

\$2.64 and \$3.80 per pound of product, respectively. Additional total manufacturing cost for producing 3,409,600 lbs. of crude phosphatides annually is 9.1¢ per pound of phosphatide. If this cost is subtracted from the market value of unbleached lecithin, which is currently 13¢ per pound, a profit of 3.9¢ per pound is realized. Converting this profit to the gossypol equivalent would result in a profit of \$1.64 per pound of gossypol. This profit, when credited to the manufacturing cost of gossypol, would reduce the total manufacturing cost from \$5.55 to \$3.91 per pound.

The direct manufacturing costs, in most cases, were found to account for the greater portion of the total manufacturing costs. Chemical requirements were determined from data obtained from laboratory and pilot-plant runs. In the calculation using recommended percentages, 95% recovery of the methyl ethyl ketone was used, and in the calculation using minimum percentages, 99% methyl ethyl ketone was used. In all cases 98% recovery of acetic acid was assumed, with no recovery allowed for the other chemicals. If the phosphatides are not to be returned to the meal, a value should be assessed the gums to be used in the process to allow for the purchase of acidulated soapstock, which would be used instead of the gums in the cottonseed meal. Therefore an additional chemical cost of \$166,320, or 4¢ per pound of gums, was allowed.

Direct labor costs were based on process labor requirements (1, 5) with the wage rates adjusted to current scales for skilled labor and foremen. Overtime and night differential rates were also applied when necessary. The labor used for Phase 1 was also used for Phases 2 and 3.

Utility costs include those for steam, electricity, process and cooling water. Unit steam cost of 50¢ per 1,000 pounds was used (5). Since steam-generating facilities are presently in use at the mill, on which this cost study is based, it was assumed that additional demands made on their boiler would not overtax its capacity.

Electric power costs were estimated by using a unit cost of 1¢ per kwh (5). City process water was used

at a cost of 20¢ per 1,000 gal. (5). Cooling water was to be supplied by a tower, currently in use, at a cost of 3¢ per 1,000 gal. (5).

General expenses are those for general administration and office overhead, financing, and sales costs.

Summary

Based on operations of a hypothetical gossypol-extraction plant, it is estimated that crude gossypol-acetic acid can be produced at a cost of \$2.64 per pound at an annual production of 113,500 lbs.; that pure gossypol-acetic acid can be produced at a cost of \$3.80 per pound at an annual production of 100,200 lbs.; and that pure gossypol will cost \$5.55 per pound at an annual production of 81,000 lbs. By marketing the phosphatides instead of returning them to the gums, the cost of producing pure gossypol will be \$3.91 per pound at an annual production of 81,000 lbs. These costs were estimated by assuming that Phase I of the process would be accomplished simultaneously with oil mill-extraction operations, and Phases II and III during remainder of the season.

It is readily apparent that gossypol or gossypol-acetic acid as produced are not inexpensive chemicals and would probably have to be produced for specialized uses, such as pharmaceuticals and the like, where the cost of these chemicals would not be prohibitive. Should product evaluation research now under way establish specific uses for gossypol and its intermediates, a demand for sizeable quantities of these materials might result.

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Report of the Uniform Methods Committee, 1958-59

THE MEETING of the Uniform Methods Committee was held at 2 p.m., April 20, in the Baronne room of the Roosevelt Hotel. The meeting was attended by six of the seven members of the committee. E. M. Sallee, our editor of Methods, was present as a member *ex officio*. Guests present were J. R. Mays Jr., V. C. Mehlenbacher, T. F. Waters, R. C. Stillman, J. C. Harris, W. T. Coleman, W. E. Link, and Harry Smith Jr. The last two were representing the Statistical Committee. The following matters were discussed, and the indicated decisions were made:

1. Fat Analysis Committee, V. C. Mehlenbacher, chairman

- a) *Congel Point. Tentative Method Cc 14-59.*
This new method was proposed for adoption as Tentative. Data on its precision and drawings of the cooling baths will be supplied. With minor additions, relative to type of burner and size of sample, the method is recommended for adoption as Tentative. *Adopted.*
- b) *Viscosity of Transparent Liquids by Bubble Time Method. Tentative Method Ka 6-59.*
This new method is proposed as a replacement for present Official Method Ka 6-55 (revised April, 1956). The

method has been carefully checked by the drying oils subcommittee under its chairman, K. E. Holt, and is judged much superior to our present Official Method. The Uniform Methods Committee commends Mr. Holt and his subcommittee for the excellence of its work. Precision data will be added. This method is recommended for adoption, as Tentative, to replace present Ka 6-55. *Adopted.*

