

moto (12) reported no remarkable change in thiamin up to 13 days of germination. At most it appears that alternating minor gains and losses of thiamin occur during germination. Ascorbic acid was absent in ungerminated beans but appeared within the first 24 hrs. of germination and reached a value of 390 micrograms per gram at the end of three days. It was found in the reduced form only (14). Previous investigators (3, 13, 15) have reported that ungerminated soybeans contained ascorbic acid and that the original value increased several-fold during the first three to five days of germination. Previous reports (3, 13) also show varying amounts of ascorbic acid present in the dehydro form. A report (14) of the modified indophenol-xylene extraction method used in the present study to estimate ascorbic acid showed that the analytical methods used by previous investigators gave high values for ascorbic acid because of interference by sulfhydryl groups. It was also shown that the ascorbic acid values reported for ungerminated soybeans and the values reported for dehydro-ascorbic acid resulted from interference associated with sulfhydryl group activity.

From several excellent studies (3, 7, 13, 15) on the increase in the vitamin content of soybeans on germination it appears that a substantial increase can be expected in two to three days. Since the over-all composition of soybeans does not change appreciably during the first 72 hrs. of germination, it appears that sprouted beans could be used as a basis for producing a high-energy, high-protein broiler feed. Hulls are easily removed from sprouted soybeans because they become loose and detached on drying. This property would facilitate removal of undesired fiber. However grinding followed by screening would have to be used because aspiration would probably remove the dried sprouts.

Summary

Changes in nitrogen and oil contents and loss in dry weight have been followed during the first six days of germination of the Hawkeye variety of soy-

beans; thiamin and ascorbic acid content have been followed for four days. During the first three days about 1.5% of the dry matter was lost; the nonprotein nitrogen increased from 3 to 6% of the total nitrogen, with no change in the petroleum ether extractables and with a decrease in free fatty acids. At the end of six days, with sprouts about 2½ in. long, only 2.6% of the dry matter was lost, the nonprotein nitrogen had increased to 13% of the total nitrogen with 2.6% loss of total nitrogen, and a 12% loss was observed in petroleum ether extractables; the free fatty acids did not increase appreciably. No change in the thiamin content occurred during the first four days of germination. Ascorbic acid was found to be absent in mature beans but appeared after the start of germination and increased rapidly during the four-day period.

An analysis of these results and of those from the literature indicates that soybeans sprouted for two to three days have possibilities for use in high-energy, high-protein broiler feed.

REFERENCES

1. Becker, H. C., Milner, R. T., and Nagel, R. H., *Cereal Chem.*, **17**, 447-457 (1940).
2. Block, R. J., and Bolling, D., *Amer. Diet. Assoc. J.*, **20**, 69-76 (1944).
3. Burkholder, P. R., and McVeigh, I., *Plant Physiol.*, **20**, 301-306 (1945).
4. Crocker, W., and Barton, L. V., "Physiology of Seeds," p. 173, Waltham, Mass., Chronica Botanica Company, 1953.
5. Dunn, M. S., Camein, M. N., Shankman, S., and Block, H., *Arch. Biochem.*, **18**, 195-200 (1948).
6. Everson, G. J., Steenbock, H., Cederquist, D. C., and Parsons, H. T., *J. Nutr.*, **27**, 225-229 (1944).
7. French, C. E., Berryman, G. H., Goorley, J. T., Harper, H. A., Harkness, D. M., and Thacker, E. J., *J. Nutr.*, **28**, 63-70 (1944).
8. Hill, J. B., Overholts, L. O., and Popp, H. W., "Botany," p. 311-312, New York, N. Y., McGraw-Hill Book Company, 1950.
9. Johnson, B. C., "Methods of Vitamin Determination," p. 52-56, Minneapolis, Burgess Publishing Company, 1948.
10. McKinney, L. L., Weakley, F. B., Campbell, R. E., Eldridge, A. C., Cowan, J. C., Picken, J. C. Jr., and Jacobson, N. L., *J. Am. Oil Chemists' Soc.*, **34**, 461-466 (1957).
11. Sasaki, S., *J. Dept. Agri., Kyushu Imperial Univ. (Japan)*, **5**, 51-116 (1936), in English.
12. Sugimoto, K., *Bull. Osaka Med. Sch.*, **1**, 1-16 (1954).
13. Wai, K. N. T., Bishop, J. C., Mack, P. B., and Cotton, R. H., *Plant Physiol.*, **22**, 117-126 (1947).
14. Weakley, F. B., and McKinney, L. L., *Absts., Am. Oil Chemists' Soc.*, Sept. 30-October 2, 1957, Paper 46.
15. Wu, C. H., and Fenton, F., *Food Res.*, **18**, 640-645 (1953).

