which depends upon the refining of the oils by the standard A.O.C.S. procedure, separating the foots, and centrifuging them in the Servall machine. A committee, under the chairmanship of J. R. Mays, was appointed and some work with this method was carried out. A report was submitted at the New York meeting.

N. W. Ziels and his small subcommittee presented a report at the New York meeting covering work with the cone-type agitator in the centrifuge method as well as a proposed modification in which both the refining and bleaching work would be carried out at room temperature. The results indicated that while it appeared possible to refine with the cone agitator with temperatures of 100° F. and below, the cold bleaching was not successful.

The results of the work on the two types of agitators during the year indicated that both gave reproducible results and that low temperature refining with the cone agitator gave reproducible results. At the New York meeting Mr. Ziels exhibited a multiple paddle machine fitted with cone agitators to be used with the centrifuge method, and Mr. Cole, the designer and builder, was present. It was decided that experimental work would be carried out with this machine in the Lever Brothers laboratories and a report made to the committee at a later date. This work is still under way, and it is not intended to make a formal report at the New Orleans meeting.

In view of the difficulty with the standard A.O.C.S. Method on degummed expeller oils, three members of the committee volunteered to check this method and to investigate the use of an intermediate excess (0.15% as 12° lye) in the refining. Mr. Mays' committee on the modified centrifuge method also agreed to run check tests on the samples of degummed expeller oils. A report covering this work, together with the results of a referee sample of degummed expeller oil in which the intermediate excess was also employed, were reported to the committee in a report issued March 24, 1949. The results showed clearly that the method was considerably improved by the use of the intermediate excess.

As a result of a vote of the members the Refining Committee recommends to the Uniform Methods Committee that a new tentative method for the refining of degummed expeller soybean oils only be established in which the excesses to be used are 0.15% and 0.20% NaOH as 12° lye.

E. M. JAMES.

Thermal Polymerization of Unsaturated Fatty Esters Normal Methyl Linoleate*[1]

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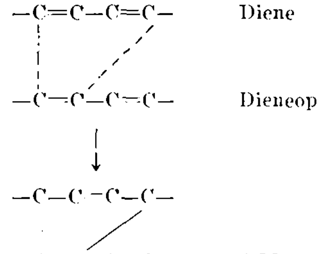
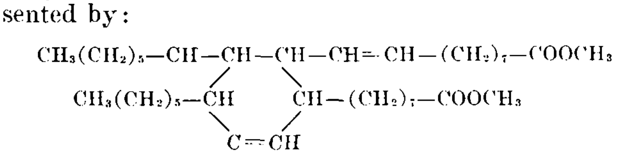
Introduction

THE knowledge of the reactions involved in the thermal polymerization of drying oils has increased considerably during the past 10 years. This has been largely due to the study of the thermal polymerization of the simple monohydric alcohol esters of the unsaturated esters of the drying oil acids. The earlier work of Kino (1), Kappelmeier (2), Steger and Van Loon (3), and Brod, France, and Evans (4) all pointed to the formation of a polymer which was largely dimer when the methyl or ethyl esters of polyunsaturated acids were polymerized by heat.

Bradley and Johnston (5) confirmed dimerization as the principal polymerization reaction of the methyl esters from olive, dehydrated castor, soybean, linseed, and tung oils. These same workers (6) subsequently isolated a methyl linoleate dimer from the polymerized methyl ester of dehydrated castor oil acids. Bradley and Richardson (7) followed the development of conjugation during thermal polymerization and found that conjugation reached a low equilibrium value when non-conjugated esters were used as starting material. This fact, together with the properties of the dimers from non-conjugated esters, was considered by Bradley and Johnston to be confirmation of the Scheiber (8) theory that non-conjugated linoleic and linolenic esters are changed to the conjugated forms before they polymerize and that the dimerization was by way of a modified 1,4-diene or Diels Alder addition, as proposed by Kappelmeier (9) for eleostearate, between two molecules of the conjugated forms. In such a diene addition, one of the conjugated systems acts as the diene, while a double bond in another molecule acts as the dieneophile. According to this theory,



the dimers from conjugated and non-conjugated esters would be essentially identical. The dimer of conjugated or non-conjugated linoleate was represented by:



this being a dimer of 9,11-methyl linoleate.

