

**Critical Solution Temperatures.** It can be observed from Figure 7 that the critical solution temperature increased with the water content of ethanol, and in each case the relationship was linear. Similar results have been reported for other oils (1, 2).

**Pressure in the System.** The maximum pressure, which was the result of both vapor and air pressure in the vessel, was recorded for different alcoholic compositions at various temperatures as shown below in p.s.i.g.

Concentration of alcoholic solution	Temperature °C.					
	50	60	70	80	90	95
95.4%.....	2	4	8	15	17	20
98.0%.....	2	4	8	15	18	
99.9%.....	2	4	10	15		

The maximum pressure to be used for alcoholic extraction of the oils is thus about 20 p.s.i.g. Similar data have been reported for other oils (1, 2) and

were determined for use in extraction-equipment design and operation rather than as vapor pressure values.

### Summary

Solubilities of babassu, coconut, olive, palm, rapeseed, and sunflower seed oils in aqueous alcoholic solutions at various temperatures were determined by a direct and simple method. Solubility curves for the six oils are presented.

The critical solution temperatures increase with the water content of the alcohol, and in each case the relationship is linear. The pressure in the system also varies with the temperature, the maximum being about 20 p.s.i.g.

### REFERENCES

1. Rao, R. K., Krishna, M. G., Zaheer, S. H., and Arnold, L. K., *J. Am. Oil Chemists' Soc.*, **32**, 420-423 (1955).
2. Rao, R. K., and Arnold, L. K., *J. Am. Oil Chemists' Soc.*, **33**, 82-84 (1956).

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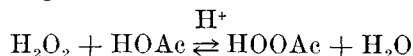
## Epoxy Fatty Acid Ester Plasticizers. Preparation and Properties<sup>1</sup>

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IN THE FEW SHORT YEARS since their introduction epoxy fatty acid ester plasticizers have achieved an important place in the vinyl plasticizer field. Based upon abundantly available and cheap raw materials, these plasticizers are produced by the epoxidation of unsaturated fatty acid esters.

### Preparation

Epoxidation reactions with peracids have been widely described in the literature (1-3, 15). The earliest method of preparation of epoxy esters utilized a preformed peracid, peracetic acid (6, 11, 17); the peracetic acid was prepared by reaction of hydrogen peroxide with acetic anhydride (16) or glacial acetic acid (9). The latter procedure in recent years has largely displaced the use of acetic anhydride for this purpose. Reaction of hydrogen peroxide with acetic acid is an equilibrium reaction:



All four components are present at equilibrium along with the strong acid employed to catalyze the reaction. The degree of conversion to the peracid is dependent upon the respective molar ratios and concentrations of acetic acid to hydrogen peroxide.

Epoxidations with a preformed peracid are generally run at relatively low temperatures, *i.e.*, below 25°C. A number of improvements have been made in this mode of epoxidation. A modified, preformed peracid procedure giving low iodine number epoxy end products has recently been described (11). This process utilizes a short high temperature reaction period, at the conclusion of a normal epoxidation run, to reduce residual unsaturation. Table I illustrates results of reactions with a preformed peracid with (modified Becco Process) and without (Standard Process) a final high temperature reaction period.

<sup>1</sup> Presented at the 28th fall meeting, American Oil Chemists' Society, October 1954, Minneapolis, Minn.

TABLE I  
Epoxidation of Unsaturated Esters with Preformed Peracetic Acid

Epoxy Ester	Standard Process <sup>a</sup> Oxirane Oxygen %	Iodine Value	Modified Becco Process <sup>b</sup> Oxirane Oxygen %	Iodine Value
Butyl Epoxystearate.	4.1	5.4	4.2	1.4
Epoxy Soybean Oil...	6.1	18.2	6.5	3.2

<sup>a</sup> A preformed peracetic acid (40%—1.04 moles) was reacted with the unsaturated ester (1.0 mole of ethylenic unsaturation) at 20°-25°C. for 4 to 5 hrs.

