Densities and Viscosities of Niacin + 3-Picoline + Sulfuric Acid + Water from (293.15 to 343.15) K

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The densities and viscosities of niacin + 3-picoline + sulfuric acid + water mixtures have been determined experimentally at temperatures from 293.15 K to 343.15 K. The apparent molar volumes of niacin were calculated from experimental measurements. Results were fit to obtain the adjustable parameters and standard deviations between the measured and fitted values.

Introduction

Niacin, also known as nicotinic acid or vitamin B_5, is an important drug, feed additive, and intermediate with wide use. We[1] have developed a new technique for the electrochemical synthesis of niacin using 3-picoline as a raw material and an aqueous sulfuric acid solution as supporting electrolytes. This synthesis is characterized by mild reaction conditions, high product purity, and reduced waste. In the synthesis and purification process of niacin, it is useful to know the physical properties of niacin + 3-picoline + sulfuric acid + water mixtures. We[2] have reported the solubility data of niacin in 3-picoline + water, and we[3] have also studied the densities and viscosities of niacin with 3-picoline + water mixtures. This paper is a continuation of our previous work. In this study, the densities and viscosities of the niacin + sulfuric acid + water ternary mixture and the niacin + 3-picoline + sulfuric acid + water quaternary mixture have been measured from 293.15 K to 343.15 K. From measurements of densities, the apparent molar volumes of niacin were calculated. Results were fit to obtain the adjustable parameters and standard deviations between the measured and fitted values. These quantities can be used to study the molecular interactions among the components of the mixture.

Experimental Section

Materials. High-grade sulfuric acid from Louyan Chemical Reagent Co. was used directly without further purification, and its purity was greater than 99% by mass. 3-Picoline obtained from Shanghai Chemical Reagent Co. was of AR grade and was further purified by distillation; the purity was determined at wavelength $\lambda = 262$ nm by UV spectrometry (type UV-2401PC, Shimadzu Co.) to be 99.7% by mass. Analytical-grade nicotinic acid (niacin) obtained from Peking Biotech. Co. Ltd. was further purified by recrystallization from aqueous solutions. After filtration and drying, its purity was determined by titration to be 99.8% by mass. Water used in the experiments was double-distilled water; the conductivity was less than 1×10^{-4} $S{\cdot}m^{-1}$.

Apparatus and Procedure. The mixtures were prepared by mass using an electronic balance (type AW120, Shimadzu Co.) and were stored in ground-glass-stoppered bottles of 200 cm^3. The balance has an uncertainty of ± 0.0001 g. It was ensured that the components were adequately mixed before being transferred to the pycnometers. The possible error in the mass fractions is estimated to be ± 0.00005.

The density was measured with five Ostwald−Sprengel-type pycnometers having a bulb volume of 25 cm^3 and an internal capillary diameter of about 1 mm. The internal volumes of the pycnometers were calibrated with pure water at each of the measured temperatures; the densities of water were taken from the literature.[4] The thoroughly cleaned and perfectly dried pycnometers were first weighed on an electronic balance and then filled with experimental liquid and immersed in a thermostat (type 501, Shanghai Laboratory Instrument Works Co. Ltd.) controlled to within ± 0.02 K. After thermal equilibrium had been achieved at the required temperature, the pycnometers were removed from the thermostat and properly cleaned, dried, and weighed. The density was then determined from the mass of the sample and the volume of the pycnometers. The readings from five pycnometers were averaged to determine the density. The standard deviations of five parallel measurements were calculated by the Bessel equation to be less than 0.97×10^{-4} $g{\cdot}cm^{-3}$. The uncertainty analysis was based upon the *International Guide to the Expression of the Uncertainty in Measurement*. Uncertainties in the density measurement were within ± 0.0002 $g{\cdot}cm^{-3}$ on the basis of the 95% confidence level. The errors were caused mainly by the weighing process, repeatability of the measurement, and glassware.

