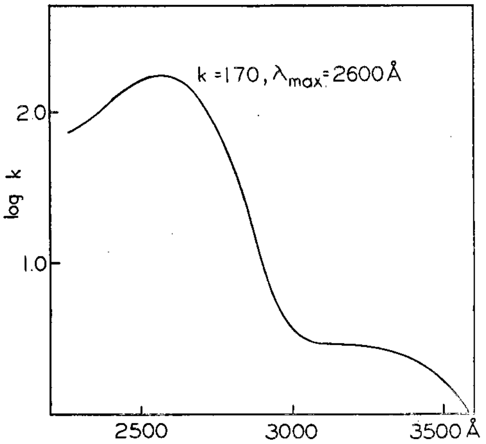

FIG. 2. Ultraviolet absorption spectrum of 2,4-dodecadienoic acid.

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[1, 2]

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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_{18} 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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[1] From a dissertation submitted by Doris Kolb to The Ohio State University in partial fulfillment of the requirements for the Ph.D. degree, August, 1953.

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TABLE I

Analytical Constants of the Fatty Acids

Acid	Iodine Value		Neut. Equiv.		Melting Point	
	obs.	theor.	obs.	theor.	obs.	ref. value
Palmitic	0.0	....	256.3	256.42	62.8°	62.9° (10)
Stearic	0.0	....	284.5	284.47	69.5°	69.6° (10)
Arachidic	0.0	....	313.0	312.52	74.7°	75.35° (10)
Behenic	0.0	....	340.3	340.57	80.0°	79.95° (10)
Oleic	89.9	89.87	282.5	282.45	13.3°	13.36° (11)
Elaidic	89.85	89.87	282.5	282.45	43.6°	43.68° (12)
Petroselinic	88.73	89.87	281.7	282.45	30–30.2°	30° (12)
Petroselaidic	89.25	89.87	282.0	282.45	53.0°	53° (12)
Eicosenoic	81.7	81.75	310.8	310.5	22.0°	22° (13)
Erucic	75.0	74.98	338.5	338.56	33.5°	33.4° (12)
Brassidic	74.97	74.98	338.6	338.56	60.0°	60° (12)
Linoleic	181.0	181.03	280.4	280.44	—5.2°	—5.2–5.0° (6)
Stearolic	89.5	181.03[a]	280.0	280.44	46.2–46.5°	48° (14)

[a] The observed iodine value is actually one-half the theoretical value since only one molecule of iodine chloride is added to the triple bond.

Linoleic acid was prepared by a modification of the method of Matthews, Brode, and Brown (6). Debromination linoleic acid, obtained from The Hormel Institute, was recrystallized seven times from petroleum ether at —62°, thus raising the melting point from —10.6° to —5.2°; the product was shown to be pure as determined by the tetrabromide yield (7).

Stearolic acid was prepared as described by Khan *et al.* (8).

Analytical Criteria of Purity. Neutral equivalents were determined by dissolving 1-3 g. of the fatty acid in neutral ethanol containing phenolphthalein, then titrating the sample against 0.1 N sodium hydroxide using a microburet. Iodine values were obtained according to the official Wijs 1-hr. method (9). Melting points were determined, using both the capillary method and the method of heating and cooling curves.

Table I lists the analytical constants of the fatty acids used in this investigation.

Purification of Solvents. All solvents were reagent grade materials and were redistilled before use. Prior to distillation some of the solvents were treated with suitable drying agents. Toluene and diethyl ether were each treated with metallic sodium; acetone and ethyl acetate were dried over anhydrous potassium carbonate, and anhydrous methanol was prepared by treating C. P. methanol with magnesium and iodine. The hydrocarbon solvents were 99 mol % materials obtained from the Phillips Petroleum Company and were distilled before use.

Constant Low Temperature Apparatus. The apparatus designed to maintain constant temperatures down to —75°, described previously by Foreman and Brown (3), was used in the present investigation with only slight modification. It consisted essentially of two monel metal compartments, one the working chamber and the other a cooling unit, filled with a mixture of methanol and ethanol and cooled by means of dry ice.