<sup>b</sup> A preformed peracetic acid (40%—1.04 moles) was reacted with the unsaturated ester (1.0 mole of ethylenic unsaturation) at 20°-25°C. for 3 to 4 hrs., completing the reaction at 50°-60°C. in an additional hour.

Although the use of a preformed peracetic acid for epoxidation is an excellent preparative method, considerable interest in recent years has been focussed on the development of *in-situ* epoxidation processes. The motivation has been essentially an economic one. When a preformed peracetic acid is employed for epoxidation purposes, the strong acid catalyst component of the peracid must be buffered to depress undesired ring opening of the epoxide. Accordingly, further formation of peracid during the reaction is negligible, and only the peracid component of the peracetic acid equilibrium mixture is effectively utilized. The hydrogen peroxide fraction (22% of the total active oxygen for the commercial 40% peracetic acid [4]) is largely wasted and discarded at the end of the reaction.

It was reasoned that if hydrogen peroxide were added to an aliphatic acid solution of an olefinic material, proper conditions could be found to effect formation of peracid accompanied by concurrent reaction of the peracid with the double bond to give the desired epoxide. Similar procedures have long been in use for hydroxylation reactions. Unfortunately this is complicated by the fact that the very conditions that favor peracid formation, notably the presence of a strong acid catalyst, high molar ratio of aliphatic acid to hydrogen peroxide, high tempera-

tures, etc., are all favorable to the ring opening of the desired epoxide.

The earliest reported *in-situ* epoxidation process was that of Niederhauser and Koroly (14), utilizing formic acid and hydrogen peroxide to effect a transition to an epoxide with a minimum of ring opening. The first *in-situ* epoxidation process using acetic acid was developed in our laboratories (7). The Becco *in-situ* epoxidation process employs a controlled molar ratio of acetic acid to hydrogen peroxide in the presence of a strong acid catalyst at what was hitherto regarded as excessively high temperatures, notably 60–70°C. The catalyst choice is flexible and may be sulfuric acid, an alkane sulfonic acid, or a sulfonic acid cation-exchange resin.

Performance of the Becco *in-situ* epoxidation process, when employed with model esters of monounsaturated fatty acids, is shown in Table II. Various catalytic systems are illustrated. The epoxidation procedure used was the same as that previously described (7). Table III illustrates the results obtained

TABLE II

In-Situ Epoxidation of Typical Monounsaturated Esters (Unsaturated esters [1.0 mole of ethylenic unsaturation] dissolved in benzene<sup>a</sup> were epoxidized with [1.1 mole] hydrogen peroxide [50%] and [0.5 mole] acetic acid in the presence of an acid catalyst<sup>b</sup> at 60–65°C.)

Epoxy Ester	Oxirane Oxygen %	Iodine Value	Reaction Time Hrs.
Propyl Epoxystearate <sup>c</sup> .....	3.8	7	12.0
Butyl Epoxystearate <sup>c</sup> .....	3.7	2	11.0
Butyl Epoxystearate <sup>c, d</sup> .....	3.7	2	3.5
Butyl Epoxystearate <sup>e</sup> .....	3.5	6	11.0
Butyl Epoxystearate <sup>f</sup> .....	3.8	2	6.0
Butyl Epoxystearate <sup>c, g</sup> .....	3.8	2	11.5

<sup>a</sup> 20%; calculated on the weight of ester.

<sup>b</sup> Catalyst concentration calculated on the combined weight of acetic acid + H<sub>2</sub>O<sub>2</sub>.

<sup>c</sup> 2% H<sub>2</sub>SO<sub>4</sub>.

<sup>d</sup> 90% hydrogen peroxide.

<sup>e</sup> 4% ethane sulfonic acid.

<sup>f</sup> Ion-exchange resin (25%), non-solvent.

<sup>g</sup> Low in polyunsaturates.

