over this same temperature range, the tetrahydrate would not be observed in industrial processing steps unless considerable effort was made to secure it.

Because of the unusual shape of the 100° C. isotherm, it would be interesting to determine if more than one crystal structure of ferrous sulfate monohydrate is formed at this temperature.

Edge (9) reports that ferrous sulfate monohydrate is completely insoluble at 290° F. (143° C). The study of this system at higher temperatures and pressures would give data for processing under these conditions.

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Received for review February 27, 1956 Accepted May 19, 1956

Liquid-Liquid Equilibrium Relations in Systems Involving Oleic Acid

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Interest in the solvent method for refining fats and oils commercially is relatively new and little published data are available. This report gives qualitatively, the mutual solubility of 35 solvents and their solubility in oleic acid, plus mutual solubility and tie-line data at 25° C. for oleic acid with five pairs of immiscible solvents—three in which propylene glycol is paired with n-heptane, cyclohexane, and xylene; and two in which furfural is paired with n-heptane and cyclohexane. Two methods for correlating tie-line data are given and evaluated.

Although refining and fractionation of fats and oils by means of solvents have been practiced for a long time, commercial application is relatively recent (9). Examples of this method include furfural extraction described by Gloyer (7, 8) and the use of liquid propane as a selective solvent—e.g., in the Solexol process (14). Because of increasing interest in solvent extraction of fats and oils commercially, and as a result of exploratory experiments, it seemed desirable to consider the use of multicomponent solvent mixtures to separate undesirable coloring material from fatty mixtures such as foots, tallow, or fatty acid stocks. A literature survey showed that reasonably promising solvent combinations could not be postulated with existing solubility data and it was necessary to determine in the laboratory some solubility relationships between fatty materials and commercial solvents.

In order to obtain reproducible data and because of the lack of information on pure fatty acids, oleic acid was selected as the fatty material. The problem was then to determine the solubility of oleic acid with two non-consolute solvents; these were selected either on the basis of solubility tables of fatty acids with non-aqueous solvents by Markley (10) or from a general list of 100 common solvents by Mellan (12). In making selections from the latter, an attempt was made to choose solvents that are commercially used with fats and oils and also to select at least two solvents in each of the classifications given by Durrans (5). After thirty-five solvents were selected, their solubility both mutually and with oleic acid was qualitatively determined in order to select pairs of solvents that were immiscible with each other but miscible with oleic acid (Table I).

From these data (Table I) 16 immiscible solvent pairs were selected, and the binodal curves of the following ternary systems were determined at 25° ± 2° C. (16):

Oleic acid -
Furfural - n-pentane
Propylene glycol - n-pentane
Furfuryl alcohol - n-pentane
Methanol - cyclohexane
Furfural - cyclohexane
Furfuryl alcohol - cyclohexane
Propylene glycol - carbon tetrachloride
Propylene glycol - cyclohexane
Propylene glycol - o-dichlorobenzene
Propylene glycol - p-chlorotoluene
Propylene glycol - benzene
Propylene glycol - ethylene dichloride
Propylene glycol - tetrachloroethylene
Propylene glycol - toluene
Propylene glycol - trichloroethylene
Propylene glycol - xylene

From these 16 ternary systems five were selected as follows for detailed study. The solubility isotherm and several tie lines were determined at 25° C.

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TABLE I. MUTUAL SOLUBILITY OF THIRTY-FIVE SOLVENTS AND THEIR SOLUBILITY WITH OLEIC ACID AT 25° C.