Another possibility which was not considered, or at least has not been emphasized, is that the dimerization might be a modified 1,4-diene addition between a conjugated form and a non-conjugated form with a double bond of the non-conjugated form acting as dieneophile. This would result in a structure similar

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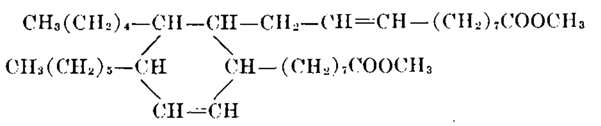
* Presented at 22nd fall meeting, American Oil Chemists' Society, New York City, Nov. 15-17, 1948.

[1] Paper No. 97, Journal Series, Research Laboratories, General Mills inc.

to the above, but with the non-cyclic double bond one carbon further removed from the ring, such as:



this being a dimer between a 9,11- and a 9,12-linoleate, with the 12 double bond acting as the dieneophile.

An analogy to this conjugate-non-conjugate dimer mechanism is found in the study of the thermal polymerization of 1,4-pentadiene by Ahmad and Farmer (10). The dimer was mainly of a structure such as would result from the diene addition of a conjugated 1,3-pentadiene with a double bond of the original 1,4-pentadiene. Recent work by Lawson and Spoerri (11) has indicated that copolymerization occurs between conjugated and non-conjugated fatty esters in their mixtures.

It was recently suggested by one of the authors that in the polymerization of normal linoleate conditions are favorable for such a conjugate-non-conjugate dimerization since the concentration of non-conjugated linoleate is quite high in proportion to that of conjugated in the early stages of polymerization.

The present work reports the results of a study of the polymerization of pure methyl linoleate at 290° and 300°, with analyses for monomer, dimer, trimer, and for normal and conjugated linoleate at various intervals of time. Also, certain binary mixtures of normal linoleate, conjugated linoleate, oleate, and stearate were polymerized in order to get some indication as to the relative dieneophilic activity of different double bond systems and to show the existence and extent of copolymerization of conjugated and non-conjugated linoleate.

Experimental

Analytical Methods. The concentrations of conjugated and of unchanged or normal linoleate were determined by spectroscopic methods, using the specific absorption coefficient ($K^{0.1\%}_{1\,\text{cm.}}$ or sp. α) of 115 at 2320 Å for conjugated linoleic acid as given by Kass (12) and using the empirical constant of 87.1 for normal linoleic acid after alkali isomerization by the method of Mitchell, Kraybill, and Zscheille (13). The normal linoleate content was calculated from the *increase* in sp. α after isomerization when conjugated linoleate was present. The amounts of monomer, dimer, and trimer were determined by analytical distillation. The bulk of the monomer was removed by vacuum distillation at 0.1 mm. in a stirred flask equipped with a short Vigreaux column, taking pot temperature to 235°. The residual dimer plus trimer, containing only a small amount of monomer was then fractionally distilled in an alembic type flask, taking small fractions and plotting refractive index vs. mean per cent distilled, as described by Cowan, Falkenberg, and Teeter (14). The monomer-dimer cut-off point was taken at the n^{30}_{D} half way between that of the main monomer fraction and that of middle point of the dimer plateau. Trimer (trimer plus any higher polymer) was taken as residue after one-half-hour hold at 300°. Free air pressure during the alembic distillation was 5-10 μ. The method was tested on known mixtures of methyl linoleate (both normal and alkali conjugated) with a pure dimer (prepared by molecular distillation in a falling film molecular still), and on a known mixture of similar dimer and trimer. These tests showed the method to be accurate to about 1% for monomer, dimer, and trimer content of the known mixtures.

