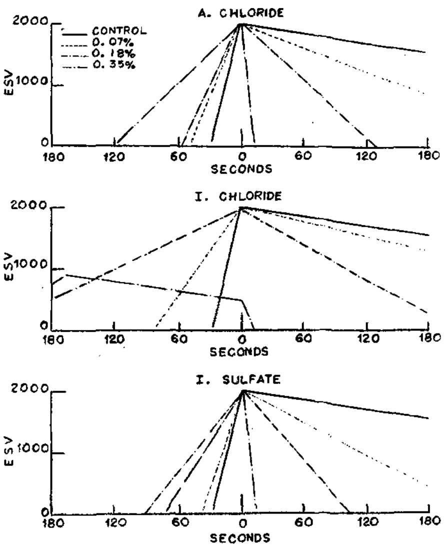

Fig. 8. Antistatic effect of softeners on Nylon.

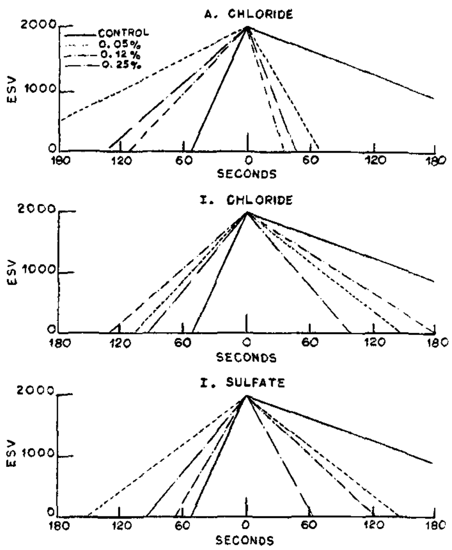

Fig. 9. Antistatic effect of softeners on Dacron.

charge to build up to a maximum of 2,000 volts, or the highest voltage attained in three minutes, whichever occurred first. The right-hand sections of the graphs illustrate the time rate of discharge. The control samples (solid lines) showed the fastest rate of build-up and the slowest rate of leak-off. Treatment of the test fabrics with the three softeners revealed two facts. As the concentration is increased, the rate of electrostatic leak-off is increased and the time required for the build-up is also increased. It should be noted that the percentages of softener on the fabrics are not the same for all four fabrics. For Dacron the wet pickup amounted to 50%, Nylon and Orlon 70%, and acetate 100%.

Figure 6 reveals that the I. chloride and sulfate are more efficient on Orlon than A. chloride. The I. sulfate in turn is a slightly better antistatic agent for Orlon than I. chloride. Figure 7 shows that the same relationship holds true also for acetate. For Nylon the I. chloride is the least effective whereas A. chloride and I. sulfate are about on a par; the I. sulfate has a slight edge over the A. compound. On Dacron (Figure 9) A. chloride is far more effective in bringing about the leak-off than either of the I. compounds. It is interesting to note however that the I. sulfate again has an edge over the corresponding chloride. It would appear that a quaternary methosulfate may be a more effective antistatic agent than the corresponding quaternary chloride having the same structure of the cation. It is obvious that the antistatic properties of a cationic agent depend not only on the chemical structure of the cation but also on the nature of the anion. Furthermore the antistatic effect is somewhat specific for individual synthetic fabrics.

Summary

The quaternary ammonium chlorides and methosulfates of a fatty amine derivative and a corresponding imidazoline derivative possess marked substantivity for cellulosic fabrics. The substantivity was determined quantitatively, and the rates of exhaustion were established. The exhaustion rate is increased with an increase of pH from 5 to 8 as well as with an increase of temperature from 70 to 90°F. Evenness of application appears to be related to slow rates of exhaustion. Evenness can be demonstrated visually by a bromphenol blue staining technique.

The amount of softener add-on has an effect upon the rate of water absorption of treated fabric; the relationship appears to be a logarithmic function. At low percentages of add-on, such as are used in the laundry industry, this is not objectionable.

