the closest agreement.

Passing now from the question of relative accuracy to that of absolute accuracy of the different methods used, there is, of course, no infallible standard by which to judge which of the first four methods (Methods I and II, both with and without the use of ammonium carbonate) gives the closest approach to the true ash content. The sulphated ash method will have to be considered separately, since in this case the ash must be calculated from the value actually obtained, by applying a correction factor.

Tables 1 to 3 show that Method II, in which the soluble salts are leached out with water and after incineration of the insoluble portion combined with it and incinerated again, has a tendency to give slightly higher results than Method I—the method of direct ashing. This seems somewhat strange, the object of Method II being to facilitate the combustion of the carbon, which would lead to high results if not removed. But it must be considered that in the operation of filtering and evaporating, the solution may easily take up dust particles which will increase the weight. In the experience of the writer, Method II has no advantage over Method I in the three samples analyzed, because it was found easy to burn off the carbon without first dissolving the soluble salts. The results obtained by Method II, without the use of ammonium carbonate, agree so closely with those obtained by Methods I and II, with ammonium carbonate, that there does not seem to be any reason for using this very slow and tedious method, except when it is found impossible to get a carbon-free ash by the shorter method.

The use of ammonium carbonate in either Method I or II has a tendency to give slightly higher results, as may be expected, the object of its use being the reconversion into carbonates of any alkaline earth oxids formed.

Considering that the relative accuracy of Methods I and II, with ammonium carbonate, is about the same, and that both give very closely agreeing results, it would appear that for general purposes Method I, using ammonium carbonate, should be given preference, except when necessity compels the use of the longer procedure. In the experience of the writer, an analysis by Method I, using ammonium carbonate, can be made just as easily and quickly as by the sulphated ash method, provided foam formation is prevented by the use of olive oil or some similar substance.

The sulphated ash method is the one most commonly used in this country in commercial work, and will therefore be discussed more in detail. The official method gives a correction factor of one-tenth, to be deducted from the actual result obtained, in order to convert it into a supposedly equivalent amount of carbonated ash. This correction factor of one-tenth has been the object of investigation on the part of a large number of workers, and an important contribution to this field has lately been made by Ogilvie and Lindfield[1]. They analyzed a number of sugars and molasses, from both beet and cane, by substantially the same methods used in the work here reported, and found that the conversion factor actually varied from 12 to 25 per cent in the case of beet sugars, averaging 15 per cent; from 12 to 17 per cent in that of beet molasses, averaging 15 per cent; from 6 to 22 per cent with cane sugars, averaging 14 per cent; and from 14 to 21 per cent with cane molasses, averaging 18 per cent. Ogilvie and Lindfield also give a summary of previous work on this question, which shows that this problem has been studied quite extensively in connection with beet products, but that very little has so far been done on cane products. Since the correction factor of one-tenth is still generally used in this country, it seems most

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[1] *Intern. Sugar J.*, 1918, **20**: 114.

important to ascertain whether this factor is correct for the cane products handled in the United States, because, if a maximum ash figure should be established as the basis of any of the official standards, then too low a correction factor might in many cases prove detrimental to the seller of these products. Let us suppose, for instance, that a maximum of 5 per cent of ash should be fixed for a certain product, and that an analysis shows 6 per cent of sulphated ash. With the correction factor of one-tenth, adopted by the association, the true ash would be found to be 5.40 per cent, ruling out this particular product. But if the true correction factor be, say one-fifth, instead of one-tenth, the corrected ash will be found to be 4.80 per cent, quite within the limit of the maximum.

Turning again to Tables 1 to 3, it will be noted that the amount of sulphated ash found increases with the quantity of sulphuric acid used. This is quite in agreement with the results of Schweizer[1], who showed that a much higher ash figure is obtained by re-treating the sulphated ash with sulphuric acid, to decompose any sulphids formed. It follows that the correction factor varies with the quantity of sulphuric acid added, and the following table shows what the correction factor is in the case of the three products analyzed in this investigation, taking the results by Method I, using ammonium carbonate, for establishing the factor.

TABLE 6.

Correction factors for sulphated ash (official samples).

PRODUCT	0.5 CC. OF ACID	1 CC. OF ACID	2 CC. OF ACID
	per cent	*per cent*	*per cent*
Sirup	16.6	18.2	19.2
First molasses	17.6	19.2	20.0
Final molasses	18.9	20.1	21.6

Four more samples, different from the above, were analyzed by E. C. Freeland, and the results are given in Table 7. The comparisons are made with results obtained by Method II, without the use of ammonium carbonate.

TABLE 7.

Corrected factors for sulphated ash (additional samples).

PRODUCT	0.5 CC. OF ACID	1 CC. OF ACID	2 CC. OF ACID
	per cent	*per cent*	*per cent*
Sirup	18.7	19.4	19.2
First molasses	16.4	17.9	18.9
Second molasses	19.3	21.1	22.3
Third molasses	17.3	17.2	21.2

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[1] *Arch. Suikerind.*, 1916, 24: 214.

The correction factor is evidently much nearer 20 than 10 per cent, as already pointed out by Ogilvie and Lindfield, and others before them. In the seven products analyzed it is rather constant, averaging about 19 per cent. But this may be accidental, and if the correction factor should in a large number of samples really show such wide variations as found by the authors just mentioned, the sulphated ash method should not be used at all in food control work concerning these products, except where the total ash content is so low that the difference between the highest and lowest correction factors falls within the limit of experimental error.

The results reported show that from the standpoint of close agreement between different analysts the sulphated ash method has no advantage over the direct ash method, and, in the writer's opinion it even has no advantage from that of ease of manipulation. It has furthermore been confirmed that the sulphated ash method, as adopted officially by the association[1], may give results widely divergent from those obtained by the direct ash method.

RECOMMENDATIONS.

It is recommended—

(1) That a study be made of the influence of different and known temperatures of incineration on the results of ash determinations in cane sirups and molasses, carrying out the incineration in both platinum and silica dishes for comparison.

(2) That a large number of samples of different grades of cane sirups and molasses be used for comparing ash determinations by the sulphate and direct methods, to determine, if possible, the proper correction factor to be applied to sulphated ash.

No report on sugar house products was made by the associate referee for the year ending November, 1919.

No report on food preservatives was presented because of the death, during the year, of A. F. Seeker, the referee

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 128.

REPORT ON COLORING MATTERS IN FOODS[1].

By W. E. MATHEWSON (Bureau of Chemistry, Washington, D. C.), *Referee.*

No work on colors was done by the association in 1918. In March, 1919, the following circular letter was sent to the collaborators:

Since the last meeting of the association a number of papers and reports have been published of interest in connection with the analysis of colored food products. Willstaetter and Schudel[2] describe a method for separating basic coloring matters from aqueous solutions, depending on the extraction of the picrates (or dichlorpicrates) by immiscible solvents. The procedure is intended especially for the separation of the anthocyans or glucoside fruit colors, and the authors give the figures in Tables 1 and 2 for the distribution ratios as found when such solutions are shaken with the solvents named.

TABLE 1.

Distribution ratios of the picrates.

SOLVENT	COLORING MATTER EXTRACTED	
	Monoglucoside picrate	Diglucoside picrate
	per cent	*per cent*
Diethyl ketone	50*	0
Amyl alcohol	80–90	30*
Amyl alcohol 2 parts plus acetophenone 1 part	100	70–80

* Approximately.

TABLE 2.

Distribution ratios of the dichlorpicrates.

SOLVENT	COLORING MATTER EXTRACTED	
	Monoglucoside dichlorpicrate	Diglucoside dichlorpicrate
	per cent	*per cent*
Ether	40	3*
Diethyl ketone	100*	60*
Amyl alcohol	100	90
Amyl alcohol — acetophenone 2 : 1	100	100

* Approximately.

No detailed description has been given, but presumably the solutions extracted are treated with an excess of the acid.

Tests for the fruit colors have usually been made in the fruit juices or other mixtures in which they occur, as no satisfactory methods for their preliminary purification have

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[1] Abstract.

[2] *Ber.*, 1918, 8: 782.

been known, and, though the various anthocyans show somewhat different behavior when treated with the common reagents, the differences are not so pronounced that tests made in such mixtures seem very conclusive. Willstaetter and his coworkers have described color reactions with ferric chlorid, aluminium salts, etc., that may be used to identify the purified coloring matter.

The preparation of dichlorpicric acid is rather a tedious and troublesome operation. Acetophenone is now on the market, however, and it is recommended that the use of mixtures of this solvent for the separation of the fruit coloring matter be tried by the collaborators.

Table 3 contains data on some of the anthocyans found in food products.

TABLE 3.

Data on some of the anthocyans occurring in food products.

ANTHOCYAN	SOURCE	PRODUCTS OF HYDROLYSIS	REFERENCE
Oenin........	Grape........	1 mol. oenidin + 1 mol. glucose.	*Ann.*, 1915, **408**: 83–109; 1916, **412**: 195–216.
Myrtillin.....	Whortleberry (blueberry)	1 mol. myrtillidin + 1 mol. galactose.	*Ann.*, 1915, **408**: 83–109; 1916, **412**: 195–216.
Idaein.......	Cranberry....	1 mol. cyandin + 1 mol. galactose.	*Ann.*, 1915, **408**: 15–41.
Prunocyanin .	Sloe.........	1 mol. cyandin + 2 mol. glucose.	*Ann.*, 1916, **412**: 164–178.
Keracyanin ..	Cherry.......	1 mol. cyandin + 1 mol. glucose + 1 mol. rhamnose.	*Ann.*, 1916, **412**: 164–178.
....	Radish.......	Glucosides of pelargonidin and cyanidin.	*Ber. pharm. Ges.*, 1915, **25**: 448.
....	Plum........	Glucoside of this group...	*Ber. pharm. Ges.*, 1915, **25**: 447.
....	Red beet.....	Glucoside of this group...	*Ber. pharm. Ges.*, 1918, **28**: 784.

TABLE 4.

Properties of the anthocyanidins derived from the glucosides named in Table 3 by hydrolysis by boiling with 20 per cent hydrochloric acid.

ANTHOCYANIDIN	PRODUCTS ON FUSION WITH ALKALI	REACTION WITH FERRIC CHLORID IN AQUEOUS OR DILUTE ALCOHOLIC SOLUTION
Pelargonidin..	Phloroglucinol + 4-hydroxybenzoic acid	No characteristic reaction.
Cyanidin.....	Phloroglucinol + 3-4-dihydroxybenzoic acid.	Violet.
Myrtillidin ...	Phloroglucinol + 3-4-5-trihydroxybenzoic acid.	Violet.
Oenidin......	Phloroglucinol + 4-methoxy-3-5-dihydroxybenzoic acid.	No characteristic reaction.

A method for the detection of butter colors has been published recently by H. A. Lubs[1]. Comment on this and on any other new methods relating to the coal-tar food colors will be of interest to the association, and may be communicated to the referee to be quoted in his report.

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[1] *J. Ind. Eng. Chem.*, 1918, **10**: 436.

The United States Tariff Commission issued a publication in 1918[1] giving the names of the dyes, some 180 in number, manufactured in America in 1917. The list contains almost all of the older dyes named in the literature as having been used as food colors. The following are not included, although no doubt several of them are now on the American market. The numbers following the names refer to Green's tables[2]. Quinoline Yellow (667), Orange IV (88), Sudan G (10), Crystal Ponceau (64), Brilliant Yellow S (89), Ponceau 6R (108), Acid Magenta (462), Rose Bengale (Dichlor.) (518), Soluble Blue (480), and Light Green S. F. Yellowish (435).

No collaborative work on the natural coloring matters was reported. Acetophenone, the more available of the two special reagents employed by Willstaetter, is at present quite expensive and probably will be used to no great extent in the analysis of food colors until it is cheaper. The properties of these coloring substances are described in convenient form in the handbook by Perkin and Everest[3] recently issued.

No very convenient methods for the separation or differentiation of Yellow A. B. and Yellow O. B. (in admixture with each other) have been published. It is believed that some attention should be given to this point by the association.

REPORT ON METALS IN FOODS.

By W. D. Collins[4] (Bureau of Chemistry, Washington, D. C.), *Referee.*

Collaborative work in past years has indicated that satisfactory results can be obtained for arsenic in many food products by the use of the Gutzeit method[5].

The referee's report for 1916[6] describes a number of changes in details of the method. It is the opinion of the present referee, and of a number of workers who have had wide experience in the use of this method, that some of the changes suggested are not helpful.

This situation brings up a matter which the present referee has desired to bring to the attention of the association for a number of years. It is the opinion of the referee that the essential features of the Gutzeit method should be adopted as official by the association. It is believed that some of the minor details might be left to the discretion and convenience of individual analysts. The essential features of the method may be summarized as follows:

The standard stains and the stains obtained from samples must be prepared under absolutely identical conditions. This includes tempera-

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[1] U. S. Tariff Com. Bull. 6: (1918).

[2] Arthur Green. A Systematic Survey of the Organic Coloring Matters. 1904. Based on the German of Schultz and Julius.

[3] A. G. Perkin and A. E. Everest. The Natural Organic Coloring Matters. Longmans, Green and Co., 1918.

[4] Present address, Geological Survey, Washington, D. C.

[5] *Assoc. Official Agr. Chemists, Methods*, 1916, 171.

[6] *J. Assoc. Official Agr. Chemists*, 1920, 3: 512.

ture, dimensions of the apparatus, volume of solution, and rate of evolution of hydrogen, and demands that the arsenic be reduced completely before the evolution of hydrogen is started. If these matters are taken care of, it is not important whether the generator bottle be of 1-ounce, 2-ounce, or 4-ounce capacity. Hydrochloric and sulphuric acids are equally good. Very few analysts, except the previous referee, have experienced any difficulty in using drawing paper saturated with 5 per cent mercuric bromid solution for the stains in preference to filter paper soaked in 1.5 per cent mercuric bromid. The strength of acid to be used in the generator depends upon the amount and quality of the zinc used. It is desirable to have the rate of evolution of hydrogen and arsine such that a heavy brown stain is obtained with a fairly definite upper limit.

The volumetric method for tin, substantially as published by the association[1], has been used for a number of years by laboratories making large numbers of determinations of tin in canned foods. There would seem to be no reason why this method should not be written up in form for final adoption as an official method of the association.

The gravimetric method for tin has given successful results in the hands of collaborators. This method should be written up for adoption as an official method.

The report of the referee for 1917[2] described work done on the Penniman method for tin in canned foods. In place of the digestion with nitric and sulphuric acids recommended in the tentative methods, the tin is extracted from the foods by digestion with concentrated hydrochloric acid. In place of the precipitation by hydrogen sulphid and solution of the tin sulphid for titration, the tin is precipitated as the metal by metallic zinc, and the resulting metallic precipitate is dissolved for titration. Some experiments which have been conducted with this method indicate that the tin can be extracted fairly completely from some canned foods, but that the precipitation by metallic zinc is not always complete. The study of this method will be continued.

RECOMMENDATIONS.

It is recommended—

(1) That the Gutzeit method for arsenic be described in its essential details, with explanation of the precautions necessary to obtain uniform results, with a view to its adoption in 1920 as an official method.

(2) That the volumetric method and the gravimetric method for tin be described in essential details for adoption as official methods in 1920.

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 173.

[2] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 172.

(3) That further study be made of the Penniman method for tin in canned foods, and, if studies now in progress justify further test of this method or modifications of it, collaborative work be done on the method.

(4) That methods for determining zinc, aluminium, and copper be made the subject of study as soon as possible.

No report on fruits and fruit products was made by the referee.

No report on canned vegetables was made by the referee.

A "Summary of Bureau of Chemistry Investigations of Poisoning Due to Ripe Olives"[1] by G. G. DeBord, R. B. Edmondson and Charles Thom (Bureau of Chemistry, Washington, D. C.) was presented by Charles Thom.

Messrs. S. L. Jodidi, S. C. Moulton and K. S. Markley (Bureau of Plant Industry, Washington, D. C.) presented a paper on "The Mosaic Disease of Spinach as Characterized by Its Nitrogen Constituents"[2].

No report on cereal foods was made by the referee.

PHYSICO-CHEMICAL METHODS FOR DETERMINING THE GRADE OF FLOUR[3].

By C. H. BAILEY (Agricultural Experiment Station, University Farm, St. Paul, Minn.).

The usual method of ascertaining the grade of a wheat flour is to determine its content of ash. This method is somewhat slow, requiring several hours for the complete combustion of all carbonaceous matter. It does not adapt itself well to plant operation in controlling processes, since the time elapsing between the drawing of the sample, and the returns from the laboratory is too great.

In an effort to develop a more rapid method, it was found that a close parallelism existed between the ash content and the specific conductivity of the water extract, when the latter was prepared under uniform conditions. A study was made of the effects of time and temperature as variables, and both of these factors were found to affect the results. A standard and uniform procedure was therefore adopted.

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[1] *J. Am. Med. Assoc.*, 1920, **74**: 1220.
[2] *J. Am. Chem. Soc.*, 1920, **42**: 1061.
[3] Published with the approval of the Director as Paper No. 243, journal series, Minnesota Agricultural Experiment Station.

Ten grams of flour at 25°C. were vigorously shaken with 100 cc. of conductivity water at 25°C. and the mixture placed in a thermostat at the same temperature. The flour was kept in suspension by intermittent shaking for exactly 30 minutes, and then thrown out of suspension by whirling rapidly in a centrifuge. A portion of the decantate was passed through a filter to remove floating particles, and at once placed in a conductivity cell, which was immersed in a water thermostat maintained at exactly 30°C. All operations up to this point have taken about 40 ± minutes. A few minutes are allowed for the contents of the cell to reach 30°C., the time being shortened, when desired, by occasional gentle agitation. The final readings can be made in such time that not more than 50 to 60 minutes total time is required to complete the test.

To conserve both time and material, a special Freas cell was constructed for the writer which has the bottom cut off. This is used as a dip electrode, the extract being placed in a vial of suitable dimensions. Several of these vials can be placed in the water thermostat, and the readings taken by shifting the dip electrode from vial to vial. When this plan is followed, it is deemed advisable to place portions of each extract in at least two vials. The electrode is rinsed off by dipping it into the first of the pair of vials, and the reading taken by transferring it to the second vial.

In Table 1 is shown the relation between the ash content of a series of flours, and the specific conductivity of their water extracts, prepared as described.

TABLE 1.

Ash content and specific conductance of a series of flour samples.

GRADE	ASH	SPECIFIC CONDUCTIVITY OF WATER EXTRACT
	per cent	$K_{30} \times 10^{-4}$
First middlings	0.44	5.36
Second middlings	0.45	5.49
Third middlings	0.55	6.26
Second break	0.58	6.67
Fifth middlings	0.61	6.69
Third break	0.67	7.59
Fourth middlings	1.17	10.00
First break	1.34	11.24

As one phase of a study of wheat flour grades, the hydrogen ion concentration and buffer action of their water extracts were determined. Again, it was found that time and temperature of extraction must be uniform to insure comparative results, especially in so far as the buffer action of the extracts is concerned. Increases in either time or temperature, within the limits studied, resulted in an increased buffer action.

A standard procedure was accordingly adopted, flour and water in the proportions of 1 to 5 being mixed, at a temperature of 25°C., and the flour maintained in suspension for 60 minutes. The mixture was then whirled in the centrifuge and the decantate pipetted.

It appears from the data in Table 1 that the P_H of the original extracts increases slightly with the ash content; that is, the higher grades yield an extract with a slightly higher hydrogen ion concentration than do the lower grades. The differences are hardly great enough or sufficiently uniform, to make the P_H a satisfactory index of grade. Moreover, this may change appreciably as the flour ages. The buffer action varies widely, however, and increases with the ash content. It appears that the data secured through the addition of 20 and 30 cc. of N/50 sodium hydroxid to 100 cc. of extract are of the most value in indicating the relative grade.

TABLE 2.

Hydrogen ion concentration in water extracts of flour before and after the addition of acid and alkali.

GRADE	ASH	ORIGINAL EXTRACT P_H	P_H AFTER ADDITION OF N/50 HYDROCHLORIC ACID TO 100 CC. OF EXTRACT				P_H AFTER ADDITION OF N/50 SODIUM HYDROXID TO 100 CC. OF EXTRACT			
			10 cc.	20 cc.	30 cc.	40 cc.	10 cc.	20 cc.	30 cc.	40 cc.
	per cent									
First middlings..	0.44	6.07	4.48	3.60	3.11	2.87	7.48	9.28	9.89	10.62
Second middlings	0.45	6.10	4.65	3.64	3.25	2.96	7.27	8.72	9.79	10.32
Third middlings.	0.55	6.22	4.94	3.96	3.45	3.04	7.22	8.59	10.00	10.35
Second break ...	0.58	6.25	5.00	4.11	3.45	3.15	7.12	8.52	9.53	10.21
Fifth middlings .	0.61	6.31	5.04	4.14	3.53	3.16	7.15	8.38	9.62	10.22
Third break.....	0.67	6.22	5.19	4.18	3.62	3.46	6.96	7.89	9.25	9.77
Fourth middlings	1.17	6.42	5.85	5.11	4.46	4.06	6.86	7.29	7.91	8.79
First break.....	1.34	6.34	5.86	5.15	4.56	4.04	6.77	7.17	7.66	8.59
Fourth break....	1.62	6.36	6.02	5.43	4.78	4.26	6.75	7.08	7.49	8.20

The data reported in Table 2 were obtained by the use of the hydrogen electrode. A colorimetric procedure can be evolved, however, which is useful in this connection. Since phenolsulphonephthalein (phenol red) is one of the most delicate indicators, it is recommended that the treatment be such that the resulting preparations are rendered sufficiently alkaline to have a P_H within the limits of color change of this indicator ($P_H = 6.8$ to 8.4). This can generally be brought about by the addition of 10 cc. of N/40 sodium hydroxid to each 100 cc. of the extract. The resulting P_H will be much higher in the case of patent flour extracts than in the extracts from the clears, owing to the lower buffer action of the former. The color of the preparation, after phenol red has been added, can be matched against a series of standards, and the P_H thus determined. In the case of patent flours containing less than 0.44 per cent of ash, it is necessary to use somewhat less than the quantity of alkali mentioned, otherwise the mixture will have a P_H greater than 8.4. It is suggested that with such flours 10 cc. of N/50 sodium hydroxid be added to each 100 cc. of extract, and a separate graph (or formula) be employed to compute the results into terms of ash content.

REPORT ON WINES[1].

By J. M. HUMBLE[2] (U. S. Food and Drug Inspection Station, U. S. Custom House, Cincinnati, Ohio), *Referee.*

In accordance with the recommendations of 1916[3], it was decided to confine the work to a study of the Rothenfusser method for glycerol. Due, no doubt, to war conditions and also to lack of interest in the subject coincident with prohibition, great difficulty was incurred in securing men willing to collaborate. The following collaborators were finally secured:

B. G. Hartmann, U. S. Food and Drug Inspection Station, 1625 Transportation Building, Chicago, Ill.

A. R. Todd, State Dairy and Food Commission, Lansing, Mich. (assisted by Ruth Hoare, analyst).

E. M. Meyer, City Chemist, Cincinnati, Ohio.

Wm. J. McCarthy, U. S. Food and Drug Inspection Station, 411 Government Building, Cincinnati, Ohio.

A copy of the Rothenfusser method, submitted herewith, was sent to the above collaborators; also, two synthetic solutions, Nos. 1 and 2, containing 0.50 and 0.75 gram, per 100 cc. of glycerol, respectively, and of the following composition:

Composition of solutions sent collaborators.

	SOLUTION 1*	SOLUTION 2*
	grams	*grams*
Alcohol, absolute	200†	200†
Lactic acid	8.0	8.0
Acetic acid	6.0	6.0
Malic acid	8.0	8.0
Tannic acid	2.0	2.0
Levulose	4.0	6.0
Potassium acid tartrate	8.0	8.0
Tartaric acid	2.0	2.0
Potassium acid phosphate	2.0	2.0
Glycerol	10.0	15.0

* Made up with water to a volume of 2000 cc.
† Cubic centimeters.

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[1] Presented by R. E. Doolittle.

[2] Present address, American Diamalt Co., Riverside, Cincinnati, Ohio.

[3] *J. Assoc. Official Agr. Chemists*, 1920, 3: 409.

ROTHENFUSSER METHOD FOR THE DETERMINATION OF GLYCEROL.

The method is based upon the following points:

(1) Removal of sugars and organic acids (lactic acid excepted) with basic lead acetate and strong ammonium hydroxid.

(2) Removal of lactic acid with stannous chlorid.

(3) Removal of lead and tin with sodium phosphate and potassium carbonate.

(4) Oxidation of glycerol in a sodium carbonate solution with potassium permanganate in the cold.

(5) Precipitation of calcium oxalate.

(6) Titration of oxalic acid with potassium permanganate.

Method I.

(Applicable to white and red wines containing up to 1 per cent of sugar.)

Treat 50 cc. of wine with 30 cc. of sodium carbonate (1 to 5) and 5 grams of crystalline stannous chlorid and, after mixing thoroughly, allow to stand for a few minutes. Make to 250 cc. with water. Filter through a double filter[1] and treat 200 cc. of the filtrate with a freshly prepared mixture of 40 cc. of lead subacetate (sp. gr. 1.24) and 20 cc. of 10% ammonium hydroxid. After shaking thoroughly, make up to 300 cc. with 10% ammonium hydroxid. Shake, filter[2] and to 200 cc. of the filtrate in a nickel dish (sugar dish) add 5 cc. of a 10% solution of sodium phosphate and 6 grams of pure sodium carbonate. Evaporate in a water bath to a volume of about 60 cc. Transfer the evaporated solution[3] to a flask with about 40 cc. of water, add 10 grams of potassium carbonate and, after cooling to about 25°C., oxidize in the cold with 2 grams of powdered potassium permanganate. Allow to stand in the cold for 45 minutes, shaking occasionally. Then add, with constant shaking, 3% hydrogen peroxid until the supernatant liquid is colorless[4]. Transfer to a 250 cc. flask with water, make to volume and filter[5]. Transfer 220 cc. of the filtrate (23.46 cc. of wine) to an Erlenmeyer, mix with 50 cc. of 30% acetic acid and bring to a boil. Treat the clear solution with 3 cc. of a 30% calcium chlorid solution and boil for about 5 minutes. Allow to stand for a short time, filter through a Gooch charged with asbestos and wash with water.

Dissolve the calcium oxalate on the felt in 200 cc. of boiling sulphuric acid (1 to 8), using very weak suction at first. Transfer the filtrate to an Erlenmeyer, heat to about 80°C. and titrate with potassium permanganate solution. Prepare the potassium permanganate solution by dissolving 8 grams of potassium permanganate in 2500 cc. of water. Allow to stand for 2 days and standardize with N/20 oxalic acid.

Calculation.—The glycerol is calculated according to the following formula:

$T \times N \times 4.26$ = grams of glycerol per 100 cc. in which

$\frac{100 \text{ cc.}}{23.46 \text{ cc.}^{6}} = 4.26$;

T = glycerol titer;

N = cc. of potassium permanganate solution required for oxidation.

Find T (the glycerol titer).

1 cc. of N/20 oxalic acid = 0.00315 gram of oxalic acid.

1 molecule of oxalic acid = 1 molecule of glycerol.

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[1] Disregard any turbidity which may subsequently form.

[2] To hasten filtration carefully sweep the wall of the filter with a policeman.

[3] Disregard the white precipitate formed.

[4] An excess of hydrogen peroxid is not harmful.

[5] Use a fluted paper.

[6] Amount of wine used.

Assuming that 24.8 cc. of potassium permanganate are required to oxidize 50 cc. of N/20 oxalic acid, $50 \times 0.00315 = 0.1575$.

1 cc. of potassium permanganate $= \frac{0.158}{24.8} = 0.00635$ gram of oxalic acid.

$\frac{(COOH)_2.2H_2O}{126} : \frac{C_3H_8O_3}{92} = 0.00635 : X.$

$X = 0.00464$ gram of glycerol.

$T = 0.00464.$

Assuming that it requires 31 cc. of potassium permanganate to oxidize the oxalic acid formed from 23.46 cc. of wine, then

$T = 0.00464.$

$N = 31.$

$\frac{100 \text{ cc.}}{23.46 \text{ cc.}}$[1] $= 4.26.$

$0.00464 \times 31 \times 4.26 = 0.61$ gram per 100 cc.

Method II.

(Applicable to sweet wines containing from 1 to 6 per cent of sugar.)

Treat 50 cc. of wine with 15 cc. of sodium carbonate solution (1 to 5), add 2.5 grams of stannous chlorid, and, after shaking, allow to stand for about 5 minutes. Make to 250 cc. with water and filter. To 220 cc. of the filtrate add 30 cc. of 5% ammonium hydroxid and 200 cc. of the lead subacetate ammonia mixture. The lead should be in excess. Filter a small portion of the mixture and test the filtrate with a drop of ammonium sulphid. Add more lead ammonia mixture, if necessary, and make to 500 cc. with 10% ammonium hydroxid. Filter, and to 250 cc. of the filtrate in the nickel dish add 5 cc. of 10% sodium phosphate and 6 grams of sodium carbonate. Evaporate on the water bath and proceed as in Method I.

Per cent of glycerol $= T \times N \times 5.16.$

Method III.

(Applicable to very sweet wines.)

Treat 50 cc. of wine with 15 cc. of sodium carbonate (1 to 5) and 2.5 grams of stannous chlorid and allow to stand for 5 minutes. Add 100 cc. of water, heat to 50°C. on a water bath, make to 250 cc., and filter. To 220 cc. of the filtrate add 50 cc. of 5% ammonia and a freshly prepared lead ammonia mixture (180 cc. of lead plus 70 cc. of 10% ammonia). Determine whether an excess of lead is present. Add 20 cc. of 10% ammonium carbonate solution, mix thoroughly, make to 600 cc. with 10% ammonium hydroxid and filter. To 300 cc. of the filtrate (22 cc. of wine) in the nickel dish add 10 cc. of sodium phosphate and 6 grams of sodium carbonate and evaporate to about 80 cc. on a water bath. Wash into a flask with about 20 cc. of water. Cool to 25 cc., add 10 grams of potassium carbonate and 1 gram of potassium permanganate. Allow to stand for 30 minutes, shaking occasionally, and proceed as in Method II.

Method IV.

(Applicable to excessively sweet wines.)

Dilute to a sugar content of about 20% (grams per 100 cc.) and proceed as in Method III.

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[1] Amount of wine used.

Method V.

(Applicable to wines containing sucrose.)

Wines containing sucrose should be inverted before attempting the determination of glycerol by one of the above methods. Neutralize before proceeding with the inverted solution.

Instructions were to follow the method for white and red wines containing up to 1 per cent of sugar, for the analyses of Solution No. 1, and to follow the method for sweet wines containing 1 to 6 per cent of sugar for analyses of Solution No. 2.

Results obtained by each collaborator, as well as by the referee, were so erratic as to hardly warrant a detailed report.

On Solution No. 1, containing 0.50 gram per 100 cc. of glycerol, analyses show a general tendency to give high results as follows: 0.74, 0.75, 0.85, 0.86, 1.26, 1.21 and 1.26, grams per 100 cc. No particular comment was made by the collaborators on Method I. The details of the method work out nicely and can be carried through in a comparatively short time, but the results obtained are entirely too high and show a wide variation.

With respect to Solution No. 2, containing 0.75 gram per 100 cc. of glycerol, and 3 per cent of sugar, on which Method II was followed, the collaborators seem to have experienced more or less difficulty. Hartmann reports "Precipitates so bulky, not considered worth while to carry analysis to completion".

E. M. Meyer "Could not secure results that would check".

The referee experienced much the same difficulty as Hartmann with respect to bulky precipitates, but finally overcame this by using the centrifuge. However, the results obtained were not consistent.

Analytical results on this sample show an even wider variation than on Solution No. 1, as follows: Glycerol present, 0.75 gram per 100 cc.; glycerol reported varied from 0.29 to 1.78 grams per 100 cc.

It would seem that the results obtained by the use of this method on wines are inaccurate. In this connection, it is interesting to note the comment of John R. Eoff, Fermentation Chemist, with William F. Jobbins, Inc., Aurora, Ill., "My experience with the Rothenfusser method does not inspire me to recommend it for anything short of fairly pure glycerol solutions". However, not enough work has been done to determine where the trouble is.

With the advent of prohibition, the question of wines becomes of rather minor importance, but it may be found that this method can be adapted to the determination of glycerol in grape juice and vinegar. The short time in which a determination can be made by this method would be a decided advantage over the present method for determining glycerol in vinegar. This, I feel, is the chief reason for recommending a further study of the Rothenfusser method.

RECOMMENDATIONS.

It is recommended—

(1) That the subject of wines be dropped from the association work on account of prohibition.

(2) That an additional study of the Rothenfusser method be made upon vinegars or grape juice.

No report on soft drinks was made by the referee.

The honorary president, H. W. Wiley, presented an address on "Problems of Today and Their Bearing Upon the Scientific World".

The meeting adjourned at 12.45 p. m. to reconvene at 2 p. m.

SECOND DAY.

TUESDAY—AFTERNOON SESSION.

REPORT ON DISTILLED LIQUORS.

By J. I. PALMORE (Bureau of Chemistry, Washington, D. C.), *Referee.*

The experiment concerning a good method for storing aldehyde-free alcohol was continued through the years 1917, 1918 and into 1919.

On May 9, 1917, two portions of freshly prepared aldehyde-free alcohol, one a 95 per cent solution (Solution 1), and the other a 50 per cent solution (Solution 2), were chilled and carbon dioxid gas from a Kipp's generator was passed into the solutions to supersaturation. The containers were sealed with paraffin and stored in a refrigerator. On May 9, 1917, before supersaturating with carbon dioxid gas, the solutions were tested for aldehyde with a sulphite-fuchsin solution of 7 days' standing, prepared as given in the directions for the analysis of distilled liquors[1]. There was a slight coloration.

Solution 1 was stored in a 2½ liter glass-stoppered bottle about three-fourths full; Solution 2 was likewise stored and the bottle was about seven-eighths full. On September 17, 1917, both solutions were tested, as in the method for the analysis of distilled liquors[1], with a sulphite-fuchsin solution, 2 days old, prepared as above. Solution 1 gave a very slight coloration, about the same as at the beginning of the experiment. Solution 2 gave a color about one-half as intense as Solution 1, both negligible. Solutions 1 and 2 were supersaturated with carbon dioxid gas immediately, the stoppers reparaffined and the containers stored in a refrigerator.

On January 31, 1918, both solutions were treated with a sulphite-fuchsin solution of 7 days' standing. Solution 1 was diluted to 50 per cent, as before, when the test was made. A very slight coloration was noticed, showing a trace of aldehyde and about the same intensity as in Solution 2, the test being made on the latter directly, *i. e.*, without dilution.

On October 24, 1919, both solutions were tested for aldehyde. Solution 1 was diluted to 50 per cent before applying the test. This test showed only a trace of aldehyde, Solution 1 being about one-half as intense as Solution 2. However, Solution 2 could be used satisfactorily as a reagent in the determination of aldehyde[1]. In this test the sulphite-

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[1] *Assoc. Official Agr. Chemists, Methods,* 1916, 244.

fuchsin solution was 3½ days old and prepared as mentioned before. Attention is called to the length of time Solutions 1 and 2 have been standing, approximately 2½ years, without any material change in the aldehyde content.

In conclusion, the referee feels justified in stating that an alcohol once freed of aldehyde[1] may be kept in a dark, cool place—a refrigerator or cold storage room—after supersaturating with carbon dioxid gas and sealing the stoppers of the containers with paraffin to avoid diffusion, for at least 2½ years and probably for an indefinite time. As a result of this experiment it may be stated that 95 per cent alcohol keeps freer of aldehydes than a 50 per cent alcohol, both stored under the conditions just mentioned.

No referee on beers was appointed and no report on this subject was presented.

REPORT ON VINEGARS.

By W. A. BENDER[2] (Douglas Packing Company, Rochester, N. Y.), *Referee.*

Some work was undertaken to find a modification of the present method for glycerol in vinegar, which, it is well known, is tedious and time-consuming. V. B. Bonney (U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, San Francisco, Calif.) cooperated with the referee in this work and also performed the analytical work.

DETERMINATION OF GLYCEROL IN CIDER VINEGAR.

By W. A. BENDER and V. B. BONNEY.

The object was to eliminate the extraction with hot 90 per cent alcohol, subsequent evaporation, transfer, and extraction with absolute alcohol and ether. It was hoped to combine all these steps in one. In each case, after securing the aliquot of glycerol solution, the tentative method[3] was followed from the point where evaporation of the absolute alcohol-ether mixture commences.

Method I.

One hundred and twenty-five cc. of vinegar were evaporated to about 5 cc., then rubbed thoroughly with 25 grams of freshly burnt lime. The resulting mixture was extracted with 100 cc. of a mixture, 2 parts absolute alcohol, 3 parts absolute ether, and a 60 cc. aliquot of this used for the determination of glycerol. The method was not satisfactory, as only about one-third of the glycerol was found, judging from determinations by the tentative method[3].

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 244.
[2] Presented by R. W. Balcom.
[3] *Assoc. Official Agr. Chemists, Methods*, 1916, 253.

Method II.

One hundred and twenty-five cc. of vinegar were evaporated to 5 cc.; this was mixed with 20 cc. of absolute alcohol, then some freshly burnt lime and sand added and the mixture well ground. Large lumps of gummy material formed, so that the method was discarded.

Method III.

One hundred and twenty-five cc. of vinegar were evaporated to 5 cc., some lime added and allowed to stand 15–30 minutes. Some sand was added and the mass ground with a pestle. Twenty cc. of absolute alcohol were added and all ground to a homogeneous mixture. The mass was transferred to a glass-stoppered Erlenmeyer flask, using 20 cc. more of absolute alcohol. Sixty cc. of absolute ether were added and the mixture allowed to stand for 3 hours with occasional shaking. Glycerol was determined in 70 cc. of the clear liquid. Only about one-third of the glycerol, as determined by the tentative method[1], was found.

Method IV.

One hundred and twenty-five cc. of vinegar were evaporated to about 5 cc., 10 cc. acetone, 17 cc. absolute alcohol, and 20 grams of lime added and ground to a homogeneous paste. The mass was transferred to a milk bottle with 23 cc. of absolute alcohol and 60 cc. of absolute ether. The material was centrifugalized for 5 minutes and glycerol determined in an aliquot of the clear liquid. About two-thirds of the glycerol present were found.

Method V.

One hundred and twenty-five cc. of vinegar were evaporated to 5 cc. This was mixed with 5 cc. of water and 10 cc. of absolute alcohol. The dish was cooled in ice and about 35 grams of lime added in small quantities with stirring. The mass was allowed to stand 10–15 minutes, then ground with a pestle and transferred to a nursing bottle containing 50 cc. of absolute alcohol. The dish was rinsed with 5 grams of lime which was transferred to the bottle. The mass was extracted with absolute alcohol, with the aid of the centrifuge, but the method did not seem to be satisfactory, as the mass did not cake well and some lime remained with the alcohol on decanting. No determination was made of the amount of glycerol recovered.

CONCLUSIONS.

The results of the work are entirely negative but it is hoped that they may serve as a basis for further work and that they may suggest some feasible modification of the present complicated procedure.

RECOMMENDATIONS.

It is recommended—

(1) That the following tentative methods[1] be made official:

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 253.

1, PHYSICAL EXAMINATION.
2, PREPARATION OF SAMPLE.
3, SPECIFIC GRAVITY.
4, ALCOHOL.
8, TOTAL REDUCING SUBSTANCES BEFORE INVERSION.
9, REDUCING SUGARS BEFORE INVERSION AFTER EVAPORATION.
10, REDUCING SUGARS AFTER INVERSION.
13, ASH.
14, SOLUBLE AND INSOLUBLE ASH.
15, ALKALINITY OF THE SOLUBLE ASH.
16, SOLUBLE AND INSOLUBLE PHOSPHORIC ACID.
17, TOTAL ACIDS.
23, PENTOSANS.

(2) That the incoming referee select the more important of the remaining methods[1] for further critical work with a view to improvement, namely:

5, GLYCEROL.
7, SOLIDS.
18, FIXED ACIDS.

REPORT ON FLAVORING EXTRACTS.

By A. E. PAUL[2] (U. S. Food and Drug Inspection Station, Transportation Building, Chicago, Ill.), *Referee.*

VANILLA EXTRACT.

Several years ago the question was raised whether the association methods for vanilla extract might not advantageously be shortened materially. There was at that time submitted for study a qualitative test for coumarin, devised by H. J. Wichmann (U. S. Food and Drug Inspection Station, Tabor Opera House Building, Denver, Colo.). In case a considerable number of samples were presented for analysis, the plan was to apply the qualitative coumarin test to the entire series, and then proceed with the complete official method, where coumarin was found to be present. In the cases in which no coumarin was found, the total ether extract would be weighed, the vanillin driven off, and the residue weighed. The difference would represent vanillin. The question of the desirability of officially introducing this preliminary examination was submitted to the collaborators, and a vote was taken, which was unanimously against the plan. The verdict may have been, in part, at least, due to the lack of definiteness in the description of the details of the method, which, however, has been amended and improved by H. J. Wichmann and J. R. Dean so as to be more readily applied.