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Solubilities of Fatty Acids and Derivatives in Acetone¹

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KNOWLEDGE of the solubilities of fatty acids and derivatives in organic solvents at various temperatures is useful in the research laboratory and in industry in connection with low-temperature fractional crystallization and liquid-liquid distribution processes. The presently available information has been covered quite completely in two reviews (1, 2). It is characterized by many gaps, largely for two reasons: a) many of the compounds are difficult to prepare in sufficiently pure form, and b) technical problems introduced by such phenomena as polymorphism and supercooling sometimes make reliable measurements difficult.

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In previous measurements, two methods have been used to determine solubilities. Brown *et al.* (2, 3) and Bailey *et al.* (1) have used a so-called analytical method, which involves withdrawal and analysis of a portion of a solution in which equilibrium has been established between the solvent phase and an excess of solute. Others have used a "synthetic" method, which is somewhat simpler and has the added advantage of using appreciably less material without sacrifice of accuracy. This method consists of observing the temperature at which the last crystal disappears when raising the temperature of a solution of known gross composition or, alternatively, of observing the temperature at which the first crystal appears when the temperature is lowered. Hoerr and Harwood (5) employed the rising temperature technique. Ward and Singleton (11), using the falling temperature method, introduced refinements involving an examination of the cooling curves at different rates of

cooling. This permitted corrections for the effects of supercooling and eliminated the necessity for visual detection of crystal formation. Using a sensitive potentiometer-galvanometer system, which detected temperature changes of the order of 0.001°C., they were able to measure accurately solubilities of 1-monostearin in the range of 0.1 to 0.2%.

The method used in our investigation was essentially an adaptation of that used by Hoerr and Harwood (5). The study was undertaken to fill gaps in the information available about solubilities in acetone for saturated fatty acids with even numbers of carbon atoms from C₆ to C₁₈ oleic, linoleic, and linolenic acids, and methyl esters, alcohols, and simple triglycerides derived from these fatty acids.

Materials

Saturated Acids and Methyl Esters. The starting materials for the preparation of purified caproic, caprylic, capric, lauric, and myristic acids were commercial grades of the free acids. The acids were esterified with methanol, using sulfuric acid as catalyst. The esters were purified by repeated distillations of center cuts through a 4-ft. Podbielniak Hyper-Cal column. Purified acids were obtained through saponification of portions of the purified esters.

Palmitic acid and methyl palmitate were derived from virgin olive oil (pressed). The C₁₆ acid fraction of the methyl esters of the olive oil fatty acids was separated by fractional distillation and hydrolyzed. The free acids were recrystallized six times from ethyl alcohol. A portion of the purified acid was esterified with methanol, using sulfuric acid as catalyst, and the product was distilled to yield methyl ester.

The purified stearic acid and methyl stearate were prepared from a commercial stearic acid (Hystrene S-97, Atlas Powder Company). The crude acid was converted to the lead salt, and unsaturated impurities were removed by the method of Philipson *et al.* (9). The free acid was regenerated, esterified with methanol, and fractionally distilled. The constant boiling ester fraction of the C₁₈ acids was purified further by crystallization from diethyl ether. The final stearic acid was then obtained through saponification of a portion of the purified ester. Analytical data on the purified saturated acids and esters are given in Table I. Melting points were determined by the capillary method.