The viscosity was measured using a commercial Ubbelohde capillary viscometer (type 1836-A, Shanghai Glass Instruments Factory, China) of 0.55-mm diameter, calibrated with double-distilled water at (293.15, 303.15, 313.15, 323.15, 333.15, and 343.15) K. A thoroughly cleaned and perfectly dried viscometer, filled with experimental liquid, was placed vertically in an insulated jacket, wherein constant temperature (± 0.02 K) was maintained by circulating water from a thermoelectric controller (type 501, Shanghai Laboratory Instrument Works Co. Ltd.) at the required temperature. After thermal stability was attained, the flow times of the liquids were recorded with an electronic digital stopwatch correct to ± 0.01 s. At least five repetitions of each datum point obtained were reproducible to ± 0.06 s, and the results were averaged. The standard

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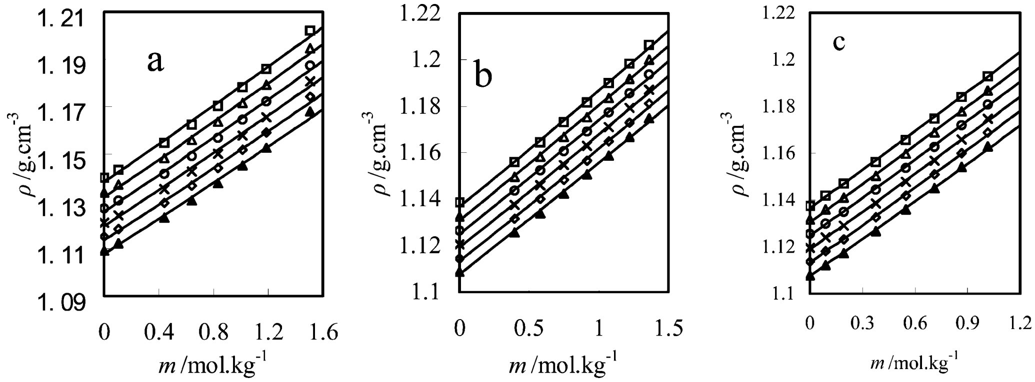

Figure 1. Variation of density with molality at □, 293.15 K; △, 303.15 K; ○, 313.15 K; ×, 323.15 K; ◇, 333.15 K; and ▲, 343.15 K for the following mixtures: (a) niacin + H_2O + 20 mass % H_2SO_4, (b) niacin + H_2O + 20 mass % H_2SO_4 + 10 mass % 3-picoline, and (c) niacin + H_2O + 20 mass % H_2SO_4 + 15 mass % 3-picoline. Solid line, calculated from eq 2.

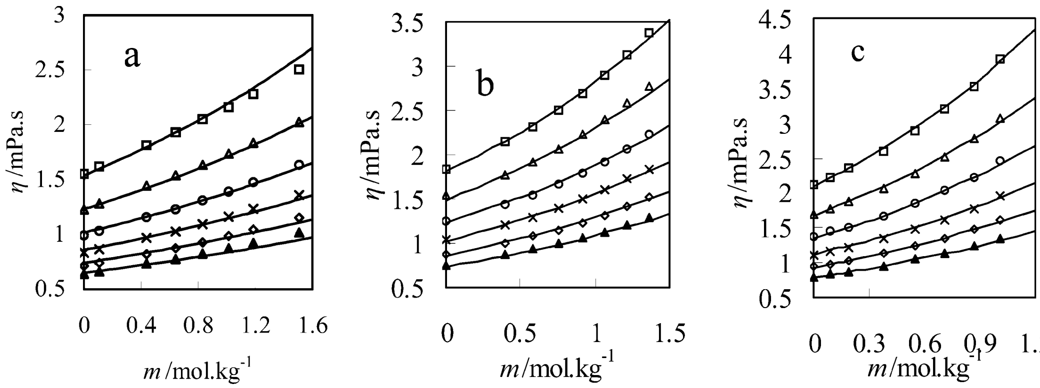

Figure 2. Variation of viscosity with molality at □, 293.15 K; △, 303.15 K; ○, 313.15 K; ×, 323.15 K; ◇, 333.15 K; and ▲, 343.15 K for the following mixtures: (a) niacin + H_2O + 20 mass % H_2SO_4, (b) niacin + H_2O + 20 mass % H_2SO_4 + 10 mass % 3-picoline, and (c) niacin + H_2O + 20 mass % H_2SO_4 + 15 mass % 3-picoline. Solid line, calculated from eq 2.