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 253.
[2] Presented by R. W. Balcom.

A colorimetric method for vanillin, devised by Otto Folin, was also submitted. The results reported by the various collaborators were most remarkably satisfactory. The method was not followed up, however, because the various determinations, according to the association method, are made on one sample, and the vanillin determination is merely a step in the process.

Recently, Wichmann submitted a revised method for the lead number determinations. It is known that the official procedure is slow and, furthermore, does not represent a complete lead precipitation. As a matter of fact, the slow precipitation is arbitrarily interrupted at the end of a given period. It seems that Wichmann's method probably more nearly represents a completed action by the lead acetate. It consists essentially in distilling off the alcohol, precipitating hot with lead acetate, and filtering.

It seems that the above suggestions may be combined into a process which would be more expeditious than the present official method. Of course, it will be remembered that a considerable number of authentic samples have been examined by the present official methods, and the results are of great value in interpreting results of analyses. It would, therefore, be undesirable to replace or change the official methods until similar data have been accumulated by any contemplated new or modified method. It was, however, appreciated that there might be an advantage in the use of a rapid method for "sorting out" the legal from the illegal samples, with the idea of checking the latter by the official procedure.

Two samples of vanilla extract were submitted to collaborators with the request that the same be examined by the methods above referred to, as well as by those now official. It was requested that, in addition to the results, an opinion be included as to the desirability of including the methods among those officially or tentatively adopted by the association.

The methods are:

WICHMANN AND DEAN'S QUALITATIVE METHOD FOR COUMARIN[1].

FOLIN'S COLORIMETRIC VANILLIN METHOD[2].

WICHMANN'S MODIFIED LEAD NUMBER METHOD.

To 50 cc. of extract in a liter flask, add 175 cc. of boiled carbon dioxid-free water, and 25 cc. of standard 8% neutral lead acetate solution. Distil over a medium flame into a 200 cc. distillation flask. Calculate the approximate alcohol content from the sp. gr. of the distillate. (For a more accurate determination, render the distillate alkaline and redistil to 100 cc.) Transfer the residue to a 100 cc. volumetric flask with carbon dioxid-free water. When cool, fill to the mark and filter.

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[1] *J. Ind. Eng. Chem.*, 1918, **10**: 536.

[2] *Ibid.*, 1912, **4**: 670.

To a 10 cc. aliquot of the filtrate add 25 cc. of water and 0.5–1.0 cc. of sulphuric acid. Stir thoroughly, add 100 cc. of 95% alcohol and again stir thoroughly. When settled clear, filter on a Gooch crucible, wash with 95% alcohol, dry at a moderate heat, ignite at low redness for 3 minutes, avoiding the reducing flame, and weigh.

Conduct a blank determination, employing water containing 4–5 drops of acetic acid in place of the sample. Calculate the number of grams of metallic lead precipitated by 100 cc. of the sample.

VANILLIN AND COUMARIN[1].

NORMAL LEAD NUMBER[2].

It will be noted that the Wichmann procedure would practically eliminate the color values, which are at present tentative. However, they have not proved to be of as much value as was originally expected. Moreover, they require the expensive Lovibond colorimeter and slides. At all events, the modified Marsh test, at present a tentative method, is far more valuable and would, of course, not be interfered with by the above-described changes.

It is claimed that the filtration of the lead precipitate is much less troublesome in the Wichmann process than in the official method, and, further, there is no necessity for obtaining any considerable amount of the filtrate, 10 cc. only being used.

The two samples submitted were the same true U. S. P. extract of vanilla, with the exception that No. 2 contained 0.02 per cent of added coumarin. The reports on these samples are given in Table 1.

TABLE 1.

Efficacy of various tests for vanilla extract.

COLLABORATOR	ALCOHOL OFFICIAL*	VANILLIN		COUMARIN		LEAD NUMBER	
		Official†	Folin	Official†	Wichmann	Official‡	Wichmann
Sample No. 1:	*per cent*	*per cent*	*per cent*	*per cent*			
F. W. Boyles, McCormick & Co., Inc., Baltimore, Md.	44.4	0.17	0.22	....	None	0.46	0.85
H. J. Wichmann.......	....	0.20	....	....	None	0.57	0.76
E. H. Berry, U. S. Food and Drug Inspection Station, 1625 Transportation Building, Chicago, Ill.	44.6	0.18	0.21	....	None	0.55	0.81
Sample No. 2:							
F. W. Boyles..........	43.3	0.16	0.22	0.03	Present	0.46	0.78
H. J. Wichmann.......	....	....	....	....	Present	....	....
E. H. Berry...........	45.2	0.16	0.20	0.03	Present	0.50	0.76

* *Assoc. Official Agr. Chemists, Methods*, 1916, 259.
† *Ibid.*; *J. Assoc. Official Agr. Chemists*, 1920, **4**: 265.
‡ *Assoc. Official Agr. Chemists, Methods*, 1916, 260; *J. Assoc. Official Agr. Chemists*, 1920, **4**: 265.

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 259; *J. Assoc. Official Agr. Chemists*, 1920, **4**: 265.
[2] *Ibid.*, 260; *Ibid.*

COMMENTS BY COLLABORATORS.

F. W. Boyles.—The Wichmann qualitative method for coumarin is very satisfactory. The Wichmann lead number method is superior to the official method and the Folin method is preferred to the official method. These methods should be included among the official methods.

H. J. Wichmann.—The lead number of the new method is about one-third higher than by the official method.

E. H. Berry.—The Wichmann qualitative method is very good; the Folin method for vanillin is accurate and short; and the Wichmann lead number appears to be more satisfactory than the official method. However, since there is no satisfactory, quick quantitative method for coumarin and the new lead number method can not well be used until some determinations have been made upon authentic vanilla extracts using this new method, it is not believed advisable that these methods be made official.

The results submitted are rather discordant. They indicate, however, that Wichmann's qualitative test for coumarin is quite valuable. It, however, is merely qualitative, and it would therefore seem that the official procedure must in any event be retained, unless Wichmann's method can be made quantitative or until some other rapid quantitative method is devised. As a matter of fact, there are enough qualitative tests for coumarin. As to the lead number, it would seem that at present it would be of very little use, if any, since results can not be interpreted in view of the absence of available results on authentic samples. Folin's vanillin method also, it would seem, would be of no use, until short methods for all the principal determinations have been devised.

While it is believed that the official methods should include none of the methods which have been proposed, it is expected that a satisfactory, simple quantitative coumarin method will be devised. At such time a large number of authentic samples should be examined by these methods, and it may then be a distinct advantage to substitute them for the present methods. It is hoped that the author of the new lead number procedure will then submit, together with his method, a sufficient number of results on authentic samples to serve as information, permitting satisfactory interpretation of results obtained on suspected samples. It is, however, the idea of the referee, that any modified lead number be designated by some entirely different term, and preferably calculated to some other basis than metallic lead. For example, if it should be found that the precipitate is due largely to organic acids, the result might be called "organic acid precipitate" and be calculated to some characteristic acid. All confusion with the Winton lead number would thus be avoided.

VANILLA SUBSTITUTES.

The food analyst is occasionally called upon to examine various vanilla preparations which are highly concentrated. They are usually thick and heavy, and contain relatively high percentages of vanillin.

These are offered for sale under various names, such as "Oleoresin", "Concentrate", etc. In many instances they are imitations entirely. These manifestly can not be satisfactorily subjected directly to the present official methods. They may be diluted and then examined as an ordinary extract, in which case the results will be accurate, with the exception of the lead number. This does not vary directly as the dilution. The questions arise: Should the lead number be determined on the dilution and then merely reported as, for example, "lead number of 20 per cent dilution"; or should it be calculated to the original substance and reported as, "lead number, calculated from 20 per cent solution"? The report should indicate also that the results are in terms of percentages, rather than grams per 100 cc. But the question also arises as to whether the lead number is a useful determination in this class of products. No definite method of manufacture of these preparations from the beans is recognized, nor is there at present available any satisfactory information as to the results to be expected from such true vanilla preparations. Collaborators were requested to give this matter all possible consideration and to submit full information concerning the manufacture and composition, together with suggestions as to the most desirable manner of examining this class of products.

The only collaborator who touched upon this question, F. M. Boyles, stated that "If they are diluted with alcohol of the same strength as that used in their preparation, the lead number would be in proportion to the dilution." According to the experience of the referee, this, however, is not the case. It is regrettable that it was found impossible to continue the work along this line, which was barely commenced in the Chicago Food and Drug Inspection Station.

It is urged that this matter, also, be studied at a later time.

LEMON AND ORANGE EXTRACTS.

The official alcohol determination in these extracts has been questioned by Julius Hortvet (State Dairy and Food Commission, St. Paul, Minn.), who calls attention to the procedure devised by Hortvet and West[1].

A sample each of lemon and orange extract, prepared from 5 per cent by volume of the respective oils, in alcohol of 90.78 per cent strength by volume, was submitted. They, therefore, contained 86.24 per cent of alcohol by volume. These were submitted to the collaborators with the request that the alcohol in each be determined by the official method and by that of Hortvet and West. Reports on this work, received from C. L. Black (U. S. Food and Drug Inspection Station, U. S. Appraiser's

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[1] *J. Ind. Eng. Chem.*, 1909, **1**: 84.

Stores, Philadelphia, Pa.), W. W. Randall (State Department of Health, Baltimore, Md.), and E. H. Berry, are given in Table 2.

TABLE 2.

Alcohol in lemon and orange extracts.

COLLABORATOR	LEMON EXTRACT		ORANGE EXTRACT	
	Official*	Hortvet and West	Official*	Hortvet and West
	per cent	*per cent*	*per cent*	*per cent*
C. L. Black	86.44	....	86.45	....
W. W. Randall	84.36	86.47–86.60	85.33	86.24–86.58
E. H. Berry	85.75	86.52	85.20	86.33

* *Assoc. Official Agr. Chemists, Methods*, 1916, 262; *J. Assoc. Official Agr. Chemists*, 1920, 4: 265.

Black was unavoidably prevented from making the determination by the Hortvet and West method. Therefore, the only collaborators were Randall and Berry, whose results by that method are quite satisfactory; in fact more so than their results by the official method.

It would seem that for an extract consisting only of oil, alcohol and water, the calculated result by Hortvet and West must be accurate to the extent that the determination of oil and specific gravity are accurate. The method commends itself because of its simplicity, and, it is believed, may well be included officially, for use in connection with extracts consisting only of oil, alcohol and water.

In connection with his report on the alcohol, Randall calls attention to his method for determining oil in these extracts[1]. His method consists essentially of the use of a small measured quantity of gasoline in connection with the precipitation method. The gasoline seems to collect the lemon or orange oil. The column of oil is read and correction made for the gasoline used. In reporting his results, Randall included the determination of oil by the polarization method, the precipitation method, and the gasoline method. The results are given in Table 3.

TABLE 3.

Oil in lemon and orange extracts.

METHOD	LEMON OIL	ORANGE OIL
	per cent	*per cent*
Precipitation	5.255	5.47
Polarization	5.037	5.046
Gasoline	5.08	5.043

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[1] *J. Ind. Eng. Chem.*, 1914, 6: 926.

As the results by the gasoline method are remarkably close to the truth, this method seems to merit further attention and study.

LEMON AND ORANGE OILS.

ALBRIGHT'S DETAILS FOR THE KLEBER METHOD FOR THE DETERMINATION OF CITRAL[1].

The collaborators who studied this question, F. M. Boyles, E. H. Berry and W. W. Randall, sent in the reports shown in Table 4.

TABLE 4.

Citral by weight in lemon and orange oils.

METHOD	BOYLES	BERRY	RANDALL
	per cent	*per cent*	*per cent*
Official*	3.68	3.44	3.60
	....	3.54	3.62
Albright's details	3.68	3.59	3.70
	....	3.46	3.61

* *Assoc. Official Agr. Chemists, Methods*, 1916, 265; *J. Assoc. Official Agr. Chemists*, 1920, **4**: 266.

COMMENTS BY COLLABORATORS.

E. H. Berry.—Albright's details work very well. However, I prefer to follow the second set of details given in the official Kleber method. Albright's details would no doubt be satisfactory in the hands of an analyst who has had considerable experience with it, but I believe that the analyst who makes the determination infrequently would get much better results with the official details.

W. W. Randall.—No clear advantage was to be noted in our work through the employment of the details of the method suggested by Albright. We must be frank to acknowledge, however, that this may have been due to lack of experience.

F. M. Boyles.—We do not find Albright's details of any help. The method is better in the description than in practice.

It is believed that no advantage could result from a change of the official method in accordance with the proposed modification.

It was suggested to collaborators that Mitchell's polarization method for the determination of oil in lemon and orange extracts[2] be studied, with especial reference to the natural variations in oils, and to the influence of dilution on the polarization of the resulting solutions. It was believed that conditions were, at the time, unfavorable for this investigation, because of the virtual interruption of importations of these oils. It was hoped, however, that those collaborators who are in a position to secure any authentic samples would make the investigations indicated.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **3**: 417.

[2] *Assoc. Official Agr. Chemists, Methods*, 1916, 262; *J. Assoc. Official Agr. Chemists*, 1920, **4**: 265.

A number of such samples, furnished by the New York Food and Drug Inspection Station, were available at the Chicago Food and Drug Inspection Station and several others were secured from importations entered through the Port of Chicago. All were of Italian origin. They were investigated by C. B. Gnadinger, formerly of the Chicago Food and Drug Inspection Station, in the manner indicated. Gnadinger's results are shown in Tables 5, 6, 7 and 8.

TABLE 5.

Polarization of 10 per cent solution of lemon oil in various solvents.

SOLVENT	ANGULAR ROTATION, 100 MM. TUBE, 28°C. (CALCULATED TO ORIGINAL OIL)
	degrees
Methyl salicylate	61.8
Benzaldehyde	60.3
Kerosene	59.9
Ethyl benzoate	59.8
Phenol	59.7
Liquid paraffin	59.6
None (original oil undiluted)	59.4
Benzol	58.9
Toluol	58.9
Chloroform	57.3
Petroleum ether	56.8
Acetic acid	56.7
Ethyl acetate	56.0
Carbon tetrachlorid	56.0
Acetone	55.8
Paraldehyde	55.8
Ethyl alcohol (99.5%)	55.7
Ethyl alcohol (95%)	55.5
Methyl alcohol	54.6

TABLE 6.

Rotation of varying amounts of lemon and orange oils in alcohol and kerosene solutions (100 mm. tube, 20°C.).

SOLUTION POLARIZED	LEMON OILS						ORANGE OIL		
	Sample No. 1			Sample No. 2			Sample No. 15		
	No solvent	Alcohol (99.5%)	Kero-sene*	No solvent	Alcohol (99.5%)	Kero-sene*	No solvent	Alcohol (99.5%)	Kero-sene*
grams of oil per 100 cc.	*degrees*	*degrees*	*degrees*	*degrees*	*degrees*	*degrees*	*degrees*	*degrees*	*degrees*
100	59.0	....	....	58.2	....	....	97.4	....	....
50	....	57.3	58.9	....	56.6	57.9	....	95.6	97.6
25	....	56.2	59.0	....	55.6	57.9	....	93.3	97.5
12.5	....	55.9	59.0	....	54.9	58.0	....	92.2	97.4
6.25	....	55.9	58.9	....	54.2	58.3	....	91.5	97.6
3.125	....	55.9	....	....	54.2	....	....	90.3	....

* Kerosene, 95 parts; absolute alcohol, 5 parts.

TABLE 7.

Rotation of lemon and orange oils in kerosene solution.*

SAMPLE NUMBER	OIL IN SOLUTION POLARIZED	POLARIZATION, AT 20°C., CALCULATED TO UNDILUTED OIL, 100 MM. TUBE	POLARIZATION OF UNDILUTED OIL, AT 20°C., 100 MM. TUBE
	per cent by volume	*degrees*	*degrees*
LEMON OIL:			
1	10	59.0	59.0
2	10	57.9	58.2
3	10	60.7	60.6
4	25	60.6	60.3
5	25	59.1	59.3
6	25	57.6	57.7
7	10	62.1	62.1
8	10	60.7	61.0
9	10	62.3	61.7
10	10	62.1	62.0
11	10	61.6	61.4
12	10	62.1	62.4
13	10	61.0	60.9
14	10	59.6	59.2
ORANGE OIL:			
15	10	97.6	97.4
16	10	97.6	97.8
17	10	98.3	98.2

* Kerosene, 95 parts; absolute alcohol, 5 parts.

TABLE 8.

Polarization of 5 per cent (by volume) solution of lemon and orange oils in 95 per cent ethyl alcohol.

SAMPLE NUMBER	POLARIZATION, 20°C., CALCULATED TO UNDILUTED OIL, 100 MM. TUBE	POLARIZATION OF UNDILUTED OIL, 20°C., 100 MM. TUBE	DIFFERENCE BETWEEN TRUE AND CALCULATED POLARIZATIONS	POLARIZATION OF 5 % SOLUTION, 20°C., 200 MM. TUBE	OIL CALCULATED FROM MITCHELL'S FACTORS*
	degrees	*degrees*	*degrees*	°V.	*per cent*
LEMON OIL:					
1	54.8	59.0	4.2	15.8	4.94
2	53.8	58.2	4.4	15.5	4.84
3	56.2	60.6	4.4	16.2	5.06
4	56.2	60.3	4.1	16.2	5.06
5	55.1	59.3	4.2	15.9	4.97
6	53.8	57.7	3.9	15.5	4.84
7	58.3	62.1	3.8	16.8	5.25
8	57.9	61.0	3.1	16.7	5.22
9	57.2	61.7	4.5	16.5	5.19
10	58.3	62.0	3.7	16.8	5.25
11	56.2	61.4	5.2	16.2	5.06
12	57.9	62.4	4.5	16.7	5.22
13	56.9	60.9	4.0	16.4	5.12
14	55.8	59.2	3.4	16.1	5.03
ORANGE OIL:					
15	91.2	97.4	6.2	26.3	5.05
16	91.9	97.8	5.9	26.5	5.09
17	91.6	98.2	6.6	26.4	5.08

* 3.2 for lemon; 5.2 for orange.

Table 5 gives the polarizations of a 10 per cent solution of a lemon oil in various solvents. The difference between the maximum and minimum readings is 7.2°. The results obtained by polarizing solutions of different concentrations are compared with the readings of the undiluted oils in Table 6. Polarizations of 10 and 25 per cent solutions of 17 oils in kerosene are compared with those of the undiluted oils in Table 7. Polarizations of 5 per cent solutions of the same oils in alcohol are given in Table 8. The difference between the true polarization and that obtained on the solution varies from 3.1° to 5.2° for lemon oil and from 5.9° to 6.0° for orange oil. The limits given in the United States Pharmacopœia for optical rotation of lemon oil are 57° to 64° in a 100 mm. tube at 25°C., equivalent to 57.7° and 64.7° at 20°C., using the temperature correction of Gildemeister and Hoffman. Assuming that an oil having a rotation of 57.7° showed a decrease of 4.1° (the average found in Table 8) on diluting with alcohol to form a 5 per cent by volume solution, only 4.8 per cent of oil would be found by the polariscopic method, using the factor 3.2. Similarly, the application of the polariscopic method to a 5 per cent by volume solution of an oil having the maximum polarization (64.7° at 20°C.) would indicate the presence of 5.5 per cent of oil. An orange oil having the minimum rotation given in the United States Pharmacopœia and showing a decrease of 6.2° on dilution would be found to contain 4.9 per cent of oil by the polariscopic method, using the factor 5.2.

CONCLUSIONS.

The specific rotation of lemon and orange oils varies with the solvent employed, kerosene solutions giving values which agree well with the true polarization. Concentrated solutions of the oils in alcohol give results nearer the true polarization than those obtained on dilute solutions. Solutions in kerosene containing 5 per cent of absolute alcohol give accurate results, regardless of the concentration. The polariscopic method for determining lemon oil in extracts gives results which may vary from −4 to +10 per cent. The average error on 14 samples of lemon extract examined was +1.2 per cent; the maximum negative error, −3.2 per cent; and the maximum positive error, +5 per cent. Three orange extracts gave an average error of +1.2 per cent.

Table 5 shows the tremendous effect which different solvents exert upon the polarization of these oils.

Table 8, last column, shows that the factors now in use in connection with the Mitchell polarization method are well chosen, and yield results within about 0.2 per cent of the truth for the oils examined.

GINGER EXTRACT.

In connection with the question of alcohol determination in extracts, a method for alcohol in ginger extract was last year submitted by F. M. Boyles. A sample was sent to those who volunteered to collaborate on the subject of flavoring extracts, for the purpose of trying both Boyles' method and the tentative method of the association. Boyles' method is as follows:

> To 25 cc. of the ginger extract add 50 cc. of water, saturate with salt, and shake out with 75 cc. of petroleum ether. Let stand 10 minutes, draw off the lower layer into a 200 cc. flask. Wash the petroleum ether with 50 cc. of saturated salt solution. This washing must be done carefully, with moderate shaking, to avoid the formation of an emulsion. A slight emulsion at this point may be broken up by pouring back and forth into two separators. Add this wash water to the 200 cc. flask, and make up to the mark with salt solution. Filter through a rapidly acting folded filter, and determine the alcohol in 100 cc. of the filtrate by distillation.

Subsequently, Boyles mentioned the objectionable feature of his method, which is that the result obtained in the analytical process must be multiplied by 8. To overcome this he modified his method as follows:

> To 30 cc. of ginger extract in a separatory funnel, add 60 cc. of a 15% salt solution, shake, add 60 cc. of petroleum ether, and let stand 20 minutes. Pipette 75 cc. of the lower layer into a distillation flask, add about 5 cc. of 10% sodium hydroxid and about 10 grams of sodium chlorid, and distil to 50 cc. The percentage of alcohol indicated by the sp. gr. of the distillate is multiplied by 2.

The extract was made from 20 per cent of ground ginger and alcohol of 95.6 per cent strength by volume. It would therefore seem that the correct result would be from 93.4 to 94.0 per cent by volume. The results reported appear in Table 9.

TABLE 9.

Alcohol (by volume) in ginger extract.

COLLABORATOR	BOYLES' ORIGINAL METHOD	BOYLES' MODIFIED METHOD	TENTATIVE METHOD*
	per cent	*per cent*	*per cent*
C. L. Black	91.12	....	93.44
F. M. Boyles	91.36	....	93.4
W. W. Randall	91.06 92.14	92.30 92.46	92.77 92.07 (92.87)
Leonard Feldstein, U. S. Food and Drug Inspection Station, Tabor Opera House Building, Denver, Colo.	92.40	....	93.36
E. H. Berry	88.96 89.76	93.40 93.96	92.80 92.72

* *Assoc. Official Agr. Chemists, Methods,* 1916, 267.

From the figures in Table 9 it would appear that the results reported by the official method are preferable to those obtained by Boyles' method.

It would seem that neither of the methods suggested by Boyles is free from serious objection. The solubility of alcohol in water is greatly reduced by the presence of salts. In fact, it may be "salted out" of water solution by saturation with extremely soluble salts. Again, absolute alcohol is freely miscible with petroleum ether. Under these circumstances, it would be expected that the results obtained by either of the proposed methods would be materially low, as was actually the case. It is possible that the author may still further modify the details so as to overcome these discrepancies. Since the official method seems quite satisfactory, however, it would seem that it should be retained.

RECOMMENDATIONS.

In view of the results submitted, it is respectfully recommended—

(1) That the rapid methods for vanillin (Folin's quantitative), coumarin (Wichmann's qualitative), and lead number (Wichmann's quantitative), while meritorious, be held in abeyance until: (*a*) Sufficient data are collated on authentic samples of vanilla extracts to enable satisfactory interpretation of analyses; (*b*) the new lead number is submitted in some form in which it will not be confused with the present official method; and (*c*), a satisfactory quantitative rapid method for coumarin has been developed. Investigations along these lines by individuals, especially the authors of the methods, are urged.

(2) That a study of methods of analysis of imitation vanilla preparations containing large quantities of coumarin and vanillin be undertaken.

(3) That Hortvet and West's method[1] for alcohol in lemon and orange extracts be adopted as an official alternative method for extracts consisting only of oil, alcohol and water.

(4) That Randall's method[2] for the determination of oil in lemon and orange extracts be studied in connection with the official method.

(5) That the methods of analysis of non-alcoholic flavoring extracts be studied.

ERRORS IN GRAVIMETRIC VANILLIN DETERMINATIONS IN VANILLA EXTRACTS.

By H. J. WICHMANN (U. S. Food and Drug Inspection Station, Tabor Opera House Building, Denver, Colo.).

When vanillin is extracted from a dealcoholized vanilla extract after the addition of lead acetate and the filtration of the resulting precipitate,

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[1] *J. Ind. Eng. Chem.*, 1909, **1**: 84.
[2] *Ibid.*, 1914, **6**: 926.

it is invariably contaminated with a brown, gummy impurity. Direct weighing gives high results. Hiltner[1] has reported that such results on vanilla extracts are sometimes too high by as much as 0.04 per cent. Purification with petroleum ether[2] is tedious and uncertain, the results frequently being too low, because of the occlusion of vanillin in the gummy matter. This defect in the method was pointed out by Hiltner[3]. The association results recorded by him bear out this statement. Since direct weighing gives high, and petroleum-ether purification generally low results, Hiltner[3] suggested that the vanillin be sublimed at 105°C. and the weight determined by difference. This suggestion was based on the assumption that vanillin was completely volatile at 105°C. and that the brown residue left represented solely the gummy matter extracted by the ether. He states that probably the volatilization method gives the more reliable figures. In this paper attention is directed to the errors of the different gravimetric methods for vanillin determinations, especially to the latter method.

When commercial samples of so-called chemically pure vanillin were sublimed in the air, invariably a yellowish-brown, more or less crystalline residue, non-volatile at 105° to 108°C., was left behind. This residue varied between 3 and 8 per cent in the samples available for examination. In an attempt to ascertain whether the residue was due to decomposition during heating or to the presence of non-volatile impurities, pure vanillin was prepared.

Commercial vanillin is produced mainly by the oxidation of acetyl eugenol with potassium permanganate. The crude product is contaminated with vanillic and vanillinic acids. Vanillin is removed from an ether solution of vanillin and vanillic acid by shaking with a strong solution of sodium acid sulphite which combines with the vanillin, leaving the vanillic acid in the solvent. By subsequent treatment of the sulphite compound with acid and extraction with a suitable solvent the vanillin is recovered. Vanillinic acid is removed from vanillin by treatment with a suspension of magnesium carbonate in water. It can also be broken up at 140°C. into carbon dioxid and vanillin. Chemically pure vanillin is produced by such methods of purification.

In an effort at further purification, chemically pure vanillin was distilled in a vacuum of from 3 to 5 mm., maintained with a vacuum pump. The receiver was ice chilled and the distillation flask heated in a sulphuric acid bath. A slight stream of dry carbon dioxid was admitted to promote distillation. Vanillin distilled at 140°C. and condensed, partly as a colorless liquid that quickly solidified and partly as "flowers" of vanillin. Any vanillinic acid present as an impurity would decompose

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[1] U. S. Bur. Chem. Bull. **152**: (1912), 135.
[2] *Assoc. Official Agr. Chemists, Methods*, 1916, 259.
[3] U. S. Bur. Chem. Bull. **152**: (1912), 135; **162**: (1913), 83.

at 140°C. into vanillin. Vanillic acid, the only other impurity likely to be present, is but slightly volatile at this temperature in a vacuum. One gram of the specially purified vanillin was dissolved in ether and shaken several times with a nearly saturated solution of sodium acid sulphite, as in the technical purification. No crystals were observed on evaporation of the ether. In a check experiment with a gram sample containing 10 mg. of vanillic acid a beautiful crop of crystals was left. This distilled vanillin, therefore, can be considered to be as pure as it is possible to get.

Fifty, 100, and 200 mg. of this pure vanillin were weighed into small platinum dishes and heated in an air oven for intervals of 1 hour at 105° to 108°C., as in the procedure recommended by Hiltner. In every case, a dirty, yellowish-brown, partly crystalline residue, similar to that left by the chemically pure commercial samples, was left. This residue was not entirely soluble in alcohol, ether or hot water, but was soluble in ammonia, and must, therefore, have been of an acid character. It is believed to be a mixture of oxidation and decomposition products of perhaps very complex nature. Its amount and composition probably vary according to the conditions of heating; namely, temperature, time of heating, quantity of vanillin heated, exposure to oxygen, and perhaps other factors. The results of the sublimation experiments are given in Table 1.

TABLE 1.

Results of sublimation of pure vanillin.

WEIGHT OF VANILLIN	TIME REQUIRED TO REACH CONSTANT WEIGHT BY HEATING FOR 1-HOUR INTERVALS	RESIDUE	RESIDUE
mg.	*hours*	*mg.*	*per cent*
50	3	2.0	4.0
50	3	2.3	4.6
100	4	4.4	4.4
200	6	11.2	5.6

Table 1 shows that pure vanillin is not entirely volatile in air at 105° to 108°C., as was assumed to be the case by Hiltner. The residues left by commercial chemically pure vanillin samples, therefore, may be considered as produced by decomposition rather than by impurities.

Results by the sublimation method, therefore, average about 5 per cent too low. In the case of an average extract containing 0.2 per cent of vanillin, the absolute error would amount to about 0.01 per cent. This seems to be almost negligible. On the other hand, the error by direct weighing and the often larger one by attempted petroleum-ether purification, as has been shown by Hiltner, are not negligible. In the case of flavors reenforced with vanillin, the error due to the non-volatile residue may become appreciable and a 5 per cent correction should be applied.

The difference between results by direct weighing and sublimation in standard extracts usually varies between 0.01 and 0.04 per cent. The average plus error due to the gummy matter will about counterbalance the minus error due to the non-volatile residue. A compensation of errors would, therefore, be introduced if the results by the two methods were averaged, and an intermediate value obtained which would probably be very close to the truth. However, it seems the more scientific to apply an experimental average correction to a known error than to compensate two errors of opposite sign, even if both methods will produce approximately the same results.

SUMMARY.

Errors due to direct weighing of vanillin extracted from vanilla extracts and attempted purification by petroleum ether, first reported by Hiltner, have again been emphasized.

Pure vanillin has been obtained.

Pure vanillin when sublimed in air at 105° C. has been shown to be not entirely volatile.

The error due to this non-volatile residue when using the sublimation method for vanillin determination, as suggested by Hiltner, has been shown to be small in the case of standard vanilla extracts, but becomes appreciable in reenforced flavors.

REPORT ON DAIRY PRODUCTS.

By **Julius Hortvet** (State Dairy and Food Commission, St. Paul, Minn.), *Referee.*

During 1918 the collaborative work was confined chiefly to a continued study of the Roese-Gottlieb method and various modifications as applied to dried milk products of varying fat content and to malted milk. Carefully prepared uniform samples representing these products were distributed among a dozen or more collaborators with instructions to proceed according to the following directions:

DETERMINATION OF FAT IN DRIED WHOLE MILK, DRIED SKIMMED MILK AND MALTED MILK.

PREPARATION OF SAMPLES.

Thoroughly mix the entire sample before weighing a portion for analysis. Weigh out 1.0–1.5 gram of sample. Mix and weigh as rapidly as possible, for these powders readily take up moisture from the air.

ALKALINE METHOD.

With the help of a camel's-hair brush and glazed paper, transfer the weighed sample to a 50–60 cc. stoppered cylindrical graduate or other stoppered cylinder, add 7–8 cc. of water, and dissolve by repeated shaking. Add 1 cc. of concentrated ammonium hydroxid and shake thoroughly; then add 5 cc. of alcohol and 15 cc. of ethyl ether and proceed as directed in the following modified form of the Roese-Gottlieb method.

ACID METHOD.

Transfer the weighed sample to a small Erlenmeyer flask, add 10 cc. of hydrochloric acid (sp. gr. 1.125), and dissolve the curd by heating nearly to boiling; then transfer to a 50–60 cc. stoppered cylinder, rinse out the material remaining in the flask with 15 cc. of ethyl ether, and proceed as in the modified form of the Roese-Gottlieb method.

ALKALINE AND ACID METHOD.

Transfer the weighed sample to a small Erlenmeyer flask, add 7–8 cc. of water and 1 cc. of concentrated ammonium hydroxid. Digest at a low heat until the material is well softened, add 10 cc. of concentrated hydrochloric acid, boil gently for 5 minutes, cool, and transfer to a 50–60 cc. stoppered cylinder. Rinse out the material remaining in the flask with 15 cc. of ethyl ether, and proceed as directed in the Roese-Gottlieb method described in modified form.

ROESE-GOTTLIEB METHOD, MODIFIED.

Shake the mixture in the cylinder thoroughly, add 15 cc. of petroleum ether, and shake for 1–2 minutes; then centrifugalize for 2 minutes at a speed of 1200 revolutions. Fit into the neck of the cylinder, or in an ordinary wash bottle, a blowing-off arrangement such as is used in the Werner-Schmidt method. Blow off by the usual method or by means of an equal-pressure bulb as much as possible of the ether-fat solution (usually 0.5–0.8 cc. will be left) into a weighed flask through a small quick-acting filter. Re-extract the liquid remaining in the tube, using the same volume of ethers, shake vigorously for 1 minute with each addition of ether, and centrifugalize for 2 minutes; then draw off the clear ether solution through a small filter into the same flask as before. Again repeat the extraction in the same manner. Evaporate the ethers slowly on a steam bath, and dry the fat in an oven at 100°C. to constant weight.

Confirm the purity of the fat residue by dissolving in a little petroleum ether. Should a residue remain, remove the fat completely with petroleum ether, dry the residue, weigh, and deduct the weight from the first weighing.

Notes.—If an emulsion occurs in the cylindrical tube, the addition of a few drops of alcohol followed by shaking and centrifugalizing usually breaks it up. Take care to make the final weighing of the dried fat under exactly the same conditions as those under which the dish was first weighed.

GENERAL INSTRUCTIONS.

Condition of samples.—Carefully note the condition of each sample. If the stopper or cap covering has become loosened the sample may have taken up moisture, in which case it is not in suitable condition for analysis.

Comparative tests.—On all three samples make the determinations according to all three methods of treatment described under "Preparation of Samples" (pages 482-3).

Duplicate determinations.—In all cases make two or more determinations by each method on all samples as directed. Report the individual results so obtained, and calculate the averages of results which are in reasonable agreement.

Experience.—In the case of all methods make preliminary trials. Repeat as many times as seems necessary in order to become adequately prepared for the regular determinations to be made on selected samples. The importance of ample experience can not be overestimated, especially in connection with the Roese-Gottlieb method.

It will be noted that certain modifications not tried out during the season of 1917 have been introduced in this outline of directions. In the first place, the Röhrig tube was replaced by a cylindrical tube provided with an attachment for drawing off the solvent ether layer. It was deemed necessary, especially in working on dried milk products, to centrifugalize in order to overcome emulsions and thereby effect a clear separation of the solvent. The plan of work was arranged to bring out as adequately as possible the relative merits of the three methods of preliminary treatment of the sample which have been under discussion frequently during recent years. In the letter of general instructions submitted to the collaborators it was made plain that if for any reason it might seem desirable to make the determinations on one or more of the samples by means of the Röhrig tube and by omitting the centrifugalizing, such a procedure might be adopted. Collaborators, however, were requested in all cases to embody in their reports a statement as to the method employed.

In due time, toward the close of the 1918 season, reports were submitted by twelve collaborators. A general comparison of the results (Table 1) leads to no satisfactory definite conclusions. Not only are very wide discrepancies evident among the results obtained on the samples of dried skimmed milk, but also great irregularities among the duplicate determinations made by certain individuals. The results obtained by the alkaline method of preliminary treatment of the sample fail to justify the recommendation of the adoption of this method for dried skimmed milk. Too few collaborators were able to report even reasonably concordant results. This statement holds true even in the case of the figures submitted by individuals whose work heretofore has been considered dependable. Moreover, results obtained by the Roese-Gottlieb method after preliminary acid treatment of the material are scarcely an improvement over the regular alkaline method. Here, again, it appears to be impossible to group results from a reasonable number of collaborators which would justify a further consideration of this modification. Only three or four among a dozen individuals report results which are fairly concordant. The remainder of the results may be described as scattering and widely divergent.

Turning to the double preliminary treatment of the sample (the alkaline treatment followed by the acid treatment as described on page 483), it is surprising to find a fairly close agreement among the results submitted by all the collaborators except two, one of whom failed to complete the work until 1919. It is fairly reasonable to assume that in a product like dried skimmed milk certain variations in actual composition may have existed, and that during the interval from one season to the next a substantial change, due probably to absorption of moisture, may have taken place. The results submitted by this single collaborator appear to confirm this explanation. On the other hand, the results by the other collaborator were much higher than the bulk of the averages based on the duplicate determinations submitted. If a conclusion may be drawn merely from the numerical results reported by ten of the collaborators, there would appear to be fairly good reason for a recommendation for the adoption of the alkaline-acid modification as a tentative method. Similar conclusions may be drawn from the reports received on the samples of dried whole milk. Only a fairly close agreement exists among the results obtained by six collaborators, nearly all of whom carried out several duplicate determinations by the regular Roese-Gottlieb procedure. It is unfortunate that some of the collaborators failed to exercise proper judgment in reporting averages. In a number of instances averages have been reported on results which should not have been included in the calculation. For example, one group of duplicate determinations comprises six results ranging from 22.89 to 28.67 per cent, some of which probably were obtained by preliminary trials, although no statement as to this fact accompanied the collaborator's report. On the other hand, a limited number of reports include results showing reasonably close agreement, as, for example, in one instance eight results which range from 27.24 to 27.72 per cent. Unfortunately, however, only a few have been able to report check results within reasonable agreement, and the returns from certain groups of individuals show wide discrepancies.

A comparison of the results obtained by the preliminary acid treatment of the sample, followed by the regular Roese-Gottlieb procedure, shows a marked improvement, nine collaborators reporting results which are within reasonable agreement. If conclusions may be drawn merely from the figures submitted, there would appear to be some justification for adopting the Roese-Gottlieb acid modification as tentative. The figures in the third column of each section of Table 1, however, do not justify a preference for this modification. About the same number of collaborators, seven or eight, report results within reasonable agreement, showing averages ranging from a minimum of 27.33 to a maximum of 27.83 per cent.

Most of the collaborators found it difficult to obtain a complete solution of the sample by means of the alkaline treatment. The ammonia treatment, however, involves certain inherent difficulties, chief of which are the tendency to form an emulsion and the difficulty of obtaining a complete solution of the powder. Any experienced analyst can readily devise some means for overcoming such well-known difficulties. The discussion on this point centers chiefly around the two modifications involving treatment of the milk powder with acid. Practically all collaborators call attention to the fact that heating the sample with acid gives rise to a marked charring and that the resulting ether extract has a more or less dark color which is annoying and difficult to remove.

There is a prevailing suspicion that material other than fat is extracted after subjecting the powder to the acid treatment. Aside from these criticisms, however, there is a marked inclination to favor the preliminary acid treatment of the sample as described on page 483. Few of the collaborators express a decided preference for the double alkaline-acid procedure, although the actual results submitted, especially in the case of the dried skimmed milk, appear to contradict some of the conclusions reported by certain individuals. It is shown in all cases that the acid method tends to yield higher results, but this conclusion appears to be materially counteracted by the observation that the material is more or less darkened and that the ether-extracted residue is not in a sufficiently purified condition for weighing.

TABLE 1.

Fat determinations on dairy products, 1918.