Materials. Methyl linoleate was prepared from tetrabromostearic acid, m.p. 115° by debromination in methanol with zinc and esterification by the addition of sulfuric acid catalyst. The recovered methyl linoleate was vacuum distilled before use. Alkali conjugated methyl linoleate was prepared from debromination linoleate, as described by Terry and Wheeler (15). Methyl oleate was prepared from commercial oleic acid by fractional distillation and repeated crystallization of the methyl ester. $n^{30}_{D} = 1.4487$; sap. no. 188.5 (theory 189.2); I.V. 84.5 (theory 85.6). Linoleate by spectral analysis = 0.12%. Methyl stearate was prepared by usual methods, I.V. = 0.86.

Polymerization of Normal Linoleate. Polymerizations were carried out on 80-g. samples in evacuated and sealed glass tubes which were heated in holes in a thermostatically controlled aluminum block, using a calibrated thermometer. Heat-up time was 30 minutes. The results of the polymerizations at 300° and 290° are shown in Table I.

In order to follow the disappearance of normal and appearance of conjugated linoleate at shorter times, another series was run using 2-g. samples in small tubes so that the heat-up time (10 minutes) would be negligible. These were analyzed on the whole sample for normal and conjugated linoleate. The results are shown in Table II. The values for conjugated linoleate will be somewhat high, especially in later stages, since dimer has a slight absorption at 2300Å. However, the values at the earlier stages should be accurate since very little polymerization has then occurred.

As shown in these tables, the disappearance of normal linoleate gives a fairly constant value of K, the velocity constant for a monomolecular or first order reaction, and the values are in fairly good agreement between the series in Table I and the series in Table II. Calculation of n, the order of reaction, from the data in Table I by a differential method (16) gave values of 1.1 and 1.2 respectively.

The following abbreviations will be used: M = monomer; D = dimer; T = trimer; N = normal linoleate; C = conjugated linoleate; X = a monomer which is not normal or conjugated linoleate.

The results are shown graphically in Figs. 1 and 2. The values below 6 hrs. at 300° and 12 hrs, at 290° are from the small runs (Table II). The line for per cent N (on log scale) vs. time is almost straight, as would be expected for a first order reaction. C reaches a maximum of 6-7% in 6 hrs. at 300° or 10 hrs. at 290° and then gradually decreases. X reaches a maximum at about the same time, but remains fairly constant, while D and T increase progressively.

This first order reaction for disappearance of normal linoleate would be expected from the generally accepted theory that normal linoleate isomerizes to conjugated linoleate which then polymerizes:

$$A \begin{cases} (1)\ \text{N} \longrightarrow \text{C monomolecular or first order reaction (relatively slow)} \\ (2)\ \text{C} + \text{C} \longrightarrow \text{D consecutive bimolecular reaction (relatively fast).} \end{cases}$$

TABLE I

Thermal Polymerization of Normal Methyl Linoleate

Hours	M%[1]	I.V.[2] Monomer	D%[3]	T%[4]	$\frac{D}{T}$	N%[5]	C%[6]	X%[7]	K[8]
300°									
6	79.1	163.9	19.6	1.4	14.0	49.9	6.9	22.2	0.116
12	53.2	154.5	41.6	5.2	8.0	27.0	6.3	19.9	0.103
24	30.2	131.7	59.9	9.9	6.1	8.4	3.0	18.8	0.100
48	19.4	100.5	62.2	18.1	3.4	1.0	1.6	17.8	0.089
Average K by plot of log N vs. time									0.100
290°									
12	74.9	163.5	23.6	1.7	13.9	48.0	6.5	20.3	0.061
24	47.3	151.5	44.2	8.4	5.3	22.7	4.5	20.1	0.062
48	27.3	129.8	59.6	13.2	4.5	6.8	3.1	17.4	0.052
96	17.6	101.6	63.5	18.4	3.5	0.9	1.4	15.3	0.042
Average K by plot of log N vs. time									0.053

[1] M = monomer.