The antistatic properties of three of the cationic agents on several synthetic nonpolar fabrics were studied by a dynamic friction procedure. The rates of charge build-up and leak-off depend upon the concentration of softener on the fabric. In general, the quaternary ammonium sulfates appear to be somewhat more effective than the corresponding chlorides. Furthermore the antistatic properties vary with the chemical structure of the cation and are somewhat specific for individual fabrics.

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Report of the Spectroscopy Committee, 1957-58

The Spectroscopy Committee met twice during the year ending with the 49th annual meeting of the Society in Memphis. The first session was held October 1, 1957, during the 31st fall meeting of the Society at the Netherland Hilton hotel, in Cincinnati, O. In the absence of the chairman, N. D. Fulton presided. The meeting was attended by five members or their duly authorized alternates: J. R. Chipault, Ralph Kelly (representing R. D. Mair), William Link, Hans Wolff, and Mr. Fulton. The following nonmembers attended: Erik von Sydow, University of Upsala, Sweden; William Ferran, Best Foods Inc.;

TABLE I
Analysis of Tung Oil and Pure Eleostearic Acids in Cyclohexane

Collaborator	A. Tung oil			B. α-Eleostearic acid			C. β-Eleostearic acid		
	α Found	β Found	Total found	α Found	β Found	Total found	α Found	β Found	Total found
	%	%	%	%	%	%	%	%	%
I[5, 6]	71.3[1] 70.6	0 0	71.9 71.7	83.1	0	82.8[1]	2.7	88.2	89.8
II[2, 6]	52.4	0	53.1	58.8	25.8	85.9	8.2	73.0	80.9
III	71.4 73.0	0 0	72.8 74.5	93.7 94.6	0 0	94.7 94.8	13.1 11.0	84.1[1] 88.9	94.3 95.2
IV[4]	76.5	0	78.8	82.1	0	83.2	13.0[1]	58.7[1]	68.6
V[3, 5]	62.2) 33.1[1]) 61.4) 35.8[1])	17.2) 12.4[1]) 15.3) 10.2[1])	—) 44.1[1]) —) 43.3[1])	Not reported	Not reported	Not reported	Not reported	Not reported	Not reported
VI[5, 6]	75.7[1] 78.8	6.4[1] 8.1[1]	77.0[1] 81.9	79.1[1] 77.7[1]	6.0[1] 5.2[1]	89.6[1] 91.0[1]	1.6[1] 0[1]	82.0[1] 80.6[1]	81.4[1] 85.8[1]
VII[5]	68.0 63.2	0 1.8	70.0 68.2	80–84 70[4]	Not reported	Not reported	7–9	94.0 90.0[4]	Not reported
VIII	73.3 73.3	0.9 0.3	75.3 75.0	97.1 97.8	1.8 1.1	99.6 99.7	0 0	97.6 97.9	97.4 97.8
IX[5, 6]	74.9[1] 75.0[1]	0 0	76.1 75.8	88.4 94.7	5.7 0	97.3 95.7	9.6 13.3	84.4 80.4	92.4 91.8

[1] Recalculated from absorbances and concentrations furnished by collaborator.

[2] Calculated from absorbances and concentrations furnished by collaborator.

[3] 2,2,4-Trimethylpentane used instead of cyclohexane.

[4] *Reported instrumental difficulties with consequent delay in making measurements after vials opened.*

[5] Slit widths not indicated, or not within range specified.

[6] Instrument not mentioned.

C. Y. Hopkins, National Research Council, Ottawa, Canada; Marvin Formo, Archer-Daniels-Midland; and R. O. Crisler, Vernon Weis, and Harry Smith from Procter and Gamble.

The second meeting of the committee was held on April 22 at the Peabody hotel in Memphis during the 49th annual meeting. Six members, or their duly authorized representatives, were present: R. R. Allen, Ralph E. Kelly for R. D. Mair, W. E. Link, R. W. Riemenschneider for Samuel F. Herb, E. W. Sallee for N. D. Fulton, and Robert T. O'Connor, chairman. Ronald C. Stillman, former chairman of the Spectroscopy Committee, attended as a visitor.