TABLE III

In-Situ Epoxidation of a Typical Polyunsaturated Ester (Soybean oil [1.0 mole of ethylenic unsaturation], dissolved in benzene<sup>a</sup> was epoxidized with [1.1 mole] hydrogen peroxide [50%] and [0.5 mole] acetic acid in the presence of an acid catalyst<sup>b</sup> at 60–65°C.)

Epoxy Ester	Oxirane Oxygen %	Iodine Value	Reaction Time Hrs.
Epoxy Soybean Oil <sup>c</sup> .....	5.8	9	12.0
Epoxy Soybean Oil <sup>c, d</sup> .....	6.1	6	4.0
Epoxy Soybean Oil <sup>e</sup> .....	5.9	5	13.0
Epoxy Soybean Oil <sup>f</sup> .....	6.5	6	6.0

<sup>a</sup> 20%, calculated on the weight of ester.

<sup>b</sup> Catalyst concentration calculated on the combined weight of acetic acid + H<sub>2</sub>O<sub>2</sub>.

<sup>c</sup> 2% H<sub>2</sub>SO<sub>4</sub>.

<sup>d</sup> 90% hydrogen peroxide.

<sup>e</sup> Ion-exchange resin (5%), non-solvent.

<sup>f</sup> Ion-exchange resin (25%), non-solvent.

with the Becco *in-situ* epoxidation process applied to typical polyunsaturated fatty acid esters, *e.g.*, soybean oil. The efficiency of conversion to epoxide is higher when ion-exchange resins are employed as the catalyst in the epoxidation of polyunsaturated acid esters whereas no marked difference appears in mono-unsaturated fatty acid esters. This performance is probably related to the greater degree of sensitivity toward ring opening of the epoxides of polyunsaturated fatty acid esters. Similar results have been observed with preformed peracids, where polyunsaturated fatty acid derivatives have generally given lower conversions to the corresponding epoxides than monounsaturated materials.

Table IV illustrates the influence of the temperature variable on the results of the Becco *in-situ* process. At temperatures of 25–30°C. the reaction is slow, and the amount of unsaturated material reacted is correspondingly low. At 60–65°C. excellent conversions to epoxide are achieved.

The influence of molar ratio on the percentage of conversion to epoxy esters is shown in Table V. It

TABLE IV

The Influence of Temperature on Reaction Efficiency (Butyl oleate [1.0 mole of ethylenic unsaturation] dissolved in benzene<sup>a</sup> was epoxidized with [1.1 mole] hydrogen peroxide [50%] and [0.5 mole] acetic acid in the presence of a 2% sulfuric acid catalyst.)<sup>b</sup>

Temperature Range °C.	Oxirane Oxygen %	Yield Epoxy Ester %	Iodine Value	Reacted %
25°–30°.....	2.3	51	33	55
35°–40° <sup>d</sup> .....	2.7	60	26	65
60°–65° <sup>d</sup> .....	3.6	80	5	93

<sup>a</sup> 20%, calculated on the weight of ester.

<sup>b</sup> Catalyst concentration calculated on the combined weight of acetic acid + H<sub>2</sub>O<sub>2</sub>.

<sup>c</sup> Reaction time, 24 hrs.

<sup>d</sup> Reaction time, 12 to 13 hrs.

TABLE V

Effect of Various Molar Ratios of Acetic Acid to Unsaturated Ester on Epoxy Ester Yield (Butyl oleate [1.0 mole of ethylenic unsaturation] dissolved in benzene<sup>a</sup> was epoxidized with [1.1 moles] hydrogen peroxide [50%] and acetic acid, in the presence of a 2% sulfuric acid catalyst,<sup>b</sup> at 55–65°C.)

Molar Ratio Acetic Acid/Unsaturated Ester <sup>c</sup>	Reaction Time Hrs.	Oxirane Oxygen %	Yield Epoxy Ester %	Iodine Value	Reacted %
15:1	3	0.0	0.0	4	95
1.7:1	8	0.2	4.4	8	89
0.5:1	13	3.6	80.0	5	93

<sup>a</sup> 20%, calculated on the weight of ester.