Comp. No.	Compound	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36
1	Acetone		S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	I	S	S	S	S	S	S	S	S	S	S	S	S	S	S
2	Amyl alcohol	S		S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	I	S	S	S	S	S	I	S	S	S	S	S	S	S	S
3	Benzene	S	S		S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	I	S	S	I	S	S	S	S	S	S	S	S	S	I	S	S	S	S
4	n-Butanol	S	S	S		S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S
5	Butyl acetate	S	S	S	S		S	S	S	S	S	S	S	S	S	S	S	S	S	I	S	S	I	S	S	S	S	S	S	S	S	S	S	S	S	S	S
6	Butyl ether	S	S	S	S	S		S	S	S	S	S	S	S	S	S	S	S	S	I	S	I	I	S	S	S	S	S	S	S	S	S	I	S	S	S	S
7	Butyl lactate	S	S	S	S	S	S		S	S	S	S	S	S	S	S	S	S	S	S	S	S	I	S	S	S	S	S	S	S	S	S	S	S	S	S	S
8	Butyl stearate	S	S	S	S	S	S	S		S	S	S	S	S	S	S	S	S	S	I	I	I	I	S	S	I	S	S	I	S	S	S	I	S	S	S	S
9	Carbon tetrachloride	S	S	S	S	S	S	S	S		S	S	S	S	S	S	S	S	S	I	S	S	I	S	S	S	S	S	S	S	S	S	I	S	S	S	S
10	Chloroform	S	S	S	S	S	S	S	S	S		S	S	S	S	S	S	S	S	I	S	S	I	S	S	S	S	S	S	S	S	S	S	S	S	S	S
11	p-Chlorotoluene	S	S	S	S	S	S	S	S	S	S		S	S	S	S	S	S	S	I	S	S	I	S	S	S	S	S	S	S	S	S	I	S	S	S	S
12	Cyclohexane	S	S	S	S	S	S	S	S	S	S	S		S	S	S	S	S	S	I	I	I	I	S	S	I	S	S	S	S	S	S	I	S	S	S	S
13	Cyclohexanol	S	S	S	S	S	S	S	S	S	S	S	S		S	S	S	S	S	S	S	S	S	S	S	S	S	S	I	S	S	S	S	S	S	S	S
14	o-Dichlorobenzene	S	S	S	S	S	S	S	S	S	S	S	S	S		S	S	S	S	I	S	S	I	S	S	S	S	S	S	S	S	S	I	S	S	S	S
15	Diethylcarbinol	S	S	S	S	S	S	S	S	S	S	S	S	S	S		S	S	S	S	S	S	I	S	S	S	S	S	S	S	S	S	S	S	S	S	S
16	Ether	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S		S	S	I	S	S	I	S	S	S	S	S	S	S	S	S	S	S	S	S	S
17	Ethyl acetate	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S		S	I	S	S	I	S	S	S	S	S	S	S	S	S	S	S	S	S	S
18	Ethylene dichloride	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S		I	S	S	I	S	S	S	S	S	S	S	S	S	S	S	I	S	S
19	Ethylene glycol	S	S	I	S	I	I	S	I	I	I	I	I	S	I	S	I	I	I		S	S	S	S	I	S	S	I	I	I	I	I	S	I	I	I	I
20	Furfural	S	S	S	S	S	S	S	I	S	S	S	I	S	S	S	S	S	S	S		S	I	S	S	S	S	S	S	S	S	I	S	S	S	S	S
21	Furfuryl alcohol	S	S	S	S	S	I	S	I	S	S	S	I	S	S	S	S	S	S	S	S		S	S	S	S	S	S	S	S	S	I	S	S	S	S	S
22	Glycerol	I	I	I	I	I	I	I	I	I	I	I	I	S	I	I	I	I	I	S	S	I		S	I	S	I	I	I	I	I	I	S	I	I	I	I
23	2-Propanol	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S		S	S	S	S	S	S	S	S	S	S	S	S	S
24	Lauryl alcohol	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	I	S	S	I	S		S	S	I	I	S	S	S	S	S	S	S	S
25	Methanol	S	S	S	S	S	S	S	I	S	S	S	I	S	S	S	S	S	S	S	S	S	S	S	S		S	S	S	S	S	S	S	I	S	S	S
26	Methyl ethyl ketone	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	I	S	S	S		S	S	S	S	S	S	S	S	S	S
27	Nitroethane	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	I	S	S	I	S	I	S	S		S'	S	I	S	S	S	S	S	S
28	Nitromethane	S	I	S	S	S	S	S	I	S	S	S	S	I	S	S	S	S	S	I	S	S	I	S	I	S	S	S		S	I	I	S	I	S	S	S
29	1-Nitropropane	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	I	S	S	I	S	S	S	S	S	S		S	S	I	S	S	S	S
30	Oleic acid	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	I	S	S	I	S	S	S	S	I	I	S		S	S	S	S	S	S
31	n-Pentane	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	I	I	I	I	S	S	S	S	S	I	S	S		I	S	S	S	S
32	Propylene glycol	S	S	I	S	S	I	S	I	I	S	I	I	S	I	S	S	S	I	S	S	S	S	S	S	S	S	S	S	I	S	I		I	I	I	I
33	Tetrachloroethylene	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	I	S	S	I	S	S	I	S	S	I	S	S	S	I		S	S	S
34	Toluene	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	I	S	S	I	S	S	S	S	S	S	S	S	S	I	S		S	S
35	Trichloroethylene	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	I	S	S	I	S	S	S	S	S	S	S	S	S	I	S	S		S
36	Xylene	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	S	I	S	S	I	S	S	S	S	S	S	S	S	S	I	S	S	S	

