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Solubilities of 3-Carboxy-3-hydroxypentanedioic Acid in Ethanol, Butan-1-ol, Water, Acetone, and Methylbenzene

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S Supporting Information

ABSTRACT: The solubility of 3-carboxy-3-hydroxypentanedioic acid in ethanol, butan-1-ol, water, acetone, and methylbenzene has been measured, respectively, at temperatures ranging from (288.15 to 333.15) K by a static analytical method. The concentrations of 3-carboxy-3-hydroxypentanedioic acid in saturated solution were analyzed by an Abbe refractometer. A modified Apelblat equation was used to correlate the experimental data.

INTRODUCTION

3-Carboxy-3-hydroxypentanedioic acid (CAS No. 77-92-9) is of great importance commercially in that it is widely used in the manufacturing of food industry as food additives, environmental protection as desulfurizing agents,[1] chemical industry as analytical agents,[2] a cosmetics ingredient, medicine, and so forth. Moreover, the derivatives[3] of 3-carboxy-3-hydroxypentanedioic acid are important fine chemicals. The solubility of solids in liquids is one of the most important parameters and is significant for the development of the solution theory. However, limited solubility data of 3-carboxy-3-hydroxypentanedioic acid have been reported in the literature. In this paper, the solubility of 3-carboxy-3-hydroxypentanedioic acid in ethanol, butan-1-ol, water, acetone, and methylbenzene was systematically measured by a static analytical method.

EXPERIMENTAL SECTION

Chemicals. Analytically pure grade butan-1-ol, dehydrated ethanol, acetone, and methylbenzene were purchased from Shanghai Sinopharm Chemical Reagent Co. Ltd. All of the above solvents were refluxed over freshly activated CaO for 2 h and then fractionally distilled. The mass fractions of purities of the organic solvents were determined by gas chromatography (GC-17A, Shimadzu, Japan), 99.9 % in mass fraction. Laboratory deionized water was used in this work. 3-Carboxy-3-hydroxypentanedioic acid (> 99.5 %, mass fraction) obtained from Shanghai Sinopharm Chemical Reagent Co. Ltd. was purified by recrystallization twice from water and dried at 393.15 K. The sample was kept in a desiccator with dry silica gel.

Apparatus and Procedures. The experimental solubility of 3-carboxy-3-hydroxypentanedioic acid in solvents in the temperature range from (288.15 to 333.15) K was measured by a static analytical method described in other literature[4] and explained briefly here. The experimental saturated solutions were prepared by excess solute, 3-carboxy-3-hydroxypentanedioic acid, in glass beakers containing the solvent. Solubilities were determined by equilibrating the solute with solvent in a water jacketed beaker with magnetic stirring (Zhengzhou Greatwall Scientific Industrial and Trade Co. Ltd., P. R. China) in a constant-temperature water bath (± 0.05 K) (Shanghai Experimental Instrument Co. Ltd., P. R. China) for at least 48 h. Attainment of equilibrium was verified both by repetitive measurement after a minimum of 1 additional day and by approaching equilibrium from supersaturation by pre-equilibrating the solutions at a higher temperature. The actual temperature in the glass vessel was monitored by a mercury thermometer with an uncertainty of 0.05 K. Portions of 3-carboxy-3-hydroxypentanedioic acid saturated solutions were transferred from the internal glass tube to the volumetric flasks to determine the amounts of samples diluted quantitatively with solvent mixtures by an Abbe refractometer (Shanghai Precision & Scientific Instrument Co. Ltd., P. R. China). The relative uncertainty of the experimental solubility values is less than ± 0.05.

RESULTS AND DISCUSSION

An Abbe refractometer was chosen to determine the compositions of a saturated solution of 3-carboxy-3-hydroxypentanedioic acid in the solvent. To check the reliability of the experimental method, known masses of 3-carboxy-3-hydroxypentanedioic acid were completely dissolved in the five solvents, and the concentrations of solution were measured by the Abbe refractometer.[5] The relationships between refraction rate and mole fraction of solutions are linear, respectively. The correlation coefficients of the relationships are more than 0.99 .