Six 250-ml. Erlenmeyer flasks containing solvent plus excess of the fatty acid being studied were placed in the working chamber of the bath. In the first part of our work the mixtures were stirred individually with mercury-sealed electric stirrers. Later the equilibration procedure was simplified by the installation of a mechanical rocker powered by a continuous-duty speed reducer motor. The flasks were rocked at 30 cycles per minute. After the rocker was installed, the bath could be covered completely with a sheet of plexiglas, thus greatly reducing the tendency for water and ice crystals to form on the flasks and other exposed surfaces.

Attainment of Equilibrium. Samples were not withdrawn for analysis until the solutions had been given sufficient time to reach equilibrium. Test data for several fatty acid-solvent systems taken at time intervals during the approach to equilibrium are given in Table II. The test temperature was approached first from room temperature and then from the temperature of a dry ice-cooled bath. The flasks were placed in the bath at —30° and stirred rapidly for about 5 hrs. Samples 1 and 2 were taken at 5-hr. intervals. The flasks were then allowed to remain (unstirred) at —30° overnight. On the following day stirring was resumed, and samples 3 and 4 were withdrawn at successive 5-hr. intervals. The data indicate that equilibrium had been attained for these representative systems within a 2-day period. They also demonstrate that equilibrium tends to be reached more rapidly when it is approached from the cold side, presumably because of the tendency of fatty acids to form supersaturated solutions. Thus in most subsequent work the flasks were cooled (by immersion in the cooling unit) before being placed in the constant temperature chamber. The first sample was usually removed after 1½-2 days in the bath, and then a second sample was withdrawn on the following day. Agreement of results for the two samples was taken as evidence that equilibrium had been attained. The temperature of the bath was then lowered 10°, and a second set of solubility data were taken on the same acid-solvent mixtures. This process was repeated until the desired temperature range was covered.

TABLE II

Test Data Showing Change of Solubility with Time
(Solubilities in grams acid per 100 g. solution)

Acid-Solvent Mixture	Temperature, —30° approached from	Sample No.			
		1	2	3	4
Oleic acid— Acetone	Warm side	1.74	1.70	1.68	1.68
	Cold side	1.62	1.68	1.68	1.68
Erucic acid— Ethyl acetate	Warm side	0.203	0.160	0.121	0.112
	Cold side	0.095	0.110	0.113	0.111
Petroselinic acid— n-Heptane	Warm side	0.063	0.051	0.041	0.040
	Cold side	0.037	0.039	0.040	0.040
Elaidic acid— Methanol	Warm side	0.077	0.065	0.064	0.063
	Cold side	0.061	0.064	0.065	0.064
Stearic acid— Diethyl ether	Warm side	0.055	0.053	0.050	0.051
	Cold side	0.051	0.051	0.052	0.051

Withdrawal and Analysis of Samples. Samples were removed from the flasks in the bath by means of a sintered glass inverted filter and a special withdrawal pipette. (See Figure 1.) A sample of satu-

