Phase Equilibria Data for the System *n*-Propyl Alcohol–Water-Nitroethane

JAMES W. MALONE and ROY W. VINING¹ Louisiana Polytechnic Institute, Ruston, La.

> The ternary-liquid solubility and equilibria data for the system *n*-propyl alcoholwater-nitroethane are reported at 25° C. The solubility data were determined by the "cloud point" method. Weight percentages of components on the binodal curve vs. both density and refractive index are presented along with the distribution curve.

THE PHASE equilibria data for the ternary liquid system n-propyl alcohol-water-nitroethane were determined experimentally as the initial phase of a continuing fundamental inquiry into liquid-liquid equilibria involving commercial nitroparaffins as solvents. A literature review of published ternary systems and solubility tables (3, 5-9) shows a need for more solubility data with these compounds as solvents.

MATERIALS

The *n*-propyl alcohol used in these determinations was Fisher certified reagent grade with a boiling range of 97.0° to 97.4°C. The nitroethane used was commercial grade of essentially 93.9% purity as produced in bulk at the Commercial Solvents Corp. plant at Sterlington, La. Impurities were nitromethane (2.3%) and 2-nitropropane (3.8%). No attempt was made to purify the nitroethane further, as any large scale extraction application would be made with commercially available solvent rather than with extremely high purity material. Distilled water was used as the third component. The refractive indices (n_D 25° C.) were 1.3871 and 1.3890 for *n*-propyl alcohol and nitroethane, respectively.

PROCEDURE AND RESULTS

Solubility Data. A measured amount of nitroethane was added to a tared, 50-ml. Erlenmeyer flask fitted with a glass stopper and titrated to the cloud point with distilled water. To each succeeding sample of nitroethane a measured quantity of n-propyl alcohol was added, and the mixture was titrated to the cloud point with distilled water. As the cloud point was closely approached, in order to overcome any heat effects, the sample was allowed to stand at room temperature. This procedure was followed until approximately equal weights of nitroethane and water were present. The procedure was then reversed using mixtures of water and n-propyl alcohol and titrating with nitroethane. Weight measurements were made using an analytical balance. Titrations were performed using 5-ml. certified microburets graduated in 0.01-ml. divisions. The temperature of the room was maintained at $25^{\circ} \pm 1^{\circ}$ C. by air conditioning. Densities were determined by use of 10-ml. pycnometers (with thermometer) calibrated at 25°C. The pycnometer containing the sample was placed in a bath maintained at 22°C. A hypodermic assembly was used to maintain the sample level in the pycnometer arm. The pycnometer was removed from the bath, wiped with lens paper, and warmed to just below 25° C. Weighings were made at 25° C. An Abbé refractometer using cooling water from a bath maintained at $77^{\circ} \pm 0.02^{\circ}$ F. (25° C.) was used for refractive index readings.

¹Present address: Texas Division, Dow Chemical Co., Freeport, Tex.

Ta	ble	1. C	Composit	ion, De	ensit	y, a	nd Refr	active	
ndex	at	the	Binodal	Curve	for	the	System	n-Prop	yl
	Α	lcoh	ol–Wate	er-Nitro	betha	ane	at 25°	С.	

We	eight Per Co	Density.		
n-Propyl alcohol	Water	Nitroethane	Grams/Ml. at 25° C.	Refractive Index, $n_{\rm D}^{25}$
0.00	0.86	99.14	1.0424	1.3873
3.78	1.24	94.98	1.0299	1.3880
7.70	1.90	90.40	1.0195	1.3874
11.55	3.08	85.37	1.0065	1.3863
15.45	4.32	80.23	0.9956	1.3859
19.25	5.98	74.77	0.9843	1.3852
22,94	7.75	69.31	0.9744	1.3848
26.61	9.96	63.43	0.9642	1.3832
29.70	12.20	58.10	0.9569	1.3822
32.66	15.42	51.92	0.9488	1.3811
35.06	18.59	46.35	0.9429	1.3790
36.83	23.68	39.49	0.9378	1.3774
37.50	29.90	32.60	0.9352	1.3750
36.47	37.62	25.91	0.9372	1.3701
34.58	43.22	22.20	0.9406	1.3682
31.96	49.39	18.65	0.9457	1.3649
29.39	55.01	15.60	0.9509	1.3624
25.84	61.66	12.50	0.9586	1.3583
22.93	67.09	9.98	0.9645	1.3564
19.46	73.06	7.48	0.9714	1.3536
15.63	78.79	5.58	0.9785	1.3496
11.70	83.07	5.23	0.9852	1.3461
7.68	87.52	4.80	0.9912	1.3427
3.92	91.25	4.83	0.9968	1.3388
0.00	94.95	5.05	1.0027	1.3353

Table II. Experimental Tie Line Data at 25° C.