S = Soluble in all proportions
I = Insoluble in some proportions

Oleic acid -

Propylene glycol - n-heptane
Propylene glycol - cyclohexane
Propylene glycol - xylene
Furfural - n-heptane
Furfural - cyclohexane

CHEMICALS

Oleic acid, the purest commercially available, was obtained from two suppliers—Armour & Co. furnished an acid reported to be 90% oleic acid with impurities consisting of approximately 10% saturated acids and a trace of linolenic acid (15); Emery Industries, Inc., supplies Emersol, 233 LL Elaine, reported to be 90% oleic acid with 6% saturated acid impurities, 3% linoleic acid, and 1% linolenic acid (6). By the pycnometer method the density at 25° C. of Armour's acid was found to be 0.890 gm./cc., and of Emery's acid, 0.889 gm./cc. These densities compare favorably with 0.894 gm./cc. at 18° C. recorded by Perry (3).

Eastman Kodak Co.'s purest (white label) grade propylene glycol having a density of 1.038 ± 0.001 gm./ml. at 23° C. was used; this value compares favorably to 1.038 gm./cc. at 23° C. reported by Perry (3).

A special grade of stabilized furfural was obtained from the Quaker Oats Co. Although this furfural, reported to be 99 1/2% pure (19), was stabilized against autoxidation by the addition of an inhibitor, it was quite dark in color. Before using, it was purified by distillation in a laboratory column and during use was protected from the air as much as possible. The freshly distilled furfural was almost colorless but after one week it had darkened to a light straw yellow. Furfural that had not been distilled within the previous week was not used. Its density as determined by a hydrometer was 1.158 gm./ml. ± 0.001 at 23° C. as compared to 1.155 gm./cc. at 25° C. reported by Perry (3).

The n-heptane, obtained from the Phillips Petroleum Co., was reported to be a minimum of 99 mole % pure. Because of the high purity, density of this compound, 0.684 gm./cc. at 23° C., was taken from Maxwell (11). Likewise density of the cyclohexane—0.776 gm./cc. at 23° C.— also obtained from the Phillips Petroleum Co. and reported to be a minimum of 99 mole % pure was taken from Maxwell (11).

Mallinckrodt Chemical Works' reagent-grade xylene having a density of 0.867 gm./cc. at 23° C. was used. This compares to the literature densities at 20° C. of 0.874, 0.864, and 0.862 for o-xylene, m-xylene, and p-xylene, respectively (3).

EXPERIMENTAL PROCEDURES

The binodal curves were established by drawing a smooth curve through cloud points established by mixing known amounts of two components and adding quantities of the third until the system previously consisting of two layers became homogeneous, or vice versa. Both techniques, described in the literature (17), were used in this work. Generally in an area of relatively low solvent concentration, known mixtures of the other solvent and oleic acid were prepared, and the cloud point was determined by adding the solvent until turbidity appeared. In areas where either of the solvent concentrations was larger than about 10%, the cloud points were established by adding oleic acid to known mixtures of the two solvents until turbidity disappeared. Most of the points were determined by adding oleic acid to known solutions of the two solvents; the discussion in the next 3 paragraphs refers to those points.