deviations for the viscosity of five parallel measurements were less than 1.1×10^{-2} mPa·s. Because all flow times were greater than 200 s and the capillary diameter (0.55 mm) was far less than its length (90−100 mm), the kinetic energy and end corrections, respectively, were found to be negligible. The viscosity η was then calculated from the relationship[5]

$$\frac{\eta}{\eta_w} = \frac{\rho t}{\rho_w t_w} \tag{1}$$

where η, ρ, and t and η_w, ρ_w, and t_w are the viscosities, densities, and flow time of the mixture and water, respectively. The values of the viscosity and density of pure water come from the literature.[4] The uncertainty in the viscosity measurement is estimated on the basis of the principle of error propagation to be ±0.6% at the 95% confidence level. There are three main sources of error in the measurement of the viscosity. The first is the propagation error resulting from the measurement of the density. The second is the measurement error resulting from the weighing process of the sample and the repeatability of the measurement. The third is the instrument error.

Results and Discussion

The measured densities of the 20 mass % sulfuric acid + H_2O mixture together with literature values are included in Table 1. The experimental densities and viscosities at (293.15, 303.15, 313.15, 323.15, 333.15, and 343.15) K are listed in Tables 2 to 4. It can be found that the density and viscosity increase with increasing concentration of niacin at constant temperature and decrease with increasing temperature at a fixed concentration of niacin. The dependence of density and viscosity on temperature and concentration has been calculated by means of the Vogel−Tamman−Fulcher (VTF) equation[13]

Table 1. Comparison of Experimental Densities, ρ, and Viscosities, η, of 3-Picoline, H_2SO_4 and 20 Mass % H_2SO_4 + H_2O with Literature Values

		ρ/g·cm^{-3}		η/mPa·s	
liquid	T/K	exptl	lit	exptl	lit
20 mass % H_2SO_4 + H_2O	293.15	1.1399	1.1394[6]	1.5501	1.55[7]
					1.60[8]
	303.15	1.1337	1.1335[6]	1.2293	1.23[7]
	313.15	1.1271	1.1275[6]	0.9882	0.99[7]
	323.15	1.1210	1.1215[6]	0.8315	0.83[7]
					0.835[8]
	333.15	1.1152	1.1153[6]	0.7104	0.71[7]
	343.15	1.1091	1.1087[6]	0.6412	0.64[7]
3-picoline	293.15	0.9560	0.95658[9]	0.9459	0.973[10]
	298.15		0.95178[11]		0.8661[11]
	303.15	0.9466	0.94736[9]	0.8318	
	323.15	0.9283			
	323.137		0.9296[12]		
H_2SO_4	293.15	1.8312	1.8305[6]	27.8041	27.5[8]
	313.15	1.8105	1.8107[6]		
	333.15	1.7912	1.7922[6]	8.3192	9.0[8]

$$F = P_1 \exp\left(\frac{P_2 + P_3 m}{T/\mathrm{K} - P_4}\right) \tag{2}$$

where $F \equiv$ (ρ or η), ρ and η are the density and viscosity of solution, respectively, m is the molality of niacin, T is the absolute temperature, and P_1, P_2, P_3, and P_4 the curve-fit

Table 2. Densities, ρ, Viscosities, η, and Apparent Molar Volumes, $V_{\Phi,2}$, of Niacin + H_2O + 20 Mass % H_2SO_4 Mixtures from T = 293.15 K to 343.15 K

