Solubility of *p*-Aminobenzenesulfonamide in Different Solvents from (283.15 to 323.15) K

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The solubility of *p*-aminobenzenesulfonamide in given solvents was measured at temperature ranging from (283.15 to 323.15) K at atmosphere pressure. The laser monitoring observation technique was used to determine the disappearance of the solid phase in a solid + liquid mixture. The results of these measurements were correlated by the modified Apelblat equation.

Introduction

p-Aminobenzenesulfonamide, the first generation of antibiotics, is an important intermediate of sulfa drugs and is usually used as a veterinary drug and an antiphlogistic medicine.[1] This drug is widely applied in successfully treating many major diseases such as malaria, tuberculosis, and leprosy. *p*-Aminobenzenesulfonamide is a white powdered crystal, and the chemical structure is shown in Figure 1.

Crystallization processes are the critical steps that determine the quality of the final product. The solubility of solid compounds in solvents plays a crucial role in determination of suitable solvents and the development and operation of crystallization processes.[2,3] With regard to *p*-aminobenzenesulfonamide, it is instructive to look at its structure and ask whether or not the selected solvent should be reasonable as a solvent or antisolvent during crystallizing this substance. From Figure 1, it can be seen that there are several polar bonds in *p*-aminobenzenesulfonamide, the amino group, and the sulfonamide group. Because of these groups, *p*-aminobenzenesulfonamide has some polar character. So even though the benzene ring part of *p*-aminobenzenesulfonamide is quite nonpolar, *p*-aminobenzenesulfonamide has an intermediate polarity because of the polar groups. In addition, the amino groups and the oxygen atoms in *p*-aminobenzenesulfonamide can form hydrogen bonds with alcohols and ketones, which can increase the solubility of *p*-aminobenzenesulfonamide in these types of solvents. There are some equilibrium solubility data published in the literature on the solubility of *p*-aminobenzenesulfonamide. Kienle and Sayward studied the solubility of *p*-aminobenzenesulfonamide in water at the temperatures from (296 to 323) K.[4] Jin et al. determined the solubility of *p*-aminobenzenesulfonamide in supercritical carbon dioxide with acetone as a cosolvent.[1] Literature on the solubility of *p*-aminobenzenesulfonamide in common solvents is scarce. To the best of our knowledge, there are no solubility studies on *p*-aminobenzenesulfonamide available in polar organic solvents.

In this paper, solubility measurement of *p*-aminobenzenesulfonamide in acetone, propan-1-ol, propan-2-ol, butan-1-ol, and butan-2-ol from (283.15 to 323.15) K was performed. Experimental data were correlated by the modified Apelblat equation.[5]

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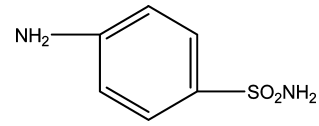

Figure 1. Chemichal structure of *p*-aminobenzenesulfonamide.

Table 1. Mole Fraction Solubility of *p*-Aminobenzenesulfonamide in Different Solvents from (283.15 to 323.15) K

T/K	x_1 10^3	$100(x_1 - x_1^{calcd})/x_1$	*T*/K	x_1 10^3	$100(x_1 - x_1^{calcd})/x_1$
Acetone			Propan-1-ol		
283.45	89.72	−0.632	283.35	1.514	−2.639
288.45	91.78	−0.120	288.55	1.973	3.099
293.35	94.52	0.673	293.15	2.286	0.054
298.15	96.92	0.719	298.35	2.715	−1.552
303.15	99.41	0.342	303.15	3.326	1.618
308.15	98.34	−0.861	308.15	3.862	−0.702
313.15	103.7	−0.755	313.15	4.521	−1.371
318.15	109.9	−0.329	318.15	5.455	1.512
323.15	115.6	0.596	323.15	6.221	−0.497
Propan-2-ol			Butan-1-ol		
283.25	0.5335	−2.986	283.15	0.7705	1.311
288.95	0.8142	−2.544	288.75	1.016	−1.566
293.65	1.053	−9.904	293.25	1.275	−0.608
298.15	1.638	4.927	298.25	1.662	1.409
303.15	2.124	−1.208	303.15	2.027	−1.194
308.15	3.095	5.879	308.15	2.553	0.635
313.15	3.835	−1.469	313.15	3.094	−0.474
318.15	4.985	−2.667	318.15	3.781	0.461
323.15	6.738	1.078	323.15	4.502	−0.286
Butan-2-ol			Butan-2-ol		
283.25	0.3543	−3.067	308.15	1.393	3.472
288.15	0.4586	−1.949	313.15	1.748	0.677
293.25	0.5973	−2.410	318.15	2.347	4.409
298.15	0.7618	−4.452	323.15	2.823	−2.712
303.15	0.9815	−5.273			

Experimental Section

The white powdered crystals of *p*-aminobenzenesulfonamide, supplied by the Beijing Chemical Reagent Factory, were used, and its mass fraction purity was higher than 99.0 %. The solvents were purchased from Tianjin Kewei Co. of China and were of analytical reagent grade.