The Determination of Alpha-, Beta-, and Total Eleostearic Acid by Ultraviolet Spectrophotometry

In connection with the committee's attempts to extend the scope of method Cd 7-48 to the determination of polyunsaturated fatty acids in the presence of large quantities of conjugated constituents (an analysis specifically beyond the present scope of Official Method Cd 7-48) it was decided at the fall meeting of the committee to test collaboratively the analysis of large quantities of preformed conjugation as exemplified by the *alpha-*, *beta-*, and total eleostearic acid in tung oil.

The committee specifically agreed collaboratively to test: (A) A Fresh *Alpha* Tung Oil; (B) A Pure *Alpha*-Eleostearic Acid; (C) A Pure *Beta*-Eleostearic Acid; and mixtures of the pure eleostearic acids prepared from the pure samples as follows: (D) 20% *Alpha*–80% *Beta*; (E) 50% *Alpha*–50% Beta; and (F) 80% *Alpha*–20% *Beta.*

Accordingly each committee member was furnished samples and asked to analyze each in duplicate from cyclohexane solution. In addition, duplicate analyses of each of the acids and mixtures were requested from

TABLE II
Analysis of Tung Oil and Pure Eleostearic Acids in Ethanol

Collaborator	A. Tung oil			B. α-Eleostearic acid			C. β-Eleostearic acid		
	α Found	β Found	Total found	α Found	β Found	Total found	α Found	β Found	Total found
	%	%	%	%	%	%	%	%	%
I[5, 6]				96.3 97.5	0 0	Not reported	5.5[1] 6.9[1]	95.8[1] 95.2[1]	Not reported
II[2, 6]				92.2	1.7	96.0	4.5	91.1	96.0
III				97.1 96.1	0 0	99.3 98.7	9.8 13.7	90.3 88.6	98.4 98.8
IV[4]	70.2	0	73.8	93.5	0	95.3	12.0	88.1	97.7
V[2, 5]				100.4 101.8	0 0	103.3 106.1	19.7 13.8	81.4 92.5	96.6 102.0
VI[5, 6]				82.8[1] 84.3[1]	9.3[1] 6.4[1]	92.0[1] 93.1[1]	8.2[1] 1.4[1]	73.4[1] 87.3[1]	82.3[1] 88.2[1]
VII[5]				70.0[4]	Not reported	Not reported	7–9	96.0[4]	Not reported
VIII				95.1 94.5	1.6 2.3	97.2 97.8	0 0	100.1 99.9	101.5 101.5
IX[5, 6]				99.0 99.4	0 0	101.2 100.8	5.4 8.4	96.3 92.7	99.7 99.5

[1] Recalculated from absorbances and concentrations furnished by collaborator.

[2] Calculated from absorbances and concentrations furnished by collaborator.

[3] 2,2,4-Trimethylpentane used instead of cyclohexane.

[4] Reported instrumental difficulties with consequent delay in making measurements after vials opened.

[5] Slit widths not indicated, or not within range specified.

[6] Instrument not mentioned.

99% ethanol solution. Results obtained showed that the pure samples of *alpha*- and *beta*-eleostearic acids had not withstood the mailing and waiting in the various collaborators' laboratories without some isomerization and probably polymerization. The data were recalculated by the chairman, using the reported analysis of the pure acids of each collaborator as the basis for the composition of the mixtures and comparing these data with the experimentally obtained results in each laboratory. Results for the fresh *alpha*-tung oil, and for the pure *alpha*- and *beta*-eleostearic acids are shown in Tables I and II for the two solvents. Results for the mixtures, with the calculated compositions based on the data of Tables I and II, are given in Tables III and IV.