m	ρ	$V_{\Phi,2}$	η	m	ρ	$V_{\Phi,2}$	η
$mol \cdot kg^{-1}$	$g \cdot cm^{-3}$	$cm^3 \cdot mol^{-1}$	$mPa \cdot s$	$mol \cdot kg^{-1}$	$g \cdot cm^{-3}$	$cm^3 \cdot mol^{-1}$	$mPa \cdot s$
	T/K = 293.15				*T*/K = 323.15		
0.0000	1.1399		1.5501	0.0000	1.1210		0.8315
0.1054	1.1432	83.66	1.6192	0.1054	1.1241	85.85	0.8646
0.4387	1.1546	81.17	1.8118	0.4387	1.1352	82.98	0.9673
0.6406	1.1624	79.40	1.9280	0.6406	1.1427	81.27	1.0274
0.8319	1.1702	77.90	2.0501	0.8319	1.1502	79.81	1.0920
1.0134	1.1781	76.43	2.1608	1.0134	1.1579	78.27	1.1622
1.1859	1.1859	75.11	2.2797	1.1859	1.1655	76.94	1.2314
1.5064	1.2022	72.22	2.5038	1.5064	1.1807	74.35	1.3588
	T/K = 303.15				*T*/K = 333.15		
0.0000	1.1337		1.2293	0.0000	1.1152		0.7104
0.1054	1.1369	84.62	1.2846	0.1054	1.1183	86.47	0.7367
0.4387	1.1482	81.77	1.4426	0.4387	1.1293	83.58	0.8212
0.6406	1.1559	80.03	1.5379	0.6406	1.1366	81.90	0.8728
0.8319	1.1636	78.54	1.6325	0.8319	1.1440	80.43	0.9282
1.0134	1.1715	77.04	1.7348	1.0134	1.1517	78.89	0.9851
1.1859	1.1792	75.67	1.8343	1.1859	1.1591	77.56	1.0429
1.5064	1.1946	73.18	2.0226	1.5064	1.1742	74.93	1.1506
	T/K = 313.15				*T*/K = 343.15		
0.0000	1.1271		0.9882	0.0000	1.1091		0.6412
0.1054	1.1303	85.24	1.0310	0.1054	1.1122	86.85	0.6620
0.4387	1.1415	82.37	1.1576	0.4387	1.1230	84.19	0.7333
0.6406	1.1491	80.65	1.2296	0.6406	1.1303	82.52	0.7748
0.8319	1.1567	79.17	1.3087	0.8319	1.1376	81.07	0.8242
1.0134	1.1644	77.66	1.3896	1.0134	1.1451	79.54	0.8712
1.1859	1.1721	76.31	1.4777	1.1859	1.1526	78.12	0.9242
1.5064	1.1874	73.76	1.6315	1.5064	1.1679	75.28	1.0172

Table 3. Densities, ρ, Viscosities, η, and Apparent Molar Volumes, $V_{\Phi,2}$, of Niacin + H_2O + 20 Mass % H_2SO_4 + 10 Mass % 3-Picoline Mixtures from T = 293.15 K to 343.15 K

m	ρ	$V_{\Phi,2}$	η	m	ρ	$V_{\Phi,2}$	η
$mol \cdot kg^{-1}$	$g \cdot cm^{-3}$	$cm^3 \cdot mol^{-1}$	$mPa \cdot s$	$mol \cdot kg^{-1}$	$g \cdot cm^{-3}$	$cm^3 \cdot mol^{-1}$	$mPa \cdot s$
	T/K = 293.15				*T*/K = 323.15		
0.0000	1.1385		1.8396	0.0000	1.1205		1.0499
0.3966	1.1559	73.16	2.1401	0.3966	1.1376	74.39	1.2080
0.5789	1.1645	71.84	2.3083	0.5789	1.1461	72.98	1.2974
0.7517	1.1731	70.48	2.4903	0.7517	1.1546	71.56	1.3911
0.9157	1.1816	69.20	2.6853	0.9157	1.1629	70.33	1.4925
1.0716	1.1899	68.06	2.8909	1.0716	1.1711	69.14	1.5991
1.2199	1.1983	66.81	3.1238	1.2199	1.1793	67.92	1.7189
1.3612	1.2065	65.67	3.3694	1.3612	1.1872	66.86	1.8427
	T/K = 303.15				*T*/K = 333.15		
0.0000	1.1324		1.5351	0.0000	1.1145		0.8749
0.3966	1.1497	73.58	1.7791	0.3966	1.1316	74.61	1.0033
0.5789	1.1583	72.18	1.9163	0.5789	1.1400	73.32	1.0753
0.7517	1.1668	70.88	2.0628	0.7517	1.1484	71.97	1.1502
0.9157	1.1753	69.55	2.2213	0.9157	1.1567	70.69	1.2340
1.0716	1.1836	68.37	2.3880	1.0716	1.1649	69.46	1.3194
1.2199	1.1918	67.22	2.5749	1.2199	1.1730	68.27	1.4146
1.3612	1.2000	66.05	2.7747	1.3612	1.1811	67.06	1.5134
	T/K = 313.15				*T*/K = 343.15		
0.0000	1.1265		1.2501	0.0000	1.1086		0.7598
0.3966	1.1437	73.98	1.4440	0.3966	1.1256	75.02	0.8681
0.5789	1.1523	72.50	1.5515	0.5789	1.1340	73.66	0.9293
0.7517	1.1607	71.27	1.6660	0.7517	1.1423	72.37	0.9920
0.9157	1.1691	69.98	1.7927	0.9157	1.1506	71.04	1.0616
1.0716	1.1774	68.75	1.9242	1.0716	1.1587	69.85	1.1335
1.2199	1.1856	67.57	2.0714	1.2199	1.1667	68.70	1.2139
1.3612	1.1937	66.42	2.2239	1.3612	1.1749	67.39	1.2965