The initial mixtures were prepared by combining volumes of each component measured from standard burets. From the binodal curve of the phase diagrams at $25^\circ \pm 2^\circ$ C., the cloud point could be closely estimated, and enough oleic acid was added to bring the composition close to the binodal curve. The flasks were mechanically shaken for about 15 minutes and were then placed in the constant temperature bath for at least one hour. The titration was carried out in several steps in order that the mixture could be frequently returned to the bath to ensure maintenance of the temperature.

Since the composition was already near that of the cloud point, the amount of oleic acid added was small; this procedure coupled with the fact that the room temperature was usually within 1° C. of the bath temperature maintained at 25.00° C. $\pm$ 0.05° C., lends reasonable assurance that the points on the binodal curve were determined at $25.0^\circ \pm 0.1^\circ$ C. To facilitate establishment of cloud points, a titration illuminator was used; as the point was approached, frequent comparisons with a standard, placed in front of the illuminator, were made.

The tie lines were determined after the binodal curve had been established, and the over-all composition of their mixtures was selected in such a manner that the tie lines on the diagram would be relatively equally spaced. A mixture of the three components corresponding to the desired over-all composition was made in a 125-ml., pear-shaped, separatory funnel by measuring known volumes from standard burets. The total weight of this mixture was 80 grams. Separatory funnels containing the mixtures were mechanically shaken for 1/2 hour and placed in the constant temperature bath for at least 24 hours. While the funnel was still in the bath, a sample of top phase taken with a pipet, was emptied into a tared 125-ml., glass-stoppered Erlenmeyer flask, and weighed. The funnel was removed from the bath, its tip flushed, and a sample of the lower phase was taken. The weight per cent of oleic acid in each of the phases was determined, and the tie line was plotted using the point established by the analysis of one of these phases and the point of over-all composition. If the tie line did not, within a small experimental error, intersect the binodal curve at the point established by the analysis of the other phase, it was completely redetermined. Thus, there was a check on the location of each tie line.

In analyzing for oleic acid, the American Oil Chemists' Society's official method Da 14-42 was used (1). This method consists of titrating the acid in a hot alcohol solvent with 0.5N sodium hydroxide, using phenolphthalein as the indicator. Because the acid was not pure, the equivalency could not be calculated but had to be determined experimentally. The amount of standard sodium hydroxide required to neutralize the oleic acid was determined each week because the normality of the base and oleic acid was found to change slowly.

The n-heptane, cyclohexane, propylene glycol, and xylene were checked to be sure that impurities took none of the standard sodium hydroxide. In every case one drop of the sodium hydroxide solution put the alcohol solution containing about 40 ml. of reagent past the end point.

It was known (4) that autoxidation of the furfural would create a certain amount of acid which would react with the sodium hydroxide. However, exclusion of air, the only preventive, was not always practical. In the furfural systems, the oleic acid concentration was still determined by titration with sodium hydroxide to a phenolphthalein end point, but the amount of oleic acid present in the samples was calculated by taking into account the amount of furfural present. On the day of the analysis the amount of sodium hydroxide required to neutralize the furfural in a 95% aqueous alcohol solution to a phenolphthalein end point was determined at three furfural concentrations. Thus, it was possible by trial and error to determine the oleic acid content in the various tie-line samples of the furfural systems.

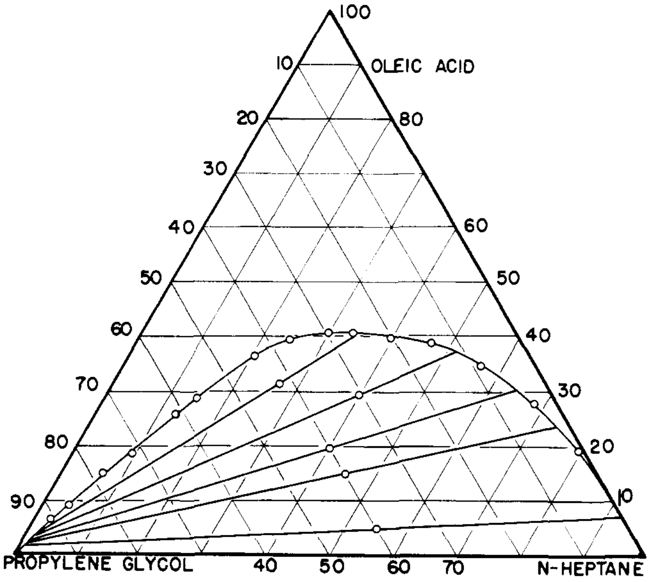

Figure 1. Solubility curve and tie lines for the oleic acid-propylene glycol-n-heptane system at 25° C.