[2] I.V. = iodine value by rapid Wijs method (Hoffman & Green, Oil and Soap *16*, 236, 1939).

[3] D = dimer.

[4] T = trimer.

[5] N = normal linoleate, by spectral analysis after alkali conjugation.

[6] C = conjugated linoleate by spectral analysis before alkali isomerization.

[7] X = monomer which is not normal or conjugated linoleate = M — (N + C).

[8] K = monomolecular reaction constant for disappearance of normal linoleate = $\frac{2.303}{t_2-t_1}\log\frac{\%N_1}{\%N_2}$.

However, it is to be noted that the value for K became smaller in the later stages of reaction; furthermore, the per cent of conjugated linoleate rose to a low value after 3 to 6 hrs. which changed relatively little during the period of rapid dimer formation and then dropped off in the later stages of reaction. These two facts suggested the possibility that a consecutive bimolecular reaction between normal and conjugated linoleate might be occurring. This would be formulated as:

$$B\begin{cases}(1)\ N \longrightarrow C\ \text{monomolecular reaction}\\ \qquad\text{(relatively slow)}\\ (2)\ C + N \longrightarrow D\ \text{bimolecular reaction}\\ \qquad\text{(relatively fast).}\end{cases}$$

(First order with respect to C and to N.)

The fairly uniform value of K for disappearance of normal linoleate could be explained by the more or less constant concentration of conjugated linoleate during the main reaction, which would result in a pseudomonomolecular reaction, first order with respect to normal linoleate. This would also explain the lower value of K in the later stages, where dilution and the lower concentration of conjugated linoleate would retard this second order reaction.

If, indeed, it could be shown that conjugated linoleate reacts readily with normal linoleate to produce a dimer, it would be reasonable to predict that the dimer formed from normal linoleate is quite largely the reaction product of a thermally produced conjugated linoleate with normal linoleate, especially in the earlier stages of reaction. Dimerization between two conjugated linoleates would be relatively slight in extent because their concentration is low while the concentration of normal linoleate is relatively high. This reasoning assumes that the formation of dimer by reaction of two molecules of normal linoleate is relatively slow or non-existent.

Copolymerization of Normal and Alkali Conjugated Linoleate. Experiments were made to determine whether the reaction of conjugated with normal linoleate did occur and to estimate how extensive this reaction might be. Polymerization of a mixture of equal parts of normal linoleate and alkali conjugated linoleate was compared to that of similar mixtures of methyl stearate with the conjugated and with normal linoleate. Results are shown in Table III.

If there was no reaction between normal and conjugated linoleate each component would act merely as a diluent of the other, and one might expect 33.9 + 4.6 = 38.5% of polymer in the N + C mixture. Actually 66.7 was found which would indicate 66.7 — 38.5 = 28.2% of polymer which could be due to the N + C ⟶ D reaction. This 28.2% of the reaction mixture represents $\frac{28.2}{66.7}$ = 42.3% of the total polymer from N + C. This probably represents a *minimum*, since any N + C reaction decreases the concentration of N and of C and would decrease the amount of their polymerization by themselves. It might be argued that if normal isomerizes to conjugated linoleate in the N + C mixture, the increased amount of polymer in the N + C mixture could still be explained solely by dimerization of conjugated linoleate

TABLE II

Thermal Polymerization of Normal Methyl Linoleate
(2-g. sample not distilled)