TABLE III

Analysis of Mixtures of α- and β-Eleostearic Acids in Cyclohexane

Collaborator	D. 20% α–80% β		E. 50% α–50% β		F. 80% α–20% β	
	Calc.[7]	Found	Calc.[7]	Found	Calc.[7]	Found
I[4, 5, 6]	Not reported		Not reported		Not reported	
II[2, 6]	% 21.6 α 60.4 β 82.0 T	% 22.1 α 68.9 β 91.4 T	% 32.5 α 50.4 β 82.9 T	% 32.3 α 46.6 β 78.7 T	% 48.2 α 35.8 β 84.0 T	% 50.9 α 32.1 β 81.7 T
III	28.6 α 28.7 α 69.1 β 68.9 β 97.7 T 97.6 T	32.1 α 29.7 α 67.8 β 70.6 β 97.3 T 97.1 T	53.2 α 53.5 α 43.1 β 42.8 β 96.3 T 96.3 T	57.0 α 59.4 α 41.7 β 40.7 β 97.8 T 98.6 T	77.9 α 78.1 α 17.1 β 17.0 β 95.0 T 95.0 T	83.4 α 84.4 α 12.6 β 12.7 β 96.6 T 97.1 T
IV[4]	26.9 α 46.9 β 73.7 T	28.0 α 59.0 β 84.0 T	47.3 α 29.4 β 76.7 T	51.9[1]α 28.3[1]β 79.9 T	67.9 α 11.9 β 79.8 T	74.2[1]α 11.5[1]β 86.5 T
V[5]	Not reported		Not reported		Not reported	
VI[5, 6]	16.3 α 66.2 β 82.5 T	20.0 α 17.0 α 64.4 β 62.6[1]β 83.2 T 87.0 T	39.6 α 43.4 β 83.0 T	42.7 α 44.0 α 38.3 β 43.4 β 85.6 T 88.1 T	62.9 α 20.7 β 83.6 T	63.0 α 63.4 α 26.2 β 30.0 β 89.5 T 91.0 T
VII[5]	Not reported		Not reported		Not reported	
VIII	19.4 α 19.5 α 78.4 β 78.6 β 97.8 T 98.1 T	18.2 α 16.7 α 70.8 β 72.0 β 89.4 T 89.0 T	48.5 α 48.8 α 49.7 β 49.6 β 98.3 T 98.4 T	42.9 α 45.4 α 49.5 β 46.6 β 92.9 T 92.5 T	77.7 α 78.2 α 21.0 β 20.5 β 98.7 T 98.7 T	74.4 α 74.0 α 19.0 β 19.7 β 93.3 T 94.0 T
IX[5, 6]	26.7 α 67.2 β 94.0 T	19.7 α 76.9 β 95.6 T	50.4 α 43.8 β 94.1 T	40.0 α 57.5 β 97.7 T	Not reported	

[1] Recalculated from absorbances and concentrations furnished by collaborator.

[2] Calculated from absorbances and concentrations furnished by collaborator.

[3] 2,2,4-Trimethylpentane used instead of cyclohexane.

[4] Reported instrumental difficulties with consequent delay in making measurements after vials opened.

[5] Slit widths not indicated, or not within range specified.

[6] Instrument not mentioned.

[7] Calculations take into account the % α in β- and the % β in α-eleostearic acid.

The results are, in most cases, not entirely satisfactory. At the spring meeting of the committee in Memphis it was decided that this work should be repeated with the samples furnished in a more stable form. It is believed that urea adducts of the two pure acids will be sufficiently stable for collaborative study, and these samples will be submitted to each collaborator just as soon as they can be made available. So that every sample will be identical the chairman will prepare the three synthetic mixtures. The samples will be analyzed in ethanol with the special precaution that the sample must a) be analyzed promptly and b) it must be analyzed immediately after it is dissolved in the ethanol solvent. This collaborative work is planned early in the coming year.

Determination of "Trans" Acids as "Elaidic" by Means of Infrared Absorption

At both committee meetings considerable interest was expressed in the need for a standard or official method for the determination of *trans* isomers by means of infrared absorption. Following collaborative work of the committee last year (1) it was decided that: a) if all collaborators were to follow in strictest detail a very specific set of experimental procedures, good collaborative agreement might be expected; b) each infrared spectrophotometer to be used for the quantitative determination of *trans* isomers must be calibrated individually with a suitable standard or standards; and c) for analysis of acids, the standard must be elaidic acid; for esters, an elaidate; and for triglycerides, trielaidin.

From discussion at the spring meeting in Memphis it appears that suitable standards may now be available. These are being collected by the chairman, who will prepare specific procedures in the format of the A.O.C.S. Official Methods for the analysis of acids, esters, and triglycerides. With each type of sample an elaidic acid, methyl elaidate, or trielaidin primary standard will be supplied each collaborator for instrument calibration. The collaborative work is expected to lead to recommendation for the tentative methods of analysis for these types of materials.