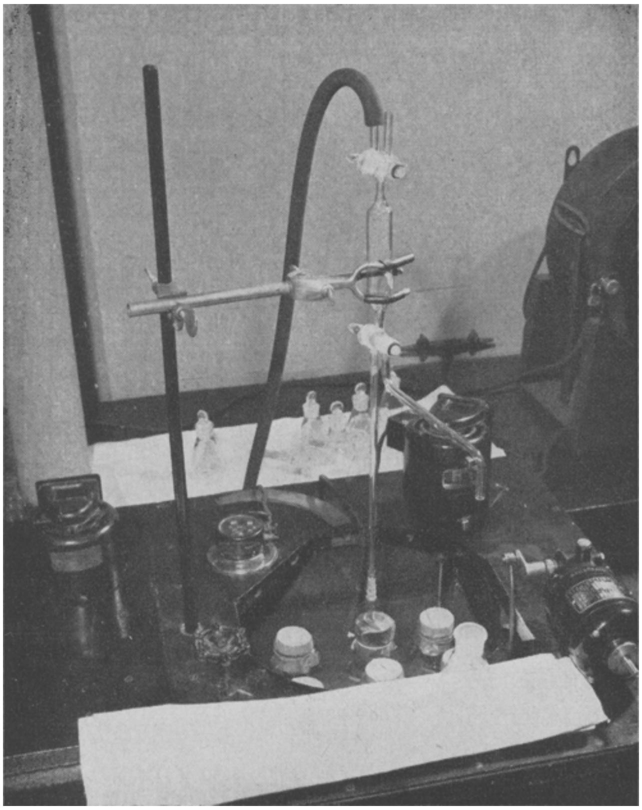

FIG. 1. Constant temperature apparatus with special pipette for withdrawing specimens.

rated liquid was drawn up into the bulb of the pipette by gentle suction and, by proper manipulation of the two-way stopcocks, allowed to flow into a tared 125-ml. glass-stoppered flask. The flask was allowed to reach room temperature and weighed to the nearest 0.01 g. The solvent was then evaporated, and the quantity of acid residue was determined both gravimetrically and by titration against standardized sodium hydroxide. Because solvent removal was often incomplete, the gravimetric results were not so dependable as those obtained volumetrically and were used mainly to check the magnitude of the volumetric results.

Results and Discussion

Some of our results are presented in Table III. These data are important primarily as physical constants which serve to extend our knowledge of the physico-chemical properties of an important class of long-chain organic compounds.

If each fatty acid in a mixture dissolved independently and if there were no mutual solubility effects, it would be possible, by use of the data presented here, to separate these compounds very readily by crystallization. The values given here however indicate only simple solubilities of pure fatty acids. No information is given as to how these same acids behave when they are present in mixtures. It is, in fact, well known that fatty acids do show mutual solubility effects, but the degree to which they influence one another varies greatly, depending both upon the solvent and upon the nature of the fatty acid mixture. Singleton (4) showed, for example, that oleic acid has a rather large solubilizing effect upon palmitic or stearic acid but that the saturated acids have relatively little influence on the solubility of oleic acid.

TABLE III

Solubility Data on Fatty Acids in Various Solvents
(All values in grams acid per 100 g. solution)

