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Solubilities of 1-Hexyl-3-methylimidazole Nitrate and 1-Octyl-3-methylimidazole Nitrate in Selected Solvents

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ABSTRACT: Solubilities of two ionic liquids, 1-hexyl-3-methylimidazolium nitrate ([HMIM][NO_3]) and 1-octyl-3-methylimidazolium nitrate ([OMIM][NO_3]), in 10 organic solvents (*n*-pentane, *n*-hexane, cyclohexane, *n*-heptane, *n*-octane, benzene, toluene, *o*-xylene, *p*-xylene, and *m*-xylene) were measured in the range between (278.15 and 343.15) K using a static analytical gravimetric method, respectively. The logarithm of the mole fraction solubilities of these two ILs in the 10 selected solvents was correlated by a linear function of inversed temperatures.

■ INTRODUCTION

Ionic liquids (ILs) have been studied widely in many branches of chemistry these years[1] because of their interesting physical and chemical properties. They can be used as solvents for new syntheses[2] and electrolytes in electrochemical devices.[3−6] This is mainly due to many of their fascinating properties, such as high thermal and chemical stability at high temperatures, negligible vapor pressure, nonflammability, and high loading capacity. The properties of the ILs can be adjusted by altering the cation or anion, the chain length of the alkyl rests, and the substituent, so ILs are also known as "designer solvents".[7]

1-Hexyl-3-methylimidazolium nitrate ([HMIM][NO_3]) (CAS Registry No. 203389-26-8) and 1-octyl-3-methylimidazolium nitrate ([OMIM][NO_3]) (CAS Registry No. 203389-27-9) are two halogen-free ILs and thus more environmentally friendly than the halogenated ones.[8] The physicochemical information of ILs either in pure forms or as binaries with organic or inorganic compounds is of importance for their industrial application. The study on their phase behavior with organic solutes is of interest. In view of the industrial importance of phase equilibria involving ILs such as vapor−liquid equilibria, liquid−liquid equilibria, and solid−liquid equilibria, the solubilities of a number of ionic liquid in different solvents have been investigated, for example, 1-ethyl-3-methylimidazolium hexafluorophosphate ([emim][PF_6]),[9] 1-ethylpyridinium hexafluorophosphate ([EPy][PF_6]),[10] and didecyldimethylammonium nitrate ([DDA][NO_3]).[11] However, solubility data of [OMIM][NO_3] and [HMIM][NO_3] are not found in literature.

In this study, solubilities of two ILs, [HMIM][NO_3] and [OMIM][NO_3], in 10 selected solvents (*n*-pentane, *n*-hexane, cyclohexane, *n*-heptane, *n*-octane, benzene, toluene, *o*-xylene, *p*-xylene, and *m*-xylene) are measured.

■ EXPERIMENTAL SECTION

Materials. 1-Hexyl-3-methylimidazolium nitrate (99.5 %) and 1-octyl-3-methylimidazolium nitrate (99.5 %) were purchased from the Shanghai Chengjie Chemical Reagents Company. The chemical shifts for the ^1H NMR spectrum (parts per million, C_3D_6O) appeared as follows: [HMIN][NO_3], δ: 9.07 (s, 1H, CH−N), 7.73−7.66 (d, 2H, CH═CH−N), 4.16−4.12 (t, 2H, CH_2Me), 3.84 (s, 3H, CH_3N), 1.79−1.74 (t, 2H, CH_2N), 1.26−1.22 (m, 6H, $(CH_2)_3$), 0.87−0.84 (t, 3H, CH_2CH_3); [OMIN][NO_3], δ: 9.07 (s, 1H, CH−N), 7.74−7.67 (d, 2H, CH═CH−N), 4.15−4.12 (t, 2H, CH_2Me), 3.84 (s, 3H, CH_3N), 1.80−1.76 (t, 2H, CH_2N), 1.26−1.23 (m, 10H, $(CH_2)_5$), 0.86−0.84 (t, 3H, CH_2CH_3). All of the solvents in the measurement were purchased from different chemical reagent producers as listed in Table 1. Their mass fraction purities are as follows: *n*-pentane (0.990), *n*-hexane (0.990), cyclohexane (0.995), *n*-heptane (0.985), *n*-octane (0.990), benzene (0.995), toluene (0.995), *o*-xylene (0.985), *p*-xylene (0.950), and *m*-xylene (0.950). The water content in each solvent was less than 0.20 %. All the reagents in this work were analytical reagent (AR) grade and used without further purification.