ANALYST	MALTED MILK			DRIED WHOLE MILK			DRIED SKIMMED MILK		
	Roese-Gottlieb Alkaline Method	Roese-Gottlieb Acid Method	Roese-Gottlieb Alkaline-Acid Method	Roese-Gottlieb Alkaline Method	Roese-Gottlieb Acid Method	Roese-Gottlieb Alkaline-Acid Method	Roese-Gottlieb Alkaline Method	Roese-Gottlieb Acid Method	Roese-Gottlieb Alkaline-Acid Method
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
Daphne Dodd, State Food and Drug Department, Lansing, Mich.	8.48	8.35	8.42	27.94 27.90 27.74	27.88	27.83	1.84	1.90	1.86 ....
C. L. Clay, State Board of Health, New Orleans, La.	8.27 8.17	8.29 8.67 8.39 8.69	9.06 8.99	27.34 27.22	27.78 27.74	26.92 26.67 26.75	0.81 0.93	1.12 1.01	1.13 1.36 ....
I. R. Howlett, State Dairy and Food Commission, Madison, Wis.	8.20 8.22 8.19	8.36 8.36 8.25	8.03 7.93 7.80	27.50 27.29 27.44	27.72 27.73	27.43 27.38	....	....	....
W. A. Brannon, State Dairy and Food Commission, Madison, Wis.	....	....	....	....	....	....	1.18 1.08 1.25	1.24 1.29 1.37	1.27 1.35 1.30 1.33
E. R. Lyman, U. S. Food and Drug Inspection Station, 4145 Arcade Building, Seattle, Wash.	8.17 8.09	9.457 8.710	8.777 8.710	29.67 29.18 28.53 28.29 27.93	27.51 28.00 28.04	27.74 27.78	0.67 0.51	1.26 1.31	1.374 1.370 ....
D. H. McIntire, U. S. Food and Drug Inspection Station, 4145 Arcade Building, Seattle, Wash.	7.71 8.01 8.00	6.21 6.68 6.34	8.88 8.92	22.89 24.49 26.54 26.21 26.32 28.67	15.52 18.71 24.01 22.83 24.53	27.25 27.58 27.16	2.61 1.97 1.12	1.28 1.51 1.55	1.404 1.72 1.42 ....
A. S. Wells, State Dairy and Food Commission, Portland, Ore.	7.89 7.89	8.11 8.13	8.76 8.81 8.78	28.03 28.20	27.41 27.26 27.11	27.57 27.40	1.09 1.12	1.13 1.16	1.13 1.18 1.38
O. C. Kueffner, State Dairy and Food Commission, St. Paul, Minn.	8.38 8.58	8.38 8.36	8.24 8.40	27.78 27.62	27.49 27.30	27.78 27.71	1.44 1.42	1.22 1.30	1.35 1.34
J. T. Keister, Bureau of Chemistry, Washington, D. C.	8.63 8.57	8.36 8.49	8.89	27.40 27.13	27.84 28.15	28.31 29.29	1.10	1.00 1.02	1.73 1.40 1.06 1.03

TABLE 1.—Concluded.

ANALYST	MALTED MILK			DRIED WHOLE MILK			DRIED SKIMMED MILK		
	Roese-Gottlieb Alkaline Method	Roese-Gottlieb Acid Method	Roese-Gottlieb Alkaline-Acid Method	Roese-Gottlieb Alkaline Method	Roese-Gottlieb Acid Method	Roese-Gottlieb Alkaline-Acid Method	Roese-Gottlieb Alkaline Method	Roese-Gottlieb Acid Method	Roese-Gottlieb Alkaline-Acid Method
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
A. M. Easton, Merrell-Soule Co., Syracuse, N. Y.	8.00	8.28	8.60	27.72	27.62	27.66	1.18	1.34	1.26
	7.72	8.50	8.56	27.70	27.78	27.50	1.22	1.40	1.24
	8.00	8.26	7.68	27.48	27.60	27.48	1.32	1.20	1.22
	8.16	8.24	7.90	27.60	27.50	27.46	1.24	1.18	1.32
	7.86	8.50	8.00	27.24	27.62	....	1.26	1.12	1.34
	7.92	....	8.02	27.37	27.64	....	....	1.20	1.28
	7.74	....	8.74	27.50	....	....	....	1.20	1.32
	7.86	....	8.74	27.52	....	....	....	1.24	1.28
J. H. Bornmann, U. S. Food and Drug Inspection Station, 1625 Transportation Building, Chicago, Ill.	8.42	8.53	8.64	27.84	28.08	28.05	0.99	1.30	1.40
	8.43	8.65	8.65	27.66	27.75	27.97	0.92	1.38	1.30
E. H. Berry, U. S. Food and Drug Inspection Station, 1625 Transportation Building, Chicago, Ill.	7.85	7.80	8.10	26.10	26.40	27.10	0.80	0.85	1.10
	7.80	7.60	8.10	26.10	26.25	27.20	0.95	0.95	1.05
	7.85	....	....	26.80	26.80	....	....	....	....

The third sample submitted to the collaborators consisted of a well-known brand of malted milk. From the results in Table 1 it is impossible to draw a safe conclusion favoring any one of the modifications which were tried. Not more than six of the collaborators obtained concordant results by any one of these methods, and only four report results obtained by all these methods agreeing within reasonable limits.

The general trend of the comments on the various methods is about the same as that of the comments on these methods as applied to dried milk. Although most of the collaborators show a disposition to favor the regular Roese-Gottlieb procedure, a comparison of the results obtained by the alkaline treatment does not justify such an attitude. The usual difficulty experienced in preventing the formation of emulsions in the cylinder or Röhrig tube after the required shaking was in practically all cases overcome by centrifugalizing. Too much charring or darkening of the resulting extracts was noted. Some of the collaborators express a preference for the acid mode of treatment, because of the ease in obtaining a complete solution of the sample, sharp separation of the solvent, and good concordant results which, in many instances, are higher than those obtained by the regular alkaline treatment. No special preference is expressed for the alkaline-acid procedure, where, again, the criticism is frequently made that the treatment with acid results in the extraction of impurities which are weighed with the fat and therefore yields too high results.

James Henderson (Horlick Malted Milk Company, Racine, Wis.) has devoted a great deal of special study to the analysis of malted milk products. Many of his criticisms have proved valuable in relation to this subject.

As a result of correspondence with Henderson, a plan was outlined for additional collaborative work on malted milk. Uniform samples were distributed among a dozen collaborators, with instructions to proceed according to the following brief outline:

DETERMINATION OF FAT IN MALTED MILK.

ROESE-GOTTLIEB METHOD (REGULAR ALKALINE).

Proceed as described on pages 482-3.

ROESE-GOTTLIEB METHOD (NEUTRAL).

Proceed as described on pages 482-3, omitting the ammonia.

DIRECT ETHER EXTRACTION.

Weigh about 0.8 gram of the uniform sample into a small dish, and dissolve in 6–7 cc. of water. To make the solution, first add sufficient water to make a thick paste, then add the remainder of the water, stir, and heat on the water bath to insure complete solution. Transfer the solution completely to ignited fibrous asbestos in a cartridge (the last traces of solution in the dish being removed by asbestos which is added to the cartridge), and heat in an oven at 100°C. until perfectly dry. Then extract for 6–7 hours with dry sulphuric ether, evaporate the ether, heat the flask to constant weight at 100°C., and weigh.

Results have been submitted by only six collaborators (Table 2). It is impossible to draw any definite conclusions from the figures in Table 2 except that results by the alkaline method are, in general, much lower than those obtained by the neutral method. Results obtained by the neutral method are apparently quite satisfactory, except the figures of one collaborator which fall below the averages submitted by the others by from 1.0 to 1.5 per cent. Nevertheless, the results obtained by the neutral method are not only uniformly higher than those obtained by the alkaline method, but they are more uniform. Two collaborators obtained results by the direct ether-extraction method which compare very well with those obtained by the neutral method. In all other cases, however, the results are far too low, and the very low results reported by two of the Pacific Coast laboratories are difficult to explain. Yet, in spite of irregularities shown in the results obtained by the direct ether-extraction method, there is much encouragement to continue a study of this method during the coming season.

TABLE 2.

Fat determinations on malted milk, 1919.

ANALYST	ALKALINE METHOD	NEUTRAL METHOD	DIRECT ETHER-EXTRACTION METHOD
	per cent	*per cent*	*per cent*
D. H. McIntire	7.52	8.21	6.92
	7.90	8.29	6.87
	7.83	8.26	....
	....	8.28	....
Daphne Dodd	7.30	7.71	7.56
	7.30	7.63	7.43
	6.83	7.70	7.29
	6.94	7.50	7.45
E. H. Berry	7.85	7.90	8.12
	7.85	8.00	8.19
	....	....	8.12
A. S. Wells	7.38	8.09	6.71
	7.48	8.01	6.63
	7.48	8.33	6.67
	....	8.23	6.60
	....	....	6.62
J. T. Keister	8.138	6.71	8.44
	7.99	6.26	8.256
	7.908	....	8.276
	7.90	....	8.448
	....	....	8.201
E. O. Huebner, State Dairy and Food Commission, St. Paul, Minn.	8.05	8.35	7.63
	8.09	8.48	7.47
	7.94	8.40	....

The collaborative work done during 1918 and 1919 did not yield results sufficiently conclusive to justify the adoption of any particular modification of the Roese-Gottlieb method as applied to dried milk products and malted milk. Sentiment among the collaborators, however, favors a continued study of a number of these modifications, and the results in Tables 1 and 2 appear to justify the following recommendations.

RECOMMENDATIONS.

It is recommended—

(1) That a further study be made of the alkaline-acid modification of the Roese-Gottlieb method as applied to dried milk products of various fat content.

(2) That a further study be made of the Roese-Gottlieb neutral extraction method as applied to malted milk.

(3) That a further study be made of the direct ether extraction method as applied to malted milk.

(4) That methods for the determination of moisture in various dairy products be referred to a special referee on that subject for study during the coming year.

No report on the separation of nitrogenous substances in milk and cheese was made by the associate referee.

THE CRYOSCOPIC EXAMINATION OF MILK.

By Julius Hortvet (State Dairy and Food Commission, St. Paul, Minn.), *Referee on Dairy Products.*

During the month of February, 1919, a plan of routine investigation was instituted for the purpose of arriving at a definite conclusion regarding the actual character and composition of market milks distributed in St. Paul and Minneapolis. In addition to the routine determinations of specific gravity, fat, total solids and ash, the freezing-point determination was made. Although neither recent in origin nor new in principle, this determination has, nevertheless, been applied to only a comparatively limited extent in food and dairy laboratories in the United States. This method was investigated by Beckmann[1] and by Winter[2] as early as 1894 to 1896. At a somewhat later time it was applied extensively in Australia, and was then followed up by investigations along the same line by government chemists in Continental Europe and in various parts of the British Empire. Attention should be called to the fact that a constant or only slightly variable freezing point is characteristic of various natural animal gland secretions. The following tabulation will illustrate in a general way the applicability of freezing-point determinations to a number of fluids, including cows' milk:

FLUID	FREEZING POINT
	— 0°C.
Human gland secretions	
Bile	0.560
Gastric juice	0.550
Pancreatic juice	0.470
Human blood	
Normal	0.560
Variations due to dietary causes	0.510–0.620
Cows' milk	0.540–0.560

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[1] *Milch Ztg.*, 1894, **23**: 702.
[2] *Arch. ges. physiol.*, 1896, **8**.

The Queensland food law provides that milk shall not only be pure, clean and from healthy cows, and comply with certain limits for fat and other solids, but it shall also meet a definite freezing-point test of not higher than 0.550°C. below zero. From a theoretical consideration of the relation between the osmotic pressure of the blood and of the milk which the animal secretes, it soon became apparent that an upper limit to the osmotic pressure of the milk corresponding with the (constant) osmotic pressure of the blood may reasonably be expected. In other words, the freezing point of the milk will never fall below that of the blood in a healthy animal. Further investigations have confirmed and strengthened the practical reliability of the freezing-point determination as a means of detecting adulteration with water.

In 1911 Henderson[1] reported that during 1909 every milk sample containing less than 8.5 per cent of solids not fat was found by the determination of the freezing point to have been watered, and that in 1910 the same result was obtained in all but two cases. In 1913 Henderson and Meston[2] reported, as a result of freezing-point determinations on pure fresh milk from herds of cows in southern Queensland, no greater variation in freezing point than from $-0.55°$ to $-0.56°$C. They conclude that the freezing point determines with accuracy the proportion of water added to any fresh milk from a herd. In 1914 the same writers[3] discuss at length the essential features of their investigations on the freezing-point determination, concluding that the freezing point of fresh milk from herds is practically constant and that the maximum variation in results represents only about 2 per cent of added water, while the working error of the process is less than 0.5 per cent.

Substances which are not in solution, like fat, do not affect the freezing point. Therefore, as fat is the most variable component of milk, the most varying factor is removed from the result. A colloid solution, such as the albuminoids, affects the freezing point either not at all or only slightly and, in any case, owing to the high molecular weight of the albuminoids, the effect would be negligible. The lactose content of milk is fairly regular, but here again a substance of high molecular weight is encountered, and hence with a correspondingly small effect on the freezing point of the solution. These conclusions have been further confirmed by Winter and Parmentier[4], and others. Also, in general, the freezing point of milk is independent of breed, individuality, amount of fat, milking period, etc., and a very small amount of added water at once has a marked disturbing effect.

In 1917, the entire subject of the freezing-point determination, as

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[1] *Aus. Assoc. Adv. Sci.*, 1911, **13**: 88.

[2] *Proc. Roy. Soc.* (*Queensland*), 1913, **24**.

[3] *Chem. News*, 1914, **110**: 259.

[4] *Rev. gen. lait*, 1903–4, **3**: 193, 217, 241, 268.

applied to milk, was thoroughly investigated in the Bureau of Chemistry, of the United States Department of Agriculture[1]. The following conclusions are all that need to be noted here:

> The freezing-point figure of milk is the most constant one yet obtained and the safest basis upon which to draw conclusions as to the presence or absence of added water; the presence of water added to fresh milk in excess of 5% can be detected with certainty by the freezing-point measurement; the method is practical in milk control work in that the test need be applied only to samples of doubtful character; the freezing-point test should be applied only on normal or reasonably fresh milks, owing to the fact that an increase of acidity in excess of 0.15% counteracts the rise in the freezing point and may mask the presence of a substantial amount of added water.

Snell[2] states that the constancy of the freezing point of milk and the value of this determination as a test for added water have become generally recognized. Attention is also called to the usual technical difficulties incident to the accurate carrying out of this determination, and consideration is given to the fact that milk from individual cows may occasionally exhibit abnormal results with respect to general composition, with the consequent slight depression or elevation of the freezing point. For example, a cow which for a time has been underfed or existed under a diseased condition may yield a milk exhibiting abnormal chemico-physical properties. Absolutely no question is raised regarding the reliability of the freezing-point test as applied to mixed milk of herds or to market milk drawn from healthy cows properly fed and kept, and no serious uncertainty attends the application of the method to milk from individual normal cows, properly fed and kept.

In 1918, Durand and Stevenson[3] called attention to the fact that the cryoscopic method gives excellent results but occupies too much time. They express a hope for a quicker method of procedure. Snell[2] also calls attention to the urgent need of economizing in the time needed for a single determination which was placed by Durand at 45 minutes for a duplicate determination. The hope was expressed for the development of a device which will materially shorten the time and even permit a number of determinations to be made simultaneously. This is a very important point in view of the large number of routine samples of market milk submitted to a food laboratory in the ordinary course of inspection.

Obviously, the common type of Beckmann apparatus and other similar devices are not serviceable from the standpoint of economy in time and convenience in carrying out a large number of determinations, many of which must be made in duplicate. A new type of freezing-point apparatus devised by the referee within the past two years has fulfilled

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[1] *J. Ind. Eng. Chem.*, 1917, **9**: 862.
[2] *Can. Chem. J.*, 1918, **2**: 200.
[3] *J. Ind. Eng. Chem.*, 1918, **10**: 26.

the important requirement respecting economy of time, not to mention the great improvement in convenience and ease of manipulation. It has, therefore, been possible during the past eight months to apply the freezing-point determination to somewhat over 500 samples, including milks of a suspicious character, as well as to a large number of samples which, according to results of ordinary routine analysis, appeared to be normal. For the purpose of a survey on the properties of milk known to be pure, produced in the locality of St. Paul and Minneapolis, and for the purpose of establishing a basis on which to judge the purity of samples of market milk, careful analyses have also been made on samples of milk of known origin taken chiefly from individual cows and in a few instances from mixed herds.

TABLE 1.

Composition of samples of known pure milk.

(Minnesota dairy farm.)

BREED	SPECIFIC GRAVITY AT 60°F.	FAT	TOTAL SOLIDS	SOLIDS NOT FAT	FREEZING POINT
		per cent	*per cent*	*per cent*	*— 0°C.*
Guernsey	1.0320	4.00	12.94	8.94	0.544
Jersey	1.0330	5.90	15.49	9.59	0.560
Jersey	1.0330	4.80	14.15	9.35	0.542
Holstein	1.0302	3.25	11.59	8.34	0.543
Holstein	1.0312	3.40	12.02	8.62	0.538
Holstein	1.0318	3.80	12.65	8.85	0.540
Holstein	1.0300	3.10	11.37	8.27	0.541
Jersey	1.0330	3.80	13.05	9.25	0.547

TABLE 2.

Composition of samples of known pure milk.

(Inspectors' samples.)

BREED	SPECIFIC GRAVITY AT 60°F.	FAT	TOTAL SOLIDS	SOLIDS NOT FAT	FREEZING POINT
		per cent	*per cent*	*per cent*	*— 0°C.*
Known genuine samples:					
Common herds	1.0320	3.80	12.70	8.90	0.542
Common herds	1.0346	5.30	15.08	9.78	0.542
Common herds	1.0347	6.70	16.70	10.00	0.557
Common herds	1.0350	4.80	14.13	9.33	0.544
Common herds	1.0332	3.70	12.69	8.99	0.551
Common herds	1.0320	4.50	13.79	9.29	0.561
Common herds	1.0318	3.80	12.64	8.84	0.544
Common herds	1.0309	4.50	13.28	8.78	0.556
Holstein	1.0296	3.30	11.51	8.21	0.546
Jersey	1.0334	4.30	13.65	9.35	0.555
Eden Valley creamery	1.0311	3.30	11.88	8.58	0.544
Part skimmed	1.0338	1.35	10.21	8.86	0.544
Skimmed	1.0327	0.03	8.37	8.34	0.539

Tables 1 and 2 show the composition of these known-pure milks, as well as the freezing-point result obtained on each individual sample. The composition of these milks corresponds very well with the range in composition of known-pure milks tabulated by Brown and Ekroth[1], Lythgoe[2], and others. Also the range in freezing point does not materially overlap the values reported by investigators already mentioned. In a few exceptional instances, a milk may test 0.001° higher than the minimum of 0.540°. In one instance, a sample tested 0.001° below the maximum of 0.560°, fairly confirming the range of ±0.006 reported by the Queensland laboratory.

Table 2 shows also the results of the analysis of two samples of skimmed milk. It is significant that while complete skimming removes all of the fat and produces no material change in the other milk solids, substantially no effect is produced upon the freezing point. The same result is indicated in the sample of partially skimmed milk.

To a sample of skimmed milk manufactured from a skimmed milk powder was added the theoretical amount of water necessary to restore the powder to its original fluid condition. After beating the mixture up thoroughly, the sample was analyzed, and a freezing-point determination made. As might be expected, an abnormal freezing-point result was obtained, indicating the addition of approximately 12 per cent of water. The sample was left in an ice box overnight, and the next day subjected to freezing-point test, with no material change in the result. Plainly, however, the freezing-point result depends largely upon the amount of water incorporated, and it is possible that an excessive amount of water was used in preparing the artificially made skimmed milk sample.

Combining the theoretical amount of water with a dried skimmed milk, however, could not be expected to restore the product to its original condition. Nevertheless, this point has not been investigated sufficiently to warrant any positive conclusions.

A mixture consisting of equal volumes of distilled water and standard evaporated milk gave a freezing-point test of −0.568°C., indicating that an amount of water insufficient to restore the sample to its original whole milk condition was added. A mixture consisting of 60 per cent water with 40 per cent of evaporated milk gave a freezing-point test of −0.442°, indicating excess water amounting to 19.6 per cent.

These experimental trials show that the freezing-point determination may serve as a criterion indicating the correct theoretical amount of water to be combined with a given sample of evaporated milk. In the case of any sample of dried milk powder or evaporated milk, the original product may be duplicated, providing a determination of the correct percentage of water to be incorporated may be made. Table 3 contains

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[1] *J. Ind. Eng. Chem.*, 1917, **9**: 297.

[2] Mass. State Board Health, Monthly Bull., 1910, new ser., 5: 419.

a number of illustrations, based on actual analyses, of the changes which occur in a sample of milk to which skimmed milk or water or both have been added in various ways. These results confirm again the fact that the freezing-point test constitutes a reliable means of determining the addition of water. Inspection of the analytical figures alone might lead to the conclusion that certain samples of so-called adjusted or standardized milk are actually normal. Take, for example, the sample showing 3.0 per cent butter fat and a specific gravity of 1.0298. While comparison with charts showing analyses of known-pure milks makes it obvious that this sample has been adulterated by the addition of skimmed milk, there is no evidence of the addition of water. The application of the freezing-point test, however, indicates about 11 per cent of added water, the amount actually incorporated in the sample when it was made up in the laboratory.

TABLE 3.

Effect of the addition of skimmed milk and water.

DESCRIPTION OF SAMPLE	SPECIFIC GRAVITY AT 60°F.	FAT	SOLIDS	SOLIDS NOT FAT	FREEZING POINT	ADDED WATER FOUND
		per cent	*per cent*	*per cent*	*— 0°C.*	*per cent*
Whole milk	1.0314	3.9	12.68	8.78	0.545	....
Skimmed milk	1.0350	0.01	8.91	8.90	0.539	....
3.9 per cent fat milk*	1.0321	3.1	11.89	8.79	0.541	....
Whole milk	1.0323	4.0	13.04	9.04	0.550	....
4 per cent fat milk†	1.0298	3.0	11.19	8.19	0.490	10.9

* Reduced to 3.1 per cent fat by adding 20 per cent of skimmed milk.

† Reduced in fat by adding 15 per cent of skimmed milk and 11 per cent of water.

TABLE 4.

Freezing-point tests on known pure milks containing known percentages of added water.

FREEZING POINT OF ORIGINAL MILK	ADDED WATER	FREEZING POINT OF MIXTURE AFTER WATER WAS ADDED	WATER FOUND
— 0°C.	*per cent*	*— 0°C.*	*per cent*
0.550	4.5	0.524	4.7
0.550	9.5	0.494	10.2
0.549	17.6	0.450	18.2
0.540	3.5	0.532	3.3
0.540	6.5	0.512	6.9
0.551	10.0	0.494	10.2
0.545	8.0	0.509	7.5
0.550	11.0	0.490	10.9
0.545	5.0	0.521	5.2

"Skimmed milk" made from dried skimmed milk and water.

Specific gravity at 60°F.	1.0340	
Fat	0.09	per cent
Solids	8.74	per cent
Solids not fat	8.65	per cent
Freezing point	– 0.485°	
Added water indicated	11.8	per cent

TABLE 5.

Composition of two samples of milk taken at Farm School Dairy, September 26, 1919.

DESCRIPTION OF SAMPLE	SPECIFIC GRAVITY AT 60°F.	FAT	TOTAL SOLIDS	SOLIDS NOT FAT	ACIDITY	ASH	FREEZING POINT
		per cent	*per cent*	*per cent*	*per cent*	*per cent*	*— 0°C.*
Holstein milk*	1.0270	3.1	10.61	7.51	0.09	0.55	0.543
Jersey milk†	1.0306	6.1	15.11	9.01	0.18	0.61	0.482

* First portion drawn from udder, representing only a partial milking. Contains no added water.
† Complete milking from udder, supposed to contain added water—approximately 10 per cent.

TABLE 6.

Composition of two samples of milk taken at Farm School Dairy, October 14, 1919.

DESCRIPTION OF SAMPLE	SPECIFIC GRAVITY AT 60°F.	FAT	TOTAL SOLIDS	SOLIDS NOT FAT	ASH	FREEZING POINT
		per cent	*per cent*	*per cent*	*per cent*	*— 0°C.*
Holstein milk*	1.0292	1.8	9.6	7.8	0.94	0.545
Composite sample†	1.0296	5.8	14.51	8.71	0.72	0.516

* First portion drawn from udder, representing only a partial milking. Contains no added water.
† Complete milking from four Jersey cows. Supposed to contain 5 per cent added water.

The practical application of the freezing-point determination is further illustrated by the results (Table 4) of tests made on samples of known-pure milk to which various percentages of water were actually added. It is important to bear in mind the fact that the freezing-point result obtained on a sample of milk containing a known amount of added water will depend largely upon the freezing-point test of the original whole milk. For example, in the case of two milks, each known to contain 10 per cent added water, very different results may be obtained, depending upon whether the freezing point of the original milk registered in the neighborhood of −0.540° or somewhere near −0.560°. The narrow range in freezing-point variations, therefore, makes it necessary in estimating percentages of added water to take into account a correction factor which may fairly be given as approximately 2 per cent.

Tables 5 and 6 give the results of analysis of two samples of milk drawn from Holstein cows. One showed a fat content of 3.1 per cent, as well as a very low specific gravity, and solids and ash content. The general composition of the other sample was practically the same, except for the butter fat, which was very low, viz, 1.8 per cent. Judged by ordinary rules, both of these samples might be suspected of adulteration with water. Information accompanying the samples was to the effect that each had been obtained as the first portion drawn from the udder of the cow (about one pint in both instances), representing only a partial milking; in other words, that portion of the milking which would be discarded as not constituting a representative whole milk. Nevertheless, on application of the freezing-point test to these samples very normal results were obtained, in the one case −0.543° and in the other −0.544°. Thus it appears that the freezing-point test indicates freedom from added water in both samples, while the ordinary analytical results lead to a suspicion that they are adulterated by means of added water. The other two samples included in Tables 5 and 6 were obtained from Jersey cows. In each of these samples the content of butter fat and total solids is very high, the other values being correspondingly fairly normal. So far as the analytical results can indicate there is nothing abnormal in the composition of these milks, but the results of freezing-point tests indicate substantial amounts of added water, in the one instance approximately 12 per cent and in the other over 6 per cent.

RECOMMENDATIONS.

Practical results obtained by many investigators show that the cryoscopic method is dependable as a means of determining, with a reasonable degree of accuracy, percentages of added water in milk. As a result of a careful study of this question during the past two years and after an extensive amount of experience with the application of the freezing-point method to a large variety of samples of market milks, as well as milks of known origin and composition, it is recommended—

(1) That this method, to be known as the cryoscopic method for the determination of added water in milk, be adopted as a tentative method by this association.

(2) That the cryoscopic method for the determination of added water in milk be subjected to further study with a view to its adoption as an official method.

If these recommendations are adopted in the manner suggested above, a plan of collaborative work can be arranged which will aim to serve the following purposes:

(1) To confirm the reliability of the cryoscopic method as a means of determining percentages of added water in milk.

(2) To develop and standardize the essential details in carrying out the determination.

(3) To arrive at definite conclusions regarding error factors and necessary corrections to be applied under various conditions.

(4) To perfect the details of a standard experimental outfit to be devised for the purpose of making freezing-point determinations on samples of milk, milk products and other food products to which the cryoscopic test may be applied.

REPORT ON MEAT AND MEAT PRODUCTS[1].

By RALPH HOAGLAND (Bureau of Animal Industry, Washington, D. C.), *Referee.*

The official methods for the examination of meats and meat products which were adopted by the association in 1916[2] are very much in need of further revision in order to bring them up to date. For several reasons such a revision proved to be impracticable. Because of personal interest in two methods, however, the referee undertook a study of: (1) A method for the determination of sugar in meat; and (2) a comparative study of two methods for the determination of moisture in meat. A report of the work follows:

STUDY OF THE PHOSPHOTUNGSTIC ACID METHOD FOR THE ESTIMATION OF DEXTROSE IN MEAT.

The following method, which has been used successfully by the referee for several years, was sent out to a number of chemists for cooperative work. Reports were received from four.

ESTIMATION OF TOTAL SUGAR.

REAGENTS.

Phosphotungstic acid.—Dissolve 100 grams in water, and make up the solution to 100 cc.

PREPARATION OF WATER EXTRACT.

Weigh 100 grams of the finely ground sample into a 600 cc. beaker, add 200 cc. of water, heat to boiling, and boil gently for 5 minutes. Stir the contents of the beaker frequently during this and subsequent extractions to prevent bumping. When several samples are extracted at the same time a mechanical stirring device is practically a necessity. Remove the beaker from the flame, allow the insoluble matter to settle, and decant the clear liquid on an asbestos mat in a 4-inch funnel. Filter with the aid of suction. Add 150 cc. of hot water to the residue in the beaker, boil gently for 5 minutes, let settle, and decant the clear liquid as before. Repeat the operation, and finally transfer the contents of the beaker to the funnel, wash with 150–200 cc. of hot water, and press the meat residue as dry as possible. Transfer the contents of the filter flask to an evaporating dish, and evaporate on a steam bath to a volume of about 25 cc., but not to dryness. Transfer the extract to a 100 cc. volumetric flask, taking care that the volume of liquid does not exceed 60 cc. Add 25–35 cc. of phosphotungstic acid, shake vigorously, let stand a few minutes for gas bubbles to rise to the surface, make to volume, shake, and either filter or centrifugalize. The use of a centrifuge is to be preferred since a larger volume of liquid is obtained. Test a portion of the filtrate with dry phosphotungstic acid for complete precipitation. If an appreciable precipitate forms, take an aliquot portion of the filtrate, add 5–10 cc. of phosphotungstic acid, make to volume, filter, and test the filtrate for complete precipitation. The filtrate should also show not more than a slight reaction for creatinin by Jaffe's test[3].

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[1] Presented by W. C. Powick.
[2] *Assoc. Official Agr. Chemists, Methods*, 1916, 271.
[3] O. Hammarsten. Textbook of Physiological Chemistry. 1915, 696.

DETERMINATION.

Transfer 50 cc. of the clarified extract to a 100 cc. volumetric flask, add 5 cc. of concentrated hydrochloric acid, and invert by one of the official methods. Cool the solution, neutralize to litmus, cool, make to volume and filter. To the filtrate add enough dry powdered potassium chlorid to precipitate the excess of phosphotungstic acid, filter, test for complete precipitation, and determine the reducing sugar by the method of Munson and Walker[1]. Estimate the reduced copper by Low's iodid method[2]. Calculate the total sugar as dextrose.

Table 1 shows the results obtained by the several analysts:

TABLE 1.

Estimation of dextrose in meat by phosphotungstic acid method.

ANALYST	DEXTROSE IN MEAT	DEXTROSE ADDED	TOTAL DEXTROSE	DEXTROSE FOUND	ERROR	RECOVERY
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
L. H. Almy, Food Research Laboratory, 1833 Chestnut Street, Philadelphia, Pa.	0.178	0.792	0.970	0.880	−0.09	90.7
	0.178	1.361	1.539	1.304	−0.235	84.7
R. Hoagland	0.122	0.177	0.299	0.300	+0.001	100.4
	0.122	0.318	0.440	0.428	−0.012	97.3
	0.122	0.742	0.864	0.794	−0.070	91.9
	0.122	0.980	1.102	1.071	−0.031	97.2
	0.122	1.274	1.396	1.335	−0.061	95.6
	0.356	0.177	0.533	0.539	+0.006	101.1
	0.356	0.318	0.674	0.680	+0.006	100.9
	0.356	0.742	1.098	1.060	−0.038	96.5
	0.356	0.980	1.336	1.344	+0.008	100.6
	0.356	1.274	1.630	1.590	−0.040	97.5
R. C. Holder, Food Research Laboratory, 1833 Chestnut Street, Philadelphia, Pa.	0.180	0.186	0.366	0.362	−0.004	98.9
	0.180	0.334	0.514	0.496	−0.018	96.5
	0.180	1.038	1.218	1.042	−0.176	85.6
W. D. Richardson, Swift and Company, Chicago, Ill.	0.1395	0.1947	0.3342	0.3520	+0.0178	105.1
	0.1457	0.3505	0.4962	0.5038	+0.0076	101.5
	0.1580	0.8178	0.9758	0.9630	−0.0128	98.7
	0.1580	1.0885	1.2465	1.2803	+0.0338	102.7
	0.1580	1.4049	1.5629	1.6520	+0.0891	105.7
J. J. Vollertsen, Morris and Company, Chicago, Ill.	0.2625	0.1805	0.4430	0.4612	+0.0182	104.1
	0.2373	0.3372	0.5745	0.5566	−0.0179	98.6
	0.2373	0.7871	1.0244	0.9580	−0.0664	93.5
	0.2373	1.047	1.2843	1.2449	−0.0394	97.0
	0.2373	1.352	1.5893	1.5466	−0.0427	97.3

These results may be considered as fairly satisfactory, particularly in the determination of the smaller quantities of sugar. The method is much superior to any other method which the referee has been able to find for the estimation of sugar in meat. Additional cooperative work with it undoubtedly will serve to give a higher degree of accuracy.

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 86.

[2] *Ibid.*, 96.

DETERMINATION OF MOISTURE IN MEAT BY DRYING AT 100°C. AS COMPARED WITH DRYING IN VACUO OVER SULPHURIC ACID AT ROOM TEMPERATURE.

Considerable work has already been done along this line, and the following data are offered simply as additional information on the subject.

Triplicate samples of the meat were dried in 2¼-inch aluminium dishes provided with friction covers to prevent absorption of moisture by the dried samples on weighing. Samples were dried in an electric oven at 100°C. and in 6-inch Scheibler desiccators containing sulphuric acid. The desiccators were evacuated by a Geryk pump which gave a pressure of less than 1 mm. on the manometer next the pump, but a pressure of 4 to 5 mm. inside the desiccator, as indicated in a small manometer. The desiccators were rotated frequently during the drying so as to mix the absorbed water with the acid. Weighings were made at 24-hour intervals. In many cases constant weight was obtained on the third weighing or at 72 hours, indicating that the samples were dry at the end of 48 hours. In practically all cases, constant weight was obtained at the fourth weighing. Table 2 shows the results obtained.

TABLE 2.

Moisture content of meat.

PRODUCT	DRIED AT 100°C. IN ELECTRIC OVEN	DRIED AT 3–5 MM. PRESSURE OVER SULPHURIC ACID IN DESICCATOR	
	per cent	*per cent*	*difference*
Fresh beef	75.66	75.50	−0.16
Fresh beef	73.50	73.37	−0.13
Calf brains	82.68	82.30	−0.38
Calf kidney	77.61	77.54	−0.07
Knockwurst sausage	55.82	56.16	+0.34
Frankfurter sausage	54.34	54.28	−0.06
Liver sausage	66.55	65.71	−0.84

The results obtained indicate slightly greater losses by drying at 100°C. than by drying in vacuo. Where a high degree of accuracy is desired, and particularly when fat is to be extracted from the dried residue, the method of drying in vacuo over sulphuric acid is to be preferred.

RECOMMENDATIONS.

It is recommended—

(1) That as soon as means are provided for the prompt publication of the proceedings of the association, a thorough revision be made of the methods for the examination of meat and meat products.

(2) That the method for the estimation of sugar in meat, previously

described, be adopted tentatively in place of the present tentative method[1].

(3) That one referee and two associate referees on meat and meat products be appointed as at present, but that the work of the associates be not designated.

No report on the separation of nitrogenous compounds in meat products was made by the associate referee for the year ending November, 1918.

REPORT ON THE SEPARATION OF NITROGENOUS COMPOUNDS IN MEAT PRODUCTS.

By L. C. MITCHELL[2] (Wilson & Co., Chemical Laboratory, Chicago, Ill.),

Associate Referee[3].

The work outlined by the associate referee consisted of a study of the Schlösing-Wagner method for the determination of nitrates in meat and meat products. As the time was very short, it was decided to limit the study of this method to the determination of nitrates in beef extract.

METHOD.

PREPARATION OF SAMPLES.

Sample A.—A high-grade beef extract, in which no sodium nitrate was used in the manufacture. This beef extract shows no qualitative or quantitative test for nitrates. It was used in the preparation of Samples B, C and D.

Sample B.—High-grade beef extract containing 0.1 per cent of added C. P. sodium nitrate.

Sample C.—High-grade beef extract containing 0.3 per cent of added C. P. sodium nitrate.

Sample D.—High-grade beef extract containing 0.5 per cent of added C. P. sodium nitrate.

Sample E.—Low-grade beef extract made from cured meats, therefore containing an unknown amount of commercial sodium nitrate.

REAGENTS.

Ferrous chlorid solution.—Dissolve nails, tacks, or other small pieces of iron, in concentrated hydrochloric acid, keeping an excess of iron present until the evolution of gas ceases. This is conveniently done by setting in a warm place a 2-liter Florence flask containing 400 grams of iron and 1 liter of concentrated hydrochloric acid. Close the flask with a stopper containing a Bunsen valve to keep out the air. Keep the solution in 50 cc. glass-stoppered bottles entirely filled. Employ only freshly opened bottles of the reagent for the determination.

Standard sodium nitrate solution.—Dissolve 2 grams of C. P. sodium nitrate in 1 liter of recently boiled water. Take 50 cc. (equivalent to 0.1 gram of sodium nitrate) and

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 278.

[2] Present address, U. S. Food and Drug Inspection Station, Federal Office Building, Minneapolis, Minn.

[3] Associate referee for the year ending November, 1919.

determine the amount of nitric oxid as given in the following method. One-tenth gram of sodium nitrate should give 26.36 cc. of nitric oxid at 0°C. and 760 mm. pressure.

APPARATUS.

Clamp a 500 cc. Kjeldahl flask with a 2-holed rubber stopper to an iron stand. Through one of the holes pass the stem of a 100–125 cc. open-top cylindrical separatory funnel having a glass stop cock and into the other fit a delivery tube leading downward at an angle from the flask into a trough containing water. Terminate the upper end of the delivery tube just below the rubber stopper in the flask, and place the lower end, which is bent slightly upward and covered with rubber tubing, under the surface of the water in the trough, the exit being immediately beneath the mouth of an inverted measuring tube (50 cc. plain eudiometer tube) filled with 40% sodium hydroxid solution. Midway on the delivery tube between the flask and the measuring tube place a short length of rubber tubing and a pinch cock. This pinch cock, however, has proved to be dangerous, and most analysts will find it safer to do without it.

DETERMINATION.

To 10 grams of the beef extract in a casserole add 30 cc. of boiling water, and stir until thoroughly mixed. Introduce 50 cc. of the ferrous chlorid solution and 50 cc. of 10% hydrochloric acid into the flask, close the stop cock of the funnel, move the end of the delivery tube so that escaping air will not pass into the measuring tube, and boil the contents of the flask until the air is expelled, as indicated by a slight pressure against the fingers when the rubber tubing is compressed after momentary removal of the flame. Place the exit end of the delivery tube beneath the measuring tube. Introduce the beef extract solution into the flask, a little at a time, through the funnel tube, and boil the contents of the flask at intervals to force the nitric oxid gas into the measuring tube. Finally rinse the casserole and the funnel tube with a little boiled water, add the rinsings to the contents of the evolution flask in the manner described, and boil until the nitric oxid no longer passes over into the measuring tube. Calculate the volume of nitric oxid at 0°C. and 760 mm. pressure. One cc. of nitric oxid at 0°C. and 760 mm. pressure is equivalent to 0.0037935 gram of sodium nitrate. Also calculate the percentage of sodium nitrate from the volume of nitric oxid obtained from the sample with the volume obtained from 0.1 gram of C. P. sodium nitrate, both being measured at room temperature.

RESULTS OF COOPERATIVE WORK.

The results of this work expressed as percentage, appear in Table 1.

TABLE 1.

Nitrogen in beef extract.

ANALYST	CALCULATED FROM SODIUM NITRATE STANDARD					CALCULATED FROM NITRIC OXID				
	A	B	C	D	E	A	B	C	D	E
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
W. D. Richardson, Swift & Co., Chicago, Ill.	None	0.078	0.24	0.44	0.98	None	0.075	0.23	0.43	0.94
E. A. Schlesser, Wilson & Co., Chicago, Ill.	None	0.09	0.27	0.45	0.98	....	....	....	....	....
P. Dunne, Wilson & Co., Chicago, Ill.	....	0.08	0.19	0.47	....	....	....	....	....	....
M. Janowsky, Wilson & Co., Chicago, Ill.	....	0.09	0.21	0.41	....	....	....	....	....	....
R. Hoagland, Bureau of Animal Industry, Washington, D. C.	None	....	0.27 0.23	0.39 0.41	0.85 0.92	None	....	0.22 0.20	0.32 0.33	0.72 0.80
H. C. Kershner, Bureau of Animal Industry, Kansas City, Kans.†‡	0.194* 0.095 0.109	0.102 0.087 0.109 0.139	0.346 0.247 0.283 0.318	0.296 0.425 0.345 0.554	1.12 1.01 1.05 1.08 1.06	0.17 0.08 0.106	0.095 0.08 0.106 0.133	0.30 0.20 0.269 0.340	0.25 0.35 0.335 0.539	0.98 0.91 1.02 1.04 1.02

* Run with water in trough, the sodium hydroxid becomes too dilute; evidently it contains carbon dioxid.

† First two results based on standards using 0.1 gram of sodium nitrate with corrections for vapor pressure.

$$V = v\,\frac{(\quad 1 \quad)}{760\,(1 - 0.00367t)} \times P\text{-}p$$

(1) 23.04 cc....................0.087 gram
(2) 25.43 cc....................0.094 gram
(3) 21.22 cc....................0.080 gram
(4) 22.35 cc....................0.085 gram

‡ Last three results based on standards using 0.1 gram of sodium nitrate without correction for vapo pressure. $V = \frac{P' V' T}{PT'}$

(1) 25.63 cc. ....0.097 gram
(2) 25.45 cc....................0.096 gram

COMMENTS OF ANALYSTS.

