and immediately fix in place an air condenser-Ascarite tube (see illustration). Heat the reaction mixture in an oil bath at 100 to 105°C., and allow it to reflux for 45 min. Run two blanks in the same way.

To titrate, add one drop of 0.2% phenolphthalein solution and seven drops of benzene. Stir the solution with a stream of nitrogen bubbles and titrate with 0.5N HCl, using a Gilmont Ultramicro burette.

Iodine Number

Weigh between 3 to 5 mg. of oil to ±0.02 mg. into a 30 x 50-mm. weighing bottle and dissolve in 1 ml. of chloroform. Using a 1-ml. tuberculin syringe, weigh rapidly, to ±0.02 mg., 1 ml. of 0.2N Wijs solution; add it to the oil and seal the bottle by applying 15% KI solution to the stopper. Let stand for 30 min. in the dark, then add 0.5 ml. of 15% KI solution and 2 ml. of water. While the contents of the bottle are stirred magnetically, titrate the excess iodine with 0.01N sodium thiosulphate solution, using Thyodene indicator. Run two blanks with the samples.

Solid Fatty Acids

Into a 50-ml. graduated contrifuge tube, weigh a 50-mg. sample of fatty acids and into another tube 0.15 g. of lead acetate. To each tube add 7.5 ml. of 95% ethanol and boil in a water bath. Add the lead acetate solution to the sample, letting it boil for a further 60 seconds, mixing well with a stirrer. (A convenient stirrer that can be left in the centrifuge tube can be made from a 15-cm. length of 16-gauge Nichrome wire.)

Cool the mixture to room temperature, then place it in an ice bath for 2 hrs. Centrifuge at 3,000 r.p.m. for 3 min., and then decant the supernatant layer. Wash the precipitate 3 times with cold 95% ethanol, mixing well with a stirrer and centrifuging each time. Test filtrate with sulfuric acid for excess lead acetate.

Add 1 drop of acetic acid and boil until the precipitate has dissolved. Cool to room temperature and place in an ice bath for 2 hrs. or in a refrigerator overnight. Centrifuge and decant, then wash with 10 ml. cold 95% ethanol, centrifuge and decant three more times.

Transfer the precipitate to a 30-ml. separatory funnel, washing with 10 ml. ether and finally with 2 ml. of 1:4 nitric acid. Separate the water and wash repeatedly with 7-ml. volumes of water until methyl orange shows no acid present. Transfer to a tared flask and carefully strip under vacuum at 40°C. until almost dry. Dry in an oven at 100°C. until weight is constant.

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Report of the Spectroscopy Committee 1955-56

DURING THE PAST YEAR the Spectroscopy Committee has been concerned with three problems:

a) modifiations of the present A.O.C.S. Tentative Method Cd 7-48, both to simplify it and to extend its scope. At the beginning of the year work on this problem was entering its final phase;

b) investigations to establish a satisfactory method for the quantitative determination of polyunsaturated fatty acids in samples containing large quantities of conjugated constituents. This problem had advanced to the stage where we were about ready for active collaborative measurements;

c) proposed investigation of an infrared absorption method for the determination of *trans*-acids in the presence of nonconjugated *cis*-unsaturated and of saturated constituents. This problem was, at the beginning of the year, still in the planning stages.

Accomplishments During the Year

Recommended Changes in Method Cd 7-48. Collaborative results in the annual report of the Spectroscopy Committee for 1954–55 had shown that the present A.O.C.S. Tentative Method Cd 7-48 could be simplified and its scope extended. If a sample is known to have no constituent more highly unsaturated than diene, measurements and calculations can be restricted to the diene region; if no more unsaturated than triene, to the triene and diene regions, etc. The scope of the method can be increased to include the determination of pentaenoic acids by means of 21% alkali reagents. Preliminary drafts of the proposed modifications in the method to achieve these purposes were prepared and submitted to each committee member for comment, correction, and modification. Based on suggestions received, a final draft was submitted to each member for approval for recommendation to the Uniform Methods Committee for adoption. By a vote of 8–0, with one member not voting, the nine-man Spectroscopy Committee approved submission of the proposed modification to the Uniform Methods Committee for incorporation into the present A.O.C.S. Tentative Method Cd 7-48.

Collaborative Work on Attempts to Establish a Satisfactory Method for Samples Containing Large Quantities of Preformed Conjugation. To test a recently published procedure for the determination of polyunsaturated acids in the presence of large quantities of preformed conjugation (J. Am. Oil Chemists' Soc., *30*, 182–186 [1953]) a sample of *alpha*-tung oil and two samples of mixtures of known proportions of *alpha*- and *beta*-tung oils were submitted to each committee member with specific instructions for collaborative measurements and calculations of linoleic and *alpha*- and *beta*-eleostearic acids. Included with these three samples were urea adducts of both *alpha*- and *beta*-eleostearic acids. Collaborative reports were received from eight of the nine-man committee. However, before a composite tabulation of the results could be compiled, reports had been received from two laboratories of committee members which indicated that the absorptivities for the determinations of *alpha-* and *beta-*eleostearic acids should be modified. Before any further effort of the entire committee was requested, it has appeared essential to recheck these constants. When this has been completed, data received from each committee member will be recalculated, and tabulations of both sets of calculations will be tabulated and submitted with requests for recommendations, from each member, for future activity on this problem.

