

1. Mg. of  $\text{SiO}_2$  from calibration curve  $\times 0.1 \div$  gm. of sample in final aliquot = %  $\text{SiO}_2$ .

Example:

Aliquot contains 0.01 gm. sample  
Colorimeter reading = 76  
Mgs.  $\text{SiO}_2$  from calibration curve — 0.435  
 $(0.435 \times 0.1) \div 0.01 = 4.35\% \text{SiO}_2$ .

2. Slope of curve  $\times$  colorimeter reading  $\times 0.1 \div$  gm. of sample in final aliquot = %  $\text{SiO}_2$ .

Example:

Slope of curve =  $57.25 \times 10^{-4}$   
Aliquot contains 0.01 gm. of sample  
Colorimeter reading = 76  
 $0.005725 \times 76 \times 0.1 \div 0.01 = 4.35\% \text{SiO}_2$ .

### Notes

1. This method may of course be applied to samples containing no phosphate. In such a case the citric acid bleach may be eliminated from the procedure. If this is done, the calibration curve should be prepared without the citric acid. However, if a laboratory plans to use the method for various products, some of which may be built with phosphates, it is simpler to prepare a single calibration curve using citric acid and then use the citric acid procedure for all samples.

2. The recommended use of this method is with a photoelectric colorimeter. However as a semi-quantitative test to check the limits on a product, the silico-molybdate color may be compared visually with two ampules of picric acid solution representing the upper and lower limits of silica allowed. Twenty-five and six tenths mg. of vacuum dried picric acid per liter is equivalent to 50 mg. of  $\text{SiO}_2$  per liter. Standard silica solution may be used as a standard, but the color has no permanence. Picric acid solutions, if protected from evaporation, may be used indefinitely.

3. Advantage may be taken of the double color development, before the addition of citric acid, quantitatively to determine orthophosphate. Measurement of the color should be made, before the addition of citric acid, at 420  $m\mu$ . After the final measurement is made of the bleached color, the difference between the two colorimeter readings may be taken as the color due to orthophosphate. A calibration curve should be prepared using standard  $\text{NaH}_2\text{PO}_4$  solution and the standard silicate solution.

It must be remembered that the phosphorus color will be due to orthophosphate only and that the color will not be stable when polyphosphates are present. The color development takes place in acid medium which favors hydrolysis.

### Summary

Water-soluble silica is determined rapidly and accurately by measuring the color produced after the development of silico-molybdic acid. Citric acid is used to eliminate any interference from phosphate builders. The entire procedure requires not more than 30 minutes and is applicable to both soaps and synthetic detergents. Semi-quantitative estimation between limits may be carried out even more rapidly by using picric acid standards and a visual comparator.

### Acknowledgment

The author expresses grateful acknowledgment to Procter Thomson who initiated this work by pointing out the need for such a method.

### REFERENCES

1. W. H. Hadley, *Analyst*, **66**, 486 (1941).
2. W. H. Hadley, *Ibid.*, **67**, 5 (1942).
3. H. W. Knudson, *et al.*, *Ind. Eng. Chem., Anal. Ed.*, **12**, 270 (1940).
4. M. C. Schwartz, *Ibid.*, **6**, 364 (1934).

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## Report of the Uniform Methods Committee

1. At the 23d Fall Meeting in Chicago, November 1, 1949, the Seed and Meal Analysis Committee recommended for adoption as tentative, a new Method Ad 1-48, for sampling tung fruit, and new Methods Ad 2-48, Ad 3-48, Ad 4-48, and Ad 5-48, for analysis of tung fruit and kernels. These methods were adopted as tentative.

2. The Soap Analysis Committee decided to change the esterification flask in Method Da 12-48 (A-1) from 150 ml. to 125 ml. capacity as the latter size is more readily obtainable. Approval of this change was voted by the Society.

3. The Refining Committee, in response to a request from the Department of Commerce, Bureau of the Census, submitted the following as a proposed definition for a refined vegetable oil:

A refined vegetable oil or fat is one which has been processed so as to reduce the coloring matter and remove substantially all of the free fatty acids, phosphatides, gums, and insoluble impurities.

This definition represented a composite or consensus of definitions submitted by individual members of the Refining Committee and agreed upon by a subcommittee consisting of E. M. James, T. C. Smith, and J. R. Mays Jr., chairman. An overwhelming majority

of both the Refining and Uniform Methods Committee members approved this definition for adoption by the Society and submission to the Bureau of the Census. However there was minority opposition to it in both committees. For this reason, and because of our uncertainty concerning the use to which it might be applied, the Uniform Methods Committee requested authorization to inquire further of the Bureau of the Census regarding the purpose for which the definition is required, also authority to modify its phrasing as seemed advisable in the light of the information obtained. The final definition to be recommended to the Society must have the approval of the Refining and Uniform Methods Committees. This permission was granted.

At the 41st Annual Meeting in Atlanta, May 3, 1950, a brief report of this investigation was made. From the reply to our inquiry it was apparent to the Uniform Methods Committee that the desired definition must either be so comprehensive as to include all oils, or a multitude of definitions must be formulated to cover individual types and sources, with processing procedures which become extremely complex. The definition submitted by the Refining Committee

would apply only to certain vegetable oils of edible grade. This would not constitute a satisfactory solution to the problem. Neither a comprehensive definition nor an endless number of restricted definitions appears at all practicable.