Temp.	Solvent					
	Methanol	Ethyl Acetate	Diethyl Ether	Acetone	Toluene	Normal Heptane
			Stearic Acid			
10°	0.26	0.58	2.40	0.54	0.390	0.080
0°	0.090	0.13	0.95	0.11	0.080	0.018
−10°	0.031	0.027	0.38	0.023	0.015	0.004
−20°	0.011	0.006	0.15	0.005	0.003	......
−30°	......	......	0.051	......	......	......
			Oleic Acid			
−20°	4.02	5.95	......	5.20	......	2.25
−30°	0.86	1.90	......	1.68	3.12	0.66
−40°	0.29	0.62	5.15	0.53	0.96	0.19
−50°	0.10	0.20	1.80	0.17	0.28	0.050
−60°	0.03	0.057	0.61	0.055	0.075	0.011
−70°	......	......	0.21	......	......	......
			Elaidic Acid			
0°	......	......	......	......	......	0.59
−10°	0.48	......	......	......	0.86	0.19
−20°	0.18	0.29	1.40	0.26	0.20	0.060
−30°	0.064	0.10	0.60	0.092	0.056	0.019
−40°	0.020	0.027	0.23	0.029	0.013	0.007
−50°	0.010	0.008	0.10	0.009	......	......
			Linoleic Acid			
−50°	3.10	4.40	......	4.10	......	0.98
−60°	0.90	1.38	......	1.20	......	0.20
−70°	0.25	0.39	......	0.35	......	0.042
			Arachidic Acid			
10°	0.080	0.14	0.90	0.13	0.12	0.028
0°	0.028	0.036	0.38	0.035	0.026	0.005
			Eicosenoic Acid			
−20°	0.80	1.30	......	1.10	......	......
−30°	0.35	0.60	3.90	0.54	1.10	0.45
−40°	0.15	0.26	1.70	0.27	0.30	0.15
−50°	0.06	0.11	0.68	0.12	0.07	0.048
−60°	0.02	0.04	0.22	0.05	......	0.01
			Petroselinic Acid			
−10°	......	......	......	......	......	0.50
−20°	0.48	0.73	3.52	0.78	0.98	0.13
−30°	0.18	0.30	1.68	0.31	0.23	0.040
−40°	0.060	0.11	0.76	0.11	0.046	0.009
−50°	0.018	0.040	0.31	0.035	0.008	......
−60°	......	......	......	......	......	......
			Petroselaidic Acid			
0°	0.50	......	......	......	1.40	0.22
−10°	0.20	0.36	......	0.32	0.19	0.030
−20°	0.082	0.14	......	0.13	0.060	0.008
−30°	0.028	0.050	0.51	0.050	0.020	0.002
−40°	0.010	0.018	0.19	0.019	0.008	......
−50°	......	......	0.070	......	......	......
			Stearolic Acid			
0°	......	......	......	......	......	......
−10°	0.65	1.15	4.85	1.26	1.70	0.050
−20°	0.25	0.45	2.00	0.52	0.40	0.010
−30°	0.103	0.17	0.78	0.19	0.083	0.006
−40°	0.040	0.065	0.32	0.070	0.018	......
			Palmitic Acid			
10°	1.30	......	......	1.60	1.41	0.30
0°	0.46	0.52	2.95	0.66	0.36	0.08
−10°	0.16	0.18	1.35	0.27	0.086	0.02
−20°	0.050	0.060	0.56	0.10	0.018	0.005
−30°	......	0.018	0.21	0.038	......	......
			Behenic Acid			
10°	0.019	0.055	0.48	0.050	0.040	0.012
0°	0.007	0.016	0.18	0.014	0.010	0.002
−10°	0.002	0.004	0.068	0.004	0.002	......
			Erucic Acid			
−10°	0.49	......	......	......	......	0.35
−20°	0.19	0.31	......	0.28	0.68	0.11
−30°	0.068	0.11	1.20	0.10	0.16	0.030
−40°	0.024	0.040	0.49	0.037	0.044	0.008
−50°	0.007	......	0.18	......	......	......
			Brassidic Acid			
10°	0.27	0.70	......	0.68	0.62	0.20
0°	0.094	0.26	......	0.24	0.18	0.058
−10°	0.035	0.096	0.78	0.065	0.050	0.016
−20°	0.010	0.028	0.28	0.024	0.013	0.005
−30°	0.003	0.01	0.10	......	......	......

Although it must be kept in mind that such mutual solubility effects do exist, the solubility curves for the pure acids are nevertheless valuable as guides for determining optimum conditions for attempting

TABLE IV

Fatty Acid Solubilities in Various Hydrocarbon Solvents
(All values expressed in grams acid/100 g. solution)

Stearic Acid

Temp.	n-Heptane	Methyl Cyclohexane	n-Pentane	Isopentane
10°	0.080	0.20	0.089	0.096
0°	0.018	0.06	0.015	0.040
—10°	0.004	0.021	0.003	0.017
—20°	......	0.005	......	......

Temp.	Diisopropyl	Neohexane	2-Methyl pentane	Isooctane
10°	0.070	0.04	0.086	0.051
0°	0.015	0.010	0.03	0.020
—10°	0.004	0.003	0.01	0.008
—20°	......	......	0.003	0.003

Oleic Acid

Temp.	n-Heptane	Methyl Cyclohexane	Diisopropyl	Isooctane	Neohexane	2-Methyl pentane
—40°	0.19	0.34	0.212	0.162	0.13	0.19
—50°	0.050	0.11	0.112	0.070	0.050	0.08

Linoleic Acid

Temp.	n-Heptane	Methyl Cyclohexane	Diisopropyl
—50°	0.98	2.06	0.94
—60°	0.20	0.38	0.17
—70°	0.042	0.072	0.032

the separation of given fatty acid mixtures, or for deciding whether or not low temperature crystallization is at all feasible for separating a particular mixture.