coefficients; the values are listed in Table 5, along with the standard deviations. The standard deviation is defined by

$$\sigma = \left[\sum_{i=1}^{p} \frac{(Y_i^{\text{exptl}} - Y_i^{\text{calcd}})^2}{p - n}\right]^{1/2} \tag{3}$$

where p is the number of experimental points and n is the

Table 4. Densities, ρ, Viscosities, η, and Apparent Molar Volumes, $V_{\Phi,2}$, of Niacin + H_2O + 20 Mass % H_2SO_4 + 15 Mass % 3-Picoline Mixtures from T = 293.15 K to 343.15 K

m	ρ	$V_{\Phi,2}$	η	m	ρ	$V_{\Phi,2}$	η
$mol \cdot kg^{-1}$	$g \cdot cm^{-3}$	$cm^3 \cdot mol^{-1}$	$mPa \cdot s$	$mol \cdot kg^{-1}$	$g \cdot cm^{-3}$	$cm^3 \cdot mol^{-1}$	$mPa \cdot s$
	T/K = 293.15				*T*/K = 323.15		
0.0000	1.1374		2.1196	0.0000	1.1196		1.1118
0.0902	1.1418	70.26	2.2217	0.0902	1.1240	70.77	1.1614
0.1930	1.1469	69.61	2.3472	0.1930	1.1291	70.10	1.2208
0.3754	1.1563	68.19	2.6029	0.3754	1.1385	68.64	1.3415
0.5480	1.1656	66.81	2.8784	0.5480	1.1478	67.21	1.4725
0.7116	1.1747	65.57	3.1909	0.7116	1.1569	65.95	1.6139
0.8669	1.1839	64.15	3.5298	0.8669	1.1660	64.58	1.7744
1.0144	1.1928	62.96	3.9086	1.0144	1.1748	63.42	1.9485
	T/K = 303.15				*T*/K = 333.15		
0.0000	1.1315		1.6949	0.0000	1.1137		0.9377
0.0902	1.1359	70.43	1.7742	0.0902	1.1181	70.93	0.9778
0.1930	1.1410	69.77	1.8727	0.1930	1.1232	70.26	1.0266
0.3754	1.1504	68.34	2.0620	0.3754	1.1326	68.78	1.1247
0.5480	1.1597	66.94	2.2833	0.5480	1.1419	67.35	1.2316
0.7116	1.1689	65.58	2.5158	0.7116	1.1510	66.07	1.3485
0.8669	1.1780	64.27	2.7833	0.8669	1.1600	64.79	1.4765
1.0144	1.1867	63.22	3.0744	1.0144	1.1689	63.52	1.6175
	T/K = 313.15				*T*/K = 343.15		
0.0000	1.1255		1.3799	0.0000	1.1078		0.8024
0.0902	1.1299	70.60	1.4420	0.0902	1.1122	71.10	0.8351
0.1930	1.1350	69.93	1.5183	0.1930	1.1173	70.42	0.8762
0.3754	1.1444	68.49	1.6714	0.3754	1.1267	68.93	0.9560
0.5480	1.1537	67.08	1.8386	0.5480	1.1360	67.48	1.0448
0.7116	1.1628	65.82	2.0250	0.7116	1.1451	66.19	1.1402
0.8669	1.1719	64.47	2.2267	0.8669	1.1541	64.90	1.2459
1.0144	1.1808	63.24	2.4504	1.0144	1.1629	63.70	1.3599