Apparatus. A jacketed equilibrium cell with a working volume of 100 mL was immersed in a circulating water bath with a thermostat (type 50 L, Shanghai Laboratory Instrument Works Co. Ltd.) with a precision of ± 0.05 K as described by Shang et al.[12] An analytical balance (type TG328B, Shanghai Balance Instrument Works Co.) with an uncertainty of ± 0.1 mg, a magnetic stirrer, and a calibrated thermometer with a precision of 0.1 K were used in the experiment. The equilibrium cell was sealed to prevent the evaporation of solvent or solute during the experiment.

Solubility Measurement. The solubilities were measured by a gravimetric method.[13] For each measurement, an excess mass of IL was added to a known mass of solvent. Then the equilibrium cell was heated to a constant temperature with continuous stirring. After at least 1 h (once the temperature of water bath approached a constant value, the actual value of temperature was recorded), the stirring was stopped, and the solution was kept still until it was clear and the excess mass of IL was in the lower portion. About 2 mL of the clear upper portion of the solution

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Table 1. Boiling Points of the Solvents Used in This Work

solvent	boiling point/K	producers	mass of residue after evaporation/%
n-pentane	309.25	[d]	0.050
n-hexane	342.15	[a]	0.001
cyclohexane	353.89	[b]	0.002
n-heptane	371.55	[a]	0.002
n-octane	398.15	[a]	0.002
benzene	353.25	[b]	0.001
toluene	383.75	[b]	0.001
o-xylene	417.15	[c]	0.005
m-xylene	412.45	[c]	0.005
p-xylene	411.15	[c]	0.005

[a] Tianjin Fuchen Chemical Reagent Co., Ltd. [b] Beijing Chemical Works. [c] Sinopharm Chemical Reagent Co., Ltd. [d] Acros Organics (Geel, Belgium).

was withdrawn to another previously weighed measuring vial (m_0) with a preheated on−off injector. The vial was quickly and tightly closed and weighed (m_1) to determine the mass of the sample ($m_1 - m_0$). The vial was uncovered and then covered with a piece of filter paper to prevent dust contamination. After the solvent in the vial had completely evaporated, the vial was dried and reweighed (m_2) to determine the mass of the residue IL ($m_2 - m_0$). The IL concentration of the sample solution in mole fraction, x, is determined from eq 1

$$x = \frac{(m_2 - m_0)/M_1}{(m_2 - m_0)/M_1 + (m_1 - m_2)/M_2} \qquad (1)$$

where M_1 and M_2 are the molar mass of the IL and solvent, respectively.

A different dissolution time was tested to determine a suitable equilibrium time. It was found that 1 h was enough for [OMIM][NO_3] and [HMIM][NO_3] to reach equilibrium in the solvent. Three parallel measurements were performed at the same composition of solvent at each temperature, and an average value was given. The maximum relative deviation of each triplicate data is 0.25 %, and the minimum is 0.15 %. The estimated relative uncertainty of the solubility values based on error analysis and repeated observations is within 2 %. The uncertainty in the experimental solubility is due to uncertainties in the temperature measurement, weighing procedure, and instabilities of the water bath. The temperature range for the solubility measurement in different organic solvents should be below the boiling point of the respective solvent. The evaporation temperatures of the solvents used in this work are listed in Table 1.

RESULTS AND DISCUSSION

Solubilities of [HMIM][NO_3] and [OMIM][NO_3] in 10 selected organic solvents were measured in the range between (278.15 and 343.15) K. The mole fraction solubility data of these two ILs at different temperatures are presented in Table 2.

Correlation. Since in the temperature range studied the molar enthalpy of IL solutions can be considered temperature-independent, the experimental solubility data can be correlated by the following equation:[14−16]

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

Table 2. Mole Fraction Solubilities (x) of ILs in Pure Organic Solvents at Different Temperatures

ILs	solvent	T/K	10^3x	$(x - x^{\text{calcd}})/x$
[HMIM][NO_3]	*n*-pentane	278.15	0.359	−0.0005
		283.15	0.397	−0.0042
		288.15	0.442	0.0056
		293.15	0.484	−0.0011
		298.15	0.534	0.0053
		303.15	0.578	−0.0051
	n-hexane	288.15	0.306	−0.0006
		293.15	0.362	0.0014
		298.15	0.420	−0.0244
		303.15	0.492	−0.0062
		308.15	0.575	0.0090
		313.15	0.693	0.0264
		318.15	0.794	0.0211
		323.15	0.895	−0.0003
		328.15	0.995	−0.0064
		333.15	1.129	−0.0213
	cyclohexane	293.15	0.304	0.0106
		298.15	0.357	0.0251
		303.15	0.400	−0.0002
		308.15	0.446	−0.0277
		313.15	0.516	−0.0118
		318.15	0.592	−0.0025
		323.15	0.674	0.0044
		328.15	0.751	−0.0079
		333.15	0.857	0.0092
		338.15	0.970	0.0199
	n-heptane	313.15	0.268	0.0046
		318.15	0.358	−0.0099
		323.15	0.490	0.0010
		328.15	0.629	0.0081
		333.15	0.841	−0.0035
		338.15	1.093	−0.0002
		343.15	1.419	−0.0001
	n-octane	293.15	0.203	−0.0142
		298.15	0.234	−0.0089
		303.15	0.268	0.0138
		308.15	0.299	0.0101
		313.15	0.344	0.0153
		318.15	0.380	0.0038
		323.15	0.421	−0.0072
		328.15	0.458	−0.0133
		333.15	0.518	0.0004
		338.15	0.568	−0.0091
		343.15	0.631	−0.0010
	benzene	298.15	0.219	−0.0059
		303.15	0.263	−0.0006
		308.15	0.308	−0.0100
		313.15	0.382	0.0044
		318.15	0.454	0.0087
		323.15	0.536	0.0058
		328.15	0.610	0.0014
		333.15	0.714	−0.0099
		338.15	0.832	0.0018
		343.15	0.962	0.0032