Hours	%N	%C	%X	I.V. Mixture	n_D^{30}	K, hrs.$^{-1}$
300°						
0	100.8	0.3	—1.0	172.3	1.4582	....
0.75	93.4	2.0	2.0	170.9	1.4585	0.101
1.5	86.1	3.3	5.0	167.4	1.4587	0.107
3	74.8	5.8	8.4	163.3	1.4596	0.094
6	52.6	8.9	16.0	149.9	1.4621	0.117
12	27.8	8.8	16.6	122.2	1.4668	0.106
24	7.1	6.0	17.1	98.5	1.4715	0.114
48	0.4	3.7	15.3	86.1	1.4738	0.120
Average K by plot of log % N vs. time						0.111
289°						
0	96.4	0.4	3.2	172.8	1.4578	....
1.5	88.7	2.0	5.0	170.5	1.4582	0.055
3	83.1	3.4	6.0	168.9	1.4588	0.043
6	70.0	5.8	10.2	162.8	1.4601	0.058
12	46.8	8.1	19.1	146.3	1.4631	0.067
24	25.2	7.4	14.7	121.2	1.4674	0.051
48	7.5	4.7	14.1	99.8	1.4718	0.051
96	1.3	3.0	13.3	89.2	1.4742	0.037
Average K by plot of log N vs. time						0.054

TABLE III

Polymerizations at 290° (50-50 mixtures) 12 hrs. of Normal and Conjugated Linoleate Mixtures

Components	% D + T	D/T	n_D^{30} of dimer*
N + St	4.6	10.0	1.4760
C + St	33.9	3.4	1.4744
C + N	66.7	4.5	1.4760
(N alone)	25.3	14.1	1.4762
(C alone)	80.9	2.9	1.4753

*From mid-point of dimer plateau on alembic distillation.

with itself. However, the refractive index of the dimer from conjugated linoleate is considerably lower than that from N or from the N + C mixture and is a qualitative indication that the dimer formed from normal linoleate is not the result of dimerization of thermally generated conjugated linoleate but is largely the reaction of such a conjugated linoleate with a normal linoleate.

Dilution Polymerization of Normal Linoleate. Since the above data indicated the probability of a bimolecular reaction between normal and conjugated linoleate, it was thought that dilution of normal linoleate with an inert material such as methyl stearate should minimize the effect of this bimolecular reaction and by extrapolation and afford a measure of the true monomolecular reaction constant for disappearance of normal linoleate without the effect of any bimolecular reactions.

Thus if scheme A was the principal mechanism, the value for K, the first order velocity constant, should not be affected by dilution since no collision with another molecule of linoleate is necessary for reaction A (1), the only reaction by which N disappears. However, if scheme B occurs to any very appreciable extent, the apparent value of K should be lower on dilution since the bimolecular reaction B (2) which involves N would be tremendously slowed down by dilution, theoretically completely stopped at infinite dilution, since the two molecules N and C would never collide even though this reaction might be relatively fast when they are present in relatively high concentrations.

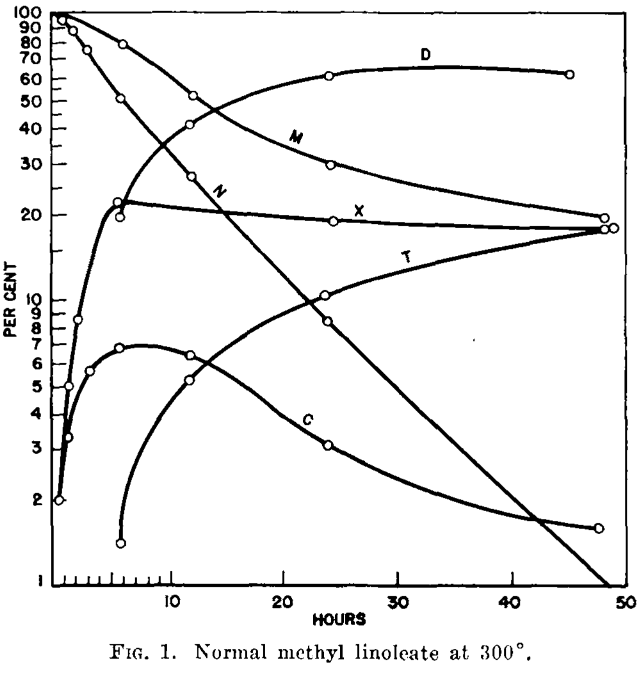


FIG. 1. Normal methyl linoleate at 300°.