Unsaturated Acids and Methyl Esters. Virgin olive oil (pressed) was used as a starting material for the preparation of oleic acid and methyl oleate. The methyl esters of the C₁₈ acid fraction of olive oil methyl esters was obtained by fractional distillation. Methyl oleate was obtained from these esters by repeated low temperature-fractional crystallizations from acetone and was fractionally distilled for further purification. Purified oleic acid was obtained through saponification of a portion of the purified ester. Analytical values for the purified ester were as follows: iodine value (Wijs) 86.0, n_D²⁰ 1.4445.

Linoleic acid and methyl linoleate were derived from safflower oil fatty acids. The linoleic acid was concentrated by urea fractionation of the safflower oil acids and was further purified by repeated low-temperature crystallizations from Skellysolve F. The concentrate was esterified with methanol, and the esters were fractionally distilled. A portion of the

TABLE I
Physical Properties of Normal Saturated Compounds

Substance	Melting point (capillary method)	Refractive index
	(°C.)	(°C.)
Caproic acid.....	-3.5	1.4170/20
Caprylic acid.....	16.2	1.4241/30
Capric acid.....	30.4	1.4287/40
Lauric acid.....	44.2	1.4328/45.3
Myristic acid.....	54.4	1.4329/55
Methyl caproate.....	1.3990/35
Methyl caprylate.....	1.4152/25
Methyl caprate.....	1.4239/25
Methyl laurate.....	1.4303/25
Methyl myristate.....	1.4350/25
Methyl palmitate.....	30.4-30.5	1.4359/35
Methyl stearate.....	38.5-38.9	1.4370/40
Caproyl alcohol.....	-47.7 to -47.2	1.4184/20
Caprylyl alcohol.....	-15.2	1.4292/20
Capryl alcohol.....	6.9	1.4372/20
Lauryl alcohol.....	24.5	1.4374/25
Myristyl alcohol.....	37.5	1.4347/50
Palmityl alcohol.....	49.3	1.4394/51.5
Stearyl alcohol.....	58.1	1.4390/60
Tricaproin.....	1.4431/25
Tricaprylin.....	1.4480/20
Tricaprin.....	29.0	1.4451/40
Trilaurin.....	43.0-43.5	1.4401/60
Trimyristin.....	54.0-55.0	1.4420/60
Tripalmitin.....	66.5	1.4387/80
Tristearin.....	70.0-71.0	1.4403/80

purified linoleate from the center cut was saponified to obtain a purified linoleic acid. Analytical values for the purified methyl linoleate were as follows: iodine value (Wijs) 171.6, n_D²⁵ 1.4695.

Linolenic acid and methyl linolenate were prepared by the method of Mathews, Brode, and Brown (8) from linolenic acid derived from linseed oil by a bromination-debromination procedure. After 12 recrystallizations from Skellysolve F at -65°C., the linolenic acid had the following analytical values: iodine value (Wijs) 273.2, n_D²⁰ 1.4805; m.p. -11.3 to -11.2°C.; conjugated diene by ultraviolet analysis 0.12%.

Because the linolenic acid showed a slight absorption at 10.33 μ in the infrared spectral region, the product was recrystallized an additional 14 times from Skellysolve F at -65°C. The slight absorption at 10.33 μ persisted and very probably was caused by *trans* isomers; the absorption was too small however to permit accurate determination of the amount of contamination with *trans* material.

A portion of the purified linolenic acid was esterified with methanol, and a purified methyl linolenate was obtained by simple distillation of the product.

Saturated Alcohols. Commercial samples of good quality were used as source materials. These were further purified by careful fractional distillation and, wherever necessary, this was followed by fractional crystallization from acetone to yield the final products. Analytical values for the unsaturated alcohols are given in Table I.

Unsaturated Alcohols. Oleyl (iodine value 93.6, n_D²⁵ 1.4580), linoleyl (iodine value 186.6, n_D²⁰ 1.4676), and linolenyl (iodine value 288.0, n_D²⁰ 1.4778) alcohol were prepared by lithium aluminum hydride reduction of the corresponding purified fatty acids by the method of Lighthelm *et al.* (7). On infrared spectrophotometric examination, carbonyl impurities could not be detected in the final products, but a small amount of isomers containing *trans* double bonds was present in the linolenyl alcohol. Conjugated diene by ultraviolet analysis was virtually nil in the final products.

