

Liquid-Liquid Equilibrium Data for the System Carbon Tetrachloride-Acetone-Water

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THE equilibrium data as a function of temperature for the immiscible system carbon tetrachloride-acetone-water is presented. This liquid-liquid system was worked out because it is easily analyzed and is noncorrosive to silver and because the lower phase possesses relatively high dielectric strength. These characteristics were required in studying the effect of ultrasonic vibrations on the rate of mass transfer across a liquid-liquid interface where a silver-coated piezoelectric quartz crystal formed the bottom of the extraction column.

Commercial sulfur-free carbon tetrachloride (Imperial Chemical Industries, Ltd.), commercial acetone (Robert Corbett, Pty., Ltd.), and tap water were used in these determinations because their purity exceeds 99% and also because they were to be used in the liquid-liquid extraction studies.

The solubility curve was established by titrating known mixtures of carbon tetrachloride-acetone and water-acetone with the third component to the first permanent turbidity (persisting for 5 minutes with shaking). The two components were carefully weighed into a stoppered constant-temperature, jacketed cylinder. After the addition of small-volume increments of the third component from a buret, the cylinder was stoppered and shaken. When the first permanent turbidity resulted, the specific gravity of the mixture was measured in situ with a Westphal balance. Knowledge of the specific gravity and the volume of the third component, and the weights of the other two, permitted accurate calculation of the mixture composition and fixed a point on the solubility curve. The specific gravities of the saturated

mixtures are plotted against acetone concentrations in Figure 1. The solubility curve is shown in Figure 2, using the conventional three-component diagram. The solubility data at 30° C. are presented in Table I. Knowledge of the specific gravity of any equilibrium phase at 30° C. immediately establishes the composition. Since the solubility curves at different temperatures are almost the same, the analysis of an equilibrium sample at any temperature can be made by determining its specific gravity at 30° C.

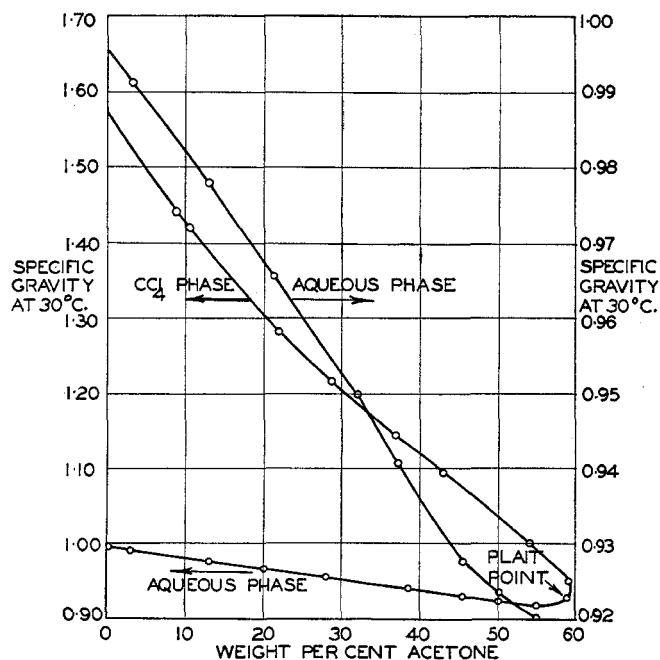


Figure 1. Specific Gravities vs. Acetone Concentration

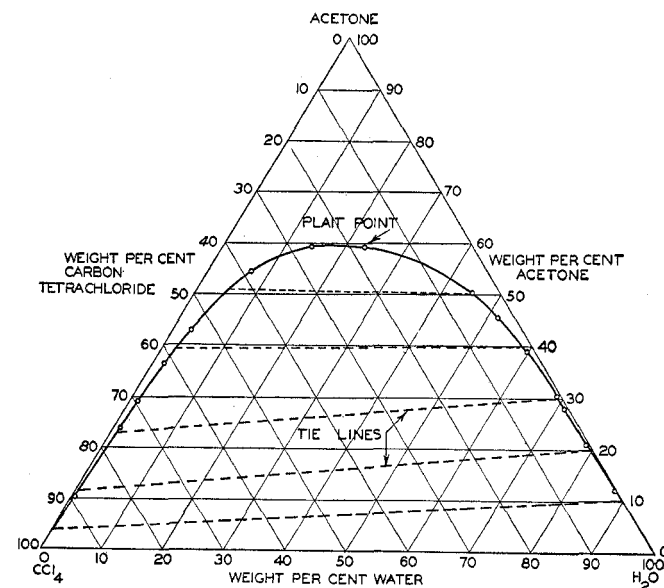


Figure 2. Solubility Curve

TABLE I. SOLUBILITY DATA FOR THE SYSTEM CARBON TETRACHLORIDE-ACETONE-WATER AT 30° C.