The solubilities of 3-carboxy-3-hydroxypentanedioic acid in butan-1-ol, ethanol, water, acetone, and methylbenzene reported in Table 1 represent an average of three measurements with a reproducibility better than 97 %. From the results, we can see that

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Table 1. Solubility of 3-Carboxy-3-hydroxypentanedioic Acid in Ethanol, Butan-1-ol, Water, Acetone, and Methylbenzene

	x_s				
T/K	butan-1-ol	ethanol	water	acetone	methylbenzene
288.15	0.05885	0.2462	0.1105	0.05552	0.05670
291.15	0.06626	0.2588	0.1169	0.06339	0.06003
294.15	0.07463	0.2708	0.1235	0.07226	0.06353
297.15	0.08385	0.2826	0.1301	0.08224	0.06720
299.15	0.09057	0.2895	0.1347	0.08960	0.06973
301.15	0.09781	0.2962	0.1393	0.09755	0.07235
303.15	0.1056	0.3027	0.1445	0.1061	0.07505
305.15	0.1139	0.3091	0.1495	0.1155	0.07783
307.15	0.1223	0.3149	0.1545	0.1254	0.08069
309.15	0.1323	0.3201	0.1594	0.1363	0.08363
311.15	0.1424	0.3253	0.1648	0.1478	0.08667
313.15	0.1534	0.3297	0.1700	0.1605	0.08979
315.15	0.1651	0.3341	0.1754	0.1740	0.09301
317.15	0.1773	0.3369	0.1808	0.1887	0.09632
319.15	0.1908	0.3407	0.1865	0.2044	0.09972
323.15	0.221	0.3458	0.1977	0.2395	0.1068
327.15	0.2538	0.3492	0.2094	0.2800	0.1143
330.15	0.283	0.3506	0.2184	0.3145	0.1203
333.15	0.3126	0.3509	0.2275	0.3527	0.1265

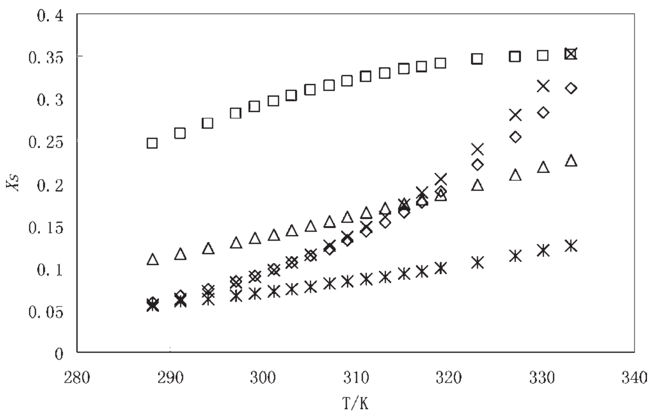

Figure 1. Mole fraction solubility of 3-carboxy-3-hydroxypentanedioic acid in different organic solvents: □, ethanol; ×, acetone; ◇, butan-1-ol; △, water; ∗, methylbenzene.

the solubility of 3-carboxy-3-hydroxypentanedioic acid in solvents increases as the temperature increases. The solubility data of 3-carboxy-3-hydroxypentanedioic acid in water are similar to the literature.[6]

The relationship between temperature and solubility of 3-carboxy-3-hydroxypentanedioic acid is correlated with the modified Apelblat equation where x_s and T are the mole fraction of the saturated solute and absolute temperature, respectively, and a, b, and c are empirical constants.[7]

$$\ln x_s = a + \frac{b}{T/\mathrm{K}} + c \ln T/\mathrm{K} \qquad (1)$$

The experimental data of mole fraction solubility in Table 1 were correlated with eq 1 and plotted as shown in Figure 1, whereas the parameter values of a, b, and c and the root-mean-square deviation (rmsd) are given in Table 2. The rmsd is defined as

Table 2. Regression Curve Coefficients in eq 1 for 3-Carboxy-3-hydroxypentanedioic Acid Solubility in Ethanol, Butan-1-ol, Water, Acetone, and Methylbenzene

solvent	a	b	c	10^3 rmsd
butan-1-ol	−68.0083	−1.4859	11.5089	0.3393
ethanol	218.6369	−10746.4158	−32.2672	0.2197
water	14.4831	−2062.8535	−1.6823	0.1022
acetone	−71.5624	−160.2348	12.2235	0.05185
methylbenzene	−33.8263	−15.9867	5.4757	0.06713

$$\mathrm{rmsd} = \left[\frac{1}{N-1}\sum_{j}^{N}(x_{s,j} - x_{s,j}^{\mathrm{calcd}})^2\right]^{1/2} \qquad (2)$$

where N is the number of experimental points; $x_{s,j}^{\mathrm{calcd}}$ is the solubility calculated from eq 1; and $x_{s,j}$ is the experimental value of solubility.

■ CONCLUSION

The solubilities of 3-carboxy-3-hydroxypentanedioic acid in ethanol, butan-1-ol, water, acetone, and methylbenzene have been measured, respectively, at temperatures ranging from (288.15 to 333.15) K by a static analytical method plotted as shown in Figure 1. The solubilities of 3-carboxy-3-hydroxypentanedioic acid in solvents increase as the temperature increases. The modified Apelblat equation was employed to correlate the experimental data with good agreement.

■ ASSOCIATED CONTENT

Ⓢ Supporting Information. This material is available free of charge via the Internet at http://pubs.acs.org.

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