- c) *Acetone Insoluble Matter (in Lecithin). Tentative Method Ja 4-46 (revised May, 1957).*
Advancement of this Tentative Method to Official status is proposed by the subcommittee on analysis of lecithin, T. C. Smith, chairman. The Fat Analysis Committee has approved, and the Uniform Methods Committee recommends that this action be taken. *Adopted.*
- d) *Fat Stability—Active Oxygen Method. Tentative Method Cd 12-57.*
A change in the end-point from a peroxide value of 125 milliequivalents to 100 milliequivalents is proposed, also several changes of a minor nature in the air-purification train, A. Apparatus. 3. These changes have been agreed upon by the Fat Analysis Committee and are recommended for adoption. *Adopted.*
- e) *Titer Test. Tentative Method L 6a-55.*
The subcommittee on analysis of commercial fatty acids,

J. L. Trauth, chairman, has recommended a considerable revision of this method in order to make it accord with Official Method Cc 12-41, which was similarly revised and adopted at the Fall Meeting, October, 1958. The changes involve chiefly the maintenance of air bath at a temperature of 15° to 20°C. below the expected titer-point, and use of an improved dry ice-ethylene glycol bath for samples with titer below 35°C. Other minor changes in apparatus are included. The Fat Analysis Committee has approved and the Uniform Methods Committee recommends adoption of the proposed revision. *Adopted.*

2. Seed and Meal Analysis Committee, M. H. Fowler, chairman Subcommittee on Determination of Oil in Cottonseed, R. T. Doughtie, chairman.

The subcommittee has arranged for a new supplier for the fuming pots required by Official Methods Aa 4-38 and Aa 7-55. These pots no longer are available from the Niloak Pottery. The Apparatus section, A-8, of Aa 4-38 should be changed to show the new supplier as "The Seedburo Equipment Company, 618-26 Jackson Blvd., Chicago 6, Ill." In Aa 7-55, E. Note 1, change the description of the clay pot to read, "... [Inside dimensions: approximately $5\frac{1}{8}$ in. diam. \times $1\frac{1}{8}$ in. deep. (The Seedburo Equipment Company, 618-26 Jackson Blvd., Chicago 6, Ill.). . . ." The Seed and Meal Analysis Committee has approved these changes. The proposed revisions are recommended for adoption. *Adopted.*

3. Spectroscopy Committee, R. T. O'Connor, chairman Polyunsaturated Acids—Ultraviolet Spectrophotometric Method, Official Method Cd 7-58

In the interests of safety the Spectroscopy Committee has proposed certain changes in B. Reagents. Sections 11-c and 11-d. These changes concern preparation of the 6.6% KOH-glycol solution and are intended to avoid the possibility of too violent expulsion of the water introduced during addition of the KOH pellets. The Uniform Methods Committee recommends adoption of these changes. *Adopted.*

4. Glycerin Analysis Committee, W. D. Pohle, chairman

a) The Glycerin Analysis Committee has proposed a revision of the statement under Section E on the title page to modernize and correct this statement to make it factual with respect to present practices in glycerin analysis. The Uniform Methods Committee concurs, with one dissent, and recommends adoption of the revised statement. *Adopted.*

b) *Total and Organic Residue at 175°C. Official Method Ea 3-58.*

Correction of several typographical errors should be made in C. Procedure, and one in D. Calculation. These are so obvious that the Uniform Methods Committee unhesitatingly recommends their adoption. *Adopted.*

c) *Statements of Precision*

The Glycerin Analysis Committee, at the 1958 Fall Meeting, proposed that statements of the precision to be expected be added to Methods Ea 3-58, Ea 6-51, Ea 7-50, Ea 8-58, Ca 14-56, Cd 11-57, and Da 23-56. Since our Statistical Committee was already working on a method for expressing the precision to be expected from an analytical method in a standard format, our approval for these additions was delayed, pending the submission of the standard format by the Statistical Committee. This has been done, and there no longer seems to be any reason for further delay. Data are available, and the chairman of the Glycerin Analysis Committee will be requested to present his statements of precision for these methods in accord with the prescribed standard format. The Uniform Methods Committee recommends that these revised additions be made to the methods indicated. *Adopted.*

d) *Standard Salt Lye Crude Glycerin*

Our present Standard Crude Glycerin sample has long outlived its usefulness as at least two of the analyses were made by methods now considered obsolete. For more than two years the Glycerin Analysis Committee has been engaged in the preparation and standardization of a new standard sample to be used as a replacement. Their work is now completed, with the standard sample and Analysis Certificate ready to be issued. The certificate shows ten analyses with mean value, and 95% confidence limits for a single analysis, by A.O.C.S. Methods.