Table 2. Continued

ILs	solvent	T/K	10^3x	$(x - x^{\text{calcd}})/x$
	toluene	298.15	0.278	0.0040
		303.15	0.334	0.0006
		308.15	0.390	−0.0141
		313.15	0.482	−0.0037
		318.15	0.585	0.0214
		323.15	0.686	0.0111
		328.15	0.776	0.0006
		333.15	0.904	−0.0162
		338.15	1.061	−0.0023
		343.15	1.227	0.0034
	o-xylene	298.15	1.332	−0.0156
		303.15	1.450	−0.0110
		308.15	1.599	0.0082
		313.15	1.732	0.0127
		318.15	1.866	0.0136
		323.15	1.983	0.0038
		328.15	2.133	0.0075
		333.15	2.266	0.0014
		338.15	2.400	−0.0062
		343.15	2.533	−0.0151
	m-xylene	298.15	2.392	−0.0055
		303.15	2.715	0.0049
		308.15	3.032	0.0028
		313.15	3.368	−0.0011
		318.15	3.764	0.0046
		323.15	4.127	−0.0058
		328.15	4.578	−0.0010
		333.15	5.051	0.0011
		338.15	5.534	−0.0009
		343.15	6.070	0.0010
	p-xylene	298.15	0.795	−0.0178
		303.15	0.896	−0.0127
		308.15	1.024	0.0091
		313.15	1.140	0.0085
		318.15	1.277	0.0168
		323.15	1.409	0.0138
		328.15	1.550	0.0109
		333.15	1.686	−0.0001
		338.15	1.823	−0.0149
		343.15	1.995	−0.0144
[OMIM][NO_3]	*n*-pentane	278.15	0.687	−0.0029
		283.15	0.816	0.0053
		288.15	0.955	0.0042
		293.15	1.103	−0.0035
		298.15	1.266	−0.0131
		303.15	1.493	0.0098
	n-hexane	288.15	0.683	0.0160
		293.15	0.780	−0.0283
		298.15	0.919	−0.0091
		303.15	1.129	0.0120
		308.15	1.282	−0.0096
		313.15	1.624	0.0190
		318.15	1.844	0.0273
		323.15	2.056	−0.0121
		328.15	2.316	−0.0119
		333.15	2.706	−0.0046

Table 2. Continued

ILs	solvent	T/K	10^3x	$(x - x^{\text{calcd}})/x$
	cyclohexane	293.15	0.775	−0.0014
		298.15	0.901	−0.0225
		303.15	1.056	−0.0036
		308.15	1.244	0.0197
		313.15	1.462	0.0131
		318.15	1.685	0.0149
		323.15	1.909	−0.0003
		328.15	2.121	−0.0072
		333.15	2.440	−0.0072
		338.15	2.754	−0.0061
	n-heptane	293.15	0.342	0.0247
		298.15	0.407	−0.0115
		303.15	0.488	−0.0082
		308.15	0.586	−0.0009
		313.15	0.728	0.0039
		318.15	0.856	−0.0102
		323.15	1.010	−0.0224
		328.15	1.188	−0.0013
		333.15	1.427	0.0112
		338.15	1.664	0.0084
		343.15	1.932	0.0055
	n-octane	293.15	0.355	0.0377
		298.15	0.411	−0.0157
		303.15	0.502	−0.0096
		308.15	0.605	−0.0106
		313.15	0.733	−0.0002
		318.15	0.866	−0.0095
		323.15	1.009	−0.0275
		328.15	1.230	0.0057
		333.15	1.455	0.0133
		338.15	1.681	0.0021
		343.15	1.976	0.0127
	benzene	298.15	0.303	−0.0002
		303.15	0.357	0.0046
		308.15	0.410	−0.0045
		313.15	0.490	−0.0047
		318.15	0.572	0.0031
		323.15	0.661	−0.0019
		328.15	0.751	0.0078
		333.15	0.860	−0.0042
		338.15	0.981	−0.0001
		343.15	1.114	−0.0001
	toluene	298.15	0.371	0.0111
		303.15	0.430	−0.0074
		308.15	0.500	−0.0026
		313.15	0.598	0.0016
		318.15	0.699	0.0135
		323.15	0.806	0.0098
		328.15	0.892	−0.0044
		333.15	1.025	−0.0106
		338.15	1.173	0.0016
		343.15	1.329	−0.0021
	o-xylene	298.15	4.998	−0.0048
		303.15	5.290	−0.0010
		308.15	5.581	0.0013

