

**B. DCC Evaluations.** Thin films were cast from a solution of the plasticizer and a resin in an appropriate solvent. These films were stacked in a Carver press and pressed to 0.05-in.-thick sheets. Tables III and IV present, respectively, results of evaluations of DCC in ethyl cellulose resin (Ethocel 20 cps, Dow) and in vinyl butyral resin (Butvar H. V., Monsanto). Dioctyl phthalate and dibutyl sebacate were used for comparison.

Table V summarizes the results of the evaluation of DCC in an acrylonitrile-butadiene rubber of the following formulation:<sup>1</sup>

	Parts by weight
Paracril 18.....	100
Zinc oxide.....	5
Stearic acid.....	1
Agerite resin D.....	60
Altax.....	1.5
Sulfur.....	1.5
Plasticizer.....	15

Dioctyl azelate was used for comparison.

<sup>1</sup>This evaluation was carried out through the courtesy of a Department of Defense testing facility.

## Summary

A number of ester-amides and diester-amides of aminoalcohols were prepared and evaluated as plasticizers. Screening tests established the necessity of a secondary amine group in the aminoalcohol to impart the desired degree of compatibility with vinyl resins.

Optimum plasticizer properties were attained with the C<sub>8</sub> and C<sub>10</sub> diester-amides of diethanolamine. The mixture of caprylic and capric acids as obtained from coconut oil was convenient and economical.

## Acknowledgment

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# Isolation of 2,4-Dodecadienoic Acid from the Seed Oil of *Sebastiania Lingustrina*

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**A**N INVESTIGATION of the oil of the seed of *Sebastiania lingustrina* by Hanks and Potts (1) revealed the presence in this oil of a chromophoric fatty acid of about 12 carbons chain length. The absorption spectrum of the oil was very similar to that of the oil of *Sapium sebiferum* (Stillingia or Chinese Tallow Seed oil) in which Hilditch (2) has found the chromophoric fatty acid to be 2,4-decadienoic acid. It was of interest therefore to isolate the chromophoric fatty acid from *Sebastiania lingustrina* and to identify it.

The method of obtaining the seed oil and its characteristics are given in an earlier publication (1). The oil was converted to methyl esters by direct alcoholysis, using sodium methylate. The esters were then distilled in a Todd fractionating column, and the fraction boiling between 86 and 90°C. at 1 to 2 mm. Hg. was collected. This was the first substance to distill and was clearly separated from the remaining high boiling esters.

The free acids obtained from this fraction had an equivalent weight of 193, indicating it to be composed largely of 12 carbon acids. The low-boiling, very fragrant methyl ester fraction was found to exhibit strong absorption in the ultraviolet region with a maximum of 2680 Å. The 1.007 g. of the methyl ester fraction took up 237 ml. of hydrogen. Assuming the average molecular weight of the methyl esters to be 207, the average unsaturation was 2.18 double bonds per molecule. The hydrogenated esters after saponification yielded acid which had an equivalent weight of 200.4 (lauric acid is 200.3).

The C<sub>12</sub> methyl ester fraction (240 mg.) was next subjected to displacement chromatography on a coupled filter column having a capacity of 30 ml., using 1:2 Darco G60 charcoal-Hyflo Supercel filter aid as adsorbent, 85% ethanol as solvent, and 1% ethyl erucate as displacer. Details of the technique are described elsewhere (3). The light absorption of the effluent was measured as it passed through a 0.2-mm. quartz cell in the Beckman spectrophotometer. Measurement was made at 2900 Å, representing a position to the side of the maximum such that the density throughout the experiment remained within the scale of the instrument. The portion of the eluate shown between the two arrows in Figure 1 was kept, and the

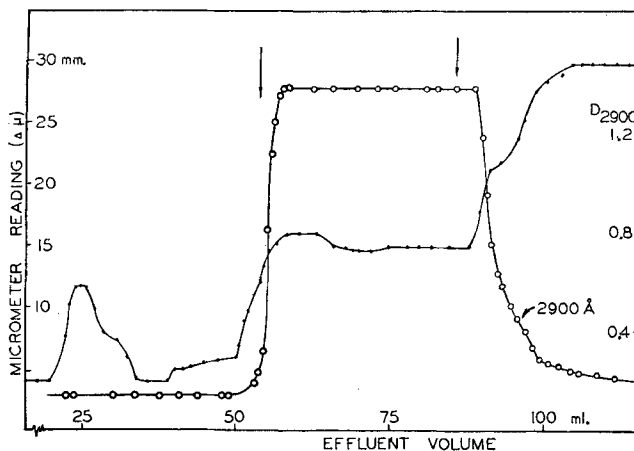


FIG. 1. Displacement diagram for chromatographic isolation of 2,4-dodecadienoic acid.

