Greek:

γ activity coefficient

Subscripts:

1 more volatile component

Literature Cited

(1) Ewell, R. H., Harrison, J. M., Berg, L., *Ind. Eng. Chem.*, **36**, 871 (1944).
(2) Hala, E., Pick, J., Fried, V., Vilim, O., "Vapour-Liquid Equilibrium", 2d English ed, Pergamon Press, London, 1967, p 123.
(3) Hala, E., Wichterle, I., Polak, J., Boublik, T., "Vapour-Liquid Equilibrium Data at Normal Pressures", Pergamon Press, London, 1968.
(4) Herington, E. F. G., *J. Inst. Pet. (London)*, **37**, 457 (1951).
(5) Horsely, L. H., "Azeotropic Data", American Chemical Society, Washington, D.C., 1952.
(6) Ward, S. H., Ph.D. Thesis, University of Texas, 1952.
(7) Wiessberger, A., Proskaners, E. D., Riddlick, T. A., Toops, E. E., "Organic Solvents", 2d ed, Interscience Publishers, London, 1955.
(8) Wohl, K., *Trans. Am. Inst. Chem. Eng.*, **42**, 215 (1946).

Received for Review December 15, 1975. Accepted August 11, 1976.

Solubility in the System Sodium Orthophosphate, Acetone, and Water

Zvi Nirenberg, Beatrice Solenchyk, and Isaac Yaron*

Research & Development Authority, Ben-Gurion University, Beer-Sheva, Israel

Equilibrium in the ternary solid–liquid system of sodium orthophosphate, acetone, and water has been determined. Solubility isotherms for 10.0, 20.0, 31.5, and 40 °C are presented.

There has been considerable interest recently in processes of selective separation of inorganic salts from aqueous solutions through the addition of polar organic solvents. Very few data are available on the solid–liquid equilibrium of ternary and quaternary systems containing a polar organic component. Even less data are available for systems containing such a cheap and readily recoverable solvent as acetone. Thus, the well-known compilations of Linke and Seidel (*5*) and Stephen and Stephen (*6*), which comprise thousands of solubility tables, contain hardly a dozen or so such tables for systems including acetone. Emons et al. (*2*), Emons et al. (*3*), Emons et al. (*4*), Winkler and Emons (*7*), Cohen et al. (*1*), and others have determined equilibrium data for acetone-containing ternary and quaternary systems in relation to development of specific processes. In the course of our work on the selective separation of phosphorus compounds, we have investigated and present solid–liquid equilibrium data for the system sodium orthophosphate, acetone, and water at various temperatures.

Experimental Section

Materials. Analytical grade $Na_3PO_4 \cdot 12H_2O$ from Merck and acetone from Frutarom were used. Purity of the reagents was rechecked. In all experiments doubly distilled water was used.

Procedure

Samples of acetone–water mixtures (50 ml) with varying acetone/water ratios were introduced into rubber-capped 100-ml flasks and thermostated in a Haake KT33 bath. Temperature was controlled within 0.1 °C. In experiments conducted at 31.5 °C small known amounts of sodium orthophosphate were progressively added to the acetone–water mixtures until saturation was reached, as indicated by the first appearance of a solid phase. When acetone/water ratios exceeded 2/3, $Na_3PO_4 \cdot 12H_2O$ was added as a 9% aqueous solution in aliquots of 0.5 ml with a precision syringe. In other experiments, conducted at 10, 20, and 40 °C, excess $Na_3PO_4 \cdot 12H_2O$ was always taken.

The saturated systems were equilibrated for 24 h and agitated periodically. Preliminary tests indicated that this duration was sufficient to reach equilibrium. Samples of the liquid phases (4.68 ml) were withdrawn with a calibrated syringe, and diluted immediately. They were analyzed for Na^+, P_2O_5, and acetone. Specific gravity of the liquid systems was determined. X-ray diffraction showed that the solid phase was $Na_3PO_4 \cdot 12H_2O$ and remained unaltered.

Phosphorus was determined as P_2O_5 by the vanadomolybdate method, using a Bauch and Lomb Spectronic 200 UV spectrophotometer at 426 mμ. Accuracy of determination of P_2O_5 was ± 0.25 ppm at concentrations of 50 ppm. Concentration of acetone was measured by iodometric titration with an accuracy of ± 0.048 mg of acetone. Sodium was determined using a Pye Unicam SP90 atomic absorption spectrometer with an accuracy of ± 2 ppm at 200 ppm full scale.

Results

Solubility data for sodium orthophosphate in acetone–water mixtures are summarized in Table I. Solubility isotherms at 10, 20, 31.5, and 40°C are presented in Figure 1. Compositions of the liquid phases in equilibrium with solid $Na_3PO_4 \cdot 12H_2O$ are

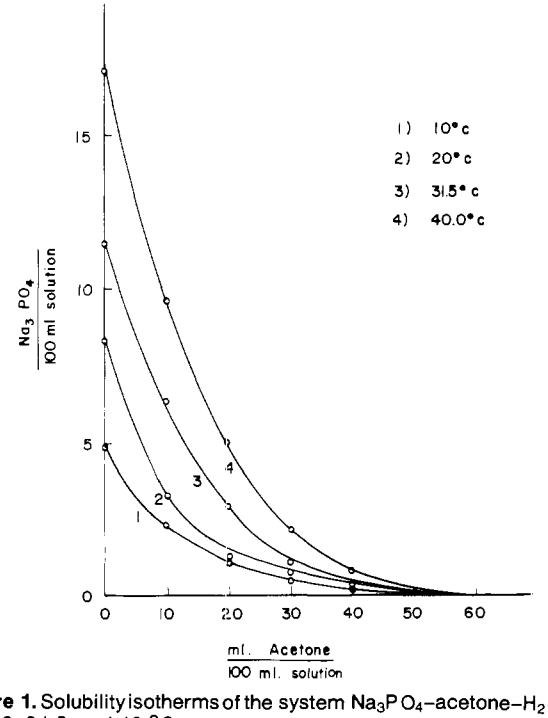

Figure 1. Solubility isotherms of the system Na_3PO_4–acetone–H_2O for 10, 20, 31.5, and 40 °C.