The Uniform Methods Committee, by unanimous vote, recommended that the following resolution be approved by the Society:

WHEREAS, in our opinion, the desired definition for refined oil is not within the province of our Society, we recommend that the Bureau of the Census be so advised. We further recommend that the Bureau of the Census refer this problem to the various trade associations concerned and suggest that they may be able to solve the question by "spelling out" in their instructions all of the various types of oils involved and specifying under which category they wish each to be reported.

This recommendation was approved by the Society and the adopted resolution, with an appropriate explanation of the reasons for the position taken, has been transmitted to the Bureau of the Census.

4. The Oil Color Committee, under the chairmanship of G. W. Agee, has performed a truly incredible amount of labor on improving our present photometric method for measurement of oil colors. Much of the credit for the evaluation of the proposed method should be given to a subcommittee headed by Dr. Beall of Swift and Company though valuable contributions were made by the technical staffs of several organizations. As a result of exhaustive tests the Oil Color Committee recommended a four-factor photometric method to replace our present Tentative Method Cc 13-49. The Uniform Methods Committee, by unanimous vote, concurred with this recommendation and added to it the following:

1. The Oil Color Committee should be continued and requested to work actively toward simplification of the manipulative procedure proposed in their 1950 report.

2. They should be requested to develop an independent procedure for the evaluation of the green color in oils.

3. They should be requested to work toward the ultimate development of an objective method for the measurement of color in oils based on physical principles and not on an arbitrary system.

The proposed tentative method and the three additional recommendations by the Uniform Methods Committee were approved by the Society.

5. The Committee on Analysis of Commercial Fats and Oils (F.A.C.) recommended a slight change in the dimensions of the condenser shown in the figure which accompanies Official Method Ca 2a-45, for moisture in oils by the distillation method. The distance between the bottom of the condenser jacket and the top of the ground joint, now 76 mm., should be changed to  $40 \pm 2$  mm. This is done in the interest of apparatus uniformity to bring it into accord with comparable A.S.T.M. methods. It complies with Corning standard unit No. 2360.

Since the same figure is used in conjunction with Methods Da 2b-41 and F 1a-42, for moisture in soaps and sulfated or sulfonated oils, respectively, the same change should be made in these methods. These changes were approved by the Uniform Methods Committee and authorized by the Society.

6. The Glycerin Analysis Committee recommended that our present Tentative Methods Ea 7-46 and Ea 8-46, for specific gravity at  $25^{\circ}/25^{\circ}\text{C}$ . and for moisture in glycerin by the Karl Fischer method, respectively, be made official. On approval by the Uniform Methods Committee the Society voted that this action be taken.

7. The Gossypol Committee, in its 1950 Report, has shown that Tentative Method Ba 7-49, for the determination of free gossypol, is both rapid and yields highly accurate results. It recommends that this method be made official and that the committee be discharged. The Uniform Methods Committee approved this recommendation, with the additional suggestion that the thanks of the Society be tendered to Mr. Tenent and his committee for an excellent accomplishment. This action was authorized by the Society.

The present method requires pure gossypol as a standard. It is our understanding that requests for this material will be honored if addressed to the Executive Secretary of the American Oil Chemists' Society, 35 East Wacker Drive, Chicago 1, Illinois.

8. The Seed and Meal Analysis Committee reported that the methods for sampling and analysis of tung fruit and kernels, adopted as tentative last November, have been edited by the Committee to bring them into the desired style and format and are now ready for publication in our methods. This committee recommended that our present Tentative Method Be 1-49, for sampling soyflour, be made official. Tentative Method Ab 4-47, for the determination of nitrogen, ammonia, and protein in peanuts, uses the same apparatus, reagents, and procedure as employed for cottonseed. This too should be made official. These recommendations were approved by the Uniform Methods Committee and the Society.

The Seed and Meal Analysis Committee is exercising its functions smoothly and efficiently through the agency of five subcommittees. While this form of organization is not unusual the Uniform Methods Committee commends it to other technical groups whose problems may be handled effectively by this procedure.

9. The Vitamin Committee has cooperated with the U.S.P. and A.O.A.C. in the development of methods for Vitamin A, and these methods will be available in forthcoming publications of these organizations. No methods were recommended for adoption by the A.O.C.S. The Uniform Methods Committee shares the conviction of the Vitamin Committee that these methods of analysis should be left in the hands of the U.S.P. and A.O.A.C. At their request discharge of the Vitamin Committee was voted by the Uniform Methods Committee and the Society, with our thanks for their valuable services.

10. Standard Bleaching Earths. With increased usage of standard bleaching earths, especially in the soybean industry, our problems in satisfying the demand adequately have multiplied. This problem is one which belongs logically in the province of the Bleaching Methods Committee. It is the consensus of the Uniform Methods Committee that the Bleaching Methods Committee be requested:

1. To study aggressively the problem of a reliable source of uniform bleaching earth, of United States origin, to serve as a single standard in place of the natural and activated bleaching earths now required by our Methods.