A.O.C.S. Method Cd 7-48 for the Determination of Polyunsaturated Fatty Acids

The Uniform Methods Committee has adopted the minor revisions recommended by the Spectroscopy Committee in last year's report (1). Recommendation that this method be changed from "tentative" to "Official" has also been approved. These changes will appear in revisions of the Official Methods for 1958.

The Uniform Methods Committee has called the attention of the Spectroscopy Committee to a potentially dangerous step in this procedure. Some laboratories have experienced trouble with the preparation of the 6.6% potassium hydroxide—glycol solution in the 25-min. isomerization. The danger arises from the violent disengagement of water when KOH pellets are added to the glycol at 150°C. One isolated incident of an explosion has been reported during this operation.

At the spring meeting in Memphis the Spectroscopy Committee considered this matter and, after considerable discussion with members who are familiar with the method, decided that safety would be assured if the KOH pellets were added to the glycol at a somewhat lower temperature. It was recommended specifically that the method be amended as follows:

(B) 11 c: Change last sentence from

> Lower the bath and allow the bath temperature to drop to 150°C.
>
> to
>
> Lower the bath and allow the bath temperature to drop to 120°C.

(B) 11 d: Change first sentence from

> When the bath temperature reaches 150°C., carefully add 60 g. of 85% KOH, A.C.S. grade pellets to the glycol, keeping the solution under nitrogen.

TABLE IV
Analysis of Mixtures of α- and β Eleostearic Acids in Ethanol

Collaborator	D. 20% α–80% β		E. 50% α–50% β		F. 80% α–20% β	
	Calc.[7]	Found	Calc.[7]	Found	Calc.[7]	Found
	%	%	%	%	%	%
I[4, 5, 6]		19.0 α		21.6 α		61.6 α
	26.4 α	19.5 α	49.1 α	21.2 α	79.6 α	62.7 α
		73.5 β		50.3 β		20.7 β
	74.2 β	73.9 β	50.3 β	51.3 β	18.2 β	20.0 β
		92.0 T		72.0 T		83.2 T
	100.6 T	93.1 T	99.4 T	72.9 T	97.8 T	83.2 T
II[2, 6]	20.2 α	14.6 α	50.2 α	35.7 α	74.6 α	61.2 α
	75.2 β	60.8 β	44.4 β	46.0 β	19.8 β	19.5 β
	95.4 T	75.9 T	94.7 T	81.9 T	94.3 T	82.4 T
III	28.6 α	32.9 α	54.0 α	59.7 α	79.5 α	84.3 α
	27.5 α	28.8 α	52.2 α	54.6 α	78.4 α	82.9 α
	71.6 β	68.3 β	44.9 β	40.3 β	18.0 β	14.6 β
	72.8 β	72.4 β	46.8 β	45.5 β	19.2 β	14.9 β
	100.3 T	99.1 T	98.9 T	99.7 T	97.5 T	99.9 T
	100.4 T	99.2 T	99.0 T	99.2 T	97.6 T	101.1 T
IV[4]	28.3 α	28.3[1]α	52.5 α	57.7[1]α	77.0 α	79.3[1]α
	70.5 β	71.5[1]β	44.3 β	41.1[1]β	17.8 β	18.0 β
	98.8 T	97.0 T	96.8 T	97.6 T	94.8 T	98.3 T
V[5]		30.0 α		61.7 α		88.2[1]α
	33.7 α	33.0[1]α	58.9 α	75.3 α	83.6 α	86.0[1]α
		69.0 β		39.0 β		11.9 β
	69.2 β	71.3 β	43.0 β	30.7 β	17.1 β	15.1 β
		97.2[2]T		100.6[2]T		100.5[2]T
	102.9 T	101.8[2]T	101.8 T	107.3[2]T	100.7 T	105.2[2]T
VI[5, 6]		23.2 α		45.8 α		66.9 α
	20.6 α	20.3[1]α	44.2 α	47.2 α	67.8 α	69.0 α
		62.2 β		41.5 β		22.2 β
	65.8 β	68.4 β	44.1 β	42.8 β	22.4 β	21.7 β
		88.1 T		89.4 T		90.6 T
	86.4 T	88.0 T	88.3 T	88.0 T	90.1 T	89.5 T
VII[5]	Not reported		Not reported		Not reported	
VIII	19.0 α	17.7 α	47.5 α	46.6 α	76.1 α	76.2 α
	19.0 α	18.7 α	47.3 α	47.0 α	75.7 α	76.5 α
	80.3 β	80.2 β	50.8 β	51.5 β	21.3 β	21.1 β
	80.4 β	80.8 β	51.0 β	51.4 β	21.8 β	21.3 β
	99.3 T	99.6 T	98.3 T	99.4 T	97.4 T	98.1 T
	99.3 T	100.8 T	98.4 T	99.8 T	97.4 T	98.7 T
IX[5, 6]	Not reported		Not reported		Not reported	