Acetone, Wt. %	CCl ₄ , Wt. %	Water, Wt. %	Gravity, 30°/4° C.
00.00	100.00	0.00	1.5750
09.14	90.78	0.08	1.4410
10.58	89.19	0.23	1.4230
22.17	76.98	0.85	1.2852
29.32	69.61	1.07	1.2120
36.40	61.98	1.62	1.1470
42.63	54.67	2.70	1.0936
54.34	38.73	6.93	1.0000
59.46	26.15	14.39	0.9524
59.04	18.01	22.95	0.9330 Plait point
54.83	7.57	37.60	0.9197
50.27	4.69	45.05	0.9235
45.77	2.73	51.50	0.9292
37.32	1.36	61.32	0.9411
31.58	0.69	67.73	0.9494
21.08	0.40	78.52	0.9657
19.82	0.39	79.78	0.9680
12.83	0.28	86.90	0.9780
3.22	0.22	96.56	0.9910
0.00	0.00	100.00	0.9956 (Tables give 0.9960)

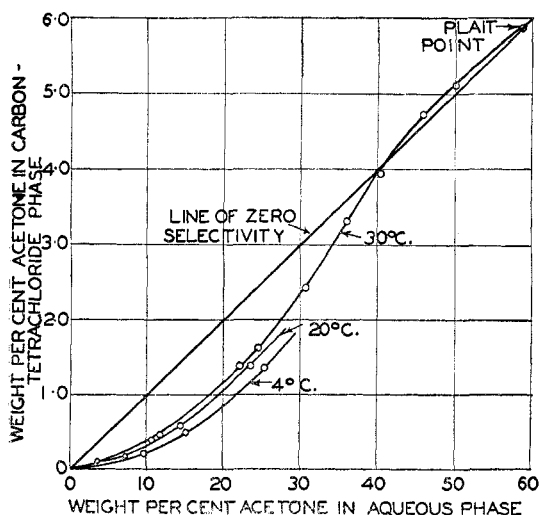


Figure 3. Distribution of Acetone between Carbon Tetrachloride and Water with Temperature

In the tie line determinations, the three components were weighed carefully into the same constant-temperature jacketed cylinder, the relative amounts being chosen to cause the interface to be near the middle. After 5 minutes of vigorous shaking, the phases were settled and the specific gravity of both determined in situ with a Westphal balance. Near the plait point, where the specific gravities of the two phases are almost identical, 3 or 4 hours were required for settling. The specific gravities of the two equilibrium phases were converted to tie line concentrations with Figure 1. The accuracy of the data is verified when the over-all, and the two equilibrium compositions on the triangular graph, fall on the same straight line. A few tie lines are shown in Figure 2. The distribution of acetone between the two

TABLE II. EQUILIBRIUM DISTRIBUTION OF ACETONE BETWEEN CARBON TETRACHLORIDE AND AQUEOUS PHASES

Temp., ° C.	Acetone-CCl ₄ Phase, Wt. %	Acetone-Water Phase, Wt. %
30	0.80	3.35
30	3.95	10.30
30	4.60	11.50
30	13.90	22.30
30	16.50	24.40
30	24.20	30.40
30	33.30	35.80
30	39.60	40.20
30	47.20	46.00
30	51.90	49.80
30	59.04	59.04 Plait point
20.8	2.10	6.90
20.8	6.20	14.30
20.8	13.80	22.50
4	2.21	9.50
4	4.90	14.90
4	9.00	20.60
4	13.80	25.30

phases is shown in Figure 3 at varying temperatures. The equilibrium data are presented in Table II.

This system is solutropic (1) since the distribution favors the aqueous phase at low acetone concentrations, the reverse being true at high concentrations. In Figure 3, the change in distribution between 20° and 30° C. is small and is probably negligible in comparison with the experimental error. When the direction of mass transfer is unimportant as in many liquid-liquid extraction studies, simple operation can be achieved by extracting acetone from the carbon tetrachloride with water, recycling the raffinate, and discharging the extract to the sewer.

LITERATURE CITED

- (1) Smith, A. S., *IND. ENG. CHEM.*, **42**, 1206 (1950).

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Vapor-Liquid Equilibria at 760 Mm. Pressure

2-PROPANOL-METHANOL, 2-PROPANOL-ETHYL ALCOHOL, 2-PROPANOL-PROPANOL, AND 2-PROPANOL-2-BUTYL ALCOHOL SYSTEMS

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ADVENT of the various processes for oxidation of hydrocarbons has posed numerous problems of separation of the components present in the process products. Fractional distillation is one of the common separation procedures and design of fractional distillation equipment requires a knowledge of vapor-liquid equilibrium data of the systems of multicomponent mixtures produced in the oxidation processes. The data presented here are binary vapor-liquid equilibrium data on 2-propanol (isopropanol)-alcohol binaries and are the result of one of a series of investigations concerned with the experimental study of vapor-liquid equilibria of the oxidized hydrocarbons.

The materials used for vapor-liquid equilibrium determinations must be of high purity. The methanol and ethyl alcohol

were obtained pure, but the 2-propanol, propanol, and isobutyl alcohol (isobutanol) required further purification before use. They were fractionally distilled in a 5-foot, 0.75-inch diameter, glass distillation column packed with glass helices. In each case, both ends of the original charge were discarded and the middle fraction was retained until the desired purity was obtained.

Purity was determined by boiling point and refractive index. The refractive index was determined at 20° C. with a Bausch and Lomb refractometer. The boiling points were determined in a Cottrell boiling point apparatus. The total pressure was maintained at 760 mm. of mercury by the use of air pressure.

Comparison of the experimental refractive indexes and boiling points on the materials used in this investigation with values reported in the literature on pure compounds is presented in Table I.

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