Triglycerides. The triglycerides of the saturated fatty acids from C₁₄ to C₁₈ and of the unsaturated fatty acids were prepared by *trans*-esterification of

TABLE II
Solubility Data
(g. solute per 100 g. of solvent/°C.)

Caproic acid	Caprylic acid	Capric acid	Lauric acid	Myristic acid	Linoleic acid	Linolenic acid
91.6 /-31.7 60.3 /-38.8 38.5 /-46.9 24.4 /-56.6 11.6 /-70.0	138.7 /-4.9 84.2 /-11.2 62.3 /-15.4 56.1 /-17.4 59.4 /-19.3 37.2 /-22.5 21.5 /-30.7 9.42/-48.5 4.24/-58.6	106.5 /6.2 64.2 /0.6 28.6 /-7.2 11.8 /-18.9 5.55/-29.0 2.65/-38.7 1.11/-50.5 0.58/-54.5	22.8 /8.6 9.84/0.6 6.94/-3.5 2.05/-18.1 0.59/-31.4 0.20/-42.9	16.8 /21.3 7.51/13.0 3.27/5.1 0.91/-9.0 0.53/-12.3 0.18/-25.3 0.05/<-79.3	11.49/-37.3 5.47/-44.4 2.00/-52.1 0.69/>-79.3 0.52/<-79.3	45.1 /-36.9 25.8 /-41.3 11.4 /-49.3 7.79/-53.8 5.66/-56.3 3.22/-62.3 2.73/-66.9 1.04/>-79.3 0.77/<-79.3
Methyl caproate ^a	Methyl caprylate	Methyl caprate	Methyl laurate	Methyl myristate	Methyl palmitate	Methyl stearate
400.0 /-76.0 100.0 /-86.0 42.8 /-93.0 21.2 /-100.0 5.27/-120.0	330.0 /-44.3 43.7 /-58.0 18.1 /-65.5 8.72/>-79.3 5.74/<-79.3	330.0 /-20.0 96.4 /-27.0 43.4 /-31.8 25.1 /-35.3 18.1 /-37.5 11.6 /-41.8 5.49/-49.5 1.21/>-79.3 0.21/<-79.3	37.2 /-13.3 10.68/-21.5	32.0 /0.5 11.1 /-6.3	33.0 /12.3 10.80/6.5	5.93/13.0 1.10/ 1.5
Methyl oleate	Methyl linoleate	Methyl linolenate	Caproyl alcohol	Caprylyl alcohol	Capryl alcohol	Lauryl alcohol
208.0 /-27.5 98.0 /-29.4 44.9 /-31.0 27.1 /-33.0 12.0 /-35.3 5.42/-40.0 0.60/-55.8	200.0 /-45.7 104.0 /-47.5 48.4 /-49.8 26.1 /-51.7 11.5 /-55.3 5.77/-59.0 0.67/<-79.3	192.0 /-62.3 101.0 /-64.7 47.9 /-68.0 28.2 /-69.8 11.7 /-79.0 6.27/<-79.3	834.0 /-49.3 225.0 /-52.2 66.7 /-56.0 25.3 /-59.8 11.6 /-65.5 5.58/-70.7	14.8 /-34.8 9.08/-39.5 4.89/-46.8 1.43/-60.1 0.60/-68.9 0.43/>-79.3 0.12/<-79.3	15.7 /-15.2 6.77/-22.3 3.12/-28.1 1.51/-36.1 0.62/-47.5 0.21/-59.8 0.05/<-79.3	14.7 /0.8 7.43/-4.8 4.48/-9.5 1.54/-20.3 0.61/-28.2 0.22/-37.0 0.08/-45.3 0.04/-53.3 0.01/-69.0
Myristyl alcohol	Palmityl alcohol	Stearyl alcohol	Oleyl alcohol	Linoleyl alcohol	Linolenyl alcohol	Tricaprylin
6.22/8.0 2.22/1.0 0.94/-6.5 0.35/-13.8 0.15/-20.8 0.05/-31.6 0.02/-37.1 0.01/-46.5	7.13/22.4 5.51/19.1 2.67/14.2 2.08/12.0 1.00/8.3 0.82/7.1 0.31/0.9 0.126/-6.0 0.05/-12.2	1.07/18.6 0.43/10.8 0.16/3.3 0.07/-1.7 0.03/-16.0 0.01/>-79.3	11.8 /12.7 5.41/-17.8 2.66/-23.1 1.05/-29.5 0.55/-34.8 0.25/-41.9 0.12/-47.5	27.2 /-25.0 11.1 /-28.6 5.32/-33.3 2.62/-37.8 1.04/-45.7 0.54/-50.8 0.11/-63.3	25.0 /-38.9 11.1 /-44.0 5.48/-49.1 2.69/-55.1 1.05/-63.9 0.54/-71.0 0.18/<-79.3	19.63/-15.9 12.74/-18.3 6.61/-21.8 4.42/-24.3 2.63/-27.3 1.06/-32.7
Tricaprin	Trilaurin	Trimyristin	Triolein	Trilinolein	Trilinolenin	
5.41/1.8 2.88/-1.5 1.06/-8.0 0.51/-13.0 0.21/-19.5 0.10/-21.8	5.21/21.5 2.65/18.0 1.04/13.8 0.51/10.3 0.23/6.5 0.11/3.3	0.43/24.7 0.11/18.5 0.06/16.3	185.7 /-3.8 64.2 /-4.0 25.3 /-4.5 8.32/-6.5 1.20/-11.8 0.52/-14.8 0.20/-18.5 0.05/-25.5	25.0 /-25.0 11.48/-26.0 2.28/-30.7 0.54/-37.0 0.22/-40.7 0.05/-47.3	99.8 /-38.0 42.7 /-39.1 11.5 /-40.3 5.52/-43.3 2.11/-47.0 1.11/-50.7	