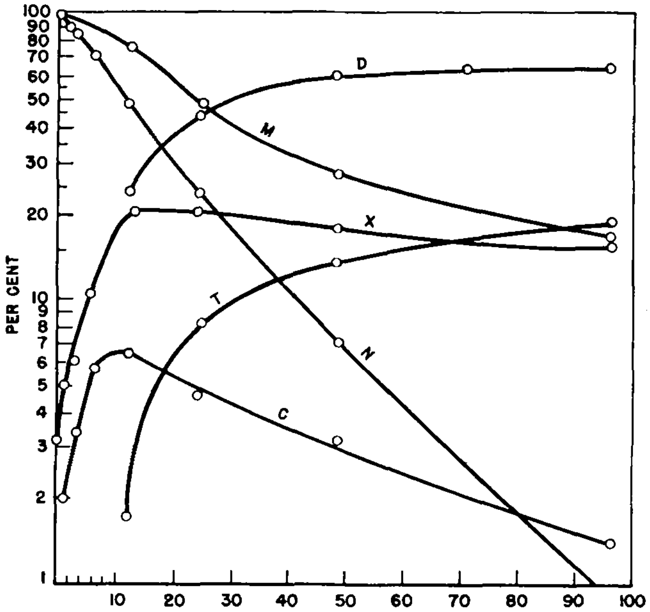


FIG. 2. Normal methyl linoleate at 290°.

Such dilution experiments were performed, diluting normal linoleate with methyl stearate to 40, 20, and 10% linoleate. The mixtures were heated for various times at 290° and 300° and analyzed for normal linoleate on the whole mixture. While individual values for K, the monomolecular rate constant, were somewhat erratic, average values, obtained by plotting log N vs. time, were obtained which are certainly fairly accurate. These are shown in Table IV.

TABLE IV

Values of K for Normal Linoleate Diluted With Methyl Stearate

% N at start	K, hrs.$^{-1}$ 290°	K, hrs.$^{-1}$ 300°
100	0.053	0.100
40	0.028	0.046
20	0.015	0.028
10	0.016	0.025

It will be seen that, with dilution, the values of K decrease to a limiting value of about one-fourth that obtained on undiluted linoleate. This very much lower rate obtained on dilution indicates that the disappearance of undiluted normal linoleate during polymerization is quite extensively due to a bimolecular or higher order reaction which is monomolecular with respect to normal linoleate. The proposed conjugated-non-conjugated dimerization reaction is qualitatively in agreement with the effects observed on dilution. However, it has not been possible so far quantitatively to account for the formation of dimer, trimer, and conjugated linoleate in a completely satisfactory manner by any treatment of the reaction constants obtained.

Moreover, there are other reactions occurring beside the isomerization and dimerization reactions discussed so far. The decreasing iodine values of the monomer to a value of about 100, as shown in Table I, indicate the formation of monomers of decreased unsaturation. The rather rapid appearance of 15-20% of X, a monomer which is neither conjugated nor normal linoleate, and its rather constant value throughout the reaction is a rather surprising phenomenon which is difficult to explain qualitatively or quantitatively.

Monomers From Normal Linoleate. A consideration of the iodine numbers of the monomers and the amounts of conjugated and normal linoleate found indicates that in the earlier stages of reaction X is largely an isolinoleate. This isolinoleate decreases in amount while the concentration of monoethenoid esters increases as the reaction proceeds. The monomers from the final samples (48 hours 300°, 96 hours at 290°) apparently consist largely of these monoethenoid esters. These two monomers were combined and crystallized three times from acetone (10 cc./g. at —60°C.) to afford 10.3% of a precipitate which appeared to be a fairly pure isomer of methyl oleate. (No precipitate formed at —30°, indicating the absence of methyl stearate.)