	Density	Weight Per Cents, Figure 2			
Sample No.	Grams/Ml. at 25° C.	n-Propyl alcohol	Water	Nitroethane	
	Ni	troethane Lay	er		
1 2 3ª 4 5 6	$\begin{array}{c} 1.0269 \\ 1.0077 \\ 0.9840 \\ 0.9591 \\ 0.9392 \\ 0.9351 \end{array}$	4.9 11.3 19.4 28.6 36.3 37.6	$1.4 \\ 3.0 \\ 5.9 \\ 11.6 \\ 22.0 \\ 30.7$	$\begin{array}{c} 93.5 \\ 85.8 \\ 74.6 \\ 59.7 \\ 41.6 \\ 31.7 \end{array}$	
		Water Layer			
1 2 3ª 4 5 6	0.9951 0.9881 0.9840 0.9801 0.9739 0.9660	5.0 9.8 12.4 14.7 18.1 22.1	90.2 85.5 82.5 79.8 75.0 68.5	4.8 5.0 5.2 5.6 6.9 9.4	

^a Isopycnic.



Figure 1. Solubility diagram and tie lines at 25° C.



Figure 2. Weight percentages of components as functions of density along the binodal curve at 25° C.





Tie Lines. The three components were added one at a time to a tared, 50-ml. Erlenmeyer flask fitted with a glass stopper until approximately equal volumes of the two liquid phases were obtained. The mixture was then agitated vigorously and allowed to stand. It was then again agitated and allowed to separate into phases. The two layers were removed by use of hypodermic assemblies. Density and refractive index determinations were then made as outlined previously. Densities were used to determine compositions of the equilibrium phases. Refractive index readings were used primarily to identify phases, particularly in the region near the isopycnic (4), and secondarily to check compositions from density measurements.

Figure 1 and Table I show the compositions at the binodal curve. Figure 2 presents the variation of density of the mixtures of the components as a function of the binodal curve compositions and Figure 3 is a similar plot of refractive index. Basic data for the distribution curve, Figure 4, and the Bachman plot (2), Figure 5, were experimental tie line data (Table II). Additional points to complete



Figure 5. Bachman plot of tie line data

the distribution curve and define the plait point were extrapolated using Alders' method (1).

LITERATURE CITED

- (1) Alders, L., "Liquid-Liquid Extraction," Elsevier, New York, 1955.
- (2) Bachman, I., Ind. Eng. Chem., Anal. Ed. 12, 38-9 (1940).
- (3) Fowler, A.R., Hunt, H., Ind. Eng. Chem. 33, 90-5 (1941).
- (4) Francis, A.W., *Ibid.*, 45, 2798 (1953).
- (5) Francis, A.W., J. CHEM. ENG. DATA 11, 234-7 (1966).
- (6) Francis, A.W., "Liquid-Liquid Equilibriums," Wiley, New York, 1963.
- (7) Hankinson, R.W., Thompson, D., J. CHEM. ENG. DATA 10, 18 (1965).
- (8) Schumacher, J.E., Hunt, H., Ind. Eng. Chem. 34, 701 (1942).
- Seidell, A., "Solubilities of Organic Compounds," Van Nostrand, New York, 1941.

RECEIVED for review January 27, 1966. Accepted April 17, 1967.

Diffusivities of Some Binary Liquid Systems Using a Diaphragm Cell

MARIE JOSEPH AMOURDAM and G. S. LADDHA

Department of Chemical Engineering, Alagappa Chettiar College of Technology, Madras 25, India

Diffusivity data on 25 binary systems were obtained by using a diaphragm cell at 30 $^{\circ}$ C.

PRESENT UNDERSTANDING of diffusion is far from satisfactory, and reliable data are still scarce and fragmentary even for dilute binary solutions. The lack of a usable kinetic theory of liquids has resulted in dependence on simplified physical models and semiempirical correlations to predict diffusivities, and these can be used only with

certain reservations. There is, therefore, need for accurate experimental measurements. Because of the simplicity in construction and fairly high accuracy, a diaphragm cell has been used in many investigations for measuring binary liquid diffusivities. The present investigation reports data for 25 binary systems using a diaphragm cell at 30° C.