^a Calculated values.

the methyl esters with triacetin, using sodium methoxide as catalyst. Soap, mono- and diglycerides, and unreacted methyl ester were removed by extraction of a Skellysolve F solution of the compounds with 80% aqueous ethanol. The washed products then were crystallized from acetone, Skellysolve F, or diethyl ether.

The triglycerides of the lower saturated acids were prepared by direct esterification with glycerol, using p-toluenesulfonic acid as catalyst. The crude tri-caproin and tricaprylin were purified by high-vacuum distillation. Tricaprylin, tricaprin, and trilaurin were further purified by several recrystallizations from acetone. Saponification values were as follows: tricaproin 417.8; tricaprylin 348.3; tricaprin 302.4; trilaurin 263.5; trimyristin 235.9; tripalmitin 208.9; tristearin 189.9. Other analytical data on the saturated triglycerides are given in Table I. Triolein had an iodine value of 86.0, n_D^{20} 1.4638; trilinolein, an iodine value of 171.0, n_D^{20} 1.4750; trilinolenin, an iodine value of 260.4, n_D^{20} 1.4825.

Apparatus and Procedure

The apparatus devised for the solubility measurements in this study is shown in Figure 1 and has been

described elsewhere (10). It was virtually identical with an apparatus devised by Ward and Singleton, a description of which was published during the course of the work presented here (11). However, as mentioned previously, in our work the solubility end-point was approached from the cold side.

Although, as shown by Ward and Singleton (11), the end-point may be approached from the warm side with some materials such as 1-monostearin, this approach was found to be impractical with some of the compounds in our study because of difficulties caused by supercooling and polymorphism. In our hands the results of the synthetic method were in no way impaired by visual detection of the end-point as approached from the cold side. The temperature at which complete solution occurred was reproducible in most cases within $\pm 0.1^\circ\text{C}$.

Detection of the total disappearance of crystals was greatly facilitated by observations through a strong magnifying glass and by illumination from a pinpoint light source (12 v. AC-DC "grain of wheat" bulb), placed directly behind the sample chamber.