The Uniform Methods Committee has approved the issuance of this Standard Salt Lye Crude Glycerin, with its Certificate of Analysis, and recommends its adoption as an A.O.C.S. Official Standard. *Adopted.*

5. Soap and Synthetic Detergent Analysis Committee, J. C. Harris, chairman

The Soap and Synthetic Detergents Analysis Committee has proposed several additions to Section D of our Methods:

a) *Sampling and Preparation of Sample. Official Method Da 1-45*

Changes are proposed whereby the term "Universal food cutter" is eliminated and replaced by "... a suitable food chopper." The Uniform Methods Committee recommends that this change be adopted. *Adopted.*

b) *Moisture—Distillation Method. Official Method Da 2b-42 (revised May, 1950)*

Improvements have been made so that the size of traps and of samples, for various moisture contents in samples to be analyzed, are shown.

The Uniform Methods Committee recommends that these changes be made. *The Method will remain Official. Adopted.*

c) *New Section, or Sections, Dc, for Fatty Alkyl Sulfates and Dd, for Alkylbenzene Sulfonates. Sampling and Preparation of Sample*

These should be set up, at the discretion of the editor of Methods, in one, or perhaps two, sections. Modification of Da 1-45 should be made where indicated, to eliminate reference to "soap" and substitute "fatty alkyl sulfates" or "alkylbenzene sulfonates." Other minor changes are necessary under C (c)—"Liquids" and C (d)—"Pastes."

The Uniform Methods Committee recommends that these proposed section additions be made Tentative. *Adopted.*

d) *Under "Analysis of Alkylbenzene Sulfonates" two new methods are proposed:*

1. *Moisture—Karl Fischer Method*

2. *Moisture—Distillation Method*

Coding to be at discretion of editor of Methods. These methods have been in regular industrial use for several years and have been adopted by A.S.T.M.

The Uniform Methods Committee recommends adoption of these two new methods as Tentative. *Adopted.*

e) *Under "Analysis of Fatty Alkyl Sulfates" In addition to "Sampling and Preparation of Sample" (Da 1-45 modified, as indicated above for "Alkylbenzene Sulfonates") eight new methods for analysis of fatty alkyl sulfates are proposed:*

1. Ester SO₃

2. Unsulfated Material

3. Alcohol-Insoluble Matter

4. Moisture-Distillation Method

5. Sodium Sulfate

6. Combined Alcohols

7. Alcohol-Soluble Matter

8. Alkalinity

These methods have all been in extensive industrial use and have been adopted by A.S.T.M. Proper coding will be at discretion of the editor of Methods. None of these ten new analytical methods have precision data readily available. The chairman of the Soap and Synthetic Detergents Analysis Committee has agreed to obtain data, in the near future, which will enable a statement of the precision to be expected for each, in the prescribed standard format, to be added.

Though these methods have not been subjected to the usual cooperative testing by the committee, they have sufficient character to warrant their adoption as Tentative. The Uniform Methods Committee recommends their adoption. *Adopted.*

6. Statistical Committee, W. E. Link, chairman

One year ago the Uniform Methods Committee requested the Statistical Committee to:

a) prepare a new section for A.O.C.S. Methods on a uniform method for determining the precision of an analytical method;

b) formulate a standard method for expressing the precision of an analytical method.

Both of these assigned tasks have been performed creditably by Mr. Link and his committee. As was inevitable, in such a complex task, much constructive criticism and many suggestions were offered freely to the committee when its tentative procedure was submitted. The Uniform Methods Committee and the Statistical Committee have agreed to:

1. add to Scope "... (See 'Definition of Terms' in Section F);"

2. add to A. Experimental Design, the necessary "between samples" procedure, when replicate samples are employed,

or when several samples are used to cover the range of concentration in samples to be analyzed;

3. show procedure to be followed in B. Analysis of Data, and C. Example, when multiple samples are employed as in (2) above;
4. show in C. Example, how "agreement within (and between) laboratories" is determined. (Calculation of Confidence Limits or "T-Test");
5. add, under "References," the textbook, "Introduction to Statistics," by Dixon and Massey.