H. C. Kershner.—The figures are very unsatisfactory and apparently inaccurate, due no doubt to the rushing through of the test. The results on 0.1 gram of sodium nitrate vary beyond reason, the lowest results appearing when the gas evolution was the most uniform. It is not considered fair to the method to place much reliance on these figures, but they might be taken as a sample of what could be expected from operators unfamiliar with the method and compelled to perform the test without time for finding the proper conditions. No criticisms are made, but the idea of substituting a 300 cc. flask arises. This size appears ample, and might shorten the time used to expel the air. A 50 cc. funnel was used, as the 100 cc. appeared unnecessary and top heavy. It was found essential to add additional hydrochloric acid after the charge of extract, 10–15 cc. being added. Rubber gloves were also found to be very useful.

R. Hoagland.—The results obtained by calculating the percentage of nitrate from the volume of nitric acid at 0°C., 760 mm., are incorrect, since measured quantities of a standard solution of sodium nitrate yielded only from 80–92% of the theoretical quantity of nitric oxid. When a portion of the standard solution of nitrate was run into the reaction flask after a sample of meat extract, the quantity of nitric oxid liberated was always smaller than that obtained from a like volume of the standard solution run before the meat extract.

DISCUSSION OF RESULTS.

The results of the first five analysts show an average recovery of 82.6 per cent of the added sodium nitrate when compared with the amount of gas obtained from a standard nitrate solution. Two of the analysts show 69.4 per cent average recovery when calculated from nitric oxid at 0°C. and 760 mm.

The only explanation the associate referee can offer for the wide variation in the results obtained by the last analyst is that either all the air had not been driven from the flask before the extract was added or the apparatus was not air tight. From 0.1 to 0.19 per cent of sodium nitrate is reported in Sample A which was prepared from fresh (not cured) meat, and a negative qualitative test for nitrates is shown.

The following modifications are suggested for future work on this method:

APPARATUS.

(1) That a 40% solution of commercial sodium hydroxid be substituted for the water used in the trough so that the carbon dioxid may be effectively absorbed.

(2) That just after "into a trough containing water", the following be inserted, "The lower end of this delivery tube, should be slightly constricted."

(3) That beginning with "midway on the delivery tube, etc.", to end of paragraph, be deleted.

DETERMINATION.

(1) That "Calculate the volume of nitric oxid at 0°C. and 760 mm. * * * is equivalent to 0.0037935 gram of sodium nitrate" be deleted. Comparing the volume of nitric oxid found with the volume obtained from a known weight of pure sodium nitrate serves as a check upon the method, and the results reported indicate that this method gives a more accurate basis for calculation.

(2) That at the end of the method the following be added: "This is conveniently done by transferring the measuring tube to a tall jar containing a 40% sodium hydroxid solution (commercial). The temperature of the surrounding caustic solution will soon (10–15 minutes) be imparted to the contents of the tube, and the volume of nitric oxid is read with the tube in such a position that the level of the solution within and without the tube coincides. The caustic solution in the jar should be kept at room temperature".

Hoagland suggested a convenient device for cooling the soda solution in the trough during the determination which greatly facilitates the operation. It consists of fitting a single coil of tin tubing into the trough and passing a current of cold water through it during the determination.

It would be advisable to add the following suggestion of Hoagland as an explanatory footnote on the manipulation of the method:

> After all of the air has apparently been driven out of the apparatus, boil a short time longer after the delivery tube has been placed under the eudiometer to make certain that no air remains. Gradually introduce a measured portion of standard nitrate solution, rinse the funnel tube with 10% hydrochloric acid, and boil until all of the nitric oxid has been driven over. After the gas tube has been removed, quickly invert another tube over the delivery tube and boil a short time longer to make sure that all of the nitric oxid has been driven over. Run another portion of the standard solution into the apparatus, and repeat the determination. Then run the samples in the same way, in each case making certain that all of the nitric oxid has been driven over. After running 6–8 determinations, not counting the standards, finally run another standard. The three standards should check within 0.5 cc. on about 35 cc.

RECOMMENDATIONS.

It is recommended—

(1) That further work be done on this method, using beef extract, meat and other meat products.

(2) That the following 1916 recommendation be studied during the coming year: That the referee for next year attempt to determine the relative amounts of some of the dissociation products in water-soluble and water-insoluble meat proteins.

(3) That the title of the method be changed from "Nitrates" to "Nitrates and Nitrites (calculated as sodium nitrate)".

No report on meat extracts was made by the associate referee for the year ending November, 1918.

REPORT ON MEAT EXTRACTS.

By C. R. MOULTON (Agricultural Experiment Station, Columbia, Mo.), *Associate Referee*[1].

Nothing definite has been accomplished during 1919.

It is recommended—

(1) That an attempt be made to determine the relative amounts of some of the dissociation products in water-soluble and water-insoluble meat proteins. This probably can best be accomplished by studying certain groups of amino acids, or other protein derivatives, in meat and meat extracts, in collaboration with other referees to be appointed by the association.

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[1] Associate referee for the year ending November, 1919.

(2) That the work on the separation of some of the amino acids derived from meat proteins be continued.

(3) That the associate referee be not bound to a single method, but be left to choose as circumstances dictate and the collaborators accept.

REPORT ON EGGS AND EGG PRODUCTS[1].

By C. E. MARSH (State Department of Health, Boston, Mass.), *Referee.*

The work of the last two years consisted of the following:

(1) Testing methods for the determination of decomposition in eggs. This included a comparison of the Folin[2] and the Hendrickson and Swan method for the determination of ammoniacal nitrogen[3], and the comparison of the United States Department of Agriculture method for the determination of dextrose with that of Klein.

(2) Analyses showing the composition of both fresh and decomposed eggs.

(3) Methods for the detection of decomposition in dried eggs.

(4) Methods for the determination of heavy metals in dried eggs.

Later, it was suggested by R. W. Hilts (U. S. Food and Drug Inspection District, U. S. Appraiser's Stores, San Francisco, Calif.) that work be done on Juckenack's method on lecithin-phosphoric acid[4] with a view to reaching a suitable official method. This suggestion was adopted, and the following methods sent out to the collaborators:

FOLIN METHOD FOR AMMONIA[5].

Weigh about 20 grams of the well-mixed sample into a cylinder, add 5 cc. of saturated sodium carbonate, 2 cc. of a saturated solution of potassium oxalate, and some mineral oil to prevent frothing. Close the cylinder with a stopper containing two tubes, one of which reaches to the bottom of the cylinder, the other being of the distillation bulb and trap type to prevent any liquid passing over. The lower end of the second tube should pass into a 100 cc. flask containing about 50 cc. of water and 2 cc. of N/10 acid. If the outlet tube from the cylinder is slightly larger than the inlet, frothing will be reduced. Now blow a current of air (freed from ammonia by being passed through a sulphuric acid bottle) through the eggs, any ammonia carried over being absorbed by the acid. About 2 hours is usually required for this part of the process, but the exact time should be determined by experimentation in each laboratory. The cylinders found most convenient by the Massachusetts State Department of Health are 11½ inches tall and ⅜ inch in diameter, inside measure.

After the complete distillation of the ammonia, dilute 5 cc. of Nessler's solution with 25 cc. of water. Add this in three portions to the distillate, and dilute with water to 100 cc. Compare the colored solution, in a Duboscq colorimeter, with that produced

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[1] Presented by H. C. Lythgoe.
[2] *J. Biol. Chem.* 1912, **11**: 493.
[3] *J. Ind. Eng. Chem.*, 1918, **10**: 614.
[4] *Z. Nahr. Genussm.*, 1900, **3**: 13.
[5] *J. Biol. Chem.*, 1912, **11**: 493.

from a known amount of ammonium sulphate (usually 1 mg. per 100 cc.) treated in the same way with the Nessler reagent, and calculated as mg. per 100 grams. The standard ammonium sulphate is conveniently made of such strength that 5 cc. contain 1 mg. of nitrogen.

Preparation of the standard.—As ordinary C. P. ammonium chlorid contains pyridin and other bodies which interfere with the reaction of the Nessler solution, it is necessary to treat some of it with sodium carbonate in an apparatus similar to that described on page 507, and blow into C. P. dilute sulphuric acid until it is neutralized. Precipitate the ammonium sulphate with an equal volume of alcohol, filter, and dry. To obtain a standard solution of which 5 cc. contains 0.001 gram nitrogen, 0.471 of the purified product should be dissolved in sufficient water to make 500 cc. of solution. When a blank is distilled with this standard, at least 95% of the ammonia should be recovered; with ordinary C. P. ammonium chlorid only about 80% can be recovered.

HENDRICKSON AND SWAN METHOD FOR THE DETERMINATION OF AMMONIACAL NITROGEN[1].

Weigh 25 grams of the well-mixed sample into a tared dish, and pour into the aeration cylinder, transferring the egg adhering to the sides of the weighing vessel with the aid of four successive 25 cc. portions of ammonia-free water. Add 75 cc. of alcohol, mix, and let stand for 15 minutes. Then add 10 grams of sodium fluorid, 2 cc. of 50% potassium carbonate, and 1 cc. of kerosene. Connect the apparatus, and aerate strongly until no more ammonia comes over, then titrate at once with N sodium hydroxid. The apparatus used is essentially that of Folin, except that an aeration cylinder 50 mm. in diameter and 350 mm. high is used. Ordinary glass tubing sealed at the bottom and with small holes punctured according to the method of Folin and Farmer[2] is used in place of the special ammonia absorption tubes. It is recommended that a blank containing 1 mg. of nitrogen be run at the same time as the sample.

UNITED STATES DEPARTMENT OF AGRICULTURE METHOD FOR DEXTROSE IN EGGS[3].

After thoroughly mixing the sample, weigh 25 grams into a 100 cc. lipped beaker. Wash the sample into a 200 cc. graduated flask, using 70 cc. of distilled water. (Add about 40 cc. first, and mix the sample with the water by stirring with a rubber-tipped glass rod. After the contents of the beaker have been poured into a graduated flask, use a 20 cc. and finally a 10 cc. portion of distilled water to thoroughly wash the beaker.)

Then add 2 cc. of 5% acetic acid to the sample if it be egg white, or 1 cc. of the acid if the sample is mixed egg or egg yolk; mix thoroughly by shaking the flask, and place the flask in a water bath at 100°C. Egg should coagulate in 10 minutes. (There is danger of foaming during the first 5 minutes of heating.) After the egg material has been coagulated, place the flask in cold water until the contents are of room temperature. Then make up to the mark with alumina cream that has been washed several times to take out the dissolved salts. Shake the sample vigorously for 1 minute, allow it to stand about 5 minutes, and then shake for 1 minute.

The egg material should then filter readily, especially if folded filters are used.

The filtrate is clear and nearly colorless, and the reducing sugars determined in an aliquot should be calculated as dextrose.

Fresh egg white yields about 0.44% of dextrose;
Fresh egg yolk yields about 0.22% of dextrose; and
Fresh mixed egg yields about 0.34% of dextrose.

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[1] *J. Ind. Eng. Chem.*, 1918, **10**: 614.

[2] *J. Biol. Chem.*, 1912, **11**: 493.

[3] Personal communication from U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, Boston, Mass.

The results were obtained from composite samples, and variations of as much as 0.03 per cent seldom occur. As the egg material deteriorates, the dextrose content decreases. If the liquid egg has an excess of white or of yolk, the dextrose content can readily be calculated if a moisture or a fat determination is made.

KLEIN'S MODIFICATION OF THE BENEDICT AND LEWIS METHOD FOR THE DETERMINATION OF DEXTROSE IN EGGS[1].

ESTIMATION OF REDUCING SUBSTANCES IN FROZEN AND FRESH EGGS.

Weigh out 5 grams of eggs and wash into a 100 cc. sugar flask with about 25 cc. of water and fill up to the mark with a saturated aqueous picric acid solution. Shake the mixture thoroughly and allow it to stand for about 10 minutes. Filter the clear, yellow, supernatant liquid through a dry, double-folded filter paper. Introduce 10 cc. of the filtrate into a 50 cc. volumetric flask to which 3 cc. of saturated picric acid, 2 cc. of sodium carbonate solution (10 grams of anhydrous sodium carbonate to 100 cc. of water), and a few glass beads are added. Heat the flask on a sand bath until the solution is evaporated nearly to dryness. Care must be taken not to char the organic matter. A color will develop, varying in shade from yellow to dark red, depending on the amount of reducing matter present. Wash the neck and the sides of the flask with a few cc. of hot water, and boil the solution for about 3 minutes. Add warm water to dissolve the evaporated mass. Cool the flask to room temperature, and make up the contents to volume. If the solution is turbid, filter it through a cotton plug, rejecting the first few cc. of the filtrate.

Introduce the clear liquid into a Duboscq colorimeter chamber, and compare with a standard. The standards are made up as follows:

Prepare a solution having a color intensity equivalent to 0.004% of dextrose by dissolving 1 gram of C. P. dextrose in 500 cc. of water, diluting 20 cc. of this solution with 140 cc. of aqueous saturated picric acid solution, and making up to 200 cc. with water. Treat 10 cc. of this solution, containing 0.002 gram of dextrose, in the same way as described above. This standard is satisfactory for whites and whole eggs. For yolks and decomposed eggs, a weaker standard should be used. If the original sample contains 0.2%, or less, of reducing matter as dextrose, it is advisable to compare it with a standard equivalent to 0.2% of dextrose, or, if it contains less than 0.1% of reducing matter as dextrose, with a standard of 0.1% of dextrose. It is advisable in all cases to have about the same concentration of free picric acid in the standards as is present in the unknown solutions.

Calculations.—Dilute 5 grams of the sample to 100 cc.; finally dilute 10 cc. to 50 cc. The sugar standard with which it is compared contains 0.002 gram of dextrose in the same volume. Therefore

$$\text{Per cent of reducing matter as dextrose in sample} = \frac{\text{Reading of standard} \times 0.002 \times 200}{\text{Reading of unknown}}$$

Later in the year a sample of dried egg was sent to each collaborator for the determination of lecithin-phosphoric acid, as described by Leach[2]. Although few of the collaborators sent in any report, some gave very complete ones. The following suggestions and methods are taken from them:

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[1] Personal communication from David Klein, Division of Foods and Dairies, State Department of Agriculture, Chicago, Ill.

[2] A. E. Leach. Food Inspection and Analysis. 3rd ed., 1913, 349.

G. C. Swan (Food Research Laboratory, 1833 Chestnut Street, Philadelphia, Pa.) states:

The determinations found most valuable for the detection of decomposition in eggs are total solids, ether extract, acidity of ether extract, ammoniacal nitrogen, reducing sugar, indol and skatol, bacteria, and, of course, physical appearance. The ammoniacal nitrogen determination is of most value, for it best indicates incipient decomposition, which is perhaps the condition most commonly met. The total solids and ether extract must be known in order to properly interpret the results for ammoniacal nitrogen and reducing sugar. Practically all the ammoniacal nitrogen is found in the yolk of the egg, while the white contains over twice as much reducing sugar as the yolk. This is shown in Table 1.

Total solids.—Weigh out 3–5 grams of a well-mixed sample into a tared dish (lead bottle caps, which can be rolled up in fat-free filter paper for the ether extract, are useful), and dry in a vacuum oven at 55°C. to prevent coagulation. The vacuum should be 25 inches or over. It is recommended that weighings be made at the end of 2½ hours' drying, and thereafter at intervals of about 45 minutes. There is an appreciable gain in weight after the minimum has been reached.

Ether extract.—Extract the dry residue from the total solids determination in a Knorr extraction apparatus with absolute ether. Extraction for 3 hours is sufficient if the apparatus is working rapidly. (The rolling up of the lead dishes containing the sample in filter papers makes it unnecessary to use an asbestos plug in the bottom of the extraction tube.) Distil off the ether, and dry in vacuo for 1 hour at 55°C., cool in a desiccator, and weigh.

Acidity of ether extract.—Dissolve the ether extract in 50 cc. of neutral benzol to which 2 drops of phenolphthalein have been added, and titrate with N/20 sodium ethylate to first pink color. Express the results as the number of cc. of sodium ethylate required to neutralize 1 gram of ether extract.

Indol and skatol.—Place 200 cc. of liquid egg, 40 cc. of 5% acetic acid, and 500 cc. of water in a liter flask, and coagulate the protein in a steam bath or Arnold sterilizer. Then filter the material through a folded filter, and distil the filtrate with steam. Extract the indol in the distillate with a small amount of ether in a separatory funnel, add 1–2 cc. of water, and volatilize the ether before a fan until only a slight odor remains. Test the watery solution remaining according to the directions for the vanillin test given by V. E. Nelson[1].

Make the bacterial examination according to the directions given by the Bureau of Chemistry[2].

The following figures in Table 1 are taken from work done by Swan and are representative.

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[1] *J. Biol. Chem.*, 1916, **24**: 528.

[2] U. S. Dept. Agr. Bull. **51**: (1914).

TABLE 1.

Determination of decomposition in eggs.

(Analyst, G. C. Swan.)

QUALITY OF EGGS	DETERMINATION				
	Total solids	Ether extract	Acidity of ether extract	Ammoniacal nitrogen	Reducing sugar
	per cent	*per cent*	*cc.**	*mg. per 100 grams*	*per cent*
Tanners'	27.32	10.52	2.17	5.9	0.18
Tanners'	26.19	9.77	2.34	8.1	0.22
Sour	27.45	10.55	2.28	4.5	0.22
Partially addled	28.60	11.32	2.37	4.4	0.23
Stuck yolk	27.55	10.55	2.11	2.9	0.28
Borderline rots	26.69	10.44	1.99	3.4	0.31
Addled	29.11	11.22	3.59	12.6	0.11
Moldy, stuck, sour	27.75	11.06	6.17	13.3	0.04
Commercial whole egg	28.08	11.05	1.78	2.5	0.33
Commercial whole egg	27.57	10.65	1.81	2.4	0.36
Yolk (commercial)	44.35	23.32	1.89	4.2	0.19
Yolk (commercial)	44.31	23.40	2.04	4.0	0.14
Strictly fresh eggs:					
White	12.02	0.03	....	0.3	0.45
Yolk	46.92	24.49	....	2.8	0.20
Whole	26.18	9.97	....	1.4	0.33

* Cc. N/20 sodium ethylate required to neutralize 1 gram of ether extract.

No methods for the determination of the heavy metals have been developed. Chemical methods for the determination of decomposition in dried eggs have not proved successful. Dried egg made from material in an advanced stage of decomposition is dull yellow, and has a foul odor, but, unless it is badly decomposed, the foul odor and dull color are not apparent to the senses after drying. The ammoniacal nitrogen and educing sugar tests are of little value here, as it has been found that ammoniacal nitrogen is driven off and reducing sugar more or less destroyed on drying. The acidity of ether extract from freshly dried egg is a fairly good index, but the acidity increases on holding, and the influence of various factors, such as temperature, moisture, original grade of eggs, etc., is too uncertain to rely on this method for evidence of decomposition. The fact that so many decomposition products are volatile, and escape on drying, makes this a difficult problem, but it is one which should be vigorously attacked.

A. R. Todd (State Food and Drug Department, Lansing, Mich.) submitted the following figures and comments on them:

TABLE 2.

Determination of decomposition in eggs.

(Analyst, A. R. Todd.)

QUALITY OF EGGS	AMMONIACAL NITROGEN				DEXTROSE				FAT (BABCOCK)
	Hendrickson and Swan method		Folin colorimetric method		U. S. Department of Agriculture method		Klein method		
	Trials	Average	Trials	Average	Trials	Average	Trials	Average	
	mg. per 100 grams	*mg. per 100 grams*	*mg. per 100 grams*	*mg. per 100 grams*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
Strictly fresh	......	......	0.00032	......	....	....	....	....	....
	......	......	0.00036	....	.	...	...	....	....
	0.00181	......	0.00034	......	0.296	....	0.360	....	12
	0.00188	0.00185	0.00040	0.00036	0.324	0.310	0.368	0.364	12
Store fresh	0.00180	......	0.00072	......	0.270	....	0.304	....	13
	0.00168	0.00174	0.00065	0.00068	0.268	0.269	0.320	0.312	13
Frozen 4 months	0.00177	......	0.00046*	......	....	....	....	....	....
	0.00181	......	0.00048*	0.00047	....	....	....	....	....
	0.00200	......	0.00124	......	0.267	....	0.376	....	16.6
	0.00180	0.00185	0.00116	0.00120	0.262	0.264	0.384	0.380	16.5
Stale	0.00179	..	0.00112	......	0.234	....	0.248	....	12
	0.00173	0.00176	0.00106	0.00109	0.231	0.232	0.240	0.244	12.1
Small blood rings	0.00231	......	0.00282	......	0.162	....	0.200	....	12.2
	0.00213	0.00222	0.00264	0.00273	0.157	0.160	0.208	0.204	12.3
Green whites	0.00264	......	0.0039†	......	0.20	....	0.170	....	13
	0.00241	0.00253	0.0022	......	0.10	0.15	0.136	0.153	13
Moldy	0.00269	......	......	......	0.05	...	0.076	....	13
	0.00242	0.00256	......	......	0.07	0.06	0.072	0.074	13.5
Blood rots	0.0034	......	0.0037	......	0.078	....	0.088	....	13
	0.0031	0.0032	0.0036	0.00365	0.057	0.067	0.096	0.092	13

* Aerated 2 hours while the following determinations were allowed to run for 4–6 hours.
† Distilled water used for rinsing, causing high results.

Of the two methods for ammoniacal nitrogen, the Hendrickson and Swan method was much more satisfactory than the Folin colorimetric method. The range of variation is so narrow that it would seem to be very difficult to fix any definite dividing line for the percentage of nitrogen in edible eggs and those not fit for food.

The United States Department of Agriculture method for dextrose seems to give more uniform results and is easier to control than Klein's colorimetric method. The range of variation in the percentages of dextrose is wider than that of nitrogen, and would seem to be a more reliable indication of the age of the eggs.

FOLIN METHOD FOR AMMONIACAL NITROGEN IN EGGS.

TABLE 3.

Effect of varying the aeration period, other conditions being constant.

QUALITY OF EGGS	AMMONIACAL NITROGEN CONTENT OF EGGS AERATED			
	2 hours	4 hours	6 hours	8 hours
	mg. per 100 grams	*mg. per 100 grams*	*mg. per 100 grams*	*mg. per 100 grams*
Rots.............	0.00168	0.00162	0.00168	0.00174
	0.00156	0.00168	0.00168	0.00180
Strictly fresh.......	0.00018	0.00028	0.00034	0.00038
	0.00020	0.00030	0.00032	0.00040

As a result of the extremely low results obtained on fresh eggs by the Folin method, the series of determinations shown in Table 3 were made. In the fresh eggs, the increase in the percentage of nitrogen is very pronounced, depending on the period of aeration, while in stale eggs, the period of aeration has little effect after the first 2 hours.

R. W. Hilts gives the following suggestions regarding Juckenack's method:

Mix 30 grams of the finely ground sample with a teaspoonful of asbestos fiber rubbed through a screen. Pack this mass in a paper extraction capsule, and place in a Soxhlet extractor. Further manipulation is the same as that described by Juckenack, except that the volumetric method of determining phosphoric acid is generally employed.

Raymond Hertwig (U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, San Francisco, Calif.) sends the following report on the determination of lecithin-phosphoric acid in a sample of dried egg:

Grind the sample in a mortar to as fine a powder as possible. Extract the lecithin-phosphoric acid with hot absolute alcohol by two slightly different methods:

(1) *Juckenack method.*—Place 3 grams of the sample in a Soxhlet extractor in a paper capsule, mix with some ignited asbestos, and extract for 10 hours. Add 10 cc. of 4% alcoholic potash to the extract, and nearly evaporate the alcohol. Wash the residue into a platinum dish, evaporate, dry, and burn in a muffle at below redness for a few minutes, until all the fat is burned off. Extract the mass with hot, dilute nitric acid, and filter the solution. Ash the washed filter paper in the platinum dish to a white ash, treat this with dilute nitric acid, and filter into the first filtrate. Determine the phosphoric acid in the complete filtrate by the volumetric method[1].

(2) Heat 3 grams of the sample in an Erlenmeyer flask on the steam bath with 125 cc. of absolute alcohol for 2 hours. Place a funnel in the mouth of the flask to condense the alcohol vapors. Filter the solution, and wash the filter paper with a little hot absolute alcohol. Subsequently treat the filtrate as in Method 1.

Method 1 gave 1.323–1.328 per cent lecithin-phosphoric acid as P_2O_5, while Method 2 gave 1.312 per cent.

Method 2 gave practically the same results as Method 1. If it were possible to use Method 2 on dry products, it would have the advantages of shorter time of extraction, inexpensive apparatus and simplicity.

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 3.

H. C. Lythgoe[1] (State Department of Health, Boston, Mass.) states that strictly fresh eggs contain less than 1 mg. of ammonia per 100 grams of egg, but reasonably fresh eggs may contain as high as 2 mg. of ammonia per 100 grams of egg. When the ammonia content exceeds 4 mg. per 100 grams, the egg passes into the class of decomposed eggs. He finds that as a rule in December, January and February cold storage eggs compare favorably in ammonia content with the so-called "fresh", or "fresh western" eggs as sold in the stores at a price considerably above that asked for cold storage eggs.

As practically all of the ammoniacal nitrogen and acidity occur in the yolks, while most of the reducing sugars occur in the whites, it is necessary to know the amount of fat in broken-out eggs containing varying amounts of added yolk, in order to properly interpret the results. Table 4 gives the highest limits of ammonia and acidity allowable in broken-out eggs with varying amounts of added yolk. The acidity is calculated as cc. of N 10 acid for 100 grams of egg and the ammonia as mg. for 100 grams of egg.

TABLE 4.

Highest limits of ammonia and acidity allowable in broken-out eggs.

(Analyst, H. C. Lythgoe.)

FAT	AMMONIA	ACID
per cent	*mg. per 100 grams*	*cc. per 100 grams*
10	4.0	25.0
11	4.4	27.5
12	4.8	30.0
13	5.2	32.5
14	5.6	35.0
15	6.0	37.5
16	6.4	40.0
17	6.8	42.5
18	7.2	45.0
19	7.6	47.5
20	8.0	50.0
21	8.4	52.5
22	8.8	55.0
23	9.2	57.5
24	9.6	60.0
25	10.0	62.5
26	10.4	65.0
27	10.8	67.5
28	11.2	70.0
29	11.6	72.5
30	12.0	75.0
31	12.4	77.5
32	12.8	80.0
33	13.2	82.5

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[1] Mass. State Dept. Health, Monthly Bull., 1918, 5: 328.

In the Massachusetts Department of Health laboratory the comparisons shown in Table 5 were made.

TABLE 5.

Comparative results on different quality of eggs.

QUALITY OF EGGS	SOLIDS	FAT	AMMONIA	DEXTROSE	
			Folin method	U. S. Department of Agriculture method	Klein method
	per cent	*per cent*	*mg. per 100 grams*	*per cent*	*per cent*
In water glass:					
3 years	....	...	6.08	....	....
3 years	....	...	4.95	. .	....
2 years	....	...	7.24	....	....
Water glass	....	...	7.41	....	0.45
Water glass	....	...	7.41	....	0.53
Cold storage 2 years and considerably dried	40.84	10.	....	0.40	0.584
Water glass	26.70	14.	2.54	0.355	0.336
Commercial	28.60	16.	3.47	....	0.338
Commercial	27.62	14.	3.29	....	0.415

The referee tried both methods for ammonia, and relies on the Folin method. The principal disadvantage with the Hendrickson and Swan method is the great difficulty of cleaning the glassware. The Klein method for dextrose was found to be quicker than the United States Department of Agriculture method, and better where a large number of determinations was to be made at once. Several of the dextrose determinations by the United States Department of Agriculture method were unsatisfactory, because of faulty clarifying and filtering, but no trouble was found in the same sample using the Klein method.

No work was done by any one in the determination of heavy metals in dried eggs, although A. S. Thatcher (Loose-Wiles Biscuit Co., New York, N. Y.) suggested that the method of the Bureau of Animal Industry for gelatin might be useful.

The general opinion about tests for the determination of decomposition in dried eggs was that there were no satisfactory chemical tests. Some work done by the referee seemed to indicate that the acidity might be of some value as an indication of decomposition.

RECOMMENDATIONS.

It is recommended—

(1) That the Folin method for the determination of ammoniacal nitrogen in eggs be adopted as a tentative method.

(2) That further comparisons of Klein's method and that of the United States Department of Agriculture for the determination of dextrose be made with a view to adopting one of them as official.

(3) That further work be done on the methods as given for the determination of lecithin-phosphoric acid in dried eggs and alimentary pastes.

(4) That work be done on the determination of heavy metals in egg products.

(5) That work be done on methods for the detection of decomposition in dried eggs.

DISCUSSION OF REPORT OF REFEREE ON EGG PRODUCTS.

By H. W. Redfield (U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, New York, N. Y.).

Many of the conclusions and criticisms to be offered are based upon the results of an exhaustive investigation, conducted by the Bureau of Chemistry[1], on the correlation existing between analytical data and the quality of frozen egg products. One of the objects of this investigation was to perfect analytical methods by means of which a correct judgment might be reached as to the original condition of preserved foods, and it is recommended that the methods used for this work be designated the United States Department of Agriculture methods.

The method employed for the determination of ammonia nitrogen differs somewhat from the Folin method for ammonia, as described by the referee, page 507, resembling rather the method ascribed by the referee to Hendrickson and Swan, page 508. In practically every instance when four analysts determined the ammonia nitrogen in the same sample, results which did not vary from one another by more than a few hundredths of a milligram per 100 grams of sample were obtained, which is one of the crucial tests of a method.

In all of the 83 samples of fresh and frozen eggs examined the ammonia nitrogen was determined by the titration method[2], the Nesslerization method[3], and the phosphotungstic acid method[4]. The following conclusions were unanimously reached:

(1) The titration method for ammonia nitrogen[2] is accurate, reliable, and much to be preferred to any other where legal action may be involved.

(2) The Nesslerization method for ammonia nitrogen[3], while more

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[1] U. S. Dept. Agr. Bull. 846: (1920).
[2] *Ibid.*, 90.
[3] *Ibid.*, 92.
[4] *Ibid.*, 93

rapid, is not so accurate as the titration method. If it is employed for samples where legal action may be involved, it should be run in triplicate, and the average of the three determinations used.

(3) The phosphotungstic acid method[1] is good for rapid and approximately accurate work, but should not be used for samples where legal action may be involved.

The dextrose method described by the referee as the United States Department of Agriculture method, page 508, is almost like that used throughout the investigation with two exceptions. In the first place, the referee's method specifies the use of 1 cc. of acetic acid in the case of mixed egg. If by this he means yolky mixtures, he has neglected to state the quantity to be used for whole egg. Two cc. should be used for whole egg. Secondly, he says that the egg should coagulate in 10 minutes. Proper coagulation requires 15 minutes.

Necessary details in the method for ether extract, not mentioned in Swan's report, page 510, include the preparation of the material for extraction and the amount of vacuum in drying.

The method for indol and skatol differs from that of Swan in that the egg material is diluted before adding the acetic acid, using 40 cc. of acetic acid for white or whole egg, and 20 cc. for yolk, is taken up with water after the evaporation of the ether, and is then filtered before testing for indol or skatol. The vanillin test is a modification of the one given by V. E. Nelson[2]. It was considered essential to confirm for indol with a modification of the para-dimethylaminobenzaldehyde test and for skatol with an adaptation of the dimethylaniline test.

The directions given for the bacteriological examination[3] are not sufficiently exhaustive for egg products.

Swan's statement, page 511, that ammoniacal nitrogen tests are of little value for detecting the fact that decomposed eggs have been used in making dried egg products is open to question. Experiments made by the Illinois Division of Foods and Dairies indicate very strongly that even when dried in a vacuum of 29.6 inches on a commercial scale, practically none of the ammonia was removed from the egg material. It would seem that if ammonia could be removed by any commercial process it would be by this vacuum process.

Swan's contention, page 511, that the matter of detecting the use of decomposed eggs in making desiccated egg products should be vigorously attacked is well taken. Such an attack, however, can not be made in this country. It must be made in China. Conclusions drawn from samples prepared in the laboratory on a small scale would be of little

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[1] U. S. Dept. Agr. Bull. **846**: (1920), 93.
[2] *J. Biol. Chem.*, 1916, **24**: 528.
[3] U. S. Dept. Agr. Bull. **51**: (1914), 3.

value. The material to be studied must be handled on a commercial basis, and China is the only place now where that is being done.

The ammonia nitrogen figures, page 512, given by A. R. Todd (State Food and Drug Department, Lansing, Mich.), using both the Hendrickson and Swan method and the Folin method, for strictly fresh, store fresh, frozen 4 months, stale, small blood rings, green whites, moldy, and blood-rot eggs are all very much too low, probably because he did not use enough air pressure in aeration. The use of a cylinder $\frac{3}{8}$ inch in diameter for holding the material to be aerated, as recommended by the referee, page 507, makes it impossible to apply enough air pressure, as in a cylinder of that size all of the material would be promptly elevated out from the top if sufficient air pressure to do any good in a reasonable length of time was turned on. The cylinder should be much larger. the air pump should provide at least 10 pounds pressure per square inch, and there should be an equalizing tank between pump and cylinders to eliminate the effect of the pulsations of the pump.

The results secured by Todd, page 512, for what he calls dextrose (more properly "reducing substances calculated as dextrose") by the alleged United States Department of Agriculture method seem to be correct.

All of the substances in eggs capable of reducing Fehling's solution are not dextrose, as is well illustrated by the fact that if the reducing substances in a sample of whole egg, yolk, or white are determined, the material frozen and held for a year and the reducing substances again determined, there will be more at the end than at the start. Is that due to the formation of dextrose in the frozen material? Hardly. What probably happens is the breaking down of some of the ovomucoid, which is a glycoprotein and on hydrolysis yields a reducing substance, presumably chondroitin-sulphuric acid. This may break down into glucosamin or some similar substance. Todd's results, page 512, for dextrose by the Klein method are too high, probably because of charring on evaporation. His results for Babcock fat, page 512, are much higher than those obtained by any other analyst using the admittedly more accurate extraction method.

Whether Todd's general deduction, page 512, that the dextrose content is a more reliable indication of the age of eggs than ammonia nitrogen is true or not depends entirely upon the bacterial flora of the eggs and whether the predominating changes during aging are fermentative, proteolytic, or lipolytic. Since they may be any of these, the quality of eggs can not be judged by any one chemical determination. This is supported by Swan's statement, page 510, that the valuable methods for the detection of decomposition in eggs are total solids, ether extract, acidity of ether extract, ammoniacal nitrogen, reducing sugar, indol and skatol, bacteria, and, of course, physical appearance.

All of Todd's figures, page 512, for ammonia nitrogen, as determined by the Folin method, using different periods of aeration, in rots and strictly fresh eggs, are so low as to prove conclusively that the method prescribed by the referee, as operated by Todd, is absolutely unreliable.

H. C. Lythgoe's (State Department of Health, Boston, Mass.) statement, page 514, that, "When the ammonia content exceeds 4 mg. per 100 grams, the egg passes into the class of decomposed eggs", unquestionably should be amended to read, "has long since passed into the class of decomposed eggs".

His statement that it is necessary to know the amount of fat in broken-out eggs containing varying amounts of added yolk, in order to properly interpret ammonia nitrogen results, is true. The simplest way to do this is to plot the ammonia nitrogen results against the ether extract and calculate the formula value. Obviously this is unnecessary in the case of acidity of fat if the acidity is calculated to a per gram of fat basis.

The allowable limits set by Lythgoe, page 514, for ammonia nitrogen and acidity of fat in broken-out eggs with varying amounts of added yolk are entirely too high. His ammonia nitrogen figures give a clean bill of health to more than half of the samples known to be inedible when made up for the investigation conducted by the Bureau of Chemistry in August, when eggs are admittedly at their worst. Moreover, only two of the samples known to be inedible when made up would fall above Lythgoe's line of highest allowable results for acidity of fat; all the rest would pass. The only eggs excluded by Lythgoe's highest allowable limits for ammonia nitrogen and acidity of fat would be the most stinking of decomposed eggs; the moderately stinking eggs would not be excluded.

Some of the results reported from the Massachusetts State Department of Health laboratory, page 515, are curious. For example, 10 per cent of fat in eggs containing 40.84 per cent of solids, and 14, 16, and 14 per cent of fat, respectively, in eggs with 26.70, 28.60, and 27.62 per cent of solids seem rather unreasonable. In that laboratory, the Folin nitrogen method, as manipulated, apparently gave reasonable results, but the same can not be said of the Klein dextrose method. The statement of the referee, page 515, that his results show the Klein method to be better than the United States Department of Agriculture method seems unwarranted.

The referee's recommendation that the Folin method as described in the report be adopted as a tentative method should not be accepted by the association, because it evidently does not give concordant results in the hands of different workers, as proved by the result of the referee and Todd. The referee's recommendation that further comparison of the Klein and United States Department of Agriculture methods for dextrose be made with a view to adopting one of them as official, should not be accepted by the association unless the alleged United States Department of Agriculture method is first made correct.

REPORT ON GELATIN.

By C. R. Smith (Bureau of Chemistry, Washington, D. C.), *Referee.*

The work on gelatin for 1918 and 1919 involved a study of its polariscopic constants and a method for the determination of sulphites. The usefulness of the polariscopic study of gelatin is indicated in the study of jelly strength and certain other applications[1]. The estimation of sulphite by the present association method[2] is troublesome; the suggested method offers rapid results but needs to be tested for accuracy. Since the proportion of sulphites decreases with time, results should be obtained by the collaborator comparing the proposed diffusion method and the distillation method at the same time.

In determining the optical rotation of gelatin in equilibrium at 15°C., it is very important to control the temperature carefully for accurate results. In all cases, improvised constant temperature baths were used. In view of this, the results were fairly satisfactory.

POLARISCOPIC CONSTANTS OF GELATIN.

Prepare concentrations of 2 and 3 grams per 100 cc. of both samples, lettered S. C. S. and T. D. T., by soaking in 40–50 cc. of cold water, heating to about 50°C. for 15 minutes and making to volume at 35°C. Polarize at 35°C. in 200 mm. tubes.

Fill 100 mm. tubes of each concentration in duplicate to obtain the equilibrium rotation at 15°C. To avoid strains in the jellies, cool the solutions quickly to 10–15°C. and pour into cold dry tubes before jelly has been formed. Place the tubes in a constant temperature bath at 15°C. and leave overnight. Polarize the next day at 9 a. m., 12 m. and 4 p. m.

Tabulate the results after doubling the rotations at 15°C. to place all readings on 200 mm. tube basis and using Ventszke degrees as in saccharimetry as shown in Table 1.

In place of a constant temperature bath, place the tubes in a part of the ice chest registering between 12 and 16°C. overnight. Carefully control the temperature at 15°C. ± 0.4°C. the next day by immersion in a large volume of water maintained at 15°C. or carefully control in a dry container which is a poor conductor of heat an placed in a part of the ice chest which is near the correct temperature.

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[1] *J. Ind. Eng. Chem.*, 1920, **12:** 878.

[2] *Assoc. Official Agr. Chemists, Methods*, 1916, 150.

SULPHUR DIOXID—DIFFUSION METHOD.

Take 5 grams of powdered gelatin sample; add 100–150 cc. of ice water containing 3 cc. of 10% hydrochloric acid (1 to 3) and 10 grams of sodium chlorid. Gently mix and allow to stand in ice water for 2 minutes. Add starch paste of good quality and titrate with N/100 iodin until a blue color appears. Replace in the ice bath for 1 minute, remove, and titrate carefully until the color reappears. Repeat these operations until the color remains after standing for 1 minute with gentle agitation.

Report the number of cc. consumed and calculate the sulphur dioxid as mg. per kilo.

1 cc. N/100 I = 0.00032 gram of SO_2.

If possible, check the diffusion method with the distillation method[1], using a stream of inert gas and recovering the sulphur dioxid as barium sulphate.

TABLE 1.

Polarizations of Sample S. C. S.

ANALYST	POLARIZATION AT 35°C.		POLARIZATION AT 15°C.						ROTATION AT 15°C. / ROTATION AT 35°C.	
			9 A. M.		12 M.		4 P. M.			
	2 grams	3 grams	2 grams	3 grams	2 grams	3 grams	2 grams	3 grams	2-gram ratio	3-gram ratio
W. D. Richardson, Swift & Co., Chicago, Ill.	−13.7	−20.2	−29.7	−43.8	−29.5	−44.9	−29.1	−44.7	2.148	2.127
	.....	.....	....	−43.4	....	−43.4	....	−43.6	....	....
T. R. Tennant, United Chemical and Organic Products Co., Hammond, Ind.	−13.55	−20.5	−28.9	−43.4	−29.0	−43.5	−29.0	−43.4	2.14	2.12
R. Hertwig, U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, San Francisco, Calif.	−15.2	−20.2	−28.0	−40.4	−29.6	−44.8	−28.8	−46.0	1.89	2.15
							−31.4	−43.6	2.06	2.27
E. H. Berry, U. S. Food and Drug Inspection Station, Transportation Building, Chicago, Ill.	−13.6	−20.3	−31.0	−45.0	....	....	−25.0	−40.0	1.83	....
L. A. Salinger, U. S. Food and Drug Inspection Station, U. S. Custom House, Savannah, Ga.:										
October 4, 1919........	−13.85	−20.5	−27.7	−42.3	−28.0	−40.9	−28.7	−40.1	2.08	1.95
October 20, 1919........	−13.5	−20.75	−29.7	−45.0	−29.6	−44.2	−29.6	−43.1	2.19	2.08

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[1] *Assoc. Official Agr. Chemists. Methods*, 1916, 150.