With each compound a series of solutions was prepared at concentrations selected to provide adequate data for the accurate construction of its solubility

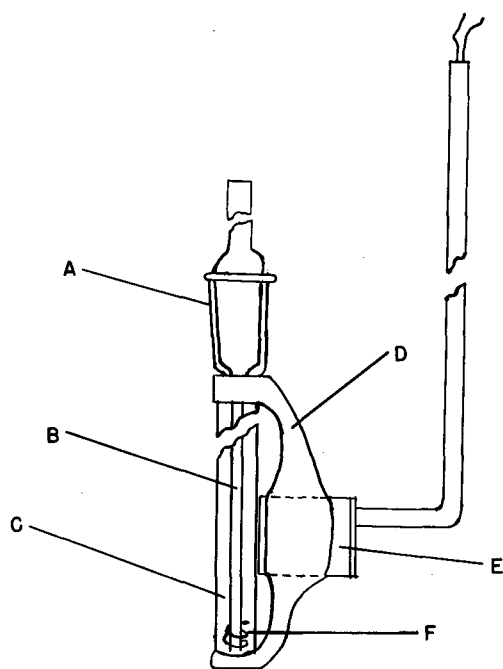


FIG. 1. Solubility apparatus: A, 14/35 ground glass joint; B, thermocouple well; C, crystallization chamber; D, support; E, small solenoid; F, tin-plated iron coil.

curve. An individual sample chamber containing about 2 ml. of solution was used for each concentration. In general, it was found convenient to conduct measurements in an order progressing from the lowest to the highest concentrations.

In each case the solution was cooled to a temperature where an appreciable number of crystals formed. It then was placed in a support and immersed in an acetone bath, the temperature of which was about the same as that of the sample. Cooling of the acetone bath was accomplished by immersing several cold elements consisting of Pyrex cylinders that contained dry ice. The bath was equipped with a stirrer to maintain a uniform temperature throughout.

The rate of warming of the bath was controlled by removing one or more of the cold elements from time to time. A magnetically operated agitator in the solution provided rapid heat distribution and thorough mixing of the solution and crystals. The temperature was allowed to rise about 1°C. per 5 minutes more or less, depending to some extent on the solubility and the temperature coefficient of solubility. Preliminary experiments indicated that this rate of temperature rise was sufficiently small so that virtual equilibrium existed at the time when the last crystals disappeared.

The temperature of the solution was measured with a calibrated, single element thermocouple constructed from No. 30 B & S ga. copper and constantan wires. The thermocouple voltage was measured with a Leeds and Northrup K-2 potentiometer used in conjunction with a Leeds and Northrup No. 2430 galvanometer. With this system, temperatures were determined with an accuracy of $\pm 0.1^\circ\text{C}$. over the temperature ranges studied.

The same procedure was used with each succeeding solution of higher concentration until the temperature range up to the melting point of the compound had been covered. The solubilities are presented in

tabular form (Table II). Figure 2 compares the data obtained for linoleic acid by our method with those reported by Foreman and Brown (4), Kolb and Brown (6), and Hoerr and Harwood (5).

Where reliable data had previously been obtained by others, only scattered determinations were made in this study, primarily as a check on the method employed here. In most cases the results of this study agree well with the data previously obtained (Figure 2).

The preparation of methyl caproate could not be crystallized from acetone at -70°C ., and the solubility values for this compound are hypothetical, obtained by extrapolation of the solubilities of the other members of the series.

The solubility of tristearin was less than 0.025% at 40°C ., and the solubility of tripalmitin was only 0.027% at 24°C .

Although most fatty materials exhibit more than one crystal modification, it is generally believed that only the most stable polymorph is formed by crystallization from common fat solvents. Such was not the case with some of the compounds in this study. Because different polymorphs of the same substance have different solubilities, as has been observed with oleic acid, for example, by Hoerr and Harwood (5), it was necessary to use special precautions in some cases to insure that the solubilities of the stable forms were being determined.

A good example of the effect of polymorphism on solubility encountered in this work was represented by the case of palmityl alcohol in hexane. The solubility depended on the previous history of the palmityl alcohol, the crystallization procedure used, and the conditioning of the sample. Palmityl alcohol previously crystallized from hexane or benzene gave entirely different solubility data than when crystallized from acetone unless the samples were properly conditioned.

In general, whenever this problem was encountered, it was necessary to treat each case individually and to find by trial and error the conditions that would give crystals in their most stable modification.