<sup>b</sup> Catalyst concentration calculated on the combined weight of acetic acid + H<sub>2</sub>O<sub>2</sub>.

<sup>c</sup> Moles of unsaturated ester refer to moles of ethylenic unsaturation present in ester.

will be noted that at high molar ratios of acetic acid, in the presence of a sulfuric acid catalyst, excellent reaction of the double bond is achieved, but comparatively all of the double bond converted to the epoxide undergoes secondary ring opening. When the ratio is reduced to 0.5 mole of acetic acid to 1 mole of ethylenic unsaturation, optimum conversions are realized.

The effect of known peracid formation catalysts (8) on epoxy ester yields is shown in Table VI. It will be noted that not all strong acid catalysts for peracid formation are satisfactory in this respect. Nitric acid and boron fluoride, for example, are not

TABLE VI

The Effect of Known Peracid Formation Catalysts on Epoxy Ester Yield (Butyl oleate [1.0 mole of ethylenic unsaturation], dissolved in benzene<sup>a</sup> was epoxidized with [1.1 moles] hydrogen peroxide [50%] and [0.5 mole] acetic acid in the presence of a 1% catalyst<sup>b</sup> at 60–65°C.)

Catalyst	Oxirane Oxygen %	Yield Epoxy Ester %	Iodine Value	Reacted %
H <sub>2</sub> SO <sub>4</sub> .....	3.6	79	6	92
HNO <sub>3</sub> .....	.....	.....	.....	53
BF <sub>3</sub> (C <sub>2</sub> H <sub>5</sub> ) <sub>2</sub> O.....	1.6	37	10	86
p-Toluene Sulfonic Acid.....	.....	.....	.....	57
Ethane Sulfonic Acid <sup>c</sup> .....	3.5	78	6	92
Ion-Exchange Resin <sup>c, d</sup> (Amberlite IR-120).....	3.8	84	4	94

<sup>a</sup> 20%, calculated on the weight of the ester.

<sup>b</sup> Catalyst concentration calculated on the combined weight of acetic acid + H<sub>2</sub>O<sub>2</sub>.

<sup>c</sup> 4% catalyst.

<sup>d</sup> Equivalent performance was obtained with strong cation-exchange resins, *e.g.*, Dowex 50X, Chempro C-20, and Permutit Q.

TABLE VII  
Comparative Evaluation of P.V.C. Milled Sheets Plasticized with Epoxy Fatty Acid Esters<sup>a</sup>

	Diocetyl-phthalate	Epoxy <sup>b</sup> Rice Oil	Epoxy <sup>b, d</sup> Menhaden Oil	Epoxy <sup>d</sup> Safflower Oil	Epoxy Acetylated Monoglycerides of:			
					Lard Oil	Oleic <sup>e</sup> Acid	Cottonseed Oil	Soybean <sup>e, d</sup> Oil
Mod. at 100%—Elg. at 25°C.....	1280	1340	1640	1530	1220	1490	1240	1490
Ult. Tensile p.s.i. at 25°C.....	2780	2230	2690	2600	2750	2900	2650	2890
Elongation, % at 25°C.....	370	280	300	320	370	350	360	340
Shore Hard., Dur. A. at 25°C.....	76	78	77	77	74	76	72	75
Efficiency, % <sup>c</sup>								
As per hardness.....	35	40	40	36	35	36	34	35
As per modulus.....	35	39	40	36	34	37	34	35
C. & B. Low Temp. Flex. °C. (5).....	-30	-35	-20	-22	-31	-31	-30	-27

<sup>a</sup> 65% resin, 35% plasticizer, 1.5% cadmium stearate stabilizer.

<sup>b</sup> 39% plasticizer.

<sup>c</sup> Hardness at 75 ± 2. Modulus at 100% elongation 1300 ± 100 p.s.i.

<sup>d</sup> Hardness at 75 ± 2. Modulus at 100% elongation 1500 ± 100 p.s.i.