Table 5. Coefficient of Equation 2 and Standard Deviation, σ, for $\rho(g \cdot cm^{-3})$ and $\eta(mPa \cdot S)$ for Different Systems

systems		P_1	P_2	P_3	P_4	$10^2\sigma$
niacin + H_2O + 20 mass % H_2SO_4	ρ	0.5560	907.1	44.23	−973.0	0.143
	η	0.07024	396.6	45.11	164.7	2.99
niacin + H_2O + 20 mass % H_2SO_4	ρ	0.2067	5530	139.6	−2951	0.0987
+ 10 mass % 3-picoline	η	0.00014	4454	209.0	−177.4	2.80
niacin + H_2O + 20 mass % H_2SO_4	ρ	0.1220	9572	204.1	−3996	0.0568
+ 15 mass % 3-picoline	η	0.00756	1345	145.3	54.08	2.27

number of parameters. Y_i^{calcd} and Y_i^{exptl} refer to the calculated values from the equation and to the experimental value. On the basis of the obtained standard deviation values, we conclude that eq 2 can be successfully used for the correlation of the investigated physical properties.

The apparent molar volume of niacin, $V_{\Phi,2}$, is given by the following equation

$$V_{\Phi,2} = \frac{M}{\rho} - \frac{10^3(\rho - \rho_0)}{m\rho\rho_0} \tag{4}$$

where M is the molar mass of niacin, ρ is the density of the solution, and ρ_0 is the density of the solvent mixture. Values of the apparent molar volume of niacin in solvent mixtures have also been given in Tables 2 to 4. The apparent molar volume increases as temperature increases at a fixed concentration of niacin and decreases with concentration at the same temperature. These values are important because they form the basis for understanding molecular interactions. $V_{\Phi,2}$ varies linearly with the mo-

Table 6. Parameters of Equation 5 and Standard Deviation, σ, for $V_{\Phi,2}$ from T = 293.15 K to 343.15 K

T / K	$V^0_{\Phi,2}$ / $cm^3 \cdot mol^{-1}$	S_V / $cm^3 \cdot kg^{-1}$	σ / $cm^3 \cdot mol^{-1}$
Niacin + H_2O + 20 Mass % H_2SO_4			
293.15	84.64	−8.14	0.12
303.15	85.36	−8.16	0.091
313.15	85.99	−8.17	0.083
323.15	86.61	−8.18	0.077
333.15	87.23	−8.19	0.072
343.15	87.80	−8.21	0.098
Niacin + H_2O + 20 Mass % H_2SO_4 + 10 Mass % 3-Picoline			
293.15	76.30	−7.77	0.056
303.15	76.69	−7.79	0.034
313.15	77.08	−7.79	0.049
323.15	77.48	−7.81	0.032
333.15	77.82	−7.84	0.072
343.15	78.21	−7.85	0.084
Niacin + H_2O + 20 Mass % H_2SO_4 + 15 Mass % 3-Picoline			
293.15	71.11	−7.96	0.11
303.15	71.25	−7.95	0.081
313.15	71.43	−8.01	0.079
323.15	71.60	−8.03	0.071
333.15	71.76	−8.06	0.065
343.15	71.92	−8.08	0.054

lality of niacin over the range studied and can be analyzed by fitting to the following equation

$$V_{\Phi,2} = V^0_{\Phi,2} + S_v m \quad (5)$$

where $V^0_{\Phi,2}$ is the infinite dilution apparent molar volume that is equal in value to the standard partial molar volume and S_v is the experimental slope. The values of $V^0_{\Phi,2}$ and S_v obtained, by least-squares analysis, for niacin in the solvent mixtures are listed in Table 6, along with their standard deviations.

From Table 6, we find that the infinite dilution apparent molar volumes are positive and increase as temperature increases for all systems examined; however, S_v is negative and decreases as temperature increases. The positive values of $V^0_{\Phi,2}$ indicate that solvent molecules are loosely attached to the solute, which expands with increasing temperature, thus resulting in higher values of $V^0_{\Phi,2}$ at higher temperature. At a given temperature, $V^0_{\Phi,2}$ decreases with increasing mass % of 3-picoline for all systems under investigation, suggesting that the solute−solvent interaction decreases with increasing 3-picoline content. However, because the increase in $V^0_{\Phi,2}$ with increasing temperature is attributed to an increase in solvation, on raising the temperature some solvent molecules may be released from the loose solvation layer of solute in solution.

The negative sign of S_v for all systems investigated reveals weaker solute−solute interactions. S_v decreases with rising temperature, which is attributed to more violent thermal agitation at higher temperature, resulting in diminishing force of the solute−solute interactions.

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