The general trends in fatty acid solubility behavior indicated by the data in Table III are generally what would be expected. Solubility is enhanced by decrease in chain length and by unsaturation. The introduction of a double bond greatly increases solubility, the increase being greater the farther the point of unsaturation is removed from the carboxyl group. Changing an acid with a *cis* double bond to its *trans* isomer reduces its solubility considerably, as does converting its olefinic function to a triple bond.

The solubilities listed for oleic acid refer in each case to the more stable form of the acid in that system. The dimorphic character of oleic acid causes it to exhibit two solubility curves in some solvents. With the "synthetic" technique of solubility measurement used by the Armour group, it has been possible to observe portions of the unstable curves in some solvents (5). However the analytical method followed in the present study involved long waiting periods to permit the establishment of equilibrium, and, since the transformation from the unstable to the stable form occurs rather rapidly, the oleic acid data listed here can be assumed to refer to the stable modification of the acid in all cases.

The postulation by Ralston and Hoerr (15) and by Bailey (16) that solubility is closely linked with melting point is supported by these data, at least in a qualitative sense. In general, the order of the solubility curves for the various acids is the same for all six of the solvents studied; however one peculiar reversal was noted. The solubility of stearolic acid appears to change relative to erucic and elaidic acids in the following manner; in acetone, methanol, and ethyl acetate, stearolic > erucic > elaidic; in ether and toluene, erucic > stearolic > elaidic; and in n-heptane, erucic > elaidic > stearolic. On the basis of comparative melting points alone, stearolic acid would be expected to behave in all solvents as it does in n-heptane; that is, stearolic acid (m.p. 46.5°) should be less soluble than elaidic acid (m.p. 43.7°), which is in turn less soluble than erucic acid (m.p. 33.5°). The proportionately greater enhancement of solubility of stearolic acid by polar solvents (as compared

TABLE V

Comparison of Solubilities with Data Previously Reported by Other Investigators
(All solubilities expressed in grams acid per 100 g. solution)

	10°	0°	—10°	—20°
Stearic Acid				
Methanol	0.26 0.259[F]	0.090 0.092[F]	0.031 0.032[F]	0.011 0.010[F]
Acetone	0.54 0.469[F] 0.79[R]	0.11 0.219[F] 0.21[R] 0.103[S]	0.023 0.038[F] 0.021[S]	0.005 0.004[S] ....

	—20°	30°	—40°	—50°	—60°
Oleic Acid					
Methanol	4.02 3.9[H]	0.86 0.708[F] 0.9[H]	0.29 0.329[F] 0.3[H]	0.10 0.089[F]	0.03 0.051[F] ......
Ethyl Acetate	5.95 10.9[H]	1.90 4.3[H]	0.62 1.6[H]	....	....
Acetone	5.20 5.60[S] 4.9[H]	1.68 1.42[F] 2.05[S] 1.4[H]	0.53 0.516[F] 0.66[S] 0.5[H]	0.17 0.189[F]	0.055 0.061[F] ....
Diethyl Ether	....	....	5.5 4.37[F] 1.2[H]	....	....

	—20°	—30°	—40°
Eicosenic Acid			
Methanol	0.80 1.06[F]	0.35 0.348[F]	0.15 0.129[F]
Acetone	1.10 1.61[F]	0.54 0.458[F]	0.27 0.147[F]

	10°	0°	—10°	—20°	—30°
Palmitic Acid					
Methanol	1.30 1.31[F] 1.3[R]	0.46 0.396[F] 0.8[R]	0.16 0.146[F]	0.050 0.063[F]	....
Ethyl Acetate	....	0.52 0.8[R]	....	....	....
Acetone	1.60 1.77[F] 1.90[R]	0.66 0.715[F] 0.60[R] 0.65[S]	0.27 0.280[F] 0.289[S]	0.10 0.134[F] 0.094[S]	0.038 0.048[F] 0.035[S] ......

	—50°	—60°	—70°
Linoleic Acid			
Methanol	3.10 2.52[F] 3.2[H]	0.90 0.925[F]	0.25 0.394[F] ......
Ethyl Acetate	4.40 5.3[H]	....	....
Acetone	4.10 4.82[F] 3.2[H]	1.20 1.42[F]	0.35 0.519[F] ......