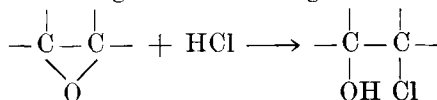
<sup>e</sup> Equilibrium mixtures.

very effective. Strong sulfur-containing acids are, in turn, comparatively efficient and interchangeable.

Catalyst choice in the Becco *in-situ* process will largely be dependent upon the nature of the raw material being employed and the processing facilities available.

### Properties

Epoxy fatty acid esters generally possess good over-all plasticizing properties and are likewise distinguished by a number of individual attributes all their own, *e.g.*, the epoxy group in the chain confers stabilizing properties on the polyvinyl chloride resin being plasticized. This is most important since polyvinyl chloride, like other chlorinated polymers, tends to degrade under heat and light action. The resultant dehydrochlorination gives rise to darkening and yellowing of the product. The epoxy group functions as a scavenger, picking up the hydrochloric acid released by the resin (as per the equation below) and in turn decreasing the rate of degradation.



Dependent upon the raw material being employed, other properties, such as low temperature flexibility and permanence, can be built into the epoxy fatty acid ester plasticizers.

Current practice employs epoxy fatty acid ester plasticizers as a partial replacement for primary plasticizers, such as dioctyl phthalate. Because of their stabilizing action, the amount of relatively expensive metallic stabilizer, normally used, is decreased approximately one-half. When end-use conditions are of a moderate nature, the metallic stabilizer may be omitted.

Epoxy plasticizers based upon monohydric and polyhydric alcoholic esters have been described (10). A number of additional epoxy esters of polyunsaturated fatty acids have been evaluated for plasticizer performance. These are shown in Table VII.

Acetylated monoglycerides have recently been shown to be secondary plasticizers for polyvinyl chloride (13). It was thought that the corresponding epoxy derivatives would be of interest as vinyl plasticizers. Several of these have been synthesized in our laboratories. Evaluations of these epoxy acetylated mono- and di-glycerides as polyvinyl chloride plasticizers are also illustrated in Table VII. Similar work has been described by Knight *et al.* (12). The epoxy acetylated glycerides have similarly been evaluated as polyvinyl acetate plasticizers for cast films and exhibited good over-all properties.

The analytical data for the above esters are given in Table VIII.

TABLE VIII  
Analytical Data for Epoxy Fatty Acid Esters

Epoxy Ester	Oxirane Oxygen %	Iodine Value
Epoxy Menhaden Oil.....	6.4	9.1
Epoxy Rice Oil.....	4.5	1.1
Epoxy Safflower Oil.....	6.9	4.5
Epoxy Acetylated Monoglycerides of		
Lard Oil.....	2.5	1.0
Oleic Acid <sup>a</sup> .....	3.3	0.6
Cottonseed Oil.....	3.7	7.0
Soybean Oil <sup>a</sup> .....	4.6	3.0

<sup>a</sup> Equilibrium mixtures.

In common with most epoxy plasticizers the heat and light stability of the aforementioned is very good, as may be noted from Table IX.

### Summary

The preparation of epoxy fatty acid ester plasticizers has been reviewed. These plasticizers can be prepared by epoxidation reactions, utilizing a preformed peracid or an *in-situ* technique. A new *in-situ* epoxidation process, utilizing hydrogen peroxide and acetic acid in the presence of strong acid catalyst, has been described. Excellent conversions to epoxide have been achieved with a series of monounsaturated and polyunsaturated esters.