[1] Recalculated from absorbances and concentrations furnished by collaborator.

[2] Calculated from absorbances and concentrations furnished by collaborator.

[3] 2,2,4-Trimethylpentane used instead of cyclohexane.

[4] Reported instrumental difficulties with consequent delay in making measurements after vials opened.

[5] Slit widths not indicated, or not within range specified.

[6] Instrument not mentioned.

[7] Calculations take into account the % α in β- and the % β in α-eleostearic acid.

to

> When the bath temperature reaches 120°C, carefully add 60 g. of 85% KOH A.C.S. grade pellets to the glycol and swirl the flask until the alkali is completely dissolved with nitrogen sweeping vigorously (not less than 50 to 100 ml. of nitrogen per minute) through the flask.

These changes will be recommended to the Uniform Methods Committee for adoption.

Spectroscopy Committee as a Central Collection Agency for Infrared Spectra of Fats and Oils

At the fall meeting it was suggested that the Spectroscopy Committee could perform a valuable service to the fat and oil industry by acting as a central agency for the collection, examination, and dissemination of infrared spectra of fatty materials from all sources. This proposal was discussed in some detail at the spring meeting. Two plans were considered. Under the first plan, spectra which must meet specifications of the committee, designed to guarantee only spectra of sufficiently high caliber, would be submitted to a central location. These spectra would then be reproduced by some nonprofit agency such as the Coblentz Society or by some commercial groups interested in reproducing spectra. The second plan would provide that pure fat and oils and related materials would be submitted to a designated laboratory where the spectra would be obtained under uniform conditions. The donor of the pure compound would be required to certify as to its purity. The spectra, which would have the advantage of being all identical in format, would then be submitted as before for reproduction and distribution by a nonprofit organization or a commercial firm.

Discussion at the spring meeting in Memphis resulted in the opinion that the collection of spectra by either of the proposed methods would be quite feasible. Reproduction and distribution however might become a bit more of a problem as most groups distributing spectra would want to reproduce these spectra in their own format and several of them are not interested in sale of specific spectra but only in subscriptions to all of the spectra they distribute. Details for activating this proposal will be investigated during the coming year, and, if at all feasible, some method for making infrared spectra available will be worked out.

Determination of Polyunsaturated Acids in the Presence of Cis and Trans and Positional Isomers Analysis of Hydrogenated Oils

The extension of the scope of Method Cd 7-48 to hydrogenated oils containing *cis* and *trans* and positional isomers has been frequently suggested as a pressing problem. The committee agreed that a need exists for this extension of application of method Cd 7-48. It was decided that the recommended order of approach is a) look for a better isomerization agency and b) look to longer isomerization times. However this problem now requires research in an individual laboratory. Once a proposal is made, the committee would be ready to test it in collaborative study. However it is not within the scope of the Committee's activities to conduct research to devise new methods.

Acknowledgment

The Spectroscopy Committee gratefully acknowledges the cooperation and assistance of several individuals in the completion and interpretation of the collaborative work. The chairman, in particular, wishes to acknowledge the considerable assistance offered by Miss Dorothy Heinzelman in recomputing and compiling the collaborative data on the tung oil and eleostearic acid samples.

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REFERENCE

1. J. Am. Oil Chemists' Soc., *34*, 600–603 (1957).