TABLE 2.

Polarizations of Sample T. D. T.

ANALYST	POLARIZATION AT 35°C.		POLARIZATION AT 15°C.						ROTATION AT 15°C. / ROTATION AT 35°C.	
			9 A. M.		12 M.		4 P. M.			
	2 grams	3 grams	2 grams	3 grams	2 grams	3 grams	2 grams	3 grams	2-gram ratio	3-gram ratio
W. D. Richardson	−13.8	−21.1	−25.6	−39.2	−25.2	−38.7	−25.8	−39.5	1.847	1.856
	....	.....	−25.0	−40.0	−25.2	−38.6	−26.6	−39.0	....	....
T. R. Tennant..	−13.6	−20.5	−25.8	−39.0	−26.0	−39.0	−26.0	−39.0	1.89	1.90
R. Hertwig.....	−13.8	−20.2	−26.0	−38.8	−26.1	−39.3	−26.4	−39.9	1.913	1.975
	....	.....	....	....	....	....	−26.3	−39.3	1.906	1.945
E. H. Berry....	−13.7	−20.2	−27.0	−40.0	....	....	−24.0	−36.0	1.71	....
L. A. Salinger:										
October 4, 1919*	−14.0	−20.65	−26.0	−41.3	−26.2	−39.7	−25.7	−37.4	1.83	1.79
October 20, 1919	−14.1	−21.0	−26.5	−39.0	−27.1	−40.6	−27.2	−39.7	1.93	1.93

* Average.

TABLE 3.

Determination of sulphur dioxid in Sample S. C. S. by the diffusion method.

ANALYST	N/100 IODIN	SULPHUR DIOXID
	cc.	*mg. per kilo*
W. D. Richardson..............................	2.54 2.68	152.5 171.6
E. H. Berry....................................	2.9	185.8
R. Hertwig.....................................	3.5	220.0
L. A. Salinger (October 20, 1919)..................	1.2	76.8

Comparison can not properly be made between the results of different collaborators made at different times. The result of Salinger was obtained last and shows that over half of the sulphur dioxid had disappeared.

Richardson reported the sulphur dioxid on a good grade of glue as 4440, 4141, 4347 and 4231 mg. per kilo as compared with 3212, 3212, 3166 and 3102 mg. by the diffusion method.

COMMENTS.

Hertwig reports that the polariscope he used for the 35°C. readings did not read farther than −20°V. He estimated the 35°C. readings given for the 3-gram concentrations as probable values.

Salinger reports that warm, humid weather made readings difficult.

Richardson had no suitable cooler to maintain the required temperature.

Berry reports irregularity in ice chest temperature during the night and difficulty in making readings at the lower temperature.

REPORT ON EDIBLE FATS AND OILS.

By R. H. KERR (Bureau of Animal Industry, Washington, D. C.), *Referee.*

The work has consisted of the examination and criticism of the report of the Committee on Fats and Oils of the American Chemical Society[1]. This committee has recommended a set of uniform methods for the sampling and analysis of fats and oils to be used by members of the American Chemical Society. The report shows divergence in some respects from the methods adopted by this association but appears unlikely to lead to any actual inconvenience or confusion except in one respect. This is in the method proposed for the determination of the iodin number. The committee has adopted the Wijs method for the determination of the iodin number[1] instead of the well-tried Hanus method[2], now official in this association and in the American Society for Testing Materials. The committee appears to consider that the Wijs method has certain advantages which make its adoption advisable, even at the cost of causing the confusion which will inevitably result from the use of two methods which do not always give identical results by the different societies. In the opinion of your referee, the advantages of the Wijs method are not substantial and the adoption of the Hanus method by this association was justified in the light of present information as well as information available at the time of its adoption, and such advantages as are possessed by the Wijs method are more than overbalanced by the disadvantages of having two different official methods. Since, however, the Wijs method has been definitely adopted by the Fats and Oils Committee and will be official in the American Chemical Society, this condition of confusion must inevitably occur and can not be prevented by this association. The following recommendations are offered for the purpose of reducing this confusion as much as possible:

RECOMMENDATIONS.

It is recommended—

(1) That the Hübl method for the determination of the iodin number[3] be dropped from the official methods.

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[1] *J. Ind. Eng. Chem.*, 1919, **11**: 1161.
[2] *Assoc. Official Agr. Chemists, Methods,* 1916, 305.
[3] *Ibid.*, 304.

(2) That the use of the Wijs method, as adopted by the American Chemical Society, be made optional under the official methods.

(3) That all reports of iodin numbers specify the method used and where no method is specified it shall be understood that the determination was made by the Hanus method.

REPORT ON SPICES.

By H. E. SINDALL[1] [2] (Austin, Nichols & Co., Inc., New York, N. Y.), *Referee on Spices and Other Condiments.*

The work has been a continuation of the study of the referee's modification of the distillation method for water in whole spices and the tentative method for the determination of volatile oil in mustard seed[3].

MOISTURE IN PEPPER AND CLOVES.

Samples of Zanzibar cloves and Lampong pepper were sent to six collaborators, with a copy of the method, a photograph of the apparatus used by the referee, and instructions to follow the method as written and to determine the moisture also by the official method[4]. Reports were received from three collaborators: M. B. Porch, H. J. Heinz Co., Pittsburgh, Pa.; W. B. Smith, Armour & Co., Kansas City, Kans.; and F. M. Boyles, McCormick & Co., Inc., Baltimore, Md. The following method was sent:

Place 50 grams of whole spice in a distillation flask with 150 cc. of kerosene; whirl the flask several times to bring the oil in contact with each particle of spice. Place the flask on an asbestos board, cut so that the bottom of the flask extends below the surface. Place a wire gauze with an asbestos center about ½ inch below the bottom of the flask. The object is to keep the flame from direct contact with the flask. The asbestos board serves to keep the heat uniform. Connect the flask directly with the vertical condenser. Insert a thermometer through the stopper of the distillation flask extending into the oil. Adjust the flame so that about 20 minutes will be required to reach the temperature of 170°C., and collect the distillate in a graduated cylinder or burette. Extinguish the flame, after which the thermometer will show a slight gradual increase in temperature. As soon as the water stops dropping from the condenser tube, which usually requires 4–6 minutes, the operation is complete. Multiply the volume of the water layer by 2 to obtain the percentage of moisture. The results obtained are shown in Table 1.

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[1] Presented by L. C. Mitchell.
[2] Present address, Francis H. Leggett & Company, New York, N. Y.
[3] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 149.
[4] *Assoc. Official Agr. Chemists, Methods*, 1916, 79.

TABLE 1.

Moisture in pepper and cloves.

ANALYST	MODIFICATION METHOD		OFFICIAL METHOD*	
	Cloves	Pepper	Cloves	Pepper
	per cent	*per cent*	*per cent*	*per cent*
M. B. Porch	6.4	7.4	8.94	9.98
W. B. Smith	7.2 7.4	7.0 7.2	9.13 6.21	12.30 10.09
F. M. Boyles	7.5 7.5	8.2 8.5	8.94	11.59 ....
H. E. Sindall	7.4	8.1	8.72	9.62

* *Assoc. Official Agr. Chemists, Methods,* 1916, 79.

DISCUSSION AND CONCLUSIONS.

These results show a difference of 1.1 per cent in both cloves and pepper by the distillation method, and a difference of 2.92 per cent in cloves and 2.68 per cent in pepper by the official method[1]. These results demonstrate that analysts can obtain closer results by the distillation method than by the official method.

VOLATILE OIL IN MUSTARD SEED.

Samples of ground California brown mustard seed and ground charlock seed were sent to five collaborators with the request that they be examined by the method printed in Service and Regulatory Announcement No. 20[2] and also by the method given in Leach[3]. The following method was used:

> Place 5 grams of the ground seed (No. 20 powder) in a 200 cc. flask, add 100 cc. of water, stopper tightly, and macerate for 2 hours at about 37°C. Then add 20 cc. of U. S. P. alcohol (95%), and distil about 60 cc. into a 100 cc. volumetric flask containing 10 cc. of 10% ammonium hydroxid solution, taking care that the tip of the condenser dips below the surface of the solution. Add 20 cc. of N/10 silver nitrate solution to the distillate, set aside overnight, heat to boiling on a water bath (in order to agglomerate the silver sulphid), cool, make up to 100 cc. with water, and filter. Acidify 50 cc. of the filtrate with about 5 cc. of concentrated nitric acid and titrate with N/10 ammonium thiocyanate, using 5 cc. of 10% ferric ammonium sulphate solution for an indicator. Each cc. of N/10 silver nitrate consumed equals 0.004956 grain of allyl isothiocyanate. The results obtained are shown in Table 2.

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[1] *Assoc. Official Agr. Chemists, Methods,* 1916, 79.
[2] U. S. Dept. Agr., S. R. A., Chemistry, **20**: (1917), 59.
[3] A. E. Leach. Food Inspection and Analysis. 3rd ed., 1913, 457.

TABLE 2.

Volatile oil in mustard seed.

ANALYST	S. R. A. METHOD		LEACH	
	California seed	Charlock seed	California seed	Charlock seed
	per cent	*per cent*	*per cent*	*per cent*
W. B. Smith	0.95 0.95	0.258	0.81	0.17 ....
F. M. Boyles	0.856 0.856	0.192 0.192	0.893 0.916	0.207 0.197
Louis Schwartz, U. S. Food and Drug Inspection Station, Transportation Building, Chicago, Ill.	0.86	....	0.81	....

RECOMMENDATIONS.

It is recommended—

(1) That the modification of the distillation method for the determination of water in whole spices be further studied for one year, with particular reference to temperature necessary to drive over all the water, and length of time required.

(2) That the tentative method for the determination of volatile oil in mustard seed be adopted as an official method.

No report on cacao products was made by the referee.

REPORT ON COFFEE.

By H. A. LEPPER (Bureau of Chemistry, Washington, D. C.), *Referee.*

At the last meeting of the association, the Fendler-Stüber method for caffein in coffee was tentatively adopted with the further recommendation that the method be studied with a view to its adoption as official[1]. Before adoption as an official method, it was deemed advisable that the method be studied in detail, as well as through the analyses of collaborative samples. The important steps in the procedure, the extraction including the filtration and the manipulation of the filtrate, the oxidation with potassium permanganate and subsequent treatment with hydrogen peroxid, and the drying of the caffein, were studied. These steps were considered in their reverse order as verification of each succeeding last step was necessary before the preceding step could be studied.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 216.

DRYING THE CAFFEIN.

Conflicting statements have appeared in the literature regarding the drying of caffein previous to weighing. Beitter[1] recommends drying at 85°C., owing to a loss of caffein at higher temperatures. Hartwich and Du Pasquier[2] found in drying caffein at 100°C. a loss of 0.4 per cent per hour. Blyth[3] claims that caffein begins to sublime at 78.8° to 79.4°C. and hence can not be dried at 100°C. Many authors[4], however, give data showing that there is practically no loss when a chloroform solution of caffein is evaporated to dryness and dried for 30 minutes in a water oven, as directed in the method.

In view of these conflicting claims, experiments were conducted to determine if the procedure of drying, as directed in the method, showed a loss of caffein. Caffein dried to constant weight in a water oven was weighed and dissolved in 150 cc. of chloroform in a tared flask. After the chloroform was evaporated off on the steam bath, the residue was dried for 30 minutes in the water oven. Three separate quantities of caffein, 0.3304, 0.3095 and 0.2004 gram gave a weight of 0.3303, 0.3096 and 0.2004 gram, respectively. It was found that the chloroform could be evaporated to a small volume and the solution transferred to a small beaker with chloroform, the evaporation completed and the residue dried for 30 minutes in the water oven, without loss of caffein. The length of time used in drying was always found to be sufficient to obtain constant weight. This procedure of drying has also been recommended recently by Power and Chesnut[5]. The advantage of transferring the residue to a small beaker allows the weighing to be conducted in a small vessel, which is of special advantage on a humid day. The method was modified to include this procedure. All weighings of caffein reported in the remainder of this report were so conducted.

THE OXIDATION WITH POTASSIUM PERMANGANATE.

Fendler and Stüber[6] adopted the permanganate purification of Lendrich and Nottbohm[7] and accepted the results of their investigation as to the negative action of potassium permanganate on caffein without further verification. The latter authors were the first to use permanganate for the purification of caffein but had some doubt as to their priority. Their doubt was founded on a note[8] which stated that a method of Markownikoff presented to the Russian Chemical Society used man-

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[1] *Ber. pharm. Ges.*, 1901, **11**: 348.

[2] *Apoth. Ztg.*, 1909, **24**: 120.

[3] M. W. Blyth. Foods, Their Composition and Analysis. 6th ed., 1909, 324.

[4] *Ber. pharm. Ges.*, 1902, **12**: 250.
Pharm. J. Trans., 1892–3, 3rd ser., **23**: 213.
Pharm. Review, 1905, **23**: 305.
Forschb. über Lebensm., 1897, **4**: 78.

[5] *J. Am. Chem. Soc.*, 1919, **41**: 1298.

[6] *Z. Nahr. Genussm.*, 1914, **28**: 9.

[7] *Ibid.*, 1909, **17**: 241.

[8] *Bull. soc. chim.*, 1877, **27**: 266.

ganese as a purifying agent in the determination of thein in tea. However, a similar note[1] states that the method of Markownikoff used magnesia. Neither of these notes made reference to an original article and Lendrich and Nottbohm state that they were unable to consult original Russian publications to clarify the discrepancy. An article by Markownikoff[2], published in 1876, on the determination of thein in tea was found by the referee in which the use of magnesia was recommended. Other attempts to find publications wherein the action of dilute potassium permanganate on caffein in neutral solution was studied met with no success. In view of the fact that the work of Lendrich and Nottbohm appeared to be the only record of study of this procedure, it was deemed advisable to verify, if possible, their conclusions. Accordingly, a solution of 4 grams of dried caffein in 1 liter of water was prepared. Three 50 cc. aliquots were treated with 20 cc. of 1 per cent potassium permanganate for 15 minutes at room temperature, as directed in the Fendler-Stüber method. After the excess of permanganate was destroyed with hydrogen peroxid and the solution heated, it was filtered hot with suction and the filter washed with hot water. The solution was then extracted with seven 25 cc. portions of chloroform and the combined extractions evaporated and weighed. Two 50 cc. portions were similarly extracted and weighed to establish a blank on the caffein solution. In the case of the untreated solutions, 0.2004 and 0.2003 gram of caffein was recovered, while in the case of the treated solutions 0.1979, 0.1977 and 0.1972 gram of caffein was recovered. In each case the manganese dioxid which was filtered off was dissolved in acetic acid, the acid neutralized and the solution extracted with chloroform to determine if caffein was retained in the manganese dioxid. No weighable residue was obtained in any case. Moreover, the solutions of caffein that had been treated and extracted were re-extracted with four, 25 cc. portions of chloroform and no weighable residue was obtained. It is indicated, therefore, that a loss of from 2.1 to 2.8 mg. of caffein was due to oxidation which, on the calculated quantity present before treatment (0.2000 gram) is about 1 per cent. A loss of this magnitude in the quantity of caffein present in coffee would mean that the result would be 0.01 to 0.015 per cent too low.

Permanganate, as is well known, acts on a great number of organic compounds to a greater or less degree, depending on the concentration, temperature, time and the reaction of the menstrum. Accordingly, experiments were performed to establish if the loss observed above was a real loss. As the purification of the caffein is carried out in neutral solution, no study was made in acid or alkaline menstrums. The concentration used in the method is just sufficient to give an excess of per-

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[1] *Ber.*, 1876, **9** (II): 1312.
[2] *J. Russ. Phys. Chem. Soc.*, 1876, **8**: 226.

manganate with the quantity of sample used and therefore no study was made with a view to changing this factor.

Weighed quantities of caffein were dissolved in 80 cc. of water, treated with 20 cc. of 1 per cent potassium permanganate and allowed to react at room temperature for different periods of time. At the end of the time hydrogen peroxid was used, the caffein extracted and weighed. The results are given in Table 1.

TABLE 1.

Effect of time on action of potassium permanganate on caffein.

TIME OF REACTION	CAFFEIN USED	CAFFEIN RECOVERED	LOSS
hours	*gram*	*gram*	*gram*
¼	0.2013	0.1982	0.0031
½	0.2021	0.1960	0.0061
¾	0.2016	0.1959	0.0057
1	0.2016	0.1927	0.0089
48	0.2000	0.1319	0.0681
48	0.2000	0.1389	0.0611

Weighed quantities of caffein in 80 cc. of water were also treated with 20 cc. of 1 per cent potassium permanganate at the temperatures of ice and steam baths. After reacting for the time specified, the excess of potassium permanganate was destroyed with hydrogen peroxid, extracted and weighed. The hydrogen peroxid was added to the cold or hot solution, as the case might be. The results are given in Table 2.

TABLE 2.

Effect of temperature on action of potassium permanganate on caffein.

CONDITION OF REACTION	TIME	CAFFEIN USED	CAFFEIN RECOVERED	LOSS
	minutes	*gram*	*gram*	*gram*
Ice bath	15	0.2000	0.1999	0.0001
Ice bath	15	0.2000	0.2005	0.0000
Ice bath	30	0.2000	0.2004	0.0000
Ice bath	60	0.2000	0.2000	0.0000
Ice bath	60	0.2000	0.2000	0.0000
Ice bath	30*	0.2000	0.1998	0.0002
Steam bath	15	0.2000	0.1350	0.0650
Steam bath	15	0.2000	0.1403	0.0597

* Used 40 cc. of potassium permanganate.

The results given in Tables 1 and 2 show conclusively that permanganate of the concentration used does act on caffein, especially when the temperature is higher or the time of reaction longer than directed in the method. This would indicate that the loss found when the reaction is allowed to progress, as directed in the method, is a real loss. This conclusion is not in harmony with that of Lendrich and Nottbohm. Results of their investigation of the action of permanganate on caffein in water solution indicate no action at room temperature for 30 minutes. However, the procedure adopted by them differed from the one used in this investigation, as they calculated their caffein recovery on the nitrogen determination only and did not weigh the dry residue. It is possible that the action of permanganate is such that the nitrogen content is not changed, in which case the caffein calculated from nitrogen would indicate a theoretical recovery. Moreover, the loss reported in this article is of a magnitude such as Lendrich and Nottbohm, judging from their results, considered within the limits of error of the nitrogen determination.

Although the error introduced by the purification of the caffein at room temperature is well within the experimental error of the method, it seemed advisable to try the modification providing for the purification at the temperature of the ice bath, as at this temperature no loss was detected. The value of this modification will be discussed with the discussion of the collaborative results.

EXTRACTION OF THE CAFFEIN FROM THE COFFEE.

The method as adopted in 1917 recommends the extraction of 10 grams of coffee after the addition of 10 grams of 10 per cent ammonium hydroxid with 200 grams of chloroform by shaking for 30 minutes with subsequent filtration and weighing of 150 grams of the filtrate. Fendler and Stüber give no data to show that the 150-gram aliquot is equivalent to 7.5 grams of the sample. To accomplish the weighing of 150 grams of the filtrate without evaporation and coincident concentration of the filtrate is tedious. Evaporation of the filtrate, of course, would cause high results. It appeared to be simpler and more accurate to catch and weigh the filtrate in a flask previously weighed with stopper. In order to test the accuracy of this procedure, two portions of caffein of 0.2 gram each were dissolved in 200 grams of chloroform and the entire solution in each case was poured, after chilling in ice, on a 24 cm. folded filter and covered with a watch glass, the filtrate being collected in a tared flask surrounded by ice. After the continuous running of the filtrate ceased, the flask was stoppered and weighed. The caffein, after evaporation of the chloroform, was dried and weighed. The caffein as weighed and as calculated from the weight of the chloroform aliquot is given in Table 3 with the difference between the theoretical and practical results. Also, two samples of coffee were extracted with 200 grams of chloroform and the filtrate collected as just described. The residue, with filter paper in each case, was transferred to a Soxhlet extractor, care being exercised to transfer all of the excess of chloroform which was not collected as filtrate. The residue was then extracted for 12 hours with chloroform. The caffein in the weighed filtrate and in the extract was then determined as directed in the method. Results were obtained which gave the total caffein in the coffee, the portion in the filtrate and the portion in the extract. From the weight of the aliquot filtrate the proportion of caffein which should be in the filtrate could be calculated and compared with the quantity actually found. As the residues were of doubtful purity, nitrogen was determined by the Kjeldahl-Gunning-Arnold method and the caffein calculated. The results are given in Table 4.

TABLE 3.

Recovery of caffein from chloroform solution by the modified method of collecting the chloroform filtrate.

WEIGHT OF FILTRATE	WEIGHT OF CAFFEIN	CALCULATED WEIGHT OF CAFFEIN	DIFFERENCE
grams	*gram*	*gram*	*gram*
194.7	0.1942	0.1947	−0.0005
195.1	0.1955	0.1951	+0.0004

TABLE 4.

Recovery of caffein from coffee by the modified method of collecting the chloroform filtrate.

WEIGHT OF FILTRATE	WEIGHT OF CAFFEIN*			CALCULATED WEIGHT OF CAFFEIN IN FILTRATE	DIFFERENCE
	In filtrate	In Soxhlet extraction	Total in coffee		
grams	*gram*	*gram*	*gram*	*gram*	*gram*
132.28	0.0747	0.0328	0.1075	0.0744	+0.0003
138.45	0.0777	0.0328	0.1105	0.0765	+0.0012

* Calculated from nitrogen by factor 3.464.

Consideration of the results given in Tables 3 and 4 leads to the conclusion that the method of extraction, filtration and manipulation of the filtrate, as given in the preceding paragraph, gives a representative aliquot of the total caffein present. Moreover, no appreciable error will be caused through concentration of the chloroform extract by evaporation if the procedure is carefully followed. The method was accordingly modified to provide this procedure.

COLLABORATIVE RESULTS.

Two samples of coffee, a roasted Bogata and a green Santos, were sent to the collaborators with a copy of the method modified as indicated. An explanatory sheet explaining the changes, with suggestions on manipulation, accompanied the method. The manner of filtration of the excess of manganese dioxid after the purification of the caffein was changed to provide for filtration on a Gooch crucible with suction instead of on paper. This was found to be quicker and more satisfactory for washing the precipitate. Caution was requested with respect to having the hydrogen peroxid acetanilid free. This chemical is soluble in chloroform and also contains nitrogen. High results would be caused by the introduction of acetanilid with the hydrogen peroxid. If an acetanilid-free reagent can not be obtained, an extraction with chloroform should be made to remove the objectionable substance. The chloroform used in this investigation was found to leave considerable residue on evaporation. Distillation removed this objectionable feature.

TABLE 5.
Results of collaborators.

COLLABORATORS	CAFFEIN IN ROASTED COFFEE			CAFFEIN IN GREEN COFFEE		
	Gravimetric	N × 3.464	Difference	Gravimetric	N × 3.464	Difference
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
M. L. Offutt, Bureau of Chemistry, Washington, D. C.	1.38	1.24	0.14	1.43	1.30	0.13
	1.41	1.28	0.13	1.44	1.36	0.12
D. B. Scott, Bureau of Chemistry, Washington, D. C.	1.23	1.17	0.06	1.26	1.08	0.18
C. S. Purcell, U. S. Food and Drug Inspection Station, U. S. Appraiser's Stores, Boston, Mass.	1.35	1.21	0.14	1.42	1.37	0.05
	1.34	1.21	0.13	1.43	1.35	0.08
Louis Schwartz, U. S. Food and Drug Inspection Station, Transportation Building, Chicago, Ill.	1.11	0.97	0.04	1.26	1.06	0.20
	1.12	1.01	0.11	1.26	0.99	0.27
H. R. Smith, U. S. Food and Drug Inspection Station, Park Avenue Building, Baltimore, Md.	1.17	1.07	0.10	1.37	....	....
	1.17	1.09	0.08	1.26	1.13	0.13
	1.10	....	....	1.25	1.16	0.07
	....	....	....	1.31	1.16	0.15
H. A. Lepper...........	1.18	1.13	0.05	1.36	1.27	0.09
	1.20	1.15	0.05	1.32	1.25	0.07
C. P. Lathrop, U. S. Food and Drug Inspection Station, Old Customhouse, St. Louis, Mo.	1.18	1.14	0.04	1.28	1.25	0.03
	1.25	1.18	0.07	1.29	1.24	0.05
F. L. Elliott, U. S. Food and Drug Inspection Station, U. S. Customhouse, New Orleans, La.	1.27	1.22	0.05	1.33	1.29	0.04
	1.25	1.17	0.08	1.32	1.26	0.06

These results show conclusively that the purity of the caffein when judged from the nitrogen determination is not equal to that of the caffein obtained in 1917[1] when the temperature of the purification with permanganate was not reduced with ice. One of the chief objects of the collaborative work this year was to determine if the modification calling for lowered temperature would produce a caffein of high purity. The error of 0.01 to 0.015 per cent caused by carrying out the purification at room temperature is within the error of the method itself and well within the personal equation as shown by the results of the collaborators. In view of this fact and of the relatively low purity of the caffein obtained by the cold process, it is believed advisable to retain the details of the method in this respect as adopted tentatively in 1917[1]. However, no reason is apparent why the other modifications on the extraction and filtration of the extract and on drying the caffein should not be included to insure greater accuracy and ease of manipulation.

The method as presented to the association this year would then read as follows:

FENDLER-STÜBER METHOD FOR CAFFEIN IN COFFEE (MODIFIED).

Pulverize the coffee to pass without residue through a 1 mm. sieve. Treat a 10-gram sample with 10 grams of 10% ammonium hydroxid and 200 grams of chloroform in a glass-stoppered bottle, shake continuously by machine or hand for 30 minutes and chill in an ice bath. Pour the entire contents of the bottle on a 24 cm. folded filter, covering immediately with a watch glass. Collect the filtrate with the funnel resting directly in the neck of a flask (previously weighed with stopper) having the flask surrounded with ice. Stopper as soon as the solution ceases to run from the funnel in a continuous stream and weigh. Evaporate on the steam bath, removing the last chloroform with a current of air. Digest the residue with 80 cc. of hot water for 10 minutes on the steam bath, with frequent shaking, and let cool. Treat the solution with 20 cc. (for roasted) and 10 cc. (for unroasted) of 1% potassium permanganate and let stand 15 minutes at room temperature with occasional shaking. Add 2 cc. of 3% hydrogen peroxid (containing 1 cc. of glacial acetic acid in 100 cc.). If the liquid is still red or reddish add hydrogen peroxid, 1 cc. at a time, until the excess of potassium permanganate is destroyed. Place the flask on a steam bath for 15 minutes and add 0.5 cc. portions of hydrogen peroxid until the liquid ceases to become lighter. Cool and filter by suction through a Gooch crucible, washing with cold water. Transfer the filtrate to a separatory funnel and extract six times with 25 cc. portions of chloroform. Evaporate the combined chloroform extracts to a small volume, transfer to a tared beaker, finish evaporation, dry at 100°C. to constant weight (30 minutes is usually sufficient) and weigh the residue as caffein. The weight of the caffein, multiplied by 2000 and divided by the weight of the chloroform aliquot obtained from the first filtration, equals the percentage of caffein in the 10-gram sample. Test the purity of the residue by determining nitrogen and multiplying by the factor 3.464.

RECOMMENDATIONS.

It is recommended—

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[1] *J. Assoc. Official Agr. Chemists*, 1920, 4: 211.

(1) That the Fendler-Stüber method, as modified, page 533, be continued as a tentative method.

(2) That further study be made of the method before it is adopted as official or superseded.

F. B. Power (Bureau of Chemistry, Washington, D. C.) called attention to work conducted by him and V. K. Chesnut, also of the Bureau of Chemistry, which resulted in an improved method for the quantitative determination of caffein in vegetable material, and also to *ilex vomitoria* as a native source of caffein.

REPORT ON TEA.

By E. M. BAILEY (Agricultural Experiment Station, New Haven, Conn.), *Referee.*

CAFFEIN.

In 1915 the referee on tea and coffee confined his attention to methods for the determination of caffein. Data for this determination were presented by the Fuller, Stahlschmidt, and modified Stahlschmidt methods in case of tea, and by the Fuller, Gorter, and modified Stahlschmidt methods in case of coffee[1]. In 1916 the referee confined his attention to coffee, and in 1917 the report on tea referred only to microscopical methods as applied to tea examination.

In the work on coffee during the last two or three years particular attention has been given to a comparison of the Fendler-Stüber method with the modified Stahlschmidt method. This suggested to the present referee the advisability of comparing these two methods in the case of tea. Unfortunately, promised collaboration did not materialize, so that the data now presented are confined to results obtained in the writer's laboratory.

Twelve samples of the United States standard teas for 1918 to 1919, furnished through the courtesy of the Collector of Customs of the Port of New York, were used in the comparative trials. The results therefore are comprehensive so far as different types of teas are concerned.

The details of the modified Stahlschmidt method, as given in the present tentative method[2], were followed. They are the same as those given by J. M. Bartlett[3] (Agricultural Experiment Station, Orono, Me.), except that the initial boiling is for 2 instead of 3 hours, and the drying of the caffein residue is done at 75°C. instead of at 100°C. The Fendler-Stüber method as directed for coffee in 1917[4], was followed without modification.

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[1] *J. Assoc. Official Agr. Chemists*, 1917, **3**: 21.
[2] *Assoc. Official Agr. Chemists, Methods*, 1916, 332.
[3] *J. Assoc. Official Agr. Chemists*, 1917, **3**: 22.
[4] *Ibid.*, 1920, **4**: 213.

The results for caffein as obtained by C. E. Shepard (Agricultural Experiment Station, New Haven, Conn.), using these two methods, are given in Table 1.

TABLE 1.

Caffein determination in tea.

(Analyst, C. E. Shepard.)

NAME AND STANDARD NUMBER	MODIFIED STAHLSCHMIDT METHOD		FENDLER-STÜBER METHOD	
	Gravimetric	From nitrogen	Gravimetric	From nitrogen
	per cent	*per cent*	*per cent*	*per cent*
Formosa Oolong, 1	2.20	2.03	2.16	2.12
Foochow Oolong, 2	2.54	2.44	2.57	2.54
Congou, 3	1.97	1.89	1.97	1.93
Ceylon, 4	2.96	2.77	2.81	2.79
Gunpowder Green, 5	1.86	1.73	1.81	1.76
Young Hyson Green, 6	1.68	1.54	1.65	1.63
Pan Fired Japan, 7	2.00	1.94	2.07	2.00
Basket Fired Japan, 8	2.07	2.01	2.13	2.11
Japan Dust, 9	2.09	1.94	2.18	2.13
Scented Orange Pekoe, 10	2.71	2.63	2.82	2.73
Scented Canton, 11	2.93	2.81	2.96	2.91
Canton Oolong, 12	3.10	2.96	3.27	3.20

From the standpoint of the Fendler-Stüber method, the results are uniformly higher for caffein from nitrogen, and the gravimetric figures are generally higher. The variation does not exceed 0.1 per cent, except in five cases out of a possible twenty-four, and in only one instance does it exceed 0.2 per cent. The caffein residues are slightly purer. The inherent errors, principally the weighing of a volatile solvent under varying conditions of temperature and pressure, tend towards high results. The hydrogen peroxid used must be assayed for acetanilid, and a proper correction made if it is present.

A study of the results of collaborative work on coffee for 1917 shows, by the Fendler-Stüber method, variations in results for caffein from nitrogen about evenly distributed between plus and minus. The gravimetric results are uniformly lower. Variations exceed 0.1 per cent in thirteen cases out of a possible thirty, but in only five instances do they exceed 0.2 per cent.

Through the courtesy of H. A. Lepper (Bureau of Chemistry, Washington, D. C.), referee on coffee, it was possible to compare the Fendler-Stüber method, as modified by him this year, with the other methods in the case of two samples of standard teas. The results, obtained by Shepard, are given in Table 2.

TABLE 2.

Comparison of methods for determination of caffein in tea.

TEA NUMBER	MODIFIED STAHLSCHMIDT METHOD		FENDLER-STÜBER METHOD		FENDLER-STÜBER MODIFIED METHOD	
	Gravimetric	From nitrogen	Gravimetric	From nitrogen	Gravimetric	From nitrogen
	per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
7	2.00	1.94	2.07	2.00	2.11	2.11
8	2.07	2.01	2.13	2.11	2.15	2.13

With very few exceptions the results by the different methods agree within the limits of reasonable analytical error. Total nitrogen determinations at the hands of the same number of analysts would agree no better.

The use of any solvent other than boiling water for the extraction of caffein seems unnecessary. On this point an old edition of Allen[1] says, "No Soxhlet extractor or similar arrangement is so effective or rapid as actual boiling with water". It has been shown[2] that 84 to 96 per cent of the caffein in tea is extracted by an ordinary household method of tea infusion.

On account of its simplicity and accuracy, a procedure practically identical with the Stahlschmidt method was adopted by G. L. Spencer[3] in his investigations on tea thirty years ago. The Stahlschmidt method has been before this association for criticism for the past five or six years, during which time a number of other methods have been considered and discarded. Judging from the data herein reported, it is the opinion of your referee that the Fendler-Stüber method possesses no marked advantage over it, in so far as results are concerned, and is much less desirable with respect to the manipulation involved.

WATER EXTRACT.

The present tentative method for determining water extract in tea[4] involves filtering 200 cc. of the extract through a tared filter, and washing with about 300 cc. of water. The procedure is generally slow and tedious, because of clogging of the filter. The following adaptation of the procedure[5], successfully used in the Connecticut Agricultural Experiment Station laboratory for many years for crude fiber filtrations, was employed:

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[1] Lab. Can. Inland Rev. Dept. Bull. **24**: (1891).
[2] Conn. Agr. Expt. Sta. Bull. **210**: (1918), 185.
[3] U.S. Bur. Chem. Bull. 13, (VII): (1892).
[4] *Assoc. Official Agr. Chemists, Methods*, 1916, 335.
[5] Conn. Agr. Expt. Sta. Bull. **210**: (1918), 182.

To 2 grams of the original (unground) sample in a 500 cc. Erlenmeyer flask add 200 cc. of hot water, and boil for 1 hour, having the flask fitted with a glass tube condenser. Replace the water lost by evaporation, if necessary. Fill the flask with cold water, and allow the suspended material to settle. Siphon off the supernatant liquid, filtering it through a linen filter. (An inverted thistle tube or other suitable tube, over the orifice of which is tied a piece of fine linen, introduced into the Erlenmeyer and connected with suction, serves the purpose satisfactorily.) Transfer the residue in the flask to a tared filter, allow to drain, and wash 3 times with water. Dry in the funnel until the excess of water is removed. Finally transfer the filter and contents to a tared weighing bottle, and dry to constant weight at 100°C.

The percentage of insoluble leaf, plus the percentage of moisture in the sample, subtracted from 100 per cent, gives the percentage of water extract.

A comparison of results by the tentative and modified methods reported by C. E. Shepard and M. A. D'Esopo (Agricultural Experiment Station, New Haven, Conn.) is given in Table 3.

TABLE 3.

Water extract in tea.

(Analysts, C. E. Shepard and M. A. D'Esopo.)

SAMPLE NUMBER	TENTATIVE METHOD	MODIFIED METHOD	DIFFERENCE BY MODIFIED METHOD
	per cent	*per cent*	*per cent*
10517	38.12	38.09	−0.03
10518	35.04	35.15	+0.11
10519	29.56	29.05	−0.51
10520	32.13	32.63	+0.50
10521	35.36	35.49	+0.13
10522	36.13	36.75	+0.62
10523	33.02	33.27	+0.25
10524	35.08	35.60	+0.52

The amount of extract obtained from tea obviously depends upon the degree of fineness of the sample, the volume of water used, and the length of the boiling period. The essential conditions of the tentative method are not materially changed in the procedure as here given, and the results obtained by the two methods are found to be in satisfactory agreement. This procedure is suggested as a substitute for the present tentative method.

DETERMINATION OF TANNIN.

A comparison of the cinchonin sulphate method with the present tentative method for the determination of tannin in tea was planned, but has not been completed The subject is suggested for future study.

RECOMMENDATIONS.

It is recommended—

(1) That the modified Stahlschmidt method, as it now appears tentatively[1], with the exception that the caffein residue be dried at 100°C. instead of at 75°C., be made official for the determination of caffein in tea.

(2) That the method for the determination of water extract as given in this report be substituted for the present tentative method.

(3) That methods for the determination of tannin be studied next year. The cinchonin sulphate method[2] particularly is suggested.

REPORT ON BAKING POWDER[3].

By H. E. PATTEN[4] (Bureau of Chemistry, Washington, D. C.), *Referee.*

Due to the cancellation of the annual meeting of the association for 1918, this report covers the collaborative work done during both 1918 and 1919. The main subjects considered were the determination of lead and of fluorin in phosphates and in baking powders. For lead the Corper-Bryan electrolytic[5], and the Chittick[6] methods were used. The Wagner-Ross method for fluorin[7] presented in the referee's report for 1917[8] has been studied with a view to adoption.

LEAD.

Corper-Bryan method.—The results of the collaborators show that with experienced operators very accurate results are obtained. In spite of the cost of instruments and platinum electrodes, the method is the cleanest and best available at present. Care must be taken not to get the thin paste resulting from the acidification and hydrolysis of the baking powder too strongly acid; it should be remembered that this paste need not be converted to a clear solution in order that its lead may be deposited during electrolysis. (Inadvertently, the impression that this solution must be clear was given in the instructions sent out for this method.)

Chittick method.—The Chittick method gives very accurate results in the hands of an experienced analyst. It requires time for filtration and much alcohol for washing. The filtration may be hastened by careful selection of filter cones (alundum) or filter paper. Methyl alcohol may be used.

FLUORIN.

The Wagner-Ross method for fluorin in phosphates requires a slight

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 332.
[2] *Analyst*, 1913, **38**: 312.
[3] Presented by R. E. Doolittle.
[4] Present address, Chemical Warfare Service, Edgewood Arsenal, Edgewood, Md.
[5] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 221.
[6] *Ibid.*, 218.
[7] *J. Ind. Eng. Chem.*, 1917, **9**: 1116.
[8] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 231.

modification in its application to baking powders. The moisture present in every baking powder is, of course, fatal to the operation of this method, which depends upon the production of anhydrous silicon fluorid. Consequently, the weighed baking powder may be ignited to drive off all water and burn off the starch, loss of carbon dioxid by decomposition of carbonates and bicarbonates having no effect, and then analyzed for fluorin. Or the weighed baking powder may be treated with water to produce reaction and then dried, charred in a casserole at about 300 °C., powdered, ignited and analyzed. Exact data as to which procedure is to be preferred is not yet in hand and will form the basis of future study.

Several collaborators, W. H. Ross (Bureau of Soils, Washington, D. C.), A. Malmstrom (Wilckes-Martin-Wilckes, Camden, N. J.), A. H. Fiske (Rumford Chemical Works, Providence, R. I.), and E. W. Thornton (R. B. Davis Co., Hoboken, N. J.), have obtained excellent checks for fluorin on the special samples of phosphates submitted by use of the Wagner-Ross method. And when the above modification for baking powders was used they have secured very consistent results on collaborative baking powder samples. These results have been so good that as the result of these two years of work the referee feels justified in recommending the adoption of the Wagner-Ross method for fluorin in phosphates and baking powders as a tentative method.

DETERMINATION OF ACIDITY OF BAKING ACIDS.

Some progress has been made by collaborators in the study of neutralizing the strength of baking acids but a report is not yet in hand for presentation.

RECOMMENDATIONS.

It is recommended—

(1) That the Wagner-Ross method for the determination of fluorin in phosphate and in baking powders be adopted as a tentative method.

(2) That the referee study methods for the removal of water from baking powders to be analyzed by the Wagner-Ross method for fluorin.

(3) That the referee study the question of the addition of phosphoric acid to tartrate and to alum powders to insure deposition of lead in the electrolytic method for the determination of lead in baking powders adopted as a tentative method by the association at the 1917 meeting[1].

(4) That the referee study methods for the determination of the neutralizing strength of baking acids.

Acknowledgment is made to the various collaborators who have assisted in this work and especially to G. H. Mains (Bureau of Chemistry, Washington, D. C.).

The meeting adjourned at 5.15 p. m. for the day.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 257.

THIRD DAY.

WEDNESDAY—MORNING SESSION.

REPORT OF THE COMMITTEE ON EDITING METHODS OF ANALYSIS[1].