CORRELATION AND INTERPOLATION OF TIE-LINE DATA

Mutual liquid solubility and tie-line data in weight per cent at 25° C. are shown in Figures 1 to 5. The tie-line data were correlated (Figures 6, 7) by the method of Othmer and Tobias (13) and of Treybal (18). The possibility of using Campbell's method (2) was considered, but a log-log plot of the variables did not show the desired straight line relationship. For the method of Othmer and Tobias, $(1-a_1) / a_1$ is plotted against $(1-b_2) / b_2$ on log-log paper, where a_1 is the weight fraction of solvent in the solvent phase, and b_2 is the weight fraction of diluent in the conjugate phase. For the method of Treybal, log c_1/a_1 is plotted against log c_2/b_2, where c_1 is the weight fraction of solute in the solvent phase, c_2 is the weight fraction of solute in the diluent phase, a_1 is the weight fraction of solvent in the solvent phase, and b_2 is the weight fraction of diluent in the diluent phase. Oleic acid is the solute, the

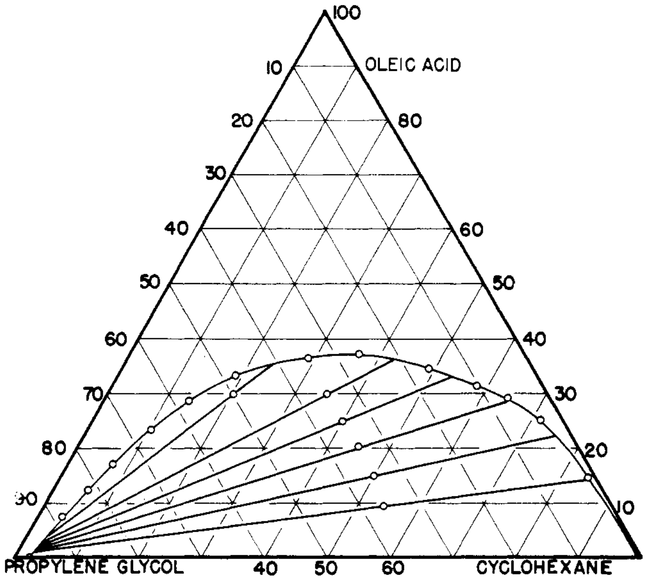

Figure 2. Solubility curve and tie lines for the oleic acid-propylene glycol-cyclohexane system at 25° C.

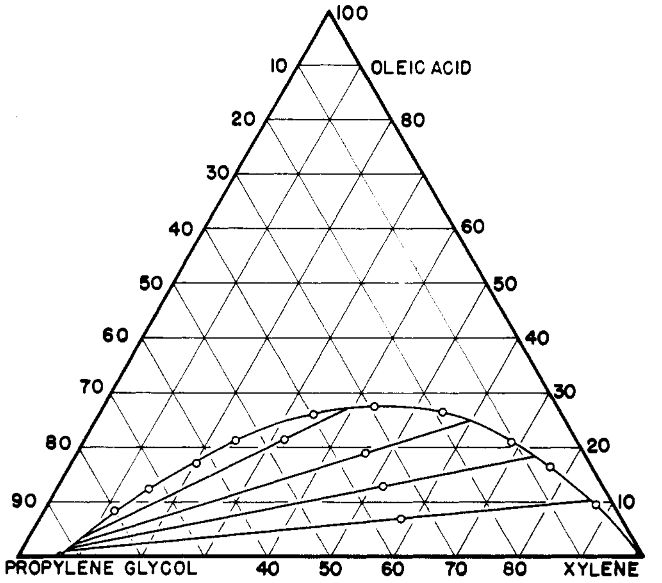

Figure 3. Solubility curve and tie lines for the oleic acid-proplene glycol-xylene system at 25° C.

hydrocarbon is the solvent, and propylene glycol or furfural is the diluent.