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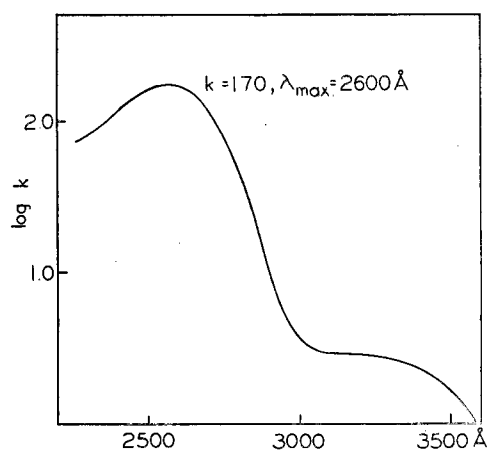


FIG. 2. Ultraviolet absorption spectrum of 2,4-dodecadienoic acid.

solvent was removed. The methyl ester was converted to the acid, and its equivalent weight was found to be 197.4. Theoretical value for 2,4-dodecadienoic acid is 196.3. The quantitative ultraviolet absorption spectrum of the purified acid is shown in Figure 2. The shape of the absorption curve and the position of its maximum are very similar to that of sorbic acid, indicating that the unsaturation is carboxyl conjugated (4, 5). It is concluded therefore that the unsaturated chromophoric fatty acid isolated is 2,4-dodecadienoic acid. To the authors' knowledge no previous report of this compound has been made.

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## Low Temperature Solubilities of Fatty Acids In Selected Organic Solvents<sup>1, 2</sup>

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SINCE THE APPEARANCE in 1937 of the first papers (1, 2) of a series by Brown and coworkers dealing with the separation of fatty acid mixtures by fractional crystallization at low temperatures, this separation technique has found wide application in the field of fat chemistry. It has proved particularly useful for separating unsaturated acids from acids with greater or lesser degrees of unsaturation and for preparing individual unsaturated fatty acids of high purity. However the utility of the method has been somewhat limited by the fact that so little information is available relating to the solubility behavior of fatty acids at low temperatures. The purpose of this investigation has been to determine the solubilities of a number of fatty acids, most of them unsaturated, in various organic solvents and within the range of temperatures obtainable with dry ice.

Whereas there has been extensive work on fatty acid solubilities at temperatures above 0°C. (saturated acids only), there have been only three quantitative investigations carried out at low temperatures. The first was that of Foreman and Brown (3), who reported the solubilities of six saturated and five unsaturated acids in methanol, acetone, and Skellysolve B. Singleton's investigations (4) described solubilities involving three-component systems. His study was limited to solutions of oleic acid with palmitic or stearic acid in acetone or hexane. More recently Hoerr and Harwood (5) have made an intensive study of the solubilities of oleic and linoleic acids in a large number of organic solvents. The latter work was not published until the present investigation was almost

completed, and the two studies tend to overlap to some extent.

The present report is essentially an extension of the work of Foreman and Brown (3). The same type of procedure for measuring solubilities has been used except that an effort has been made here to assure the establishment of equilibrium. In addition, more attention has been given to the unsaturated acids, and more solvents have been included. The acids include nine naturally occurring fatty acids plus three *trans* unsaturated compounds and an acetylenic acid. The six solvents have been chosen so that they represent different types of organic solvents, *i.e.*, an ether, an ester, an alcohol, a ketone, an aromatic hydrocarbon, and an aliphatic hydrocarbon.

#### Experimental

**Preparation of Fatty Acids.** Oleic acid was prepared from olive oil by a combination of the methods of fractional distillation and low temperature crystallization, essentially as described by Foreman and Brown (3). The following acids were prepared by an appropriate modification of the above method: erucic acid from rapeseed oil, eicosenoic acid from rapeseed oil, petroselinic acid from parsley seed oil, and palmitic acid from palm oil. Stearic acid was obtained by purification of Hystrene 97-S stearic acid, supplied by the Atlas Powder Company. The material was esterified, and the methyl esters were fractionally distilled. The C<sub>18</sub> cut was acidified, converted to the free acids, washed three times with hot water, and then recrystallized several times from petroleum ether. Arachidic and behenic acids were prepared by catalytic hydrogenation of eicosenoic and erucic acids followed by recrystallization. Elaidic, petroselaidic, and brassidic acids were obtained by isomerization of oleic, petroselinic, and erucic acids, respectively, by heating at 175-185° with 1% of powdered selenium.

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