It showed the following properties:

I.V. (rapid Wijs)	84.8 (theory 85.6)
I.V. (hydrogenation)	80.8
B.P. at 2.6 mm.	162-75° (170° for oleate)
n_D^{30}	1.4480 (1.4487 for oleate)
d^{30}	.8647 (.8679 for oleate)
Mol. refr.	91.8 (91.2 theory for oleate)
M.P. °C.	+3 to 6° (—19.9 for oleate)
sp. α 2300 Å	2.6 (0 theory)
sp. α after alk. isom. 2300 Å	1.8 (0 theory)
2680 Å	0 (0 theory)

From the solution after semi-micro I.V. by hydrogenation, methyl stearate was isolated in 81% yield, m.p. 38-38.5°, and no depression on mixture with authentic methyl stearate.

The filtrate from crystallization of this iso-oleate was recovered and hydrogenated to give a 36% yield of methyl stearate. This yield, combined with that obtained from the crystallized iso-oleate, represents a 40.7% yield of methyl stearate by hydrogenation of this whole monomer mixture. The yield of stearate expected from the estimated content of conjugated, normal, and isolinoleate is 14%. These figures indicate that this monomer contains about 26% of oleate or iso-oleates which hydrogenate to stearate, and 60% of monoethenoids which do not hydrogenate to methyl stearate. It has been suggested that these are cyclic monoethenoid isomers of linoleate. It should be mentioned that these results are on a monomer after rather extreme conditions of polymerization and that the formation of the monounsaturates is considerably less at earlier stages of polymerization.

Since the foregoing results indicated that the double bonds of normal linoleate are reactive dieneophiles, perhaps as active as those in conjugated linoleate at polymerization temperatures, it was of interest to determine whether the isolated double bond of oleate had any dieneophilic activity. Certain mixtures were polymerized, as shown in Table V.

TABLE V

Polymerizations at 300° of 50-50 Mixtures for 48 Hours

Components	D + T%	D/T
N + Ol.	42.7	6.1
N + St.	34.7	6.2
Ol. + St.	4.7	11.0
(Ol. alone)	7.0	20.0
(N alone)	80.3	3.4

Copolymerizations With Oleate. If oleate was completely inactive toward polymerization or copolymerization, we would expect the same amount of polymer from N + Ol. as from N + St. Actually 8.0% more is formed, which is $\frac{8.0}{42.7} = 18.7\%$ of polymer which is due to oleate polymer and copolymer. If correction is made for the 4.7% of polymer found in the Ol. + St. mixture, the amount of copolymer of Ol. + N (or C formed from N) is $42.7 - (34.7 + 4.7) = 3.3\%$ of copolymer in the mixture of $\frac{3.3}{42.7} = 7.7\%$ of copolymer in the polymer.

Similar mixtures involving conjugated linoleate were polymerized as shown in Table VI. There is formed 5.1% more polymer with oleate than with stearate as diluent, or $\frac{5.1}{39.0} = 13.1\%$ of total polymer which is due to oleate polymer and copolymer. This is of the same order as found for N + Ol. A greater amount of copolymer might have been expected in this case (C + Ol.) due to the high concentration of diene. However, the conditions of time and temperature were much less extreme than in case of N + Ol., and the competing reaction of conjugated diene with itself is very rapid.

TABLE VI

Polymerizations at 290° of 50-50 Mixtures for 12 Hours

Components	% D + T	D/T
C + Ol.	39.0	3.9
C + St.	33.9	3.4
(C alone)	80.9	2.9

While these demonstrations of the activity of oleate in thermal polymerization are based upon fairly small differences, they are considered accurate enough to indicate a significant polymerization activity of oleate which is of a very low order compared to normal or conjugated linoleate. Anderson and Porter (17) found that a small factor for oleate was necessary to correlate the composition of mixed glycerides with their bodying rates.