A statistical approach was used to determine if one method was significantly better than the other to correlate tie-line data for any of these five systems. The possibility that one method might, on the average, correlate the entire data better than the other was also statistically checked. The procedure, described in detail (16), can be used for determining which of several possible tie-line correlations is best for the systems involved. The analysis showed no significant difference between the ability of the two methods to correlate the data for any individual system or for the five systems considered as a group; both methods correlated the data well.

ACKNOWLEDGMENT

This work was carried out under a fellowship of the Procter and Gamble Co., Cincinnati, Ohio, whose interest and support are gratefully acknowledged.

Appreciation is also expressed to R. Payne and D. Byerly for their assistance with the experimental work.

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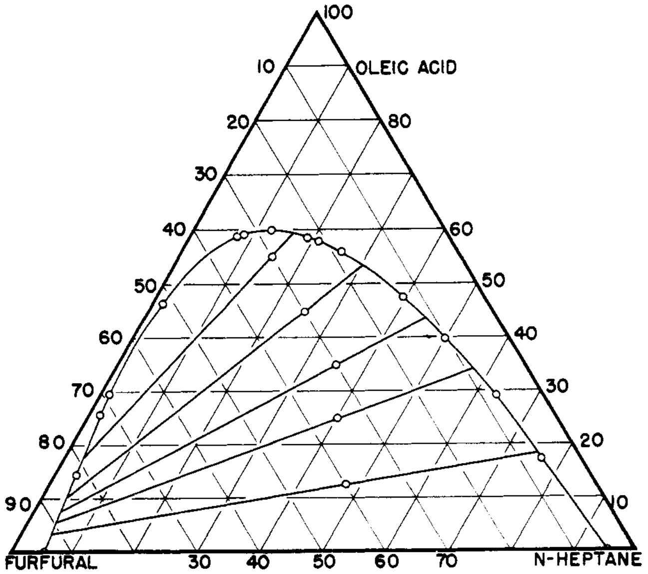

Figure 4. Solubility curve and tie lines for the oleic acid-furfural-n-heptane system at 25° C.

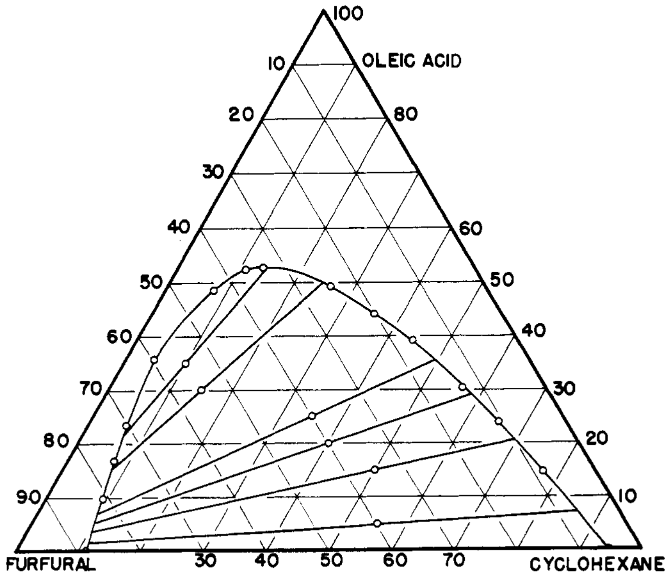

Figure 5. Solubility curve and tie lines for the oleic acid-furfural-cyclohexane system at 25° C.