Investigations of an Infrared Absorption Method. Considerable agitation has been evident for the Spectroscopy Committee to investigate and, if possible, recommend a standard method for the determination of *trans*-acids in fatty acid materials in the presence of nonconjugated *cis*-monounsaturated and *cis*-nonconjugated polyunsaturated constituents. Before any collaborative work on the method proposed by Shreve *et al.* (Anal. Chem., *22,* 1261 [1950]) and Swern *et al.* (J. Am. Oil Chemists' Soc., *27,* 17 [1950]) it has been necessary to obtain standards which can be used to calibrate infrared spectrophotometers in the laboratories of the various committee members. During the past year samples of both elaidic and oleic acids have been obtained, and present plans of the committee are to submit these two standards (if possible, also a sample of pure stearic acid) along with suitable samples collaboratively to test the published methods for determination of *trans*-acids.

Future Committee Work

During the coming year the data from the collaborative work to establish a procedure for the determination of polyunsaturated acids in the presence of large quantities of preformed conjugation should be evaluated and tabulated. From a study of these results the committee should be in a position to decide what steps are next required to achieve the object, a procedure which they can recommend as a tentative A.O.C.S. method.

With reasonably good expectation of the availability of pure samples of oleic, elaidic, and possibly stearic acids, it should be possible critically to evaluate the infrared method for *trans*-acids in fatty acid materials collaboratively. Collaborative work to achieve this purpose is planned for early next year. Another problem which has been referred to the Spectroscopy Committee is the determination of polyunsaturated fatty acids in the presence of *trans*-isomers, a determination outside the scope of Tentative Method Cd 7-48. Consideration of a procedure which could be employed for this purpose will receive attention of the committee during the coming year.

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Synthetic Detergents from Animal Fats. VII. Detergent Combinations[1]

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THE SULFATED HYDROGENATED TALLOW ALCOHOLS, RCH_2OSO_3Na, are known as effective detergents and surface-active agents with perhaps some limitations on their use because they are not very soluble in water at room temperature (5, 6). Nor are the disodium salts of α-sulfonated hydrogenated tallow acids, $RCH(SO_3Na)CO_2Na$, readily soluble. However mixtures of these two tallow-based detergents with phosphate builders, at 20% active ingredient content, form clear 0.25% solutions in hard water at room temperature. It was thus of interest to explore their detergent possibilities. The readily soluble sodium alkylbenzenesulfonate (average molecular weight corresponding to $C_{12}H_{25}C_6H_4SO_3Na$) was also included as a component in the detergent combinations.

Thus four materials were selected for a laboratory study of detergent combinations: sodium salts of sulfated hydrogenated tallow alcohols (X); disodium salts of α-sulfonated hydrogenated tallow acids (Y); sodium dodecylbenzenesulfonate (Z); and an inorganic building composition (B).

Component X was prepared to simulate hydrogenated sulfated tallow alcohols. It is an intimate mixture of the following pure sodium alkyl sulfates: 6.6% sodium tetradecyl sulfate, 27.8% sodium hexadecyl sulfate, and 65.6% sodium octadecyl sulfate (8). These proportions assume tallow to have a fatty acid composition of 6.3% myristic, 27.4% palmitic, 14.1% stearic, 49.5% oleic, and 2.4% linoleic.

Similarly the α-sulfonated hydrogenated tallow acid component, Y, is an intimate mixture of the following pure disodium salts: 6.7% disodium α-sulfomyristate, 27.9% disodium α-sulfopalmitate, and 65.4% disodium α-sulfostearate (8).

The sodium alkylbenzenesulfonate component, Z, of average molecular weight corresponding to $C_{12}H_{25}C_6H_4SO_3Na$, was prepared by sulfonation of an aromatic hydrocarbon[3] with the vapor of sulfur trioxide (3, 4). The product was a hygroscopic, cream-colored solid, with absorption maxima at 224.0, 255.0, 261.5, and 267.5 mμ. Analyses: calculated for $C_{18}H_{29}NaO_3S$, 6.60% Na, 9.20%S; found 6.70% Na, 9.14%S.

The builder, B, contained 55% $Na_5P_3O_{10}$, 24% Na_2SO_4, 10% $Na_4P_2O_7$, 10% sodium metasilicate, and 1% carboxymethylcellulose (5, 9).

Detergency

The detergency of the combinations was measured, using the Terg-O-Tometer and three different kinds of standard soiled cotton, I, II, and III.[4] Detergency was measured as the increase in reflectance, Δ R, after washing in soft (90 p.p.m.) and hard (300

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[1] Presented at the meeting of the American Oil Chemists' Society, Houston, Tex., April 1956.

[2] A laboratory of the Eastern Utilization Research Branch, Agricultural Research Service, U. S. Department of Agriculture.

[3] Neolene 400. Reference to trade name does not constitute recommendation by the U. S. Department of Agriculture of a particular product over similar products not mentioned.