2. To test immediately the uniformity of the existing supply of natural bleaching earth.

3. To search for a primary standard which will evaluate effectively the bleaching characteristics of a bleaching earth.

This basis for a Bleaching Committee program was approved by the Society. It has been transmitted to the President and Governing Board and will be sub-

mitted to the Bleaching Committee as soon as its reorganization is effected.

11. Editor of Analytical Methods. V. C. Mehlenbacher, who performed the exceedingly difficult task of revising our Analytical Methods in a highly creditable manner, and who has served for several years most efficiently as editor of our Methods has found it necessary to resign from that important post. He has earned well the appreciation and gratitude of the Society for these outstanding services. The Uniform Methods Committee, recognizing the need for a permanent editorship for Analytical Methods in order to achieve uniformity in style and format, recommended that the president appoint yearly an editor of Analytical Methods, the person selected to be approved by the Governing Board. This recommendation had the unanimous support of the Uniform Methods Committee and was approved by the Society. It has been offered to the President and Governing Board.

It is a pleasure to add that these conclusions were reached in a discussion in which all members of the

Uniform Methods Committee participated and that their decision on every question was unanimous.

Some of our technical committees have made progress reports with no recommendations for changes or additions to analytical methods. Such reports should not be interpreted as inactivity on the part of those committees. For example, the Cellulose Yield Committee has reported cooperative analytical work of its usual high quality and is planning to continue with a minimum of four samples during the coming year. Other committees are engaged in investigations whose value will become apparent when their experiments have progressed to a point where they can be reported with clear significance. It is our belief that no professional society has more active technical committees, composed of more able members, and engaged in more useful scientific effort.

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|-----------------------------|---------------|
| J. T. R. ANDREWS, chairman. |               |
| M. M. DURKEE                | R. R. KING    |
| J. J. GANUCHEAU             | T. C. LAW     |
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## Comparative Composition of Soybean and Corn Phosphatides<sup>1</sup>

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SOYBEAN "lecithin" and corn "lecithin" are the only plant phosphatides of commerce. The two are processed in a similar manner; they are similar in many of their properties, and one might expect them to be similar in composition. However very little has been published on the composition of corn phosphatides (6). Because of reports of certain superior properties of corn phosphatides, it seemed desirable that a comparison should be made between the phosphatides obtained from corn oil and those obtained from soybean oil. For this purpose, a sample of corn phosphatides has been prepared and fractionated in a manner similar to that previously used for soybean phosphatides (7). This paper reports those results and compares them with those obtained for the soybean phosphatides.

*Analytical Methods.* The analytical procedures used in this work were similar to those previously described for soybean phosphatides. Total nitrogen was determined by the Micro-Kjeldahl procedure. Phosphorus determinations reported in Table I were obtained gravimetrically as ammonium phosphomolybdate. Owing to the smaller size of the samples obtained from countercurrent distribution (Figures 1, 2, 3), phosphorus in this instance was measured spectrophotometrically by a modification of Burmaster's method for total phosphorus (2). Choline was measured by the reineckate method of Glick (5). Sugar was determined after hydrolyzing the phosphatides for approximately seven hours with 0.6 N H<sub>2</sub>SO<sub>4</sub> on a steam bath. The hydrolyzate was filtered and neutralized, and sugar was measured by the

method of Somogyi (8). Sugar was calculated as galactose in order to be comparable with the previous analysis of soybean phosphatides although other sugars are known to be present (9).

Inositol was estimated by a modification of the Atkin, Schultz, Williams, and Frey (1) microbiological method for the assay of pyridoxin. Samples were hydrolyzed with 20% HCl at 120°C. for 16 hours prior to assay.

*Preparation of Fractions.* The crude phosphatides used in this work were prepared from material obtained in the commercial degumming of corn oil. The gums were collected at the centrifuges and refrigerated immediately thereafter. This material was dissolved in ether and ether insoluble substances allowed to settle out. After drying the solution with sodium sulfate, the ether was removed under vacuum. The residue was extracted repeatedly with acetone, first stirring by hand, and, after the product became waxy, with the aid of a Waring Blendor. Analytical data on this crude acetone insoluble phosphatide preparation are shown in Table I.

The crude phosphatide preparation was subsequently separated into alcohol-soluble and alcohol-insoluble fractions in the same manner previously used with soybean phosphatides. A 150-gram portion of the phosphatides was extracted with six successive 300-ml. portions of absolute alcohol in a Waring Blendor. The alcohol-soluble portion was further fractionated by removing the solvent under vacuum at 50°C. and by again adding absolute alcohol. The alcohol was evaporated from the clear supernatant solution leaving 38.8 g., which are designated in Table I as Fraction I. A small portion, 1.6 g., which did not redissolve in the alcohol, is designated as Fraction II. Since this fraction is quite small, no

<sup>1</sup> Presented at fall meeting of American Oil Chemists' Society, October 31, November 1 and 2, 1949, in Chicago, Illinois.

<sup>2</sup> One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture. Report of a study made under the Research and Marketing Act of 1946.