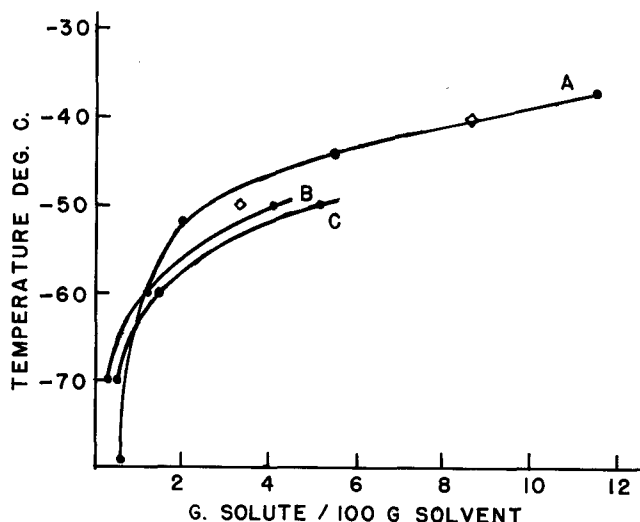


FIG. 2. Solubility of linoleic acid in acetone as obtained by different investigators: A, authors (synthetic method); B, Kolb, D. K., and Brown, J. B. (3, 6) (analytical method); C, Foreman, H. B., and Brown, J. B. (4) (analytical method); □ Hoerr, C. W., and Harwood, H. S. (5) (synthetic method).

The data obtained here extend the information on the solubilities of the fatty acids and derivatives that were studied and may be used as a guide for separation of mixtures by crystallization procedures. However, with mixtures of fatty acids in solution, the solubility of any given fatty acid is affected by the solubilizing effect of other fatty acids in the mixture, and the nature and extent of this solubilizing effect is also dependent on the nature of the solvent. Because of a paucity of data on the solubility characteristics of mixtures, it would be desirable to follow these measurements of pure individual compounds with studies of model mixtures. Nevertheless the solubility curves for the pure compounds are useful, even when fractional crystallization of mixtures is contemplated, and can be used in selecting solvents, solute concentrations, and crystallization temperatures.

Summary

An improved "synthetic" method of determining solubilities has been described which combines simplicity with accuracy.

Saturated fatty acids with even numbers of carbons from C₆ to C₁₈ and oleic, linoleic, and linolenic acids, their methyl esters, their simple triglycerides,

and their corresponding alcohols have been prepared in purified form. The solubilities in acetone of their most stable forms have been determined from ordinary room temperatures down to about -70°C. or to temperatures where they are only slightly soluble.

The precipitation of unstable polymorphs from solutions was observed in the case of palmityl alcohol.

Acknowledgment

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REFERENCES

1. Bailey, A. E., Singleton, W. S., and Feuge, R. O., *Oil & Soap*, **23**, 201 (1946).
2. Brown, J. B., *Chem. Rev.*, **29**, 333 (1941).
3. Brown, J. B., and Kolb, D. K., "Progress in the Chemistry of Fats," vol. 3, pp. 58-94. Pergamon Press, London, 1955.
4. Foreman, H. D., and Brown, J. B., *Oil & Soap*, **21**, 133 (1944).
5. Hoerr, C. W., and Harwood, H. J., *J. Phys. Chem.*, **56**, 1068 (1952).
6. Kolb, D. K., and Brown, J. B., *J. Am. Oil Chemists' Soc.*, **32**, 357 (1955).
7. Lighthelm, S. P., von Rudloff, E., and Sutton, D. A., *J. Chem. Soc.*, 3187 (1950).
8. Mathews, N. L., Brode, W. R., and Brown, J. B., *J. Am. Chem. Soc.*, **63**, 1064 (1941).
9. Philipson, J. M., Heldman, M. J., Lyon, L. L., and Vold, R. D., *Oil & Soap*, **21**, 315 (1944).
10. Privett, O. S., *Hormel Inst. Ann. Rept.*, p. 55 (1954-1955).
11. Ward, T. L., and Singleton, W. S., *J. Am. Oil Chemists' Soc.*, **32**, 172 (1955).