Table I. Solubility Isotherms of the System Na_3PO_4, Acetone, Water for 10, 20, 31.5, and 40 °C at Various Ratios of Acetone to 100 ml of Solution

	Ml of acetone to 100 ml of solut	10 °C		20 °C		31.5 °C		40 °C	
		Na_3PO_4 (g/100 ml)	Sp. gr	Na_3PO_4 (g/100 ml)	Sp. gr	Na_3PO_4 (g/100 ml)	Sp. gr	Na_3PO_4 (g/100 ml)	Sp. gr
1	0	4.86	1.04	8.35	1.08	11.50	1.07	17.05	1.13
2	10	2.37	0.98	3.30	1.02	6.39	1.05	9.67	1.10
3	20	1.05	0.98	1.25	0.98	3.02	1.00	5.11	1.04
4	30	0.47	0.97	0.72	0.95	0.61	0.96	2.25	1.00
5	40	0.19	0.95	0.16	0.94	0.32	0.93	0.87	0.97
6	50	Trace	—	Trace	—	0.18	0.90	0.26	0.94
7	60	0	—	0	—	0.07	0.90	0.08	0.91
8	100	0	—	0	—	0	—	0	—

Table II. Ternary Solid Liquid Equilibrium System Na_3PO_4, CH_3COCH_3, H_2O: Temp 10, 20, 31.5, 40 °C

10 °C			20 °C			31.5 °C			40 °C		
Na_3PO_4 % wt	Acetone % wt	H_2O % wt	Na_3PO_4 % wt	Acetone % wt	H_2O % wt	Na_3PO_4 % wt	Acetone % wt	H_2O % wt	Na_3PO_4 % wt	Acetone % wt	H_2O % wt
4.65	—	95.35	7.73	—	92.27	10.74	—	89.26	15.10	—	84.90
2.43	8.10	89.47	3.20	7.98	88.82	6.00	6.57	87.43	8.80	5.95	85.25
1.06	15.75	83.19	1.30	16.75	81.95	3.00	13.70	83.30	4.90	14.10	81.00
0.50	24.10	75.40	0.75	26.40	72.85	0.62	24.87	74.50	2.25	23.50	74.25
0.20	33.30	66.50	0.17	32.46	67.37	0.34	34.40	65.06	0.90	34.10	65.00
						0.19	42.60	57.20	0.27	44.50	55.23
						0.07	52.00	47.93	0.09	52.00	47.91

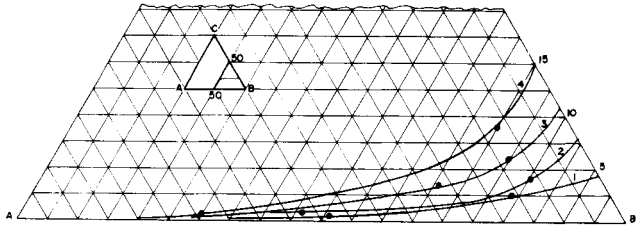

Figure 2. Ternary solid–liquid equilibrium system isotherm diagram: A, acetone; B, water; C, Na_3PO_4; 1, 10 °C; 2, 20 °C; 3, 31.5 °C; 4, 40 °C.

given in Table II from which the phase diagram of Figure 2 was drawn.

As indicated by the results, the solubility of sodium orthophosphate decreases rapidly with the addition of acetone. Increase in temperature causes increase in the solubility of $Na_3PO_4 \cdot 12H_2O$ in both water and in acetone–water mixtures.

It should be noted, that in view of the discrepancies in the published literature with regard to solubility of sodium phosphate in water at various temperatures, particular attention has been devoted in this study to the careful determination of these data.

Literature Cited

(1) Cohen, H. D., Yaron, I., Wolf, D., *J. Appl. Chem.*, **13,** 253 (1963).
(2) Emons, H. H., Dumke, D., Fortsch, M., *Wiss. Z. Tech. Hochsch. Chem., Leuna-Merseburg,* **8,** 195 (1966).
(3) Emons, H. H., Roser, H., Lauterbach, J., *Z. Anorg. Allg. Chem.*, **353,** 135 (1967).
(4) Emons, H. H., Roser, H., Roshke, E., *Z. Anorg. Allg. Chem.*, **373,** 281 (1970).
(5) Linke, W. F., Seidel, A., "Solubilities. Inorganic and Metal-Organic Compounds", Van Nostrand, Princeton, N.J., 1958.
(6) Stephen, H., Stephen, T., "Solubilities of Inorganic and Organic Compounds", Pergamon Press, Oxford, 1963.
(7) Winkler, F., Emons, H. H., *Krist. Tech.*, **4,** 511 (1963).

Received for review January 22, 1976. Accepted September 8, 1976.