Epoxy fatty acid esters are excellent plasticizers

TABLE IX  
Stability of Plasticized Films to Heat and Ultraviolet Light

	Heat Stability			Ultraviolet Light Stability 500 Hrs.
	Milled 155°C. 5 Min.	Pressed 165°C. 5 Min.	Ferro 177°C.	
Dioctyl Phthalate.....	Good	Good	Dark reddish brown after 1 ½ hrs. Light amber after 3 ½ hrs.	No discoloration, tack, or bleed. Very slight bleaching. No tack or bleed.
Epoxy Rice Oil.....	Good	Good	Very light amber after 3 ½ hrs.	Slight bleaching. No tack or bleed.
Epoxy Menhaden Oil.....	Good	Good	Very light amber after 3 ½ hrs.	No discoloration, tack, or bleed.
Epoxy Safflower Oil.....	Good	Good	Very light amber after 3 ½ hrs.	
Epoxy Acetylated Monoglycerides of:				
Lard Oil.....	Good	Good	Light amber after 3 ½ hrs.	No discoloration, tack, or bleed.
Oleic Acid <sup>a</sup> .....	Good	Good	Light amber after 3 ½ hrs.	Moderate bleaching. No tack or bleed.
Cottonseed Oil.....	Good	Good	Light amber after 3 ½ hrs.	No discoloration, tack, or bleed.
Soybean Oil <sup>a</sup> .....	Good	Good	Light amber after 3 ½ hrs.	No discoloration, tack, or bleed.

<sup>a</sup> Equilibrium mixtures.

for vinyl resins. Their performance in a series of additional epoxy plasticizers of vegetable and marine origin is described. Novel epoxy plasticizers based upon mixed acetylated mono- and di-glyceride have been prepared and are found to have excellent plasticizer performance.

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#### REFERENCES

1. Arbuzov, B. A., and Michailow, B. M., *J. prakt. Chem.*, **127**, 1 (1930).
2. Boeseken, J., and Schneider, G. C. C. C., *ibid.*, **131**, 285 (1931).
3. Boeseken, J., Smit, W. C., and Gaster, A., *Proc. Acad. Sci. Amsterdam*, **32**, 377 (1929).

4. Becco Chemical Division, F.M.C., Bull. 4, "Peracetic Acid 40%."
5. Clash, R. F. Jr., and Berg, R. M., *Ind. Eng. Chem.*, **34**, 1218-22 (1942).
6. Findlay, T. W., Swern, Daniel, and Scanlan, J. T., *J. Am. Chem. Soc.*, **67**, 412 (1945).
7. Gall, R. J., and Greenspan, F. P., *Ind. Eng. Chem.*, **47**, 147 (1955).
8. Greenspan, F. P., unpublished results.
9. Greenspan, F. P., *J. Am. Chem. Soc.*, **68**, 907 (1946).
10. Greenspan, F. P., and Gall, R. J., *Ind. Eng. Chem.*, **45**, 2722 (1953).
11. Greenspan, F. P., and Gall, R. J. (Becco), U. S. Patent 2,692,271 (1954).
12. Knight, H. B., Witnauer, L. P., Palm, W. E., Koos, R. E., and Swern, Daniel, paper presented at the American Chemical Society Meeting (1954).
13. Magne, F. C., and Mod, R. R., *J. Am. Oil Chemists' Soc.*, **30**, 269 (1953).
14. Niederhauser, W. D., and Koroly, J. E. (Rohm and Haas), U. S. Patent 2,485,160 (1949).
15. Prileschjew, N., *Ber.*, **42**, 4811 (1909).
16. Smit, W. C., *Rec. trav. chim.*, **49**, 675 (1930).
17. Terry, D. E., and Wheeler, D. H. (General Mills), U. S. Patent 2,458,484 (1949).

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## Evidence for Cyclic Monomers in Heated Linseed Oil<sup>1</sup>

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THE DISTILLABLE ETHYL ESTERS prepared from heated linseed oil have been shown by Wells and Common (24) to contain a fraction that fails to form an adduct with urea. Part of the deleterious effect of heated linseed oil on the nutrition of the rat (9, 11) is associated with this non-urea-adduct-forming distillable fraction (NAFD) (10).