The solubility of *p*-aminobenzenesulfonamide in different solvents was measured by the method which is described in the literature.[6−8] During the measurement, predetermined excess amounts of solute and binary solvents of known masses were transferred to the equilibrium vessel. The contents of the vessel

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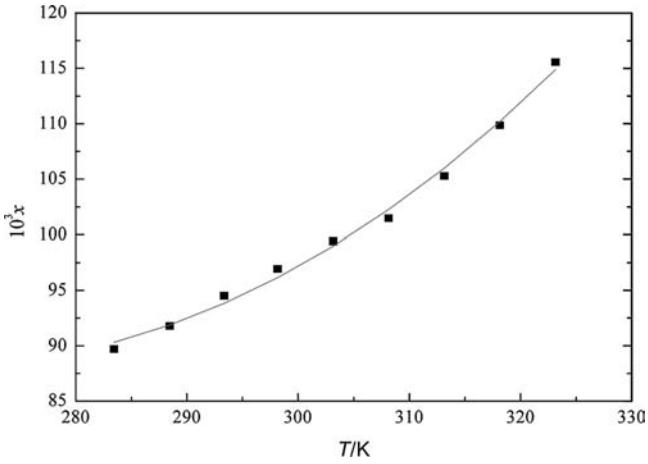

Figure 2. Mole fraction solubility of *p*-aminobenzenesulfonamide in acetone from (283.15 to 323.15) K.

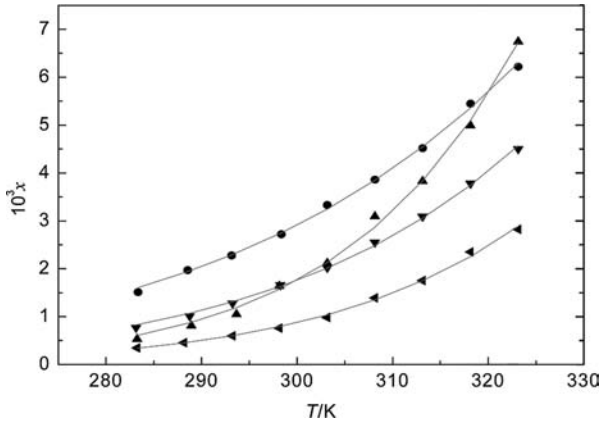

Figure 3. Mole fraction solubility of *p*-aminobenzenesulfonamide in the other four solvents from (283.15 to 323.15) K. ●, propan-1-ol; ▲, propan-2-ol; ▼, butan-1-ol; solid triangle pointing left, butan-2-ol.

Table 2. Curve-Fitting parameters of *p*-Aminobenzenesulfonamide in Different Solvents from (283.15 to 323.15) K

solvent	*A*	*B*	*C*	10^2 rsmd
acetone	−108.13	4294.7	16.039	1.716
propan-1-ol	32.750	−4464.8	−4.1550	4.524
propan-2-ol	131.50	−11089	−17.688	0.993
butan-1-ol	129.67	−9587.7	−18.242	0.993
butan-2-ol	−145.69	2162.5	23.044	0.603

were stirred at the required temperature for 30 min. Then, additional known mass solvent was introduced into the cell. When the last portion of solute just disappeared, the intensity of the laser beam penetrating the vessel reached a maximum, and the solvent mass consumed in the measurement would be recorded. Together with the mass of solute, the solubility would be determined. The mole fraction solubility of *p*-aminobenzenesulfonamide can be obtained as follows

$$x_1 = \frac{m_1/M_1}{m_1/M_1 + m_2/M_2} \quad (1)$$

In which m_1 and m_2 represented the masses of solute and solvent. M_1 and M_2 are the molar mass of solute and solvent, respectively.

All the experiments were conducted three times, and the mean values were used to calculate the mole fraction solubility. The relative uncertainty of the experimental mole fraction solubility is within 0.005.

Results and Discussion

The measured solubility of *p*-aminobenzenesulfonamide from (283.15 to 323.15) K is listed in Table 1 and graphically plotted in Figure 2 and Figure 3. x_1 expresses the experimental solubility value. x_1^{calcd} expresses the calculated solubility value. The solubility data in Table 1 are described by the modified Apelblat equation.

$$\ln x = A + B/T(\mathrm{K}) + C \ln T(\mathrm{K}) \quad (2)$$

where x is the mole fraction solubility of *p*-aminobenzenesulfonamide; T is the absolute temperature; and A, B, and C are the parameters. The values of parameters A, B, and C and the root-mean-square relative deviations (rmsd) are listed in Table 2. The rmsd is defined as

$$\mathrm{rmsd} = \left\{ \frac{1}{N} \sum_{i=1}^{N} \left(\frac{x_i^{calcd} - x_i}{x_i} \right)^2 \right\}^{1/2} \quad (3)$$

where N is the number of experimental points.

Conclusions

From Table 1, Figure 2, and Figure 3, we can draw the following conclusions: (1) The solubility of *p*-aminobenzenesulfonamide in acetone, propan-1-ol, propan-2-ol, butan-1-ol, and butan-2-ol is a function of temperature, and it increases with an increase in temperature. (2) The mole fraction solubility of *p*-aminobenzenesulfonamide in acetone is far more than the other four solvents in this work. In the other four solvents, the variety of solubility of *p*-aminobenzenesulfonamide in propan-2-ol is the biggest with the temperature change, and the solubility of *p*-aminobenzenesulfonamide is in propan-1-ol > butan-1-ol > butan-2-ol. (3) The calculated solubility of *p*-aminobenzenesulfonamide sets a good coherence with the experimental values, and the experimental solubility and correlation equation in this work can be used as essential data and models in the process of purification for *p*-aminobenzenesulfonamide.

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