	—20°	—30°
Erucic Acid		
Methanol	0.19 0.176[F]	0.068 0.087[F]
Acetone	0.28 0.352[F]	....

	10°	0°
Arachidic Acid		
Methanol	0.080 0.096[F]	0.028 0.065[F]
Acetone	0.13 0.183[F]	0.035 0.075[F]

	10°	0°
Behenic Acid		
Methanol	0.019 0.042[F]	0.007 0.010[F]
Acetone	0.050 0.051[F]	0.014 0.010[F]

[F] Data of Foreman and Brown (3).
[S] Data of Singleton (4).
[H] Data of Hoerr and Harwood (5).
[R] Data of Ralston and Hoerr (17, 18).

with erucic and elaidic acids) suggests that stearolic acid is somewhat more polar than the other two compounds.

Table IV gives solubility data for stearic, oleic, and linoleic acids in certain hydrocarbon solvents. The differences in solvent properties of the various hydrocarbons were not great, but solubilities were significantly higher in methyl cyclohexane than in the other solvents and were lowest in neohexane.

The solubility of stearic acid was also studied in dimethyl formamide. The data obtained were as follows:

Temp.	Solubility (g./100 g. solution)
10°	1.15
0°	0.38
−10°	0.102
−20°	0.024

The solubilities are higher than for most of the other solvents. Dimethyl formamide was not further studied because of its high toxicity and its low volatility, which made solvent removal difficult. It was not considered a convenient medium for use in the low temperature crystallization procedure.

For purpose of comparison Table V has been prepared. This table compares certain solubility determinations made during this study with values which have been reported by other investigators. In general, the agreement is good. However several discrepancies are apparent. The solubilities in ethyl acetate, for example, tend to be considerably lower than those of Hoerr and Harwood (5). The value listed for diethyl ether, on the other hand, is significantly higher than that of these investigators.

Attempts were made to prepare pure linolenic acid, but a product of sufficient purity for solubility measurements was not obtained in time to be included in this study. This is unfortunate since linolenic is one of the most important of the unsaturated fatty acids. Solubility studies with mixtures of linoleic and linolenic acids would also be of great practical value as our work has indicated that these compounds exhibit marked mutual solubility effects.

Summary

A number of highly purified fatty acids have been prepared and their solubilities determined in six common organic solvents within the temperature range from 10° to −70°. The acids studied were palmitic, stearic, oleic, elaidic, petroselinic, petroselaidic, linoleic, stearolic, arachidic, eicosenoic, behenic, erucic, and brassidic. The solvents used were methanol, ethyl acetate, diethyl ether, acetone, toluene, and n-heptane, representing six different solvent types. A limited study was also made with a series of hydrocarbon solvents in order to note any effects of solvent structure on fatty acid solubility. Data are discussed with respect to their application in separating various fatty acid mixtures by low temperature crystallization.

Acknowledgment

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Filtration-Extraction of Peanuts on a Bench Scale[1]

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IN THE UNITED STATES in 1952, from the 685,000 tons of peanuts produced, approximately 100,000 tons were processed to yield oil and meal products (13, 14). Most of this processing was hydraulic and screw pressing with only an insignificant portion processed by solvent extraction. Should the cost of peanut production in the United States be reduced to a level where peanut oil and meal can compete with corresponding products of cottonseed and soybeans, the amounts of peanuts grown and processed would probably increase substantially.

Peanut oil is considered one of the better quality vegetable oils and sells at a premium price. The solvent-extracted meal, besides being a cattle feed, is an excellent source of industrial and edible protein (1). The removal of oil by solvent extraction from an oilseed of high fat content, such as peanuts, poses many technical problems (5). As a solution to some of these problems prepressing is currently used prior to solvent extraction in some instances. A new direct solvent-extraction process developed at this Laboratory and called Filtration-Extraction makes prepressing unnecessary. The filtration-extraction process has been applied on a pilot plant scale to cot-

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