Wells and Common (24) have suggested that the failure of NAFD to form an adduct with urea may be due to the presence of a non-terminal ring structure. The presence of cyclic monomers among the products of the heat treatment of glycerides and fatty esters has often been suggested (*e.g.*, 5, 21), but until recently the evidence for them has been indirect. Phthalic acid has been detected, by means of color reactions, among the products of the oxidation of heated linseed oil (7). Boelhouwer, Jol, and Waterman (4), employing a graphical-statistical method of ring analysis, showed that the monomeric products obtained from the heat treatment of linseed oil contain ring compounds. Mehta and Sharma (17) concluded that intra-acyl cyclization during the early stages of the heat-bodding of linseed oil is the chief cause of the initial rapid drop in iodine number without corresponding increase in molecular weight.

Recently Paschke and Wheeler (18) obtained direct chemical evidence for the existence of six-membered rings in the monomeric products of the heat treatment of methyl eleostearate. The heat treatment of various esters, and of sunflower seed oil, yields dimeric products which have been shown by Clingman, Rivett, and Sutton (8) to be cyclic. However no such chemical evidence has been obtained for cyclic compounds in the monomeric products from unconjugated esters or glycerides.

In the present investigation evidence for cyclic monomers in the NAFD of heated linseed oil has been obtained by methods involving aromatization *via* allylic bromination and dehydrohalogenation, followed by oxidation to a simple aromatic acid (8, 18).

#### Experimental<sup>2</sup>

*Preparation of NAFD.* Alkali-refined and bleached linseed oil was stripped at 170°C. in a centrifugal

molecular still<sup>3</sup> and then heated at  $275 \pm 3^\circ\text{C}$ . for 12 hr. A stream of purified nitrogen sufficient to maintain vigorous agitation was passed through the oil during the heating period. The heated oil had the following characteristics: kinematic viscosity (Fenske viscometer), 306 centistokes at 25°C.;  $n_D^{20}$ , 1.48533; iodine number (4 hr., Benham and Klee method [14, 15]), 163. It was converted to ethyl esters by alkali-catalyzed ethanolysis, and the esters were distilled in the centrifugal molecular still. Fifty per cent of the

<sup>3</sup> Model No. CMS-5, manufactured by Distillation Products Industries. The pressure in the system was one micron when distillation was not in progress.

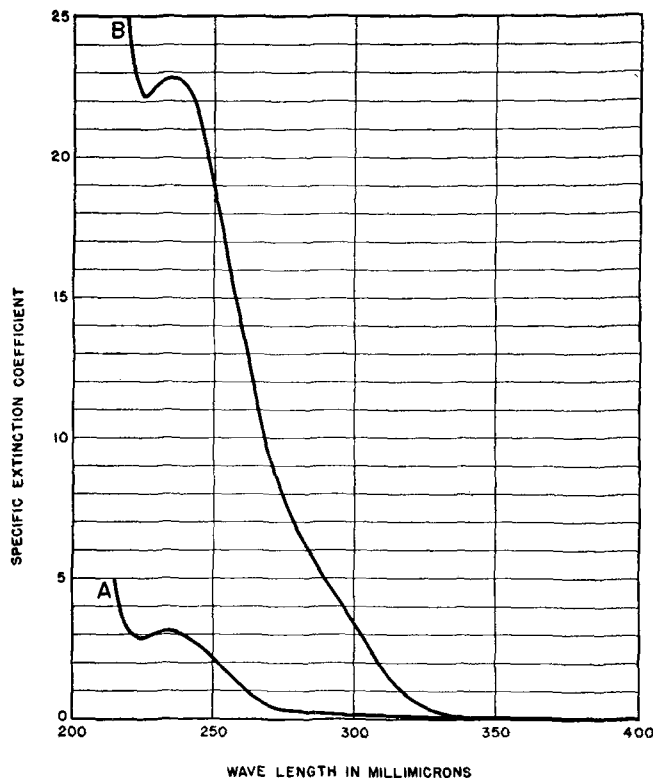


Fig. 1. Ultraviolet spectra of NAFD (Curve A) and aromatized NAFD (Curve B) in isooctane at concentrations of 0.347 and 0.404 g./l. respectively.

<sup>1</sup> Issued as N.R.C. No. 4001.

<sup>2</sup> Melting points are uncorrected.