The Statistics Committee has offered its services to any technical committee chairman in setting up an experimental design for collaborative work in determining the precision of an analytical method. They will assist also in the analysis of the analytical data obtained.

The Uniform Methods Committee has no doubt that many changes will be made during future months in this Section of Methods for "Determination of Precision and Accuracy of Test Methods." Its adoption as a Tentative Method is recommended. *Adopted.*

The Uniform Methods Committee has voted to request the Fat Analysis Committee to explore the need for methods of analysis for nitrogen derivatives of

fatty acids and, if the need is found to exist, to take appropriate action for the development and adoption of such methods as are found to be essential.

As you can see, your technical committees have been busy and their progress reports indicate that in the future there will be continued activity in the formulation of new, and the improvement of present analytical methods.

The Uniform Methods Committee wishes to thank all our technical committee chairmen and the members of their committees who have made this progress possible.

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Pharmaceutical-Grade Sterols from Tall Oil¹

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DURING RECENT YEARS there has been an increasing emphasis on medical research in the field of arteriosclerosis, popularly known as hardening of the arteries, and related disorders. Atherosclerosis, one form of arteriosclerosis, is characterized by the deposition of fatty matter on the inner walls of arteries. Atherosclerosis is the major factor in coronary artery disease and cerebrovascular accidents, popularly known as strokes. Although many theories have been advanced, the mechanism of the deposition of this fatty matter is still conjecture.

It is generally agreed among the medical profession that cholesterol, the predominant sterol found in animals, plays an important part since cholesterol is a major component of atherosclerotic deposits. Evidences have been produced which show that persons with atherosclerosis and related diseases may have a higher blood serum cholesterol content, known as hypercholesteremia, than those persons apparently free of artery disease (1). A reduction in cholesterol serum levels for hypercholesteremia patients appears desirable. However cholesterol is a very necessary ingredient for proper bodily functions. The body receives its cholesterol from two sources. Cholesterol is synthesized by the body and is absorbed from food sources found in a well-balanced diet. A lowering of serum cholesterol level sometimes can be attained by strict diet. Dieting is undesirable for the required type of diet is monotonous and unpalatable by American standards.

An oral intake of sitosterols, sterols found in vegetable and fruit sources, can be effective in reducing the level of serum cholesterol in patients with high cholesterol levels (2). The mechanism that causes this reduction is not known. One theory uses as an explanation that sitosterols interfere with absorption of cholesterol, possibly by the formation in the intestinal tract of a mixed crystal of sitosterol and cholesterol whose solubility is considerably less than that of cholesterol alone (3, 4). Other theories of the mechanism have

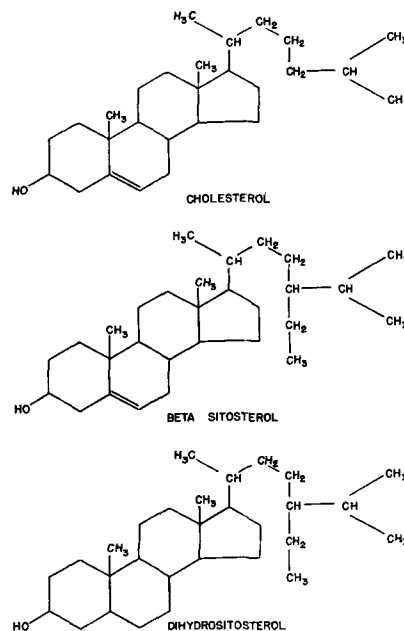


FIG. 1.

been advanced. Figure 1 shows the chemical similarity of cholesterol, betasitosterol, and dihydrositosterol.

A commercial preparation of sitosterols for oral intake has been introduced. This preparation is a 20% suspension of betasitosterol and dihydrositosterol. The introduction of this therapeutic agent to lower serum cholesterol levels became possible when commercial quantities of suitable sitosterols made from tall oil were offered by Swift and Company. Betasitosterol, which seems to be the desired sterol for this application, is found widely distributed in vegetable sources. Particularly good sources for betasitosterol are tall oil and cottonseed oil. The composition of the sterols in these oils is 80% to 85% betasitosterol, 15% to 20% dihydrositosterol, and minor amounts of other sterols.

¹ This paper won first place in the 1958-59 Tall Oil Award of the Tall Oil Division of the Pulp Chemicals Association.