It will be recalled that the 1917 report of your Committee on Editing Methods of Analysis[2] included the manuscript for the methods as revised to meet the criticisms received on the tentative report of the committee submitted at the 1915 meeting and published in *The Journal* during 1916[3]; also the additions and changes made to the methods at the 1916 meeting. The report of your committee was accepted and the methods as revised adopted, but the committee was continued with instructions to edit the 1917 changes and additions to the methods for publication as a supplement or separate report in the proceedings of the association. Soon after the 1917 meeting, the chairman of your committee collected, for consideration by the other members of the committee, the changes and additions to the methods which were made at the 1917 meeting and later, learning that the manuscript for the revised methods had not been sent to the printer, your committee undertook the task of again revising the manuscript so as to include all changes and additions made in 1917. Considerable correspondence with the secretary of the association, referees, and others was necessary in order to obtain the new methods and changes, but these data were finally secured and, upon receipt of a request from the secretary of the association early in July, 1919, for the manuscript of the revised methods, a meeting of four of the members of the committee was held in Chicago, Ill., on July 28th to 30th, inclusive. At this meeting the manuscript of the methods, revised to include all changes and additions made at the 1917 meeting, was carefully reviewed, and the manuscript as thus prepared was submitted to the secretary of the association, under date of August 4, 1919, for publication. The changes and additions which have been made to the methods since the 1917 report are as follows:

I. FERTILIZERS.

(1) It will be recalled that the Committee on Methods of Sampling Fertilizers to Cooperate with a Similar Committee of the American Chemical Society made the following recommendations at the 1917

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[1] Presented by R. E. Doolittle.
[2] *J. Assoc. Official Agr. Chemists*, 1920, 4: 258.
[3] *Assoc. Official Agr. Chemists, Methods*, 1916.

meeting[1], which were adopted by the association:

(*a*) That at least a pound of the material should constitute each official sample sent to headquarters.

(*b*) That the entire sample submitted to the chemist be passed through a 10-mesh sieve previous to its subdivision for analysis.

The Committee on Methods of Sampling Fertilizers to Cooperate with a Similar Committee of the American Chemical Society further suggests, under "Types of Samplers"[2], the use of a sampler that removes a core from the bag from top to bottom.

There is no paragraph for sampling fertilizers in the present methods and, inasmuch as the work of the Committee on Methods of Sampling Fertilizers to Cooperate with a Similar Committee of the American Chemical Society is not completed, your Committee on Editing Methods of Analysis has included the above-mentioned directions and suggestions for sampling by inserting an additional paragraph in close print to follow **2,** "Preparation of Sample"[2].

50, *Volumetric Method.*

(2) Committee A in its 1917 report recommended that the volumetric method[3] be adopted as an official method for the determination of total phosphoric acid in basic slag. This method has been designated as an official method in the manuscript of the revised methods.

(3) By motion from the floor, the association at the 1917 meeting adopted the following recommendation[4]: "That the use of potassium permanganate, wherever it appears in the Kjeldahl method, be eliminated." Your committee has accordingly made the following changes:

16 (g), *Potassium permanganate.*

This paragraph has been eliminated from the list of reagents.

18, DETERMINATION.

The sentence reading, "Remove the flask from the flame, hold it upright, and while still hot add carefully potassium permanganate in small quantities at a time until, after shaking, the liquid remains green or purple." has been eliminated; also the explanatory paragraph in close print which appears as the third paragraph in the directions for the determination.

26, DETERMINATION.

Paragraph 2, line 7.—The sentence reading, "Complete the oxidation with a little potassium permanganate in the usual way and proceed as directed under **18.**" has been changed to read: "Complete the determination as directed under **18.**"

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 287.
[2] *Ibid.*, 288.
[3] *Ibid.*, 14.
[4] *Ibid.*, 241.

(4) By motion from the floor, the association at the 1917 meeting adopted the following resolution: "That the use of 10 grams of anhydrous sodium sulphate as a substitute for potassium sulphate in the Gunning method and any modification thereof be made official."

Your committee has incorporated this change in the following paragraphs:

19, REAGENTS.

Line 1.—After "Potassium sulphate" the words "or anhydrous sodium sulphate" have been added, making the line read: "*Potassium sulphate or anhydrous sodium sulphate.*—Pulverized."

21, DETERMINATION.

23, DETERMINATION.

Line 2.—After "potassium sulphate" the words "or 15–18 grams of anhydrous sodium sulphate" have been added, making the sentence read: "Add 15–18 grams of potassium sulphate, or 15–18 grams of anhydrous sodium sulphate, etc."

28, DETERMINATION.

41 (a), *Mixed fertilizers.*

(5) On recommendation of Committee A, the association at the 1917 meeting adopted the following resolution[1], which eliminates the use of hydrochloric acid in the preparation of the solution of mixed fertilizers for the determination of potash: "That the official method for the preparation of potash solution be revised to read as follows:"

Place 2.5 grams of the sample upon a 12.5 cm. filter paper and wash with successive small portions of boiling water into a 250 cc. graduated flask until the filtrate amounts to about 200 cc. Add to the hot solution a slight excess of ammonium hydroxid and sufficient ammonium oxalate to precipitate all of the lime present, cool, dilute to 250 cc., mix, and pass through a dry filter.

This change has been incorporated by your committee.

II. SOILS.

The association at the 1917 meeting voted to substitute, as tentative methods, the methods reported by the special Committee on the Revision of Methods of Soil Analysis[2] for the methods previously published[3].

The methods presented by the special committee were referred to the Committee on Editing Methods of Analysis. The latter committee, after editing the methods, referred them to the Chairman of the Committee on the Revision of Methods of Soil Analysis for criticism. The chairman of the special committee on soils requested, under date of February 12, 1919,

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 241.
[2] *Ibid.*, 289.
[3] *Assoc. Official Agr. Chemists, Methods*, 1916, 17.

that the publication of the soil methods be deferred until the fall or winter of 1919, because considerable changes had taken place in them, and, in his opinion, it would be very undesirable to publish the methods as adopted by the association at the 1917 meeting. Under these circumstances, your Committee on Editing Methods of Analysis arranged the methods for the analysis of soils as a complete unit by themselves, so that they could be published either separately or as next to the last chapter in the book of methods, and submitted this manuscript, together with the correspondence with the chairman and other members of the special committee, to the secretary of the association for decision as to the manner in which these methods should be published.

III. INORGANIC PLANT CONSTITUENTS.

No changes or additions to these methods were made at the 1917 meeting. The substitution of the new set of methods for soils, as adopted at the 1917 meeting, however, eliminated several of the determinations to which references were made under Inorganic Plant Constituents. Your committee has, therefore, incorporated under Inorganic Plant Constituents the complete official methods for the determination of manganese, calcium, magnesium, sulphuric acid, sodium, and potassium. In this manner, all cross references to the methods for soils have been eliminated, and cross references in subsequent chapters to these determinations have been changed to refer to the determination as given under Inorganic Plant Constituents.

IV. WATERS.

At the 1917 meeting, on report of Committee A, the following recommendations were adopted by the association[1]:

(1) That the method for the determination of barium[2] be adopted as official. (First presentation.)

Your committee has inserted this method and, inasmuch as the method has been presented only once to the association, it has been designated as a tentative method, **52** and **53**.

(2) That consideration be given to the Gutzeit method for the determination of arsenic[3], with a view to having it printed in the methods for the analysis of waters (as an additional official method). (Second presentation of the method for action.) This method, **66** and **67**, has been inserted. The action taken would appear to make this an official method. The method, however, is identical with that given for the determination of arsenic in "Metals in Foods"[3], which is a tentative method. Under these circumstances, therefore, your committee deemed it advisable to

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[1] *J. Assoc. Official Agr. Chemists*, 1920, 4: 243.
[2] *Ibid.*, 86.
[3] *Assoc. Official Agr. Chemists, Methods*, 1916, 171.

insert the Gutzeit method for the determination of arsenic in waters as a tentative method, pending official action on the original method.

(3) That the method for the determination of manganese[1] be adopted as an additional official method. This method, **60** and **61**, has been inserted immediately following the present official method for manganese. As the method has been presented but once to the association, it has been designated as a tentative method.

(4) That the official reduction method for the determination of nitrogen in the form of nitrate be revised to read as outlined in the 1917 Report of the Referee on Waters[2].

The revised method, **16** and **18**, has been inserted in place of the former reduction method for the determination of nitrogen in the form of nitrate.

(5) That the methods listed below and recommended in 1916[3] for adoption as official be made official. (Second presentation of the methods for action.)

(a) Method for the determination of lithium, potassium and sodium[3].

(b) Method for turbidity, **1** and **2**[4].

(c) Method for color, **3** and **4**[4].

(d) Method for odor, **5**[4].

(e) The Schulze-Trommsdorf method for the determination of required oxygen, **22** and **23**[5].

(f) Methods I and II for dissolved oxygen, **24**, **25**, **26**, **27**, **28** and **29**[6].

(g) Method for the determination of specific gravity, **30**[7].

(h) Method for the determination of hydrogen sulphid, **37**[7].

(i) Method for temporary hardness, **70**[8].

(j) Method for alkalinity, **71**, **72**, **73** and **74**[8].

(k) Method for total hardness, **75** and **76**[9].

(l) Method for permanent or non-carbonate hardness, **77**[9].

The changes have been inserted by the committee.

(6) That the method for free carbon dioxid[10] remain tentative.

This method is given as a tentative method in the manuscript of the revised methods.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 86.
[2] *Ibid.*, 92.
[3] *Ibid.*, 3: 522.
[4] *Assoc. Official Agr. Chemists, Methods*, 1916, 35.
[5] *Ibid.*, 39.
[6] *Ibid.*, 40-1.
[7] *Ibid.*, 41.
[8] *Ibid.*, 50.
[9] *Ibid.*, 51.
[10] *Ibid.*, 42, 38.

V. TANNING MATERIALS.

No changes or additions were made to these methods at the 1917 meeting.

VI. LEATHERS.

No changes or additions were made to these methods at the 1917 meeting.

VII. INSECTICIDES AND FUNGICIDES.

The following recommendations were adopted by the association at the 1917 meeting on report of Committee A[1]:

(1) That the methods given by Roark[2] for total sulphur and for total lime be made official.

The designation of these methods as official has been inserted by your committee in the manuscript of the revised methods.

(2) That methods for the determination of the monosulphid equivalent, thiosulphate sulphur, sulphid sulphur, and sulphate sulphur under the iodin titration method[3], be made tentative.

These methods are given in the report of the Referee on Insecticides and Fungicides for 1917[3] and have been inserted as tentative methods, **74**, **77**, **79** and **81**, by your committee in the manuscript for the revised methods.

VIII. FOODS AND FEEDING STUFFS.

On report of Committee B, the following recommendations were adopted by the association at the 1917 meeting[4]:

(1) That the method for the determination of water in foods and feeding stuffs by drying in vacuum over sulphuric acid[5] be adopted as official. This change has been made in the designation of this method in the manuscript of the revised methods.

(2) That the method for the determination of water by drying over lime in vacuum[4] be adopted as a tentative method and be recommended for further study for the ensuing year.

(3) That the method for the determination of water by drying over carbide in vacuum[6] be adopted as a tentative method and be recommended for further study.

From correspondence with the Associate Referee on Water in Foods and Feeding Stuffs, it was learned that the results of study during 1918 were not nearly so favorable as those of the previous year, and it appeared that certain changes in the method were advisable. It was therefore

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 242.

[2] *Ibid.*, **3**: 354–5.

[3] *Ibid.*, **4**: 146.

[4] *Ibid.*, 247.

[5] *Assoc. Official Agr. Chemists, Methods*, 1916, 79.

J. Assoc. Official Agr. Chemists, 1920, **4**: 247.

the opinion of your Committee on Editing Methods of Analysis that the report of the associate referee for 1918 was not sufficiently favorable to warrant including in the revised manuscript the methods for the determination of water by drying in vacuum over lime and over calcium carbide.

Your committee would respectfully recommend that the association reconsider the action taken in 1917 to adopt these two methods as tentative methods and refer them back to the referee for further study.

(4) Committee C at the 1917 meeting made the following recommendation[1]:

That the methods for the hydrolysis of linamarin and the subsequent determination of hydrocyanic acid[2] be adopted as tentative methods.

Your committee has carefully considered these methods, and by reason of their general application to food products has included them as tentative methods, **68, 69** and **70,** in the manuscript of the revised methods.

IX. SACCHARINE PRODUCTS.

No changes or additions to these methods were made at the 1917 meeting.

Frederick Bates (Bureau of Standards, Washington, D. C.), however, called the attention of the Committee on Editing Methods of Analysis to the desirability of substituting the new Baumé scale for sugar solutions[3] for the old scale which was given in the official methods. After considerable correspondence with members of the association who are engaged in sugar work, your committee decided to include the new Baumé scale for sugar solutions as an additional table. It was learned through correspondence that some analysts, particularly those connected with export trade, still used the old table. It was, therefore, thought best, for the present at least, to publish both tables.

X. FOOD PRESERVATIVES.

On recommendation of Committee C, the association in 1917 made several of the tentative methods under this chapter official[4]. In accordance with this recommendation, your committee has corrected the designation of these methods in the manuscript of the revised methods.

XI. COLORING MATTERS IN FOODS.

No changes or additions to these methods were made at the 1917 meeting.

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[1] *J. Assoc. Official Agr. Chemists,* 1920, **4:** 253.
[2] *Ibid.,* 151.
[3] U. S. Bur. Standards, Technologic Paper **115:** (1918).
[4] *J. Assoc. Official Agr. Chemists,* 1920, **4:** 251.

XII. METALS IN FOODS.

No changes or additions to these methods were made at the 1917 meeting.

XIII. FRUITS AND FRUIT PRODUCTS.

No changes or additions to these methods were made at the 1917 meeting.

The attention of your committee, however, was called to the fact that the directions given under **1**(**a**), preparation of samples of fruit juices[1], was incomplete. This paragraph has been rewritten, giving in more detail the manner of preparation of samples of fruit juices for analysis.

XIV. CANNED VEGETABLES.

No changes or additions to these methods were made at the 1917 meeting.

XV. CEREAL FOODS.

No changes or additions to these methods were made at the 1917 meeting.

XVI. WINES.

No changes or additions to these methods were made at the 1917 meeting.

XVII. DISTILLED LIQUORS.

No changes or additions to these methods were made at the 1917 meeting.

XVIII. BEERS.

No changes or additions to these methods were made at the 1917 meeting.

XIX. VINEGARS.

No changes or additions to these methods were made at the 1917 meeting.

XX. FLAVORING EXTRACTS.

On report of Committee C, the following recommendations were adopted by the association in 1917[2]:

(1) That the method for the determination of benzoic acid in almond extract[2] be adopted as a tentative method.

This method has been inserted as **44**, Benzoic Acid.—Tentative.

(2) That the *Association of Official Agricultural Chemists, Methods,* 1916, 265–9, be changed as follows:

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[1] *Assoc. Official Agr. Chemists, Methods,* 1916, 177.
[2] *J. Assoc. Official Agr. Chemists,* 1920, 4: 254.

35 (a), *Phenylhydrazin solution.*

Line 2.—Eliminate the word "article" and substitute therefor the words "product in vacuo", making the sentence read: "A sufficiently pure product can be obtained by distilling the commercial product in vacuo, rejecting the first portions coming over which contain ammonia."

36, DETERMINATION.

Line 1.—After the word "Weigh" insert the words "accurately about", making the clause read: "Weigh accurately about 15 grams of the sample into a small, glass-stoppered flask;".

55, *Hortvet and West Method Modified.—Tentative.*

Change the last sentence to read as follows: "Multiply the weight of salicylic acid so found by 9.33 to obtain the per cent by volume of methyl salicylate."

These changes have been inserted in the manuscript of the revised methods by your committee.

XXI. MEAT AND MEAT PRODUCTS.

No changes or additions to these methods were made at the 1917 meeting.

XXII. DAIRY PRODUCTS.

On report of Committee B, the association at the 1917 meeting adopted the following recommendations[1]:

(1) That the Roese-Gottlieb method[2] for fat, as applied to plain ice cream, be adopted as official. (First reading.)

(2) That the Harding-Parkin method[3] for fat in ice cream be adopted as a tentative method. (First reading.)

From correspondence with the referee on dairy products, it was ascertained that no cooperative work had been done with either of these methods on any type of ice cream other than plain ice cream. It was, therefore, the intention of the referee that both the Roese-Gottlieb and the Harding-Parkin methods should apply to plain ice cream.

Under these circumstances, your committee has inserted in the manuscript of the revised methods, under the heading "Ice Cream (Plain)", the Roese-Gottlieb method, **65**, and the Harding-Parkin method for the determination of fat, **66**, both being designated as tentative methods.

(3) That the Schmidt-Bondzynski method, modified[4], for the determination of fat in cheese, be adopted as official. (First reading.)

The designation of this method as an official method has been made in the manuscript of the revised methods by your committee.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 248.

[2] *Assoc. Official Agr. Chemists, Methods*, 1916, 289.

[3] *J. Ind. Eng. Chem.*, 1913, **5**: 131.

[4] *Assoc. Official Agr. Chemists, Methods*, 1916, 297.

XXIII. FATS AND OILS.

On report of Committee C, the following recommendation was adopted at the 1917 meeting[1]:

That the method for the detection of the adulteration of lard with fats containing tristearin[2] be adopted as a tentative method.

This method, **41**, has been inserted in the manuscript of the revised methods.

XXIV. SPICES AND OTHER CONDIMENTS.

Committee C at the 1917 meeting made the following recommendation[1], which was adopted by the association:

That the method for the determination of volatile oil in mustard seed and mustard substitutes[3] be adopted as tentative.

This method has received considerable collaborative study in the various laboratories of the Bureau of Chemistry and elsewhere, and is in general use. Your committee, therefore, has inserted this method as a tentative method, **17.**

XXV. CACAO PRODUCTS.

No changes or additions to these methods were made at the 1917 meeting.

XXVI. COFFEES.

On recommendation of Committee C, the association adopted the following recommendations at the 1917 meeting[4]:

(1) That the Gorter method for the determination of caffein in coffee[5] be dropped.

(2) That the Fendler-Stüber method for the determination of caffein in coffee[6] be adopted as a tentative method.

(3) That the Stahlschmidt method for the determination of caffein in coffee[5] be not made official this year.

In accordance with these recommendations, your committee has eliminated the Gorter method and in place thereof has substituted the Fendler-Stüber method for this determination. The Stahlschmidt method remains as a tentative method in the manuscript of the revised methods.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 256.
[2] *Ibid.*, 200.
[3] *Ibid.*, 149.
[4] *Ibid.*, 257.
[5] *Assoc. Official Agr. Chemists, Methods*, 1916, 332.
[6] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 213.

XXVII. TEA.

No changes or additions to these methods were made at the 1917 meeting.

XXVIII. BAKING POWDERS.

The association at the 1917 meeting on report of Committee C adopted the following recommendation[1]:

That the modified Corper-Bryan method for the electrolytic determination of lead in baking powders[2] be adopted as a tentative method.

This method, **33** and **34**, has been inserted in the manuscript of the revised methods.

XXIX. DRUGS.

On report of Committee B, the association at the 1916 meeting adopted the following recommendations[3]:

(1) That the methods for the determination of strychnin in tablet triturates[4] be made provisional.

(2) That the method for the determination of strychnin in liquids[4] where it occurs as the only alkaloid be made provisional.

These methods, **31** and **32**, were overlooked in the 1917 Report of the Committee on Editing Methods of Analysis. They have, however, been inserted in the manuscript of the revised methods.

On report of Committee B, the association at the 1917 meeting adopted the following recommendation[5]:

That the method for the determination of atropin in tablets[6], with the following change relative to drying the alkaloidal residue, be made tentative: "Dry in vacuo to a constant weight, and weigh as atropin."

This method, **33**, has been inserted in the manuscript of the revised methods.

Respectfully submitted,

R. E. DOOLITTLE, G. W. HOOVER,
B. L. HARTWELL, A. J. PATTEN,
W. A. WITHERS.
Committee on Editing Methods of Analysis.

Adopted.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 257.
[2] *Ibid.*, 221.
[3] *Ibid.*, 3, 527.
[4] *Ibid.*, 1919, **3**: 189; 1920, **3**: 379.
[5] *Ibid.*, 1920, **4**: 249.
[6] *Ibid.*, **3**: 379.

REPORT OF SPECIAL COMMITTEE ON THE BAUMÉ SCALE[1].

Your committee begs leave to submit the following recommendation:

That the Baumé scale of the Bureau of Standards (Modulus 145), Table 31[2], be adopted as the official Baumé scale of the association, and that all Baumé tables and references thereto which are not in accordance with this scale be eliminated from the methods of the association; and further, that the Committee on Editing Methods of Analysis be authorized to make such changes in the text of the Chapter on Saccharine Products[3] as may be necessary to make this recommendation effective.

Respectfully submitted,

W. D. HORNE, R. E. DOOLITTLE,
FREDERICK BATES, PAUL RUDNICK,
E. W. MAGRUDER.

Adopted.

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[1] Presented by R. E. Doolittle.
[2] U. S. Bur. Standards Circ. 44; (1918), 151.
[3] *Assoc. Official Agr. Chemists, Methods*, 1916, 121.

REPORT OF SECRETARY-TREASURER FOR

By C. L. **Alsberg** (Bureau of Chemistry,

RECEIPTS

1916		
Nov. 12	Bank balance	$139.65[1]
1918		
Apr. 20	Dues from 3 institutions (Alabama, Massachusetts and South Dakota) received after the Treasurer's report for 1917 had been submitted	15.00
Nov. 16	Dues for the year 1918, from 71 Federal, State, Municipal and Canadian organizations	355.00
1919		
Nov. 15	Dues for the year 1919 from 68 Federal, State, Municipal and Canadian organizations	340.00
		$849.65

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[1] This balance differs from the balance noted in the Secretary-Treasurer's report of 1917, due to the fact that two checks, Nos. 84 and 85, for $2.86 and $5.00 respectively, were drawn on C. L. Alsberg's personal account in error, reimbursement for which is taken care of in Check No. 112, reported under disbursements.

THE TWO YEARS ENDING NOVEMBER 19, 1919.

Washington, D. C.), *Secretary-Treasurer.*

DISBURSEMENTS

			Amount.	Check No
1917				
Dec. 19	Tips, New Willard Hotel, 1917 meeting		$6.00	91
Dec. 19	Telephone calls, New Willard Hotel, 1917 meeting		1.90	92
1918				
Jan. 2	Delos M. Carter, to complete payment for editing May 15, 1917, number of *The Journal*		57.87	93
Jan. 5	Post office box rent for quarter ending March 31, 1918		2.00	94
Mar. 12	Postage		5.00	95
Mar. 23	Post office box rent for quarter ending June 30, 1918		2.00	96
Mar. 30	Postage		10.00	97
Apr. 3	Byron S. Adams, printing 1000 cooperative circulars		16.25	98
May 3	Chas. G. Stott & Co., printing 1000 letterheads (Secretary-Treasurer)		5.40	99
July 6	Post office box rent for quarter ending September 30, 1918		2.00	100
Sept. 24	Chas. G. Stott & Co., printing 1000 letterheads (Secretary-Treasurer)		6.25	101
Sept. 24	Post office box rent for quarter ending December 31, 1918		2.00	102
Oct. 18	Postage		5.00	103
1919				
Jan. 27	Telegrams		8.86	107
Feb. 1	C. L. Alsberg, reimbursement for checks (Nos. 105 and 106) drawn on personal account		7.00	108
Feb. 1	1000 two-cent special request envelopes		22.56	109
Mar. 21	Post office box rent for quarter ending June 30, 1919		2.00	110
Apr. 11	Postage		10.00	111
Apr. 12	C. L. Alsberg, reimbursement for checks (Nos. 84, 85 and 104) drawn on personal account		14.61	112
June 23	Post office box rent for quarter ending September 30, 1919		2.00	113
July 18	Printing 1000 letterheads		4.65	114
Aug. 21	Postage for sending out announcements of meeting		5.00	115
Sept. 18	Printing 1000 programs		37.75	116
Sept. 22	Post office box rent for quarter ending December 31, 1919		2.00	117
Nov. 5	Telegrams		12.53	118
Nov. 7	Stationery		6.10	119
Nov. 10	Postage		10.00	120
Nov. 13	2500 one-cent stamped envelopes		31.20	121
Nov. 14	Reimbursement, two telegrams		1.01	122
Nov. 15	Bank balance	$601.71		
	Less checks out	51.00		
			550.71	
			$849.65	

The undersigned committee has examined the above report and finds it correct.

G. L. Bidwell,
E. M. Bailey,
W. F. Hand.
Auditing Committee.

Adopted.

REPORT OF THE BOARD OF EDITORS.

By C. L. Alsberg (Bureau of Chemistry, Washington, D. C.), *Chairman.*

I reported at the last meeting[1] that we were in trouble with the publisher, or rather, the printer. I had thought it would be possible to adjust these differences in some manner. As a matter of fact, however, the printer has sued the association, and possibly the case will come to trial in the next two or three years in the District of Columbia. The association has been assured by its attorneys, however, that they have no doubt whatever as to the issue, so arrangements were made a few weeks ago to pay no further attention to the former printers, and another printer has been secured to print *The Journal.* The copy for the next number, Volume III, No. 2, went to the printer some weeks ago, and it was hoped that that number could be distributed before this meeting. That was not possible; in the main, because the association owed a certain responsibility or duty to its advertisers, and the former printers, the Williams and Wilkins Company, refused to give us any information concerning the status of contracts made on behalf of *The Journal* with advertisers. It was, therefore, necessary to correspond with each individual advertiser who had advertised in *The Journal* to ascertain what the status of his contract was, and to get him to submit new copy. That has been done, and the next number of *The Journal* will appear within ten days. In order to make sure that you will feel this is not a hope but a certainty, we had the printer make up a couple of sample copies of the next number of *The Journal,* and, as an evidence that work is really progressing, I beg to pass this particular copy around. There is nothing more about it, except "Here it is!" There are about 20 pages in these sample copies. There will be about 125 pages in the completed number.

According to our estimates, the printer over in Baltimore has about forty-five hundred dollars of the association's money which can not be released until the litigation, which he threatens, is decided, or, if he should change his mind, until a compromise can be effected. The association is under obligation, of course, to fulfil all duties to its subscribers and to furnish them with the four numbers for which they have subscribed, and of which only one number has been produced. Therefore, arrangements have been made with another printer by which *The Journal* will be financed quite differently than in the past. Under the new arrangement, the printer will handle *The Journal* solely as a printing proposition and get paid for the printing. Everything else will be handled by my office. This means that the association will have entire control of all money received and all business matters connected

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[1] *J. Assoc. Official Agr. Chemists,* 1920, **4**: 272.

with *The Journal.* We do not even have a contract with the printer that extends over any considerable length of time because, when I came to look into the cost of printing, I found that if we wanted to make a contract for a year or two, the printer, not knowing what labor was going to cost him, would have to hedge and charge us enough to play safe. A number of printers advised against making any contract at this time. For about a year it is going to be rather hard to finance the proposition, because we are under obligation to furnish our old paid subscribers with three numbers without additional charge.

We are also confronted with this additional difficulty, that the printer over in Baltimore has the old list of subscribers, which he refuses to turn over to us. In order to meet that difficulty, we have inserted an advertisement in the leading scientific journals requesting those who are subscribers and have paid their subscription to *The Journal* to notify us, in order that they may receive the remaining numbers of Volume III. There are probably a considerable number of others who would like to subscribe if they were certain, as I think we can promise now, that *The Journal* is going to be issued regularly in the future.

The first number to issue after publication is resumed will contain the conclusion of the 1915 proceedings and a part of 1916. That leaves a large part of the 1916 and 1917 proceedings, together with those of this meeting. It is very important that the proceedings be abbreviated as much as possible, in order to get caught up any time soon. To accomplish this, all reports will be edited and cut to the bone in my office, after which they will be sent to the authors for criticism and approval. I can not urge too strongly that all authors cooperate with us in permitting the abridged reports to be printed, provided, of course, the value of the report has not been destroyed in the condensation.

That brings me to the question of methods. We have made arrangements for financing the printing of the methods under conditions similar to those for *The Journal.* They will be handled merely as a printing proposition. The manuscript for the methods is ready to go to the printer, up to and including the 1917 meeting. We did not send the manuscript of the methods to the printer prior to the calling of this meeting, as we did the copy for *The Journal*, for the simple reason that we wanted the judgment of the Committee on Editing Methods of Analysis, as well as the judgment of the association, on the question of sending the methods to the printer without including the changes made at this meeting. If the association decides that we should send the methods to the printer without including the changes made at the 1919 meeting, then the manuscript can go to the printer within a fortnight, and the methods can appear in printed form some time in February, barring delays from strikes or accidents. We can then publish separates containing later changes and additions, in such form that they can be bound with the methods.

Now comes the cost of printing. It will be a book of over 400 pages, and it will probably not be possible to say definitely just what it will cost until the printing is nearly completed. It is obvious that the association does not want to make any money out of the methods. We want to bring the methods to the country at large at once at as low a cost as possible. The cost will depend on the size of the edition. Therefore, it is extremely important that we get as many subscriptions as possible in advance. If we find we have 1000 or 1500 subscriptions in advance, we will know that we will have to print 3000 or 4000 copies, and can get better prices than if we have only 300 or 400 books to print. Therefore, it is exceedingly important for the members to send me their subscriptions in advance.

I only want to add, in conclusion, that I think it will require pretty close figuring to finance the proposition at first, because of our money being tied up in Baltimore. But we are making a fresh and better start.

Adopted.

J. M. Bartlett: I think if these methods can be brought up to date and printed, there will be quite a large call for them.

P. F. Trowbridge: About what would you think the price ought to be?

J. M. Bartlett: If it costs no more than a good textbook usually does, there will be a good many bought by agricultural students.

C. L. Alsberg: We want to get the price down to that of a good textbook, and are making every effort to do it. It may be possible to lower the price after the issue gets started. In such case, we will want to make a rebate to the original subscribers. I think you are quite right that the price should be put as low as possible.

That brings up another point that has been raised by members of the association, whether or not separate editions of the separate chapters most in demand should be printed for the use of students. The judgment of the editors is that such a plan is not feasible, because there are so many cross-references within the chapters. Take foods, for instance; a lot of the methods that are used for foods are printed in the chapter on "Foods and Feeding Stuffs", etc. I would like the members to consider this matter very carefully.

There is also another possibility, and that is that the association might get out a college textbook which would contain about 150 pages of selected methods for the use of students. Such a book could probably be sold at a very low figure. The Board of Editors would like the judgment of the association regarding the feasibility of such a proposition. The methods are growing to huge proportions, and yet there is nothing we want to cut out. The book is becoming unwieldy for students, however, and I am inclined to think the solution would be for a special committee to compile a special textbook for the colleges.

Paul Rudnick: Does not the student usually find what he needs in the special lecture?

C. L. Alsberg: That is true, provided that is the only training he gets. But I am inclined to think that if his teacher is the right sort of a teacher, he would rather encourage the use of a small book than otherwise.

I do not suppose this association wants to compete with publishers, or private individuals, in the publication of a textbook. My own judgment is that we should do that only if nobody else were occupying that field. On the other hand, I do not think that an outline will do for the student. One of the troubles with Bureau of Chemistry Bulletin 107 was that the methods were cut down to a skeleton. The reason why the revised methods occupy over 400 pages is, in part, that they were printed in legible print, but it is due in most part to the fact that the committee has had constantly before it the making of them fool-proof, and has described very fully all the methods. So, the main thing that makes for the increase is putting in the omission of details that occurred in Bureau of Chemistry Bulletin 107.

C. L. Parsons: I am not a member of the association, but I am extremely interested in these methods, and I should like to ask whether or not the question of having them published by some outside publisher has been considered. If these methods are put out at a cost of five dollars, as they are likely to be, the sales are apt to be comparatively small. At the same time, we all know how important they are to all and how necessary it is to have a very large number of these copies available. If a large number is available, the price could be made very low. Personally, I am inclined to think—I am not making this on a basis of real knowledge—but I think it would be quite possible for you to make arrangements with some publisher to publish these methods, under the auspices of the association, in sufficient quantity to make the price very low. I am wondering if the association has thought of that method. Practically every one of the students in the Agricultural Colleges and Universities needs these methods of analysis in their college work, and the demand for them will not be confined to America if they are put out on a commercial scale. Somebody has got to take the risk.

P. F. Trowbridge: These matters will be referred to the Board of Editors.

Wm. Frear: I am sure that in laboratories for advanced students there will be a demand for a supply of these methods if they can be had at a reasonable cost, so that the students themselves can be expected to purchase them. Not for the sake of the association, but for the good of the students of chemistry in this country, I hope that the committee will consider most carefully the possibilities of making an arrangement for the stereotyping of the methods, and for their issue, whereby the cost can be kept down without too much of an initial investment. Of course, the composition does cost; that is recognized. The paper, printing and binding costs are relatively lower. And it may be that when the time comes for revision, it will be largely by addition, so that the insertion and cutting down of a word or two, here and there, can be done at a comparatively low cost, and in that way the present revision will be of relatively permanent value. I sincerely hope that the committee will make a careful canvas of the institutions of the country so that the needs of the country will be met.

R. N. Brackett: I would like to move that the president appoint a committee of the association to select methods which are most desirable to have in a book for the use of students in Agricultural Colleges. Of course, the books in the regular edition will be too large. But I would like to move that a committee be appointed by the president to select the methods which will be most suitable for students in colleges.

H. B. McDonnell: In this connection, will there be much saving? As has been stated, the principal saving will be the composition, printing, etc., but it would not cost much more to get out the complete book. It seems to me that the solution is to put out a large edition of a single book, thus bringing the cost of each book down.

P. F. Trowbridge: The motion has not been supported. It seems to the chair that it might be well for the association to take some action authorizing the Board of Editors to investigate the feasibility of these propositions, and to give them authority to put them into operation if they find them feasible. I might say that this whole matter was thrashed out in committee night before last, and the committee is very much in hopes that the methods can be brought down to three dollars.

Paul Rudnick: I move that the matter be referred to the Board of Editors with power to act.

R. N. Brackett: Second the motion.

The motion was unanimously carried.

P. F. Trowbridge: Before we pass to the next subject, I would like to ascertain, if possible, whether or not the association wishes to take any action relative to the incorporation of the changes made at this meeting in the manuscript of the methods as now prepared to send the printer.

C. L. Alsberg: Of course, we could incorporate the action taken at this meeting if it is desired, but it is my experience that to do so means going through the whole methods, from the first page to the last page, checking up and making appropriate changes in the cross-references. That is a job which might well delay the appearance of the methods from six to nine months. I do not know that anyone who has not worked with the Committee on Editing Methods of Analysis realizes the immense amount of work they have done in getting the methods in their present shape. I am convinced that it will cause a delay of six months, and maybe longer, to incorporate the 1919 changes in the present edition of the methods. It is for the association to say whether they wish that or not. My own thought was that it would be wisest to publish the methods just as soon as possible. We could print the methods up to and including the changes made in 1917 now, and later print in *The Journal*, with separate pagination so that it would not interfere with the regular pagination of *The Journal*, all the changes which have been made at this meeting. In that way, a man might take the supplementary report—which would be merely a report of the changes made at this meeting—and keep it with or paste it into the book of methods. Even the supplementary report would take considerable time to prepare. If it is ready to print in six months, we will be doing well. However, it is a matter for the association to decide.

Paul Rudnick: May I be permitted to plead with the association for as early a publication of the methods as possible. For several years there has been a demand for the methods outside of the members. The methods have not been available—nothing has been available except the 1916 revision of the methods contained in *The Journal*. Anything that will expedite the publication of the methods, I think, would be more than welcome to the many users of the methods, as well as to the members of the association themselves. The ruse of obtaining an interleaved edition of the methods would enable every user to keep the methods as nearly up to date as possible.

C. L. Alsberg: Is it your idea, Mr. Rudnick, that it would be preferable to publish the methods complete up to and including 1917, following with 1919, rather than to wait and incorporate the 1919 changes in the manuscript that is now practically ready to send the printer?

Paul Rudnick: By all means.

W. W. Skinner: I move that it is the sense of the association that the methods, revised up to and including 1917, be printed.

H. C. Lythgoe: I second the motion.

P. F. Trowbridge: The motion before you is that it be the sense of the association that the methods revised up to and including the report of the 1917 meeting, but not including the action taken at the 1919 meeting, be printed as soon as possible. Any remarks?

The motion is unanimously carried.

C. L. Alsberg: Some years ago, it is my understanding that the association voted, among other things, to drop the final "e" on all chemical terms, even such terms as "strychnine", etc. The American Chemical Society has a spelling which is quite generally used, and it occurs to me that it might be well to consider the desirability of adopting the procedure of the American Chemical Society. I should regret, however, to see any motion passed which would make it necessary to change the spelling in the manuscript of the methods, but it might be well to consider this for the spelling of future editions of *The Journal* and for the methods.

W. W. Skinner: I would like to move that this matter be referred to the Board of Editors with power to act.

Motion seconded and unanimously carried.

REPORT ON FORM OF REPORT AND RECOMMENDATIONS BY REFEREES.

By W. W. Skinner (Bureau of Chemistry, Washington, D. C.).

At the request of the secretary of the association, I have prepared the following instructions to referees and the form to be used by them in the preparation of their annual reports, and respectfully recommend that they be adopted by the association.

INSTRUCTIONS TO REFEREES.

In addition to your full report, please prepare your recommendations in triplicate in the following form, using one or more sheets for each recommendation. This is very important when a recommendation is made for the adoption of a method. A method proposed for adoption should be edited by the referee to conform to the style used throughout the Official and Tentative Methods of Analysis, and should be in the exact language in which he desires the method to be printed. If the recommendation is adverse, the method should not be given in detail, but under "Method and Remarks" should appear the suggestions for further work. For the benefit of both succeeding referee and the Committee on Recommendations of Referees, a referee should always make, if possible, quite definite suggestions as to future work. This has generally been done, but frequently is so buried in the body of the report of the referee as to escape notice and proper consideration.

A copy of all recommendations, together with abstracts of reports, must be in the hands of the Chairman of the Committee on Recommendations of Referees at least three weeks in advance of the meeting. It is also requested that duplicate copies be sent to the Chairman of Subcommittee A, B or C, as the case may be.

Committee assignments are as follows:

Subcommittee A: [Fertilizers (borax in fertilizers, preparation of ammonium citrate, nitrogen, potash), potash availability, inorganic plant constituents (sulphur and phosphorus in the seeds of plants, calcium and magnesium in the ash of seed), water, tanning materials and leather, insecticides and fungicides, and soils (sulphur in soils).]

Subcommittee B: [Foods and feeding stuffs (crude fiber, stock feed adulteration), saccharine products (honey, maple products, maltose products, sugar-house products), dairy products (moisture in cheese), fats and oils, baking powder, drugs (alkaloids, arsenicals, synthetic drugs, alkaloids of opium, medicinal plants, enzyms, essential oils), testing chemical reagents, soft drinks, and eggs and egg products.]

Subcommittee C: [Food preservatives (saccharin), coloring matters (oil-soluble colors), metals in foods, pectin in fruits and fruit products, canned foods (physical methods of examination, tomato products), cereal foods, wines, distilled liquors, limit of accuracy in the determination of small amounts of alcohol in beers, methods of analysis of near beers, vinegars, flavoring extracts, meat and meat products (separation of meat proteins, decomposition of meat products, gelatin), spices, determination of shells in cacao products, methods for the examination of cacao butter, coffee, and tea.]

RECOMMENDATION BY REFEREE.

Referee: Subject: Date:

$\left\{\begin{array}{l}\text{First}\\ \text{Second}\end{array}\right\}$ recommendation for adoption of the method given below in detail as an $\left\{\begin{array}{l}\text{official}\\ \text{tentative}\end{array}\right\}$ method of the Association of Official Agricultural Chemists. The method $\left\{\begin{array}{l}\text{has}\\ \text{has not}\end{array}\right\}$ been published in the proceedings (Ref.), as provided by By-laws 5 and 7.

METHOD AND REMARKS.

Adopted.

REPORT OF COMMITTEE ON RECOMMENDATIONS OF REFEREES.

By B. B. Ross (Alabama Polytechnic Institute, Auburn, Ala.), *Chairman.*

The reports submitted by the chairmen of Subcommittees A, B, and C have thoroughly covered the work of the Committee on Recommendations of Referees, so that the committee, as a whole, has no further report to make upon the work embodied in the reports of the referees and associate referees. The full committee, however, after holding a joint session with the members of the Executive Committee, begs to submit certain recommendations with regard to future handling of the referee work of the association.

The two committees, at their joint session, after a full consideration of the matter, reached the conclusion that it is not necessary or desirable to continue to appoint from year to year, as heretofore, referees or associate referees for each and every one of the subjects, or divisions of subjects, included in the scope of the work of the association. Many of the methods relating to the various subjects have been so well developed that it is not deemed essential to continue work along the lines of these methods, unless new conditions that may arise from time to time in the future, or new methods of apparent value or importance, justify the continuance or the resumption of work on such subjects.