A Dimer of Pure Oleate. The low yield of dimer from pure methyl oleate is interesting. This dimer was refractionated in the alembic still to obtain a dimer fraction which distilled at essentially the same pot temperature (230° at 10 μ) as linoleate dimer. It showed the following characteristics:

I.V. = 46.1 (theory 42.8 for 1 double bond)
M.W. = 493,527 (theory 593) (ebul. in CCl_4)
$d^{30} = 0.9015$
$n_D^{30} = 1.4636$

Sap. eq. 355 (theory 296.5)
Neutr. eq. of acid 336 (theory 282.5)

The I.V. is fairly close to one double bond per mole of dimer and suggests a substitutive addition of two moles of oleate with loss of only one double bond. The low values for molecular weight and high values for sap. equiv. and neutr. equiv. may indicate partial decarboxylation, or addition of a pyrolysis product to methyl oleate. The quantity isolated was too small for structural studies.

Acknowledgments

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Summary

1. Debromination methyl linoleate has been polymerized at 290° and 300° for varying periods, and analysis has been made for monomer, dimer, trimer, normal, and conjugated linoleate.
2. The disappearance of normal linoleate follows a first order reaction rate with values of $K = 0.10$ hr.$^{-1}$ at 300° and 0.05 hr.$^{-1}$ at 290°.
3. Polymerization of mixtures of normal and conjugated linoleate indicate that dimer may be formed by their reaction with each other.
4. The value for K, the first order reaction velocity constant for disappearance of normal linoleate, decreases to a limiting value on dilution with methyl stearate. This limiting value is about one-fourth that obtained on undiluted linoleate.
5. The above facts are qualitatively explained by assuming that the mechanism of dimerization of normal linoleate is extensively:

   $N \longrightarrow C$ relatively slow
   $N + C \longrightarrow D$ relatively rapid.

   Other possible reactions by which normal linoleate disappears may be:

   $N \longrightarrow$ isolinoleate
   $N + \longrightarrow$ oleate or isooleate
   $N \longrightarrow$ cyclic monomer
   $N + D \longrightarrow$ trimer
   $N + N \longrightarrow$ dimer.

6. A slight but definite polymerization functionality has been demonstrated for oleate. A dimer of methyl oleate was prepared which apparently has one double bond.

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Certain Aspects of Food Standardization After Ten Years Under the New Food and Drug Law[1]

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PROBABLY no statute is more important to the health and welfare of the people of this country than the Federal Food, Drug, and Cosmetic Act, regulating as it does the manufacture and labeling of all articles that pass our lips, be it food or drugs, as well as all cosmetics. It became a law 10 years ago (June 25, 1938) and much has been written and said in review of its first decade (1).

Chemists, and especially oil chemists, have played an important part in connection with the enactment of this law, its enforcement, and the promulgation of important regulations issued under it. While many aspects of and experiences under the statute are of possible interest to a group of this nature, this paper is limited to one provision of the Act and certain problems and questions connected with it.

I refer to Section 401 of the Act, under which the Federal Security Administrator has the power to promulgate a definition and standard of identity for any food, and, once promulgated, such definition and standard of identity has the force and effect of law. For a better understanding of the meaning and effect of this statutory provision, permit me to furnish you with what I believe to be some necessary historical and legal background.

The predecessor act of the Federal Food, Drug, and Cosmetic Act was the Food and Drugs Act of 1906 which, with amendments that were added thereto, was in effect from 1906 until the Federal Food, Drug, and Cosmetic Act became effective (2). Under the 1906 Act the Secretary of Agriculture (3) had no legal power to standardize a food although as time went on the need for such a power was recognized by many. However the Food and Drug Administration did issue what it called "advisory standards." These had no legal effect but were adopted as a guide for officials in enforcing the Food and Drugs Act. Accordingly, they were of considerable interest to industry as well as government.

These advisory standards were usually quite simple and very basic. For instance, the standard for flour read:

> The fine-ground product obtained in the commercial milling of wheat, consisting essentially of the starch and gluten of the endosperm. It contains not more than 15% of moisture, not less than 1% of nitrogen, not more than 1% of ash, and not more than 0.5% of fiber.

Another example is the advisory standard for farina, which read:

> The purified middlings of hard wheat other than durum.

By contrast, the legal standards for these two products which have been promulgated under the Federal Food, Drug, and Cosmetic Act cover in minute detail the numerous ingredients of the products and are

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