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Report of the Uniform Methods Committee, 1957-58

AT A MEETING of the Uniform Methods Committee in the Peabody Hotel on April 21, 1958, the following matters were discussed and the indicated decisions were made. The meeting was attended by all members of the Uniform Methods Committee. L. H. Hodges, J. R. Mays Jr., and H. T. Spanuth were present during part of the discussion.

1. Seed and Meal Analysis Committee,

T. H. Hopper, chairman

Subcommittee on Sampling Bulk Meal, L. H. Hodges, chairman

a) The committee recommends the adoption of additions to the Official Method for Sampling, Ba 1-38, to provide for the continuous sampling of bulk shipments of meal at point of origin. The automatic sampler is designed to take continuously a representative small portion of the entire cross-section of the meal at a point where the flow of meal is free-falling.

This method is urgently needed. The subcommittee has spent several years in its development and has demonstrated its reliability. The Uniform Methods Committee concurs with their recommendation for its addition to Ba 1-38, provided more information is included on the design and source of the automatic sampler. Under "Apparatus—A 7 (a)" the following sentence should be added: "Any automatic sampler, equal or equivalent in performance, to those offered by Standard Geary-Jennings with Type C Modified Cutter, manufactured by The Galigher Company, 545 West 8th South street, Salt Lake City 4, Utah, and Automatic Sampler by Davidson-Kennedy Company, 1090 Jefferson street N. W., Box 97, Station D, Atlanta, Ga., will be satisfactory." Furthermore a drawing, showing construction of a suitable sampler, will be included in the printed method. Adopted.

b) The Seed and Meal Analysis Committee further recommends advancement of the Tentative Method Ba 7-55, for "Free Gossypol," in cottonseed slab and sized cakes and meal, to Official status. The Uniform Methods Committee approves this proposed action. Adopted.

2. Fat Analysis Committee, V. C. Mehlenbacher, chairman

Subcommittee on Analysis of Drying Oils, J. C. Konen, chairman.

a) The Fat Analysis Committee recommends replacement of

present Tentative Method, Ka 2-55 for "Acid Value," by a complete revision. This subcommittee is a joint committee with A.S.T.M., and the proposed revision contains three references to A.S.T.M. Designations for synthetic methyl alcohol, 99% isopropyl alcohol, and industrial toluol. These specifications will be placed by our editor of Methods in Section H of A.O.C.S. Methods, with the assistance and approval of the chairman of the F.A.C., and the specifications for these solvents will be referred to in the method by their appropriate number in Section H. With these changes the Uniform Methods Committee approves replacement of Ka 2-55 by this new Tentative Method. Adopted.

b) The Fat Analysis Committee further recommends replacement of present Tentative Method, Ka 3-47, for "Color by Gardner (1933) Standard Colors," by a complete revision employing Gardner (1953) Standard Colors. Unfortunately this method also contains one reference to an A.S.T.M. Designation, which cannot be avoided without unwarranted duplication in our Methods. The caption "Composition" should be added to cover all four last columns of "Table I. Reference Standard Color Solutions." With this slight addition the Uniform Methods Committee approves adoption of this revision of Tentative Method Ka 3-47. Adopted.

c) At the Fall Meeting in Cincinnati, October 2, 1957, the Uniform Methods Committee approved, and the Society adopted, a "Continuous Flow Method for Sampling Tanks or Tank Cars During Loading or Unloading," as a replacement for C 1-47, D, (a), the present "Petcock Method." This was done with the understanding that certain criticisms made by the U.M.C. would be recognized and corrections made. This has been done, and no further action by the Society is necessary at this time. It is obvious however that a few changes are required in the drawing to show the following.

1. By enlarged insert more detail on the 45° bevel end of the bleeder line, its orientation, and a somewhat more definite angle with the horizontal for the bleeder line than "a slight downward slope" should be shown.
2. The drawing shows arrangement for sampling only while loading. To provide for sampling during unloading the drawing should be marked in appropriate places: "To Tank Car or Storage" (two places) and "From Storage or Tank Car" (one place).
3. If, as we believe, this method is applicable to tank