The further conclusion was reached that it would be desirable and advantageous to have a general referee for certain related groups of subjects, with a sufficient staff of associate referees to work with him or under his direction, in the investigation of certain important questions in the field covered by the work of the general referee. The general referee would also be expected to keep in touch with the literature dealing with the subjects embraced in his field of work, with a view to suggesting the study of such special lines of work as would be of value or importance, making recommendations along these lines at the annual meetings; or, in urgent cases, initiating investigations in the interim between meetings, whenever such action should be desirable.

Adopted.

REPORT OF COMMITTEE A ON RECOMMENDATIONS OF REFEREES.

By A. J. PATTEN (Agricultural Experiment Station, E. Lansing, Mich.), *Chairman.*

[Phosphoric acid (basic slag to cooperate with committee on vegetation tests on the availability of phosphoric acid in basic slag), nitrogen (special study of Kjeldahl method), potash, soils (nitrogenous compounds, lime absorption coefficient), inorganic plant constituents, insecticides and fungicides, water, and methods of sampling fertilizers.]

PHOSPHORIC ACID.

It is recommended—

(1) That the recommendation approved at the 1917 meeting, relative to the preparation of neutral ammonium citrate solution and the use of substitutes therefor[1], be rescinded and Recommendation 2 be substituted.

Approved.

(2) That the referee be instructed to determine the exact composition of a strictly neutral solution of ammonium citrate and report at the next meeting of the association with recommendations as to the preparation of such a solution.

Approved.

(3) That the recommendation approved at the 1917 meeting relative to a substitute for the molybdic acid method for the determination of phosphoric acid[2] be rescinded.

Approved.

(4) That the papers presented by H. D. Haskins[3] and by E. O.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, 4: 239.

[2] *Ibid.*, 240.

[3] *Ibid.*, 64.

Thomas[1], on subjects relating to the determination of phosphoric acid, be referred to the referee for 1920.

Approved.

BASIC SLAG TO COOPERATE WITH COMMITTEE ON VEGETATION TESTS ON THE AVAILABILITY OF PHOSPHORIC ACID IN BASIC SLAG.

(1) That the referee on phosphoric acid be instructed to continue the study of methods for determining the availability of phosphoric acid in slags and chemical matters pertaining thereto, so that the association may have at hand data necessary to aid it in the adoption of an availability method as soon as the results of the vegetation experiments are obtained. Sufficient reports are already in the hands of your committee to be of service to the referee on phosphoric acid in his chemical investigations. It seems unnecessary to your committee to wait until all of the vegetation results are at hand before tentative methods of analysis are submitted to the association. Your committee therefore recommends that this association instruct its referee on phosphoric acid to give prominent attention to the question of methods of determining available phosphoric acid in slags, the chemical ingredients influencing the same, and the bibliography on the subject.

Approved.

NITROGEN.

It is recommended—

(1) That the referee for 1920 study the nitrometer method (which is invariably used by the manufacturers of explosives) for the analysis of nitrate of soda.

Approved.

(2) That a further study be made of the effect of glass wool in the ferrous sulphate-zinc-soda method for nitrates.

Approved.

SPECIAL STUDY OF KJELDAHL METHOD.

It is recommended—

That the recommendations presented by the associate referee[2] be continued for further study.

Approved.

POTASH.

It is recommended—

(1) That the work on the availability of potash be referred to a special referee with the suggestion that his tenure of service be indefinite or until relieved by the association.

Approved.

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[1] *J. Ind. Eng. Chem.*, 1917, **9**: 865.

[2] *J. Assoc. Official Agr. Chemists*, 1921, **4**: 372.

(2) That the study of the perchlorate method be continued to include a study of the moist combustion method, outlined in the report of the referee[1].

Approved.

(3) That the paper presented by H. D. Haskins[2], entitled "A Modified Method for the Determination of Water-Soluble Potash in Wood Ashes and Treater Dust", be referred to the referee on potash for 1920.

Approved.

(4) That the referee for 1920 be requested to study a method for the determination of boric acid in mixed fertilizers and raw materials and present the same at the next meeting of the association.

Approved.

SOILS.

It is recommended—

That the study of the determination of total phosphorus in soil be continued with a larger number of soils and that an attempt be made to secure more collaborators.

Approved.

NITROGENOUS COMPOUNDS.

No report or recommendations.

LIME ABSORPTION COEFFICIENT.

It is recommended—

(1) That it is not advisable for the association to adopt any method for lime absorption cœfficient at this time, either tentatively or officially.

Approved.

(2) That the subject be continued for further study.

Approved.

INORGANIC PLANT CONSTITUENTS.

It is recommended—

(1) That the methods for calcium and magnesium, as outlined in the report of the referee for 1919[3], be made tentative for the ash of such material as seeds, and that further work be done on them during the coming year.

Approved.

(2) That a method be devised for the determination of iron and aluminium in the filtrate from magnesium.

Approved.

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[1] *J. Assoc. Official Agr. Chemists*, 1921, **4**: 373.

[2] *Ibid.*, 1920, **4**: 82.

[3] *Ibid.*, 1921, **4**: 392-3.

(3) That some cooperative work be done on the colorimetric method for manganese, possibly using several different oxidizing agents.

Approved.

(4) That a study of the present methods for phosphorus and chlorin be made on material corresponding to that of the ash of seed.

Approved.

INSECTICIDES AND FUNGICIDES.

It is recommended—

(1) That further cooperative work be done on the methods for the determination of lead, copper and zinc in a compound that may contain arsenic, antimony, lead, copper, zinc, iron, calcium, magnesium, etc. (Second year of cooperative work.)

Approved.

(2) That further cooperative work be done on the comparison of the zinc oxid-sodium carbonate and the blood charcoal adsorption methods with the official iodin method for the determination of the total arsenic in London purple. (First year of cooperative work.)

Approved.

(3) That further cooperative work be done on the comparison of the Gyory bromate and the Jamieson iodate methods for the titration of arsenic trioxid in hydrochloric acid solution, with the official iodin method for the determination of arsenic trioxid; and that special emphasis be placed on the temperature of the solution when titrating with potassium bromate.

Approved.

(4) That the official method for the determination of total arsenic in lead arsenate be made official for the determination of total arsenic in magnesium arsenate; and that the official method for arsenic trioxid in lead arsenate be made official for the arsenic trioxid in calcium and magnesium arsenates. (First presentation of the method for action.)

Approved.

(5) That a study be made of methods for the determination of calcium, magnesium and zinc, respectively, in calcium and magnesium arsenates and zinc arsenite.

Approved.

(6) That a study be made of methods for the determination of the soluble arsenic in calcium and magnesium arsenates and zinc arsenite.

Approved.

WATER.

It is recommended—

(1) That the method for the determination of barium, described in

the report of the referee for 1917[1], be adopted as official. (Second and final presentation of the method for action.)

Approved.

(2) That the method for the determination of manganese, described in the report of the referee for 1917[2], be adopted as an additional official method. (Second and final presentation of the method for action.)

Approved.

(3) That the method for the determination of iodin in the presence of chlorin and bromin, described in the report of the referee for 1919[3], be adopted as a tentative method. (First presentation of the method for action.) The method has not been published in the Proceedings, as provided by By-law No. 7.

Approved.

(4) That the method for the determination of bromin in the presence of chlorin but not iodin, described in the report of the referee for 1919[4], be adopted as a tentative method. (First presentation of the method for action.) The method has not been published in the Proceedings, as provided by By-law No. 7.

Approved.

(5) That the method for free and albuminoid ammonia in samples containing sulphid[5] be adopted as official. (First presentation of the method for action.)

Approved.

(6) That the methods on water be extended to cover the examination of allied products, such as brine and salt.

Approved.

(7) That continued study be given to the determination of iodin and bromin and the heavy metals and to the use of equivalents in studying the character of waters.

Approved.

METHODS OF SAMPLING FERTILIZERS.

It is recommended—

(1) That a sampler be used that removes a core from the bag from top to bottom.

Approved.

(2) That at least a pound of the material should constitute each official sample sent to headquarters.

Approved.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 86.
[2] *Ibid.*, 85.
[3] *Ibid.*, 1921, **4**: 380.
[4] *Ibid.*, 381.
[5] *Ibid.*, 387.

(3) That the entire sample submitted to the chemist be passed through a 10-mesh sieve previous to its subdivision for analysis.

Approved.

(4) That cores shall be taken from not less than 10 per cent of the bags present, unless this necessitates cores from more than 20 bags, in which case a core shall be taken from one bag from each additional ton represented. If there are less than 100 bags, not less than 10 bags shall be sampled, provided that in lots of less than 10 bags all bags shall be sampled.

Approved.

REPORT OF COMMITTEE B ON RECOMMENDATIONS OF REFEREES.

By H. C. LYTHGOE (State Department of Health, Boston, Mass.), *Chairman.*

[Foods and feeding stuffs (sugar, crude fiber, stock feed adulteration, organic and inorganic phosphorus, water), dairy products (separation of nitrogenous substances in milk and cheese), saccharine products (maple products, honey, sugar-house products), drugs (medicinal plants, alkaloids, synthetic products, balsams and gum resins, enzyms, essential oils), testing chemical reagents and microanalytical methods.]

FOODS AND FEEDING STUFFS.

No recommendations were made by the referee. The committee therefore recommends that the following 1917 recommendations be continued:

(1) That a further study be made of sulphur dioxid in bleached grain.

Approved.

(2) That the method for determining the acidity of corn, as described by Black and Alsberg[1], be considered by the referee next year with a view to its adoption as an official method, and that the method be studied to determine whether changes are necessary to make it applicable to grains other than corn.

Approved.

SUGAR.

It is recommended—

(1) That the following recommendations, left over by the former associate referee, be referred to the Carbohydrate Laboratory of the Bureau of Chemistry for study:

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[1] U. S. Bur. Plant Ind. Bull. 199: (1910).

(*a*) Further study upon the modifications proposed in 1915 for determining sucrose by acid and invertase inversion.

(*b*) Further study for the determination of small amounts of reducing sugars in the presence of sucrose.

(*c*) Further study upon the optical methods of estimating raffinose in beet products, using enzyms for the hydrolysis.

Approved.

(2) That the methods for determining copper by reduction of the oxid in alcoholic vapors be further studied.

Approved.

(3) That under the heading in the official methods, "Preparation of Sample.—Tentative" **IX**, Saccharine Products[1], the following be added:

(**d**) *Raw sugars.*—Mix thoroughly on a glass plate in the shortest possible time with spatula and glass or iron rolling pin in case of lumps, or in a large, clean, dry mortar.

Approved.

(4) That Browne's temperature formula for correcting the polarization of raw cane sugars to that of 20° be adopted:

$$P^{20°} = P^{t} + 0.0015\,(P^{t} - 80)\ (t° - 20)$$

but where the percentage of levulose is actually determined, use the formula

$$P^{20°} = P^{t} + 0.0003°S\ (t - 20) - 0.00812°L\ (t - 20).$$

Approved.

(5) That the question of the adoption of the Baumé scale of the Bureau of Standards in place of the one now in use be further considered, possibly by a committee.

Approved.

(6) That where standard quartz plates for the German or Ventzke scale are sent for certification, the Bureau of Standards be requested to certify on the old value of 100° V. = 34.657° (circular degrees) for sodium light at 20°C. in place of their new value of 34.620° until this has been adopted internationally.

Approved.

(7) That a committee be appointed to get in touch with the committee of three, appointed by the American Chemical Society, upon an international normal sugar weight; and also to ascertain the views of all members of this association upon the adoption of an international standard normal weight of 20 grams.

Approved.

(8) That the Baumé scale of the Bureau of Standards (Modulus 145), Table 31[2], be adopted as the official Baumé scale of the association, and

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 121.
[2] U. S. Bur. Standards Circ. **44**: (1918), 151.

that all Baumé tables and references thereto which are not in accordance with this scale be eliminated from the methods of the association; and further, that the Committee on Editing Methods of Analysis be authorized to make such changes in the text of the Chapter on Saccharine Products[1] as may be necessary to make this recommendation effective.

Approved.

CRUDE FIBER.

It is recommended—

(1) That further study of the preliminary digestion of the sample with pepsin be made in connection with the regular official method and the one filtration method for crude fiber.

Approved.

(2) That the use of the alundum crucible, in the final filtration for crude fiber, be compared with the asbestos pad filter.

Approved.

(3) That the one filtration method[2] be further studied.

Approved.

(4) That the matter of a uniform filtering medium be further studied.

Approved.

STOCK FEED ADULTERATION.

It is recommended that the following 1917 recommendations be continued:

(1) That the work on scratch feed be continued with a view to securing an accurate and satisfactory method of sampling.

Approved.

(2) That the work of developing a method for the quantitative determination of cottonseed hulls in cottonseed meal be continued.

Approved.

ORGANIC AND INORGANIC PHOSPHORUS.

No report or recommendations.

WATER.

It is recommended—

(1) That the method for the determination of water by drying over lime in vacuum[3] be adopted as a tentative method, and be recommended for further study for the ensuing year.

Approved.

(2) That the method for the determination of water by drying over carbide in vacuum[3] be adopted as a tentative method, and be recommended for further study.

Approved.

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 121.

[2] *J. Assoc. Official Agr. Chemists*, 1919, **3**: 256.

[3] *Ibid.*, 1920, **4**: 247.

(3) That the associate referee study the existing official general methods for water in foods and feeding stuffs with a view to rewording and fixing rigidly the conditions of temperature, pressure and other factors.

Approved.

(4) That a definite method applicable to the determination of water in dried fruits be designed and submitted to the association.

Approved.

DAIRY PRODUCTS.

It is recommended—

(1) That a further study be made of the alkaline-acid modification of the Roese-Gottlieb method as applied to dried milk products of various fat content.

Approved.

(2) That a further study be made of the Roese-Gottlieb neutral extraction method as applied to malted milk.

Approved.

(3) That a further study be made of the direct ether extraction method as applied to malted milk.

Approved.

(4) That the Roese-Gottlieb method[1] for fat, as applied to plain ice cream, be adopted as official. (Second and final reading.)

Approved.

(5) That the Schmidt-Bondzynski method[2] modified for the determination of fat in cheese be further studied.

Approved.

(6) That the cryoscopic method for the determination of added water in milk, page 491, be further studied.

Approved.

SEPARATION OF NITROGENOUS SUBSTANCES IN MILK AND CHEESE.

No report or recommendations.

SACCHARINE PRODUCTS.

No report or recommendations.

MAPLE PRODUCTS.

It is recommended—

(1) That the directions for "Preparation of Sample"[3] be amended to read as follows:

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 289.

[2] *Ibid.*, 297.

[3] *Ibid.*, 136.

53 PREPARATION OF SAMPLE.—TENTATIVE.

(a) *Maple sirup.*—Determine the moisture by one of the methods given under **54 (a)**. If sugar has crystallized out, redissolve it by warming. Shake up any sediment remaining and pour off into a casserole or beaker a suitable quantity for analysis (100 cc.). Add one-fourth the volume of distilled water. Boil to a temperature of 104° C. Filter hot through a plug of cotton wool in a funnel. After cooling redetermine the moisture according to **54 (a)** and use the result to reduce the other values determined to the dry substance basis.

(b) *Maple sugar and other solid or semi-solid products.*—Determine moisture by one of the methods given under **54 (b)** after thoroughly mixing the sample. For all other determinations prepare a sirup from about 100 grams of the material by dissolving in about 150 cc. of hot water, boiling to a temperature of 104° C. and filtering through cotton wool as in (a). After cooling determine the moisture in the sirup according to one of the methods of **54 (a)**.

Approved.

(2) That in the tentative method for the determination of moisture. **54 (a)**,[1] the refractometer method, **10**[2], be given the preference, and that the other method to be recommended in **54 (a)** and **(b)** be decided upon by the referee on saccharine products. (See Report of Associate Referee on Maple Products 1917[3].)

Approved.

(3) That the tentative method for total ash[4] be amended to read as follows:

Heat 5 grams of the prepared sirup over a low flame (an Argand burner is recommended) until completely charred. Transfer to a muffle and heat at low redness (not over 550°), until a white ash is obtained. (If desired, the ashing may be interrupted when the carbon is nearly all burned, and, after cooling, 0.5—1.0 cc. of water may be added and evaporated.) After cooling, add about 0.1 gram of ammonium carbonate, free from nonvolatile matter, and add 0.5—1.0 cc. of water. Evaporate to dryness and reheat in the muffle for 1—2 minutes. Cool in a dessicator and weigh.

Approved.

(4) That the Canadian lead method[5] be adopted as a tentative method. (First reading.)

Approved.

(5) That the caption above **IX, 63**[4], be amended to read, "Winton Lead Number.—Tentative."

Approved.

(6) That further study be given to the question of omitting the words "at least" before "3 hours" in **IX, 65,** line 3[4].

Approved.

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[1] *Assoc. Official Agr. Chemists, Methods,* 1916, 136.
[2] *Ibid.,* 126.
[3] *J. Assoc. Official Agr. Chemists,* 1920, **4**: 157.
[4] *Assoc. Official Agr. Chemists, Methods,* 1916, 137.
[5] *J. Assoc. Official Agr. Chemists,* 1921, **4**: 437.

(7) That the conductivity value method[1] be adopted as a tentative method. (First reading.)

Approved.

HONEY.

No report or recommendations.

SUGAR-HOUSE PRODUCTS.

(1) That a study be made of the influence of different and known temperatures of incineration on the results of ash determinations in cane sirups and molasses, carrying out the incineration in both platinum and silica dishes for comparison.

Approved.

(2) That a large number of samples of different grades of cane sirups and molasses be used for comparing ash determinations by the sulphate and direct methods, to determine, if possible, the proper correction factor to be applied to sulphated ash.

Approved.

DRUGS.

MEDICINAL PLANTS.

It is recommended—

(1) That work be continued to determine the value of a more extended use of volume weight determination in the analysis of crude drugs and spices.

Approved.

(2) That the subject of sublimation for the analysis of plant products, etc., be further studied.

Approved.

(3) That the methods for the macroscopic and microscopic identification of *Digitalis thapsi* (Spanish digitalis), a recent substitute for *Digitalis purpurea*, and *Hyoscyamus muticus* (Egyptian henbane), a substitute for *Hyoscyamus niger*, be studied by collaborators.

Approved.

(4) That the method for the detection of the presence of santonin in wormseed (*Artemisia cinae*), and subsequent isolation, be studied by collaborators.

Approved.

ALKALOIDS.

It is recommended—

(1) That the following be added to the method for the assay for strychnin in tablets[2], and that the method be further studied by collaborators, with a view to making it official:

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[1] *J. Assoc. Official Agr. Chemists*, 1921, **4**: 435.

[2] *Ibid.*, 1919, **3**: 189; 1920, **3**: 379.

Check the weight of the strychnin by dissolving the residue in neutral alcohol, adding an excess of N/10 sulphuric acid, and titrating back with N/50 potassium hydroxid, using methyl red as the indicator. One cc. of N/10 sulphuric acid is equivalent to 0.0334 gram of strychnin and 0.0428 gram of strychnin sulphate. The U. S. P. factor for strychnin to strychnin sulphate is 1.2815.

Approved.

(2) That the following be added to the method for the assay for strychnin in liquids[1], and the method be further studied by collaborators, with a view to making it official:

Check the weight of the strychnin by dissolving the residue in neutral alcohol adding an excess of N/10 sulphuric acid, and titrating back with N/50 potassium hydroxid, using methyl red as the indicator. One cc. of N/10 sulphuric acid is equivalent to 0.0334 gram of strychnin and 0.0428 gram of strychnin sulphate. The U. S. P. factor for strychnin to strychnin sulphate is 1.2815.

Approved.

(3) That the method submitted for the separation of quinin and strychnin[2] be further studied by collaborators.

Approved.

(4) That the methods for the assay of physostigma and its preparations[3] be studied by collaborators.

Approved.

(5) That the method submitted for the assay of fluid extract[4] of hyoscyamus be studied by collaborators.

Approved.

(6) That the comparative study of the volumetric and gravimetric methods for the assay of ipecac be subjected to collaborative investigation.

Approved.

SYNTHETIC PRODUCTS.

It is recommended—

(1) That the method of W. O. Emery and C. D. Wright for the valuation of hexamethylenetetramin tablets[5] be studied by collaborators.

Approved.

(2) That the method of W. O. Emery for the estimation of monobromated camphor in migraine tablets[6] be studied by collaborators.

Approved.

(3) That the methods for the determination of phenolphthalein be studied further with a view to submitting one or both to collaborators.

Approved.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **3**: 379.
[2] *Ibid.*, 1921, **4**: 416.
[3] *Ibid.*, 418.
[4] *Ibid.*, 419.
[5] *J. Ind. Eng. Chem.*, 1918, **10**: 606.
[6] *Ibid.*, 1919, **11**: 756.

BALSAMS AND GUMS RESINS.

It is recommended that further collaborative work be done upon the method submitted for the determination of crude fiber in gum karaya.

Approved.

ENZYMS.

It is recommended—

(1) That the tentative method for the determination of pepsin[1] be subjected to further study, with a view to making it official.

Approved.

(2) That the method developed by V. K. Chesnut for the estimation of papain[2] be studied.

Approved.

ESSENTIAL OILS.

It is recommended that the method submitted by C. W. Harrison[3] for the acetyl value of santal oil be further studied.

Approved.

TESTING CHEMICAL REAGENTS.

It is recommended—

(1) That further work be done on methods for the estimation of small amounts of impurities in chemicals.

Approved.

(2) That the study of methods for the determination of the strength of acetic anhydrid be continued.

Approved.

(3) That work on the testing of purity of immiscible solvents be continued.

Approved.

MICROANALYTICAL METHODS.

No report or recommendations.

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[1] *Assoc. Official Agr. Chemists, Methods,* 1916, 363.

[2] *J. Assoc. Official Agr. Chemists,* 1920, **3**: 391.

[3] *Ibid.*, 1921, **4**: 426.

REPORT OF COMMITTEE C ON RECOMMENDATIONS OF REFEREES.

By R. E. Doolittle (Transportation Building, Chicago, Ill.), *Chairman.*

[Food preservatives, coloring matters in foods, metals in foods, fruits and fruit products' canned vegetables, cereal foods, wines, soft drinks (bottlers' products), distilled liquors, beers, vinegars, flavoring extracts, meat and meat products (separation of nitrogenous compounds in meat products, meat extracts), eggs and egg products, gelatin, edible fats and oils, spices and other condiments, cacao products, coffee, tea, baking powder.]

FOOD PRESERVATIVES.

No report or recommendations received.

Your committee, however, submits the following suggestions, from the recommendations of the referee for 1917, for study during the coming year:

(1) That further work be done on Method II[1], for the determination of saccharin in the presence of mustard oil.

Approved.

(2) That other methods not dependent upon the sulphur component of saccharin be investigated.

Approved.

(3) That further work be done upon the determination of saccharin in baked flour preparations.

Approved.

COLORING MATTERS IN FOODS.

The report of the referee was not referred to Committee C.

(1) Your committee understands, however, that there has recently been completed in the Bureau of Chemistry some work on methods for the separation and adulteration of oil-soluble dyes. It is suggested that these methods be investigated by the referee for collaborative study.

Approved.

(2) Your committee further recommends that the referee undertake a study of the methods in the Chapter on Coloring Matters in Foods[2] for the purpose of recommending at the next meeting such methods as may be made official.

Approved.

(3) That the work on natural coloring matters be continued.

Approved.

METALS IN FOODS.

The referee submits the following recommendations[3]:

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[1] *J. Assoc. Official Agr. Chemists.* 1920, 3: 505.
[2] *Assoc. Official Agr. Chemists, Methods*, 1916, 155.
[3] *J. Assoc Official Agr. Chemists*, 1921, 4: 455.

(1) That the Gutzeit method for arsenic be described in its essential details, with explanation of the precautions necessary to obtain uniform results, with a view to its adoption in 1920 as an official method.

Your committee does not approve this recommendation. The committee is of the opinion that the various steps in the methods should be given in such detail that an analyst unfamiliar with the method can make the determinations; that essential details which can not be varied shall be so described and thus made empirical; that where slight changes or modifications may be made they be indicated in the description of the apparatus or manipulation, as the case may be.

Committee action approved by association.

(2) That the volumetric method and the gravimetric method for tin be described in essential details for adoption as official methods in 1920.

Your committee does not approve this recommendation for the reasons given above. As a substitute, your committee recommends that the gravimetric method for the determination of tin[1] be made official and that the volumetric method for the determination of tin[1] be made official. This is the first presentation of the methods to the association for adoption as official methods.

Committee recommendation approved by association.

(3) That further study be made of the Penniman method for tin in canned foods, and, if studies now in progress justify further test of this method or modifications of it, collaborative work be done on the method.

Your committee approves this recommendation.

Approved by association.

(4) That methods for determining zinc, aluminium, and copper be made the subject of study as soon as possible.

Your committee approves this recommendation.

Approved by association.

FRUITS AND FRUIT PRODUCTS.

No report of referee or recommendations received.

Your committee desires to call attention to the recommendation made in 1917 that methods for the detection of pectin from apple pomace, used in the manufacture of jellies and jams, be studied.

CANNED VEGETABLES.

No report of referee or recommendations received.

Your committee desires to call attention to the recommendation made at the 1917 meeting that the referee be instructed to study methods peculiarly adapted to the examination of canned foods, especially methods for the detection of spoilage and conditions which are likely to lead to spoilage.

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 173.

CEREAL FOODS.

No report of referee or recommendations received.

Your committee desires to call to the attention of the incoming referee the recommendations made at the 1917 meeting[1], which were as follows:

It is recommended—

(1) That the work on the determination of moisture, gluten, soluble carbohydrates, cold water extract, chlorin, and ash be continued.
Approved.

(2) That the referee study methods for the determination of fat in baked cereal products.
Approved.

WINES.

The referee submitted the following recommendations[2]:

(1) That the subject of wines be dropped from the association work on account of prohibition.

It is the opinion of your committee that it would be inadvisable to discontinue the referee on wines for reason of the necessity of work on methods for grape juice and other fruit juice beverages. It is therefore recommended that the referee on wines be continued.

Committee recommendation approved by association.

(2) That an additional study of the Rothenfusser method be made upon vinegars or grape juice.
Your committee approves this recommendation.
Approved by association.

Your committee makes the following additional recommendation:

(3) That studies be undertaken for the development of a method for the detection of methyl anthranilate, used for imparting flavor to the so-called "dealcoholized wines", grape juice preparations, and similar products.
Approved.

SOFT DRINKS (BOTTLERS' PRODUCTS).

The referee, in his 1917 report, refers to the preliminary work that has been done in his laboratory on methods for the analysis of soft drinks, and recommends that the work be continued.

Your committee approves this recommendation.
Approved by association.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 253.
[2] *Ibid.*, 1921, **4**: 463.

DISTILLED LIQUORS.

The referee has submitted no recommendations for work for the coming year. Your committee has no lines of work to suggest, and unless some member of the association has a suggestion to make as to work that might be taken up advantageously by a referee on this subject, your committee suggests that the position of referee on distilled liquors be discontinued.

Approved.

BEERS.

No report of referee or recommendations received.

Your committee submits the following suggestions for the work of the referee during the coming year:

(1) That a study be made of the method for the determination of alcohol to determine limits of accuracy.

Many of the revenue and regulatory laws now define the term "intoxicating beverage", and it is essential that the analyst should know definitely the limits of the method he is using and may be called upon to defend in court.

Approved.

(2) That methods for the analysis of cereal beverages or so-called "near beers" be studied.

Due to the prohibition laws, this class of preparations is appearing upon the market in large numbers and under various names, and it is important that the control analyst should know to what extent existing methods are applicable, and have information regarding other methods for the analysis of these preparations.

Approved.

VINEGARS.

The referee on vinegars recommends that the methods for the following determinations[1] be made official, pages 467-8:

1, PHYSICAL EXAMINATION.
2, PREPARATION OF SAMPLE.
3, SPECIFIC GRAVITY.
4, ALCOHOL.
8, TOTAL REDUCING SUBSTANCES BEFORE INVERSION.
9, REDUCING SUGARS BEFORE INVERSION AFTER EVAPORATION.
10, REDUCING SUGARS AFTER INVERSION.
13, ASH.
14, SOLUBLE AND INSOLUBLE ASH.
15, ALKALINITY OF THE SOLUBLE ASH.
16, SOLUBLE AND INSOLUBLE PHOSPHORIC ACID.
17, TOTAL ACIDS.
23, PENTOSANS.

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[1] *Assoc. Official Agr. Chemists, Methods*, 1916, 253.

Your committee finds that all of the above methods, with the exception of two, viz, "Physical Examination" and "Alcohol," were made official by the association at the 1917 meeting[1] upon recommendation of the Committee on Editing Methods of Analysis.

It is therefore recommended—

(1) That the methods for "Physical Examination" and "Alcohol"[2] be made official.

First presentation of these methods for adoption as official methods.
Approved.

(2) That the incoming referee select the more important of the remaining methods[2] for further critical work with a view to improvement, namely:

5, GLYCEROL.
7, SOLIDS.
18, FIXED ACIDS.

Your committee approves this recommendation.
Approved by association.

FLAVORING EXTRACTS.

The referee on flavoring extracts submits the following recommendations, page 479:

(1) That the rapid methods for vanillin (Folin's quantitative), coumarin (Wichmann's qualitative), and lead number (Wichmann's quantitative), while meritorious, be held in abeyance until: (*a*) Sufficient data are collated on authentic samples of vanilla extracts to enable satisfactory interpretation of analyses; (*b*) the new lead number is submitted in some form in which it will not be confused with the present official method; and (*c*) a satisfactory quantitative rapid method for coumarin has been developed. Investigations along these lines by individuals, especially by the authors of the methods, are urged.

Your committee approves this recommendation.
Approved by association.

(2) That a study of methods of analysis of imitation vanilla preparations containing large quantities of coumarin and vanillin be undertaken.

Your committee approves this recommendation.
Approved by association.

(3) That Hortvet and West's method[3] for alcohol in lemon and orange extracts be adopted as an official alternative method for extracts consisting only of oil, alcohol and water.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, 4: 264.
[2] *Assoc. Official Agr. Chemists, Methods*, 1916, 253.
[3] *J. Ind. Eng. Chem.*, 1909, 1: 84.

Your committee approves this recommendation.
Approved by association.

(4) That Randall's method[1] for the determination of oil in lemon and orange extracts be studied in connection with the official method.

Your committee approves this recommendation.
Approved by association.

(5) That the methods of analysis of non-alcoholic flavoring extracts be studied.

Your committee approves this recommendation.
Approved by association.

MEAT AND MEAT PRODUCTS.

The referee on meat and meat products submits the following recommendations, page 501:

(1) That as soon as means are provided for the prompt publication of the proceedings of the association, a thorough revision be made of the methods for the examination of meat and meat products.

Your committee calls attention to the fact that the methods for meat and meat products[2] were referred to a special committee at the 1916 meeting of the association, and that committee rendered a very exhaustive report which covered a rearrangement of the methods under this chapter, the deletion and modification of several of the methods and the introduction of new methods. The report was carefully considered by the association and adopted[3]. These changes have all been introduced in the revised manuscript submitted by the Committee on Editing Methods of Analysis. Your committee sees no reason for the adoption of a recommendation for a thorough revision of the methods for the examination of meat and meat products at this time, and therefore has not approved this recommendation.

Committee action approved.

(2) That the method for the estimation of sugar in meat, described on page 499, be adopted tentatively in place of the present tentative method[4].

Your committee approves this recommendation.
Approved by association.

(3) That one referee and two associate referees on meat and meat products be appointed as at present, but that the work of the associates be not designated.

Your committee appreciates the arguments advanced by the referee

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[1] *J. Ind. Eng. Chem.*, 1914, **6**: 926.
[2] *Assoc. Official Agr. Chemists, Methods*, 1916, 271.
[3] *J. Assoc. Official Agr. Chemists*, 1920, **3**: 563.
[4] *Assoc. Official Agr. Chemists, Methods*, 1916, 278.

for the change recommended in the designation of associate referees under meat and meat products, and it is believed that the plan suggested would tend to a correlation of the work on a given subject and a general consideration of all of the methods thereunder in much better manner. Your committee therefore approves this recommendation.

Approved by association.

SEPARATION OF NITROGENOUS COMPOUNDS IN MEAT PRODUCTS.

The associate referee submits the following recommendations, page 506:

(1) That further work be done on the Schlösing-Wagner method for the determination of nitrates, using beef extract, meat and other meat products.

Your committee approves this recommendation.

Approved by association.

(2) That the referee for next year attempt to determine the relative amounts of some of the dissociation products in water-soluble and water-insoluble meat proteins.

Your committee approves this recommendation.

Approved by association.

(3) That the title of the method be changed from "Nitrates" to "Nitrates and Nitrites (calculated as sodium nitrate)".

Your committee approves this recommendation.

Approved by association.

MEAT EXTRACTS.

The associate referee submits the following recommendations, page 506:

(1) That an attempt be made to determine the relative amounts of some of the dissociation products in water-soluble and water-insoluble meat proteins. This probably can best be accomplished by studying certain groups of amino acids, or other protein derivatives, in meat and meat extracts, in collaboration with other referees to be appointed by the association.

Your committee approves this recommendation.

Approved by association.

(2) That the work on the separation of some of the amino acids derived from meat proteins be continued.

Your committee approves this recommendation.

Approved by association.

(3) That the associate referee be not bound to a single method, but be left to choose as circumstances dictate and the collaborators accept.

Your committee does not understand that the recommendations for lines of work are to be interpreted as binding a referee to any particular method or single line of work. They are based upon work done in the past, which it is desired will be carried to completion, and are made merely as suggestions to the referees and associate referees in order to insure a continuation of work under way. However, in order that there may be no misunderstanding in the mind of the associate referee, your committee has approved this recommendation.

Approved by association.

EGGS AND EGG PRODUCTS.

The referee on eggs and egg products submits the following recommendations, page 515:

(1) That the Folin method for the determination of ammoniacal nitrogen in eggs be adopted as a tentative method.

(2) That further comparisons of Klein's method and that of the United States Department of Agriculture for the determination of dextrose be made with a view to adopting one of them as official.

(3) That further work be done on the methods as given for the determination of lecithin-phosphoric acid in dried eggs and alimentary pastes.

(4) That work be done on the determination of heavy metals in egg products.

(5) That work be done on methods for the detection of decomposition in dried eggs.

Your committee is of the opinion that it would be inadvisable at this time to adopt a single determination for a new chapter of methods. From your committee's understanding of the discussion at the 1917 meeting, it was intended that the referee should present, for the information of the members of the association, a set of methods covering the usual determinations made in the analysis of eggs and egg products. Your committee therefore recommends—

(1) That the referee for the coming year prepare and present at the next meeting a compilation of the methods used by the members of the association for the examination of eggs and egg products.

Approved.

(2) That the collaborative work and studies suggested by the referee in his Recommendations 2, 3, 4 and 5 be undertaken in so far as possible.

Approved.

GELATIN.

The referee made no recommendations.

(1) Your committee understands that it was the intention of the association that the referee should compile, for the information of the members of the association, the methods in general use for the analysis

of gelatin. This is called to the attention of the referee in order that such a set of methods may be presented at the next meeting of the association.

Approved.

(2) Your committee recommends a continuation of the work on the methods submitted by the referee in his 1919 report, page 520.

Approved.

EDIBLE FATS AND OILS.

The referee on edible fats and oils submits the following recommendations, page 523:

(1) That the Hübl method for the determination of the iodin number[1] be dropped from the official methods.

Your committee approves this recommendation. The committee had considered the reason advanced by the referee for the elimination of this method, but was of the opinion that if the Hübl method was used by any considerable number of the members of the association it should not be dropped. Upon inquiry, however, it was found that this method is not used, hence the recommendation that it be dropped.

Approved by association.

(2) That the use of the Wijs method, as adopted by the American Chemical Society, be made optional under the official methods.

Your committee recommends that the Wijs method be adopted as a tentative method and taken up by the referee for study during the coming year.

Committee recommendation approved.

(3) That all reports of iodin numbers specify the method used, and where no method is specified it shall be understood that the determination was made by the Hanus method.

Your committee approves the first part of this recommendation, viz, that all reports of iodin numbers shall specify the method used.

Committee recommendation approved.

SPICES AND OTHER CONDIMENTS.

The referee on spices and other condiments recommends, page 526:

(1) That the modification of the distillation method for the determination of water in whole spices, page 524, be further studied for one year, with particular reference to temperature necessary to drive over all the water, and length of time required.

Your committee approves this recommendation.

Approved by association.

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[1] *Assoc. Official Agr. Chemists, Methods,* 1916, 304.

(2) That the tentative method for the determination of volatile oil in mustard seed be adopted as an official method. This is the first presentation of the method for adoption as an official method.

Your committee approves this recommendation.
Approved by association.

CACAO PRODUCTS.

No report of referee or recommendations received.

(1) Your committee, however, desires to call attention to the recommendation adopted at the 1917 meeting for a continuation of the study of methods for the detection of adulteration in cocoa butters, and particularly to the recommendation made by the referee at the 1916 meeting for the further study of the method for determination of the critical temperature of dissolution with a view to its adoption as a tentative method.

(2) Also to the necessity for the development of methods for the determination of added cocoa shells to cocoa and similar products.

COFFEE.

(1) The referee on coffee suggests certain slight modifications in the Fendler-Stüber method for the determination of caffein in coffee which was adopted as a tentative method by the association in 1917[1].

Your committee recommends that the method as modified, page 533, be substituted for the method adopted by the association in 1917 and remain as a tentative method.
Approved.

(2) That further study be made of the method before it is adopted as official or superseded.

Your committee approves this recommendation.
Approved by association.

TEA.

The referee on tea recommends, page 538:

(1) That the tentative modified Stahlschmidt method[2], be made official for the determination of caffein in tea, except that the caffein residue be dried at 100° C. instead of at 75° C.

This is the first presentation of the method for adoption as an official method.

This recommendation is approved by your committee.
Approved by association.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, 4: 257.
[2] *Assoc. Official Agr. Chemists, Methods*, 1916, 332.

(2) It is further recommended by your committee that, inasmuch as the above recommendation provides for the drying of the caffein residue at 100° C. instead of 75° C. in the determination of caffein in tea, that a similar change be made in the same method which is used for the determination of caffein in coffee. It is therefore recommended that the tentative modified Stahlschmidt method for the determination of caffein in coffee[1] be changed as follows:

15, *Modified Stahlschmidt Method.—Tentative.*

Line 15.—Change "75° C." to "100° C."

Approved.

(3) That the method for the determination of water extract, page 537, be substituted for the present tentative method.

This recommendation is approved by your committee.

Approved by association.

(4) That methods for the determination of tannin be studied next year. The cinchonin sulphate method[2] particularly is suggested.

This recommendation is approved by your committee.

Approved by association.

BAKING POWDER.

The referee on baking powder submits the following recommendations, page 539:

(1) That the Wagner-Ross method for the determination of fluorin in phosphate and in baking powders be adopted as a tentative method.

Your committee approves this recommendation.

Approved by association.

(2) That the referee study methods for the removal of water from baking powders to be analyzed by the Wagner-Ross method for fluorin.

Your committee approves this recommendation.

Approved by association.

(3) That the referee study the question of the addition of phosphoric acid to tartrate and to alum powders to insure deposition of lead in the electrolytic method for the determination of lead in baking powders, adopted as a tentative method by the association at the 1917 meeting[3].

Your committee approves this recommendation.

Approved by association.

(4) That the referee study methods for the determination of the neutralizing strength of baking acids.

Your committee approves this recommendation.

Approved by association.

The meeting adjourned at 1 p. m. to reconvene at 2 p. m.

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[1] *Assoc. Official Agr. Chemists, Methods,* 1916, 332.

[2] *Analyst,* 1913, **38**: 312.

[3] *J. Assoc. Official Agr. Chemists,* 1920, **4**: 257.

THIRD DAY.

WEDNESDAY—AFTERNOON SESSION.

REPORT OF COMMITTEE TO COOPERATE WITH OTHER COMMITTEES ON FOOD DEFINITIONS[1].

Your committee submits the report of the activities of the Joint Committee on Food Definitions and Standards, of which your committee is one of the three coordinate parts. This report covers the period since the thirty-fourth meeting of this association in November, 1917[2].

CHANGES IN MEMBERSHIP.

The membership of the joint committee has changed considerably since that time. Of the committee from this association, John Phillips Street resigned in 1917 to enter the dietary and food conservation service of the Medical Corps of the Army, and was succeeded by C. D. Howard, chemist to the New Hampshire State Board of Health. Of the committee from the Association of American Dairy, Food and Drug Officials, W. F. Hand of the Mississippi Agricultural and Mechanical College, having resigned, David Klein, chemist to the Illinois Division of Foods and Dairies, was appointed his successor, but before entering upon his duties with the committee Klein also resigned to enter overseas war service, and was in turn succeeded, in 1918, by Wyatt W. Randall, chemist to the Maryland State Department of Health. Early in the present year, H. E. Barnard, in contemplation of his early retirement from the commissionership of food and drugs of the State of Indiana, tendered his resignation as a member of the committee, and F. C. Blanck, Food and Drug Commissioner of the Maryland State Department of Health, was appointed his successor.