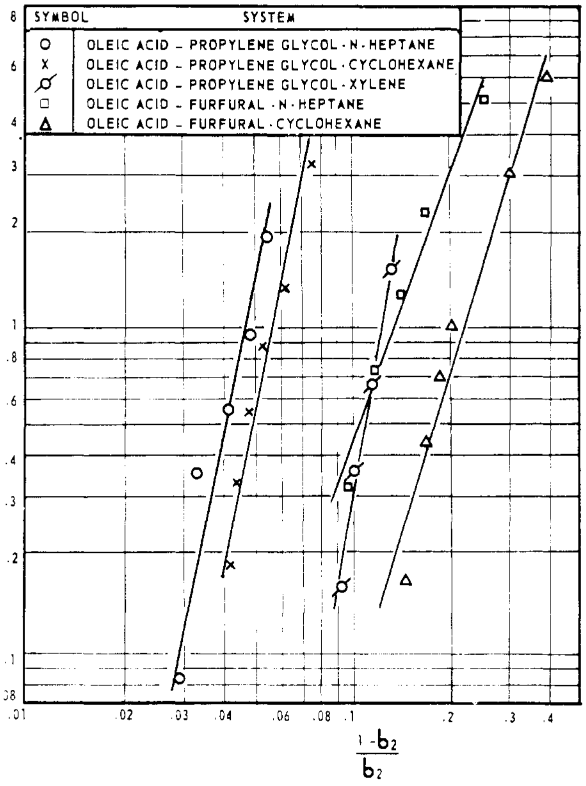

Figure 6. Othmer and Tobias tie-line correlation plot

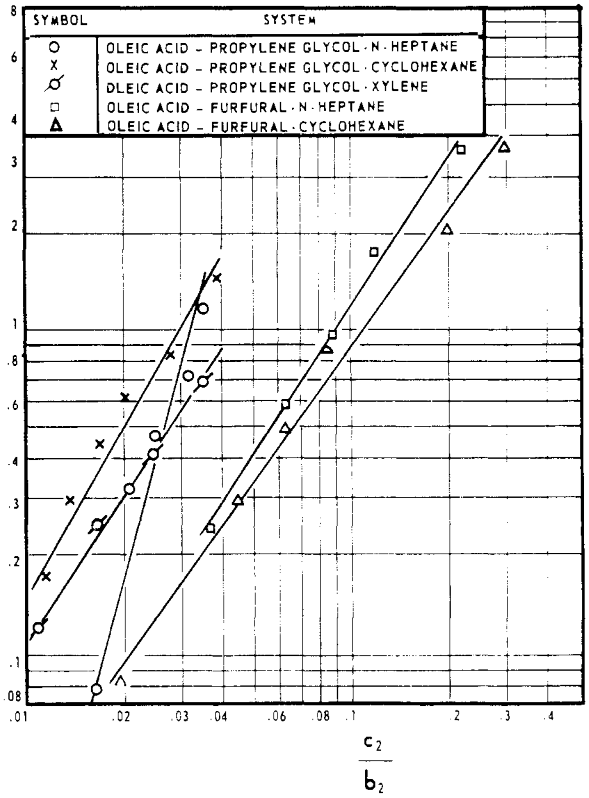

Figure 7. Treybal tie-line correlation plot

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Received for review July 6, 1955. Accepted April 30, 1956

Phase Equilibria in Dimethylhydrazine-Water System

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The design of a distillation process for separating pure asymmetrical dimethylhydrazine from aqueous solution requires vapor-liquid equilibrium data for the binary system dimethylhydrazine-water.

A series of equilibrium vapor and liquid compositions and temperatures at atmospheric pressure were determined experimentally. Plotting these values in the usual ways gave smooth, plausible curves. These data are unusual in that the activity coefficients calculated from them deviate widely from conformity with the Gibbs-Duhem equation. A few additional determinations at 102 mm. of mercury showed the occurrence of a high-boiling azeotrope at reduced pressures.

INTRODUCTION

In one process for the preparation of asymmetrical dimethylhydrazine (DMH), the reaction product consists of the desired dimethylhydrazine in dilute aqueous solution, together with various impurities. If dimethylhydrazine is to be isolated by distillation, the principal problem is separation from water.

Several trial distillations in efficient columns under various operating conditions showed that dimethylhydrazine of satisfactory purity could be taken off as distillate, but with only partial recovery. Evidently, design of an efficient distillation process requires a basic analysis of the