Your committee begs to express its great indebtedness to Street for highly valuable contributions made by him to the work of the joint committee, and also its warm appreciation of the service rendered by Barnard and Hand of the committee from our sister association.

Most of you found your normal routine largely upset by the demands of war service during 1917 and 1918. The joint committee's work was also slowed down from like cause, as well as by reason of the unusually great change in personnel during this period.

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[1] Presented by William Frear.

[2] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 275.

HEARINGS.

The following list of hearings given by the joint committee during the past two years will show, however, that its activities have not been wholly arrested:

Butter:
  June 19, 1918, St. Paul, Minn.
  June 24, 1918, Washington, D. C.
  January 20, 1919, Chicago, Ill.
Corn meal:
  December 3, 1918, Washington, D. C.
Jellies, jams, preserves, etc.:
  May 27, 1918, Washington, D. C.
  June 10, 1918, San Francisco. Calif.
  December 5, 1918, Washington, D. C.
Oleomargarine:
  December 4, 1918, Washington, D. C.
Sirups, molasses:
  August 7, 1918, Washington, D. C.
  April 4, 1919, New Orleans, La.

In addition to the foregoing it may be recalled that, at the suggestion of the joint committee, the National Canners Association, with the endorsement of the National Wholesale Grocers Association, undertook the initiative in the formulation of grade standards for the various canned foods. The chairman of your committee was designated by the joint committee to collaborate in an advisory relation with the several committees of the National Canners Association in this important work. In this relation he has attended meetings upon the following subjects:

Tomato paste:
  August, 1918, Baltimore, Md.
  January 21, 1919, Chicago, Ill.
Canned fruits:
  January 21, 1919, Chicago, Ill.
Canned corn:
  May 10, 1919, Washington, D. C.
  July 8, 1919, Chicago, Ill.
Canned tomatoes:
  November 18, 1919, Washington, D. C.

The species and grade standards formulated and approved by the National Canners Association committee for tomato paste and canned corn have been received by the Joint Committee on Food Definitions and Standards and approved, with only a few very minor changes, for publication prior to final adoption. The work of the Manufacturers Committee on canned tomatoes and canned string beans also is in a very advanced stage of formulation.

ACTION OF DEPARTMENT OF AGRICULTURE.

Since our last report the Department of Agriculture has acted upon a number of schedules previously approved by the two associations, and has adopted these schedules as approved by you except in two minor instances. In conformity, the Secretary of Agriculture has proclaimed the following food inspection decisions:

No. 172.—Condiments other than vinegars and salt. February 28, 1918.

No. 173.—Canned vegetables, canned peas, and canned pea grades. March 9, 1918.

No. 174.—Baking powder. March 15, 1918.

No. 176.—Evaporated apples. June 6, 1918.

No. 178.—Milk and cream. April 17, 1919.

The exceptions made by the Department of Agriculture affected:

(1) The method suggested for the determination of moisture in evaporated apples. This association having adopted a method for this determination, the department substituted the latter method for the trade method which the joint committee had suggested for use pending the approval of a method by this association.

(2) The definition and standard for "standardized milk" was not approved by the department, owing to an objection by its solicitor, who doubted whether the name "standardized milk" had become fully established for this widely sold product.

RECOMMENDATION.

The subjects submitted for action are cheese and butter. At the meeting in November, 1917[1], you adopted a partial schedule for cheese, then recommended to you by the joint committee. The committee has, since then, devoted much time to an extension of the schedule. It now presents for your consideration a schedule of twenty-two definitions in lieu of that with ten definitions which you approved at your last meeting. In the course of the extension a number of revisions with respect to classification of cheeses and to arrangement of subject matter were made. The following extended and revised schedule is presented to you for adoption as a substitute for the cheese schedule approved in November, 1917.

CHEESES.

Definitions and standards adopted by the Joint Committee on Definitions and Standards, September 6, 1919:

1. *Cheese* is the sound product made from curd obtained from the whole, partly skimmed, or skimmed milk of cows, or from the milk of other animals, with or without added cream, by coagulating the casein with rennet, lactic acid, or other suitable enzym or acid, and with or without further treatment of the separated curd by heat or pressure, or by means of ripening ferments, special moulds, or seasoning.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, 4: 284.

By act of Congress, approved June 6, 1896[1], cheese may also contain added coloring matter.

In the United States the name "Cheese", unqualified, is understood to mean Cheddar cheese, American cheese, American Cheddar cheese.

2. *Whole milk cheese* is cheese made from whole milk.

3. *Partly skimmed milk cheese* is cheese made from partly skimmed milk.

4. *Skimmed milk cheese* is cheese made from skimmed milk.

WHOLE MILK CHEESES.

5. *Cheddar cheese, American cheese, American Cheddar cheese,* is the cheese made by the Cheddar process, from heated and pressed curd obtained by the action of rennet on whole milk. It contains not more than thirty-nine per cent (39%) of water, and, in the water-free substance, not less than fifty per cent (50%) of milk fat.

6. *Stirred curd cheese, sweet curd cheese,* is the cheese made by a modified Cheddar process, from curd obtained by the action of rennet on whole milk. The special treatment of the curd, after the removal of the whey, yields a cheese of more open, granular texture than Cheddar cheese. It contains, in the water-free substance, not less than fifty per cent (50%) of milk fat.

7. *Pineapple cheese* is the cheese made by the pineapple Cheddar cheese process, from pressed curd obtained by the action of rennet on whole milk. The curd is formed into a shape resembling a pineapple, with characteristic surface corrugations, and during the ripening period the cheese is thoroughly coated and rubbed with a suitable oil, with or without shellac. It contains, in the water-free substance, not less than fifty per cent (50%) of milk fat.

8. *Limburger cheese* is the cheese made by the Limburger process, from unpressed curd obtained by the action of rennet on whole milk. The curd is ripened in a damp atmosphere by special fermentation. It contains, in the water-free substance, not less than fifty per cent (50%) of milk fat.

9. *Brick cheese* is the quick-ripened cheese made by the brick cheese process, from pressed curd obtained by the action of rennet on whole milk. It contains, in the water-free substance, not less than fifty per cent (50%) of milk fat.

10. *Stilton cheese* is the cheese made by the Stilton process, from unpressed curd obtained by the action of rennet on whole milk, with or without added cream. The cheese, ripened by a special blue-green mould, has a mottled or marbled appearance in section.

11. *Gouda cheese* is the cheese made by the Gouda process, from heated and pressed curd obtained by the action of rennet on whole milk. The rind is colored with saffron. It contains, in the water-free substance, not less than forty-five per cent (45%) of milk fat.

12. *Neufchatel cheese* is the cheese made by the Neufchatel process, from unheated curd obtained by the combined action of lactic fermentation and rennet on whole milk. The curd, drained by gravity and light pressure, is kneaded or worked into a butter like consistence and pressed into forms for immediate consumption or for ripening. It contains, in the water-free substance, not less than fifty per cent (50%) of milk fat.

13. *Cream cheese* is the unripened cheese made by the Neufchatel process from whole milk enriched with cream. It contains, in the water-free substance, not less than sixty-five per cent (65%) of milk fat.

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[1] U. S. Statutes at Large, 1895–7, **29**: ch. 337, 253.

14. *Roquefort cheese* is the cheese made by the Roquefort process, from unheated, unpressed curd obtained by the action of rennet on the whole milk of sheep, with or without the addition of a small proportion of the milk of goats. The curd is inoculated with a special mould (*Penicillium Roqueforti*) and ripens with the growth of the mould. The fully ripened cheese is friable and has a mottled or marbled appearance in section.

15. *Gorgonzola cheese* is the cheese made by the Gorgonzola process, from curd obtained by the action of rennet on whole milk. The cheese, ripened in a cool, moist atmosphere by the development of a blue-green mould, has a mottled or marbled appearance in section.

WHOLE MILK OR PARTLY SKIMMED MILK CHEESES.

16. *Edam cheese* is the cheese made by the Edam process, from heated and pressed curd obtained by the action of rennet on whole milk, or on partly skimmed milk. It is commonly made in spherical form and coated with a suitable oil and a harmless red coloring matter.

17. *Emmenthaler cheese, Swiss cheese,* is the cheese made by the Emmenthaler process, from heated and pressed curd obtained by the action of rennet on whole milk or on partly skimmed milk, and is ripened by special gas-producing bacteria, causing characteristic "eyes" or holes. The cheese is also known in the United States as "Schweitzer". It contains, in the water-free substance, not less than forty-five per cent (45%) of milk fat.

18. *Camembert cheese* is the cheese made by the Camembert process, from unheated, unpressed curd obtained by the action of rennet on whole milk or on slightly skimmed milk, and is ripened by the growth of a special mould (*Penicillium Camemberti*) on the outer surface. It contains, in the water-free substance, not less than forty-five per cent (45%) of milk fat.

19. *Brie cheese* is the cheese made by the Brie process, from unheated, unpressed curd obtained by the action of rennet on whole milk, on milk with added cream, or on slightly skimmed milk, and is ripened by the growth of a special mould on the outer surface.

20. *Parmesan cheese* is the cheese made by the Parmesan process, from heated and hard-pressed curd obtained by the action of rennet on partly skimmed milk. The cheese, during the long ripening process, is coated with a suitable oil.

SKIMMED MILK CHEESES.

21. *Cottage cheese, Schmierkase,* is the unripened cheese made from heated (or scalded) curd obtained by the action of lactic fermentation or lactic acid or rennet, or any combination of these agents, on skimmed milk, with or without the addition of buttermilk. The drained curd is sometimes mixed with cream, salted, and sometimes otherwise seasoned.

WHEY CHEESES.

22. *Whey cheese* (so-called) is produced by various processes from the constituents of whey. There are a number of varieties each of which bears a distinctive name, according to the nature of the process by which it has been produced, as, for example, "Ricotta", "Zieger", "Primost", "Mysost".

BUTTER.

In relation to butter we submit the following statement:

On December 20, 1904, the Food Standards Commission, collaborating with the Secretary of Agriculture under the authorization of Congress set forth in the Agricultural Appropriation Acts of 1903 to 1907, inclusive, recommended, and the Secretary proclaimed, in Circular 13, Office of the Secretary[1], the following definition and standard:

> Butter is the clean, non-rancid product made by gathering in any manner the fat of fresh or ripened milk or cream into a mass, which also contains a small portion of the other milk constituents, with or without salt, and contains not less than eighty-two and five-tenths (82.5) per cent of milk fat. By Acts of Congress approved August 2, 1886, and May 9, 1902, butter may also contain added coloring matter.

This standard the Department of Agriculture republished in 1906 in Circular 19[2] and, in 1919, in Circular 136[3] of the same series.

In connection with the study of the other definitions and standards for various milk products, the question of the need for a revision of those for butter and of the legal limitations that might hem in the committee in dealing with this product, upon which Congress had enacted more or less specific laws, arose for consideration.

REVISIONAL INQUIRIES.

The committee adopted three lines of inquiry in its work upon this subject:

(1) With the manufacturers, dealers and educational experts it raised questions as to the definition and what it should contain; the need for a butter classification; and as to the standard or standards, upon what and how many points of reference regarding composition it should be based.

(2) By the use of the inspection and investigational agencies, State and National, a survey of conditions as to raw materials, methods of manufacture and composition of product, was made. This survey was especially emphasized to cover points on which the statements of competitive commercial interests considerably differed.

(3) Collaterally, there was framed for submission to the Solicitor's Office of the Department of Agriculture, questions intended to elicit competent opinions upon all questions of law having relation to our work upon this definition and standard.

The methods followed are here stated to illustrate the care used in the gathering of facts and in the determination of the joint committee's powers. It will be impossible in this report even to summarize the representations and findings upon all the topics discussed in the hearings, briefs and inspectional reports. In essence, the trade desired a liberally

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[1] U. S. Dept. Agr., Office of the Secretary, Circ. **13**: (1904), 7.
[2] *Ibid.*, **19**: (1906), 6.
[3] *Ibid.*, **136**: (1919), 5.

inclusive definition; that water, or water and fat, the former preferably, should be the basis of the standard; and that, specifically, 16 per cent maximum of water, or 16 per cent maximum of water and 80 per cent minimum of fat should be the standard. Direct inquiry elicited no representation that water in excess of 14.5 to 15 per cent improves the butter in any physical quality. Manifestly, however, it increases the weight for which butter price is charged.

The inspectional evidence tended to the conclusion that the farm butters, and very often those made by the smaller creameries, were relatively dry, while those made by the large creameries were, not without exception, relatively wet; that the large creameries possessed the more expert management and were able to determine quite exactly and to control within 0.5 per cent, as a rule, the water content of their butter output, and that these creameries almost universally aimed to keep in, or, more exactly, to put in enough water to bring that constituent in the butter as closely up to the 16 per cent maximum as their control of operations made safe.

This 16 per cent maximum was fixed, not directly by law, but by regulation of the Commissioner of Internal Revenue to simplify his duties under the Adulterated Butter Act of 1902[1], to which reference will later be made. It was fixed to give a reasonable limit for variations that were supposedly unavoidable with the methods then in use, even when reasonable care was exercised. The improvement in factory methods has materially diminished the manufacturing risks where the oldtime dryer butter is the aim of the maker. What the regulation presumed to be occasional and accidental has now become nearly invariable and intentional on the part of most large creamery managers.

LEGAL LIMITATIONS UPON THE WORK OF THE JOINT COMMITTEE.

It is not possessed of legislative power and is, of course, strictly subject to the provisions of existing Federal food and drug laws. In addition to the Food and Drugs Act of June 30, 1906[2], with the provisions of which you are doubtless familiar, Congress has enacted three laws dealing specifically with butter or butters. These laws are as follows, as far as they relate to our problem:

> That for the purpose of this act, the word "butter" shall be understood to mean the food product usually known as butter, and which is made exclusively from milk or cream, or both, with or without common salt, and with or without additional coloring matter[3].

This definition was reaffirmed by Congress in 1902 in a section of the Adulterated Butter Act[1].

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[1] U. S. Statutes at Large, 1901–3, **32** (I): ch. 784, 194.
[2] *Ibid.*, 1905–7, **34** (I): ch. 3915, 768.
[3] *Ibid.*, 1885–7, **24**: ch. 840, 209.

* * * * "adulterated butter' is hereby defined to mean a grade of butter produced by mixing, reworking, rechurning in milk or cream, refining, or in any way producing a uniform, purified, or improved product from different lots or parcels of melted or unmelted butter or butter fat, in which any acid, alkali, chemical, or any substance whatever is introduced or used for the purpose or with the effect of deodorizing or removing therefrom rancidity, or any butter or butter fat with which there is mixed any substance foreign to butter as herein defined, with intent or effect of cheapening in cost the product or any butter in the manufacture or manipulation of which any process or material is used with intent or effect of causing the absorption of abnormal quantities of water, milk or cream.

The third Federal law established the following butter standard for the District of Columbia[1]:

Butter is adulterated if it contains more than twelve percentum of water, more than five percentum of salt, and less than eighty-three percentum of fat.

In the opinion of the Solicitor of the Department of Agriculture the Food and Drugs Act worked no repeal of these earlier butter acts. They are held to be of coordinate value.

How could pasteurized cream be required in making butter under the definition enacted in 1886 and reaffirmed in 1901? How could a harmless neutralizer be specifically admitted, if considered on economic grounds desirable, under that definition unless its use was a part of the process of manufacture usual in 1886? Even the introduction of a little water, eliminated with the buttermilk, is probably allowable under that definition only upon the assumption that it was an essential and usual part of the manufacturing method at that time, which it undoubtedly was.

The joint committee is not aware of any regionally characteristic differences in butter composition, existent at the time of the enactment by Congress of the butter standard for the District of Columbia, wherein 83 per cent is set as the fat minimum. It has no official authorization to perform the judicial function of deciding the relative legal standing of the 12 per cent water maximum in the District of Columbia butter standard as contrasted with the 16 per cent maximum fixed in the regulation of the Commissioner of Internal Revenue under the Act of 1902; still less is it authorized to set both of the limits aside and substitute a limit intermediate between 12 and 16 per cent.

Finally, allow us to note, as a matter of experience seriously deserving recognition, that to gain a conviction for violation of a food law requires a wider deviation from standard than it does in the enforcement of tax laws.

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[1] U. S. Statutes at Large, 1897–9, 30: ch. 25, 246

The joint committee therefore recommends no present change in the butter standard as it is set forth in Circular 136[1], as above quoted.

Respectfully submitted,

WILLIAM FREAR,
JULIUS HORTVET,
C. D. HOWARD.
Committee to Cooperate with Other Committees on Food Definitions.

Adopted.

REPORT OF COMMITTEE ON METHODS OF SAMPLING FERTILIZERS TO COOPERATE WITH A SIMILAR COMMITTEE OF THE AMERICAN CHEMICAL SOCIETY[2].

Shortly after the meeting of this association in 1917, your committee formulated plans and commenced operations designed to show chemical differences between samples taken from similar sources with different sampling tubes. The types of sampling tubes selected were known as the Indiana, the Massachusetts, and the Georgia. Through the courtesy of the Armour Fertilizer Works and under the direction of F. S. Lodge, Chairman of the American Chemical Society Committee, suitable amounts of a 2–8–2 and a 10–5 goods were carefully prepared, special attention being given to a thorough mixing of the several ingredients. It was planned to have these fertilizers sampled at Chicago, the place of manufacture, and portions shipped to Jeffersonville, Ind., and Atlanta, Ga., where additional samples would be drawn.

W. J. Jones, jr., late State Chemist of Indiana, arranged to have his inspector, O. S. Roberts, take all of the samples at the places indicated. This was done and the necessary sets for analysis were prepared from these official samples under the direct supervision of E. G. Proulx (Agricultural Experiment Station, La Fayette, Ind.), every precaution being taken to insure uniformity in manipulation. Each set comprised 29 samples, numbered from 1 to 29, inclusive, with the marks 2–8–2 or 10–5, as the case might be, to indicate the grade. No clue as to the method employed in securing the samples was given to any of the analysts taking part in this work.

Samples were sent to, and results received from, the following collaborators:

Virginia Carolina Chemical Company, Richmond, Va.
Fertilizer Department, Clemson College, S. C.
Department of Agriculture, Atlanta, Ga.
Agricultural Experiment Station, La Fayette, Ind.
Agricultural Experiment Station, Burlington, Vt.
Armour Fertilizer Works, Atlanta, Ga.

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[1] U. S. Dept. Agr., Office of the Secretary, Circ. 136: (1919), 5.

[2] Presented by C. H. Jones.

The results requested entailed a large amount of analytical work, about 1200 separate determinations being made, and the committee desires to express at this time most hearty thanks and appreciation for the thoroughness and ability shown by all the chemists taking part in this investigation. A detailed compilation of all the results received would be out of place in this report. It is sufficient to say that such a compilation is in the hands of the chairmen of both committees and can be reviewed by any one interested. For present purposes, the following tabulation has been prepared showing the average results obtained by all the collaborators on samples taken at Chicago, Jeffersonville, and Atlanta by the sampling methods indicated in column two.

TABLE 1.

Average results obtained by all the collaborators.

SAMPLE NUMBER	METHOD OF SAMPLING	PLACE	NUMBER OF LABORATORIES REPORTING	NUMBER OF RESULTS INCLUDED IN AVERAGE	WATER	NITROGEN	PHOSPHORIC ACID	POTASSIUM OXID
					per cent	*per cent*	*per cent*	*per cent*
Material 2-8-2:								
1 and 9	Quartering	Chicago	6	12	4.50	1.71	9.35	2.39
2 and 10	Indiana	Chicago	6	12	4.76	1.66	9.28	2.39
3 and 11	Massachusetts	Chicago	6	12	5.18	1.68	9.25	2.34
4 and 12	Georgia	Chicago	6	12	5.25	1.68	9.18	2.27
19	Indiana	Jeffersonville	6	6	4.47	1.70	9.12	2.37
17	Massachusetts	Jeffersonville	6	6	4.93	1.71	9.17	2.35
18	Georgia	Jeffersonville	6	..	4.58	1.72	9.20	2.37
24	Indiana	Atlanta	6	6	4.52	1.62	9.40	2.43
25	Massachusetts	Atlanta	6	6	4.28	1.69	9.53	2.42
23	Georgia	Atlanta	6	6	4.11	1.65	9.37	2.37
29	Cup	Atlanta	6	6	4.76	1.66	9.43	2.42
Material 10-5:								
5 and 13	Quartering	Chicago	6	12	5.01	....	10.88	4.92
6 and 14	Indiana	Chicago	6	12	4.43	....	10.84	4.91
7 and 15	Massachusetts	Chicago	6	12	5.14	....	10.84	4.90
8 and 16	Georgia	Chicago	6	12	4.51	....	10.91	4.95
22	Indiana	Jeffersonville	6	6	4.70	....	10.84	4.83
20	Massachusetts	Jeffersonville	6	6	5.25	....	10.79	4.75
21	Georgia	Jeffersonville	6	6	4.49	....	10.87	4.91
27	Indiana	Atlanta	6	6	5.85	....	10.89	5.00
28	Massachusetts	Atlanta	6	6	5.21	....	10.95	5.03
26	Georgia	Atlanta	6	6	4.42	....	11.01	5.00

The analyses of Samples 1 and 9 and 5 and 13, taken by quartering, may be assumed to represent the true composition of the two fertilizers under discussion. A review of the remaining figures indicates that it is not feasible to attempt to draw any definite conclusions as to the particular merits of the three types of samplers investigated. Any differences noted could easily be accounted for by allowable analytical error.

The most perfect agreement in the average amounts of nitrogen, phosphoric acid and potash found in the several samples shows:

(1) The uniformity of results by the official methods employed for nitrogen, phosphoric acid and potash determinations.

(2) That practically no segregation took place in the goods during their journey from Chicago to Jeffersonville, Ind., or Atlanta, Ga. This is emphasized by the results of Sample 29, taken at Atlanta, by means of a cup, from the top of eight bags.

Samples representing the Georgia-type sampler were made up of 32 cores, while the Massachusetts and Indiana types contained 16 cores, respectively. This relatively large number of separate drawings undoubtedly contributed to the similarity of the composite samples secured.

Briefly, this work clearly indicates that a sampling device that removes a core from the bag from top to bottom may secure a representative sample. All of the three types used accomplish this result, provided a sufficient number of bags representing each lot are sampled.

Apparently no particular advantage exists between the three types of samplers under consideration. In the opinion of the committee this is due to the fact that, owing to the nature of the goods, segregation was at a minimum, and the large amounts secured for the composites were sufficient to offset any differences that might have been shown had analyses been made of the cores from individual bags taken by the sampling devices employed.

It was recommended and approved at the 1917 meeting[1] that the entire sample submitted to the chemist be passed through a 10-mesh sieve previous to its subdivision for analysis. This procedure was followed in the preparation of the analytical samples sent out by your committee and the results certainly confirm the advisability of following this practice.

RECOMMENDATIONS.

It is recommended—

(1) That a sampler be used that removes a core from the bag from top to bottom.

(2) That at least a pound of the material should constitute each official sample sent to headquarters.

(3) That the entire sample submitted to the chemist be passed through a 10-mesh sieve previous to its subdivision for analysis.

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[1] *J. Assoc. Official Agr. Chemists*, 1920, **4**: 289.

(4) That cores shall be taken from not less than 10 per cent of the bags present, unless this necessitates cores from more than 20 bags, in which case a core shall be taken from 1 bag from each additional ton represented. If there are less than 100 bags, not less than 10 bags shall be sampled, provided that in lots of less than 10 bags all bags shall be sampled.

Respectfully submitted,

C. H. Jones,
E. G. Proulx,
B. F. Robertson.

Committee on Methods of Sampling Fertilizers to Cooperate with a Similar Committee of the American Chemical Society.

A TRIAL WITH TWO TYPES OF FERTILIZER SAMPLERS.

By L. D. Haigh (University of Missouri, Columbia, Mo.).

In the fertilizer inspection work of the Missouri Agricultural Experiment Station a sampler of the open type, proposed and put into use in 1908–1909 by P. F. Trowbridge, now of the Agricultural Experiment Station, Agricultural College, N. Dak., has given such satisfactory results in practice that it is desired to bring it to the attention of the members of this association. The analysis of samples obtained with it, check quite uniformly the results on samples obtained with the close type of sampler.

This sampler is made from a solid brass or steel rod about 750 mm. in length, and 12 mm. in diameter, pointed at one end, and having a solid brass head or button screwed on the other end. A groove, 7 mm. in width and 5 mm. deep, is cut out of one side of the rod, extending to about 80 mm. from each end. To use the sampler, the sack of fertilizer is placed on its side and the pointed end inserted in the end of the sack. It is then pushed in, groove down, as far as possible, turned around until the groove is upwards, and then withdrawn. The contents of the groove are emptied upon a sheet of paper and the operation repeated with a second sack. When 10 sacks have thus been sampled, there has been obtained about one-half pound of fertilizer, which is a convenient amount for analytical purposes. This sampler permits of rapid as well as accurate work. It is not cumbersome to carry and is readily made by any machinist.

A year ago this station purchased the Indiana double tube sampler and used it side by side with the Missouri sampler on trips of inspection. It was desired to discover what advantages or disadvantages would attend the use of this sampler in the practical work of inspection throughout the State and what variation in results would be obtained because of the kind of samples drawn by each sampler. Samples of a number of brands were drawn, using both samplers on each brand and using the same 10 sacks for both samples.

The accompanying table shows the results obtained:

Comparison of chemical results using Indiana closed sampler and Missouri open sampler.

BRAND NAME AND ANALYSIS	SAMPLER USED	NITROGEN		PHOSPHORIC ACID							
				Total		Insoluble		Available		Potash	
		Guaranteed	Found	Guaranteed	Found	Guaranteed	Found	Guaranteed	Found	Guaranteed	Found
		per cent	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
Darling's Grain Grower 1-9-1	Indiana	0.82	0.91	11.00	12.55	2.00	2.90	9.00	9.65	1.00	0.88
	Missouri	0.82	0.99	11.00	12.29	2.00	2.70	9.00	9.59	1.00	1.07
Read's Sp. H. G. Phosphate 0-16-0	Indiana	....	....	18.00	21.51	....	5.18	16.00	16.33	....	....
	Missouri	....	....	18.00	21.68	....	4.70	16.00	16.98	....	....
Read's Blood and Bone 2-8-2	Indiana	1.65	1.62	.....	12.88	....	3.85	8.00	9.03	2.00	1.78
	Missouri	1.65	1.55	.....	14.05	....	4.21	8.00	9.84	2.00	1.87
Swift's Diamond K Grain Grower 1-12-1	Indiana	0.82	0.86	.....	13.56	....	1.27	12.00	12.29	1.00	0.83
	Missouri	0.82	0.94	.....	13.77	....	1.26	12.00	12.51	1.00	0.86
Swift's Corn and Oats Special 2-10-0	Indiana	1.65	1.49	.....	12.60	....	1.35	10.00	11.25	....	....
	Missouri	1.65	1.43	.....	12.89	....	1.47	10.00	11.42	....	....
Swift's Diamond W Tomato and Vegetable Grower 2-8-3	Indiana	1.65	1.43	11.00	12.37	3.00	3.50	8.00	8.87	3.00	2.62
	Missouri	1.65	1.35	11.00	12.39	3.00	3.64	8.00	8.75	3.00	2.64
Swift's Bone Meal and Phosphate 1-13	Indiana	0.82	0.74	20.00	21.69	7.00	7.81	13.00	13.88	....	....
	Missouri	0.82	0.68	20.00	21.31	7.00	7.60	13.00	13.71	....	....

While some difference may be observed in certain cases, there is only one result—that on potash in the first sample—which means a difference of being over or under the guaranteed value.

Some practical difficulties have also been observed with the double tube sampler, which should be mentioned. The diameter of the Indiana sampler is about 27 mm., which makes it difficult and at times utterly impossible to insert in a sack of fertilizer of a sticky nature, such as acid phosphate. Then, again, the amount of fertilizer removed is such that, when 10 sacks of the brand have been sampled, the amount is so large that it must be mixed and quartered before a sample of convenient size can be obtained to be carried away. With the Missouri sampler, all the sample obtained is carried away and no quartering is necessary. In using the double tube sampler, fine particles of fertilizer work in between the two tubes, and after the tube has been once inserted in a sack and withdrawn with its load it must be pulled apart and all of this fine material shaken out with the sample before it can be used a second time. This fine material scratches the tubes severely, making them work more and more loosely together, thus admitting more fertilizer between them and increasing the difficulty of using the sampler.

An effort has also been made to adapt the double tube sampling idea to the Missouri sampler. Such a sampler is now being tried out, and it is thought that with the removal of one or two mechanical difficulties it may be freed from some of the practical difficulties of the Indiana sampler. These data are presented at this time to show that the Missouri sampler is free from the objection of the other open types of samplers, such as the butter tryer type, which fills as it is pushed in and empties itself when withdrawn. The pointed end of the Missouri sampler pushes away the fertilizer from the groove as it enters, and when the sampler is withdrawn the contents of the groove can not be pushed out, as the end of the groove is not open in the longitudinal direction. In this way it resembles the operation of the double tube sampler.

It is recommended that a further study be made of samplers and methods of sampling, as it is believed the discrepancy in results in the analysis of fertilizers has largely grown out of the difference in the samples obtained.

THE DETERMINATION OF BORAX IN FERTILIZER MATERIALS AND MIXED FERTILIZERS.[1]

By G. F. Lipscomb[2], C. F. Inman[3] and J. S. Watkins[4] (Agricultural Experiment Station, Clemson College, S. C.).

Owing to the large amount of damage during the past season to growing crops in this State, apparently from some ingredient in commercial fertilizers, and in consequence of the wide-spread belief that the damage was caused by borax known to be present in some of our American potash salts, it became necessary to develop some method for the determination of borax in fertilizer materials and in mixed fertilizers.

There are two well-known methods in use for the determination of boron: A gravimetric method worked out by Rosenbladt and Gooch; and a volumetric method, sometimes called the glycerol or mannitol

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[1] Presented by R. N. Brackett.
[2] Present address, University of South Carolina, Columbia, S. C.
[3] Present address, Greenville, S. C.
[4] Present address, Department of Agriculture, Commerce and Industries, Columbia, S. C.

titration method. Many trials of the Gooch method, and combinations of this method with the titration method, seemed to show that distillation methods were not suitable or convenient for routine work.

The volumetric method takes advantage of the fact that boric acid reacts neutral to methyl orange, but is acid to phenolphthalein, and may be quantitatively titrated in the presence of mannitol or glycerol, which prevents the hydrolysis of sodium borate. This method is not, however, applicable in the presence of certain impurities, notably phosphates, ammonium salts and organic matter, and hence can not be used with fertilizer materials or with mixed fertilizers containing one or more of these impurities. This volumetric method appeared to afford the best basis for the development of a rapid and accurate method for the determination of borax in fertilizer materials, and in mixed fertilizers, since it offered a means of removing the interfering substances without loss of boric acid.

The following method has been developed:

REAGENTS.

(a) *Saturated solution of lime water.*

(b) *Standard solution of pure borax, or of pure boric acid.*

(c) *N/10 solution of sodium hydroxid.*—Carefully standardized against the borax solution.

(d) *Hydrochloric acid about N/10.*

(e) *Neutral glycerol or neutral mannitol.*

(f) *Methyl red 0.1 gram in 100 cc. of hot 50% alcohol.* (Acknowledgment is made to W. H. Ross, Bureau of Plant Industry, Washington, D. C., for suggesting the use of methyl red in place of methyl orange.)

(g) Phenolphthalein 1 gram per 100 cc. of alcohol.

PREPARATION OF SOLUTION.

Weigh 10 grams of the sample and transfer to a 500 cc. graduated flask, add 200 cc. of water, and boil the contents of the flask for 10 minutes, after which cool the flask and make the contents to volume. Or, weigh 2½ grams and wash on a filter paper with hot water, collect the washings in a 250 cc. graduated flask, cool the flask and contents and make to volume.

If the total boron is desired, digest 10 grams of the sample in a 500 cc. graduated flask for about 30 minutes with 200 cc. of water and 15 cc. of hydrochloric acid (1 to 1), then cool the flask and make to volume.

(a) *Materials free from ammonium salts, phosphates, and organic matter.*—Transfer an aliquot corresponding to 1 gram of the sample to a wide-mouthed 200 cc. flask, acidify with N/10 hydrochloric acid, using methyl red as the indicator, connect the flask with a reflux condenser and boil the contents for 10 minutes to remove carbon dioxid. Cool, make neutral with sodium hydroxid, in the presence of methyl red, add 30 cc. of neutral glycerol, or 1 gram of mannitol, and titrate with N/10 sodium hydroxid in the presence of phenolphthalein[1].

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[1] W. W. Scott. Standard Methods of Chemical Analysis. 2nd ed., rev., 1917, 76.

(b) *Materials containing ammonium salts, phosphates, and organic matter.*—Bring the sample into solution as described in (a).

To insure the complete removal of ammonium salts, make the aliquot, corresponding to 1 gram of the sample, alkaline with sodium hydroxid and boil nearly to dryness, then dilute with water and repeat the same operation at least twice. Take up the residue with water and make the solution slightly acid with hydrochloric acid (1 to 10), using methyl red as the indicator. Then make the solution alkaline with lime water, and, after stirring vigorously, filter off the phosphates, if any, and wash five or six times with warm water. The solution must not be boiled after the addition of the lime water, as insoluble calcium borates are formed. Collect the filtrate and washings in a 200 cc. wide-mouthed flask. Evaporate the solution to dryness in a porcelain or platinum dish on a water bath, and ignite over a Bunsen burner to remove the organic matter. Dissolve this residue in a little dilute hydrochloric acid, make alkaline with lime water, filter, make the filtrate acid with N/10 hydrochloric acid in the presence of methyl red and wash into a 200 cc. wide-mouthed flask, finally making to a volume of about 150 cc. Connect the flask with a reflux condenser and boil the contents 10—15 minutes to remove carbon dioxid. Then cool the flask and contents, make the contents neutral with N/10 sodium hydroxid, using methyl red as the indicator, add 30 cc. of glycerol, or 1 gram of mannitol, and titrate the solution with N/10 sodium hydroxid, using phenolphthalein as the indicator. Then add an additional 20 cc. of glycerol, or 0.5—1 gram of mannitol and continue the titration to conclusion.

A blank should, of course, be run with the reagents and deducted. This blank may amount to as much as 0.12 per cent.

No report was made by the Committee on the Revision of Methods of Soil Analysis.

R. N. Brackett: I would like to ask a question. The association has changed the assignment of referees' work and has appointed referees and, in some cases, associate referees, and they have been assigned certain topics. Are the associate referees to send a statement to the general referee of what they are doing, or is the general referee to send them a statement of what he wants them to do? Just what relation does the associate referee bear to the referee?

C. L. Alsberg: The idea, as I understand it, is this: The general referee will be a man who will keep in touch with the progress of the work covered by this particular subject during the year. He will be an older and more experienced man and will keep his finger on the pulse of developments. If any condition arises in his line of work which demands investigation of the type conducted by the association, he will have full authority to appoint an associate referee who will do it under his direction. If, on the other hand, he does not feel that the subject is urgent and that it must be investigated at once, he should make a report to the association at its next annual meeting that such and such a subject should be studied. His report may be that nothing new has turned up in the year that is worthy of the attention of the association. It is also important that he shall direct and supervise the work of his associate referees. The idea is to get away from having, year after year, a referee who feels himself under obligations to report on a subject in which, perhaps, nothing new has arisen. Under the old procedure he would merely report that nothing special had turned up and that he had no recommendations to make. The idea is to get away from this perfunctory work.

P. F. Trowbridge: The referee is in charge, then, as I understand it and is at liberty to appoint associate referees to assist him in his work should circumstances arise during the year to warrant such action.

C. L. Alsberg: Yes.

REPORT OF COMMITTEE ON RESOLUTIONS[1].

Resolved, That the thanks of this association be extended to the New Willard Hotel for the use of rooms for meetings, and the excellent arrangements for conferences and committee meetings.

Resolved, That the association hereby expresses to President Trowbridge its appreciation for his unfailing courtesy in conducting successfully the affairs of his office.

Respectfully submitted,

W. D. Collins,
H. B. McDonnell,
Julius Hortvet.

Adopted.

President Trowbridge: I desire to announce to you thus officially the passing of five of our active members since we met last in regular annual session as an association. Please rise and do honor to them as I call their names:

Wallace C. Burnet.
Albert F. Seeker.
Cyril G. Hopkins.
James H. Shepard.
Martin N. Straughn.

ALBERT FREDERICK SEEKER.

The subject of this sketch, Albert Frederick Seeker, died on August 19, 1919, from complications following an operation for appendicitis. At the time of his death, he was Chief of the New York Food and Drug Inspection Station of the Bureau of Chemistry. Mr. Seeker was born at Brooklyn, N. Y., in 1878. His parents died during his early childhood, and he was obliged to earn his way through the various educational institutions he attended. After completing the grammar school course in Brooklyn,

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[1] Presented by H. B. McDonnell.

ALBERT FREDERICK SEEKER.

(See Page 602.)

he entered the New York College of Pharmacy with the intention of adopting pharmacy as a profession. His studies at that institution developed an interest in chemistry, which became his life work. Upon completing the course in the College of Pharmacy, he entered the Brooklyn Polytechnic Institute, from which he received the degree of B. S. in 1902. After engaging in tutoring for about a year following his graduation, he entered the laboratory of the Brooklyn Navy Yard in April, 1904, as assistant chemist, and in June, 1905, at his own request, was transferred to the New York Food and Drug Inspection Laboratory of the Bureau of Chemistry, with which laboratory he was associated during fourteen years of the most able service ever rendered by a public official.

Throughout life Mr. Seeker was a constant student of chemistry; few men have a more intimate knowledge of every branch of the science. His interests did not stop with chemistry, however. He was an inveterate reader, not only of the chemical literature of France, Germany, England and America, but of all good literature. As an analyst, he can only be described as brilliant. He was an adept in devising methods of analysis, and a long series of methods in daily use in food and drug laboratories in this and other countries attest his ability in this direction. During the fourteen years of Mr. Seeker's connection with the Bureau of Chemistry he was a tireless worker in the Association of Official Agricultural Chemists, serving nearly the entire time as referee or associate referee on various food products. Perhaps his most valuable contribution to the association was as a member of the recent Committee on Editing Methods of Analysis. The value of his services in connection with this publication can not be overestimated. His thorough knowledge of the principles of analytical chemistry, and his personal familiarity with many of the methods themselves, was such that his suggestions and advice prevailed throughout the work, and it is not too much to say that whatever value can be claimed for the revision is due to a great extent to the contribution of his time and experience. Mr. Seeker had an enormous capacity for work, and his activities in connection with the revision were carried on without interrupting the task of administering the largest laboratory unit in the Bureau of Chemistry.

Mr. Seeker was a member of the American Chemical Society, the Society of Chemical Industry and the American Association for the Advancement of Science, and took an active interest in these organizations. He served for many years as an abstractor for Chemical Abstracts. He collaborated with John C. Olsen in the preparation of Van Nostrand's Chemical Annual, compiling for that publication many of the tables of physical and chemical constants. He also contributed the Chapter on Coloring Matters in Foods to the Fourth Edition of Allen's Commercial Organic Analysis, Volume V. His publications in the various chemical journals cover the whole field of food analysis.

Although Mr. Seeker's tastes and training were primarily in the direction of analytical chemistry, the necessities of the service threw him more and more into administrative lines as time went on, and in these he showed a capacity hardly less marked than in the lines of laboratory work. He was a most able chemical witness. His testimony was clear and concise, and continually earned the approbation of Federal judges and United States attorneys.

Above and beyond all else, his character as a man has made his loss a source of the profoundest sorrow to all who knew him. It was said of him that he was "good, true, brave, clean, honest, able; that no one ever asked for help, or was in trouble, but he gave unselfishly to the utmost of his great ability." His home life was characterized by a tender and loving devotion to his sister, the only surviving member of his family. He was the soul of honor in all his dealings, sympathetic to those in trouble, a loyal friend, a wise and conscientious adviser, possessed of a deep sense of humor, a delightful companion. No man in the Federal service was ever accorded more ungrudging personal affection, and no chemist in the service was ever held in higher appreciation by reason of his scientific instincts and attainments. He believed and proved that the best his ability had to offer was not too much to put into the service of the public. His best will always be an unfailing source of pride to those with whom he was associated.

R. E. DOOLITTLE.
P. B. DUNBAR.

C. L. Alsberg: I can not urge too strongly upon referees the importance of sending their annual reports to the secretary's office considerably in advance of the meeting, in order that they may be circulated among the members with a view to later discussion at the meeting.

It was moved, seconded and adopted that the time and place of the next meeting be left to the Executive Committee with power to act.

The convention adjourned.

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PROCEEDINGS OF THE THIRTY-FOURTH ANNUAL CONVENTION, 1917, AND OF THE THIRTY-FIFTH[1] ANNUAL CONVENTION, 1919.

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[1] No meeting was held in 1918 other than a meeting of the Executive Committee.

Association of Official
Analytical Chemists
Journal