Table 2. Continued

ILs	solvent	T/K	10^3x	$(x - x^{\mathrm{calcd}})/x$
		313.15	5.873	0.0026
		318.15	6.164	0.0030
		323.15	6.456	0.0027
		328.15	6.747	0.0017
		333.15	7.039	0.0002
		338.15	7.330	−0.0017
		343.15	7.622	−0.0040
	m-xylene	298.15	5.256	−0.0102
		303.15	5.655	−0.0022
		308.15	6.054	0.0029
		313.15	6.453	0.0056
		318.15	6.852	0.0063
		323.15	7.251	0.0056
		328.15	7.650	0.0035
		333.15	8.049	0.0004
		338.15	8.448	−0.0036
		343.15	8.847	−0.0083
	p-xylene	298.15	3.076	−0.0103
		303.15	3.310	−0.0058
		308.15	3.576	0.0046
		313.15	3.826	0.0076
		318.15	4.076	0.0083
		323.15	4.309	0.0035
		328.15	4.576	0.0047
		333.15	4.826	0.0008
		338.15	5.076	−0.0040
		343.15	5.326	−0.0097

where A and B are correlation parameters for each solvent. The correlation constants obtained from the fitting of IL solubility data are listed in Table 3. The relative standard deviations (RSDs) for each solvent, defined by eq 3, are also presented in Table 3.

$$\mathrm{RSD} = \left[\frac{1}{N}\sum_{1}^{n}\left(\frac{x_i - x_i^{\mathrm{calcd}}}{x_i}\right)^2\right]^{1/2} \qquad (3)$$

where superscript calcd stands for the calculated values and N is the number of experimental points. The plots of ln x versus $1/T$ of two ILs are shown in Figures 1 and 2, respectively. From these figures it can be seen that, within the temperature range of the measurements, the ILs $[\mathrm{HMIM}][\mathrm{NO_3}]$ and $[\mathrm{OMIM}][\mathrm{NO_3}]$ are partially soluble in the nonpolar or weak-polar solvents studied and their solubilities in the selected solvents increase with temperature. The logarithm of mole fraction solubilities of these two ILs in the 10 selected solvents were correlated by a linear function of inversed temperatures, and the solubilities calculated from the correlation are in good agreement with the experimental data.

For the straight-chain alkanes, the mole fraction solubilities of these two ILs are ranked as *n*-pentane > *n*-hexane > *n*-heptane > *n*-octane, which may result from the difference in their molecular mass. For the three aromatic solvents investigated, the mole fraction solubilities of these two ILs are ranked as xylene > methylbenzene > benzene, which were consistent with their polarity order. For the three xylene isomers, the order of solubility is: *m*-xylene > *o*-xylene > *p*-xylene, which is also related to their polarity order. In general, a relatively lower solubility of the two ILs in these solvents was observed. The alkyl chain increases in the cations of these ILs which can influence the

Table 3. Parameters of Equation 2 and Relative Standard Deviations (RSDs) of the Measured Solubility of Solutes in Selected Solvents from the Calculated Results

ILs	solvent	A	B	RSD
$[\mathrm{HMIM}][\mathrm{NO_3}]$	*n*-pentane	−2.1160	−1617.3	0.0042
	n-hexane	1.6950	−2820.0	0.0153
	cyclohexane	0.5356	−2534.1	0.0149
	n-heptane	0.7757	−2693.8	0.0093
	n-octane	−0.7799	−2260.6	0.0102
	benzene	2.8474	−3360.9	0.0062
	toluene	3.1202	−3371.0	0.0104
	o-xylene	−1.7060	−1460.9	0.0106
	m-xylene	6.7903	−4047.7	0.0035
	p-xylene	−0.1242	−2085.8	0.0129
$[\mathrm{OMIM}][\mathrm{NO_3}]$	*n*-pentane	1.9711	−2573.1	0.0075
	n-hexane	3.0027	−2970.1	0.0167
	cyclohexane	2.4004	−2803.0	0.0120
	n-heptane	4.0424	−3533.4	0.0123
	n-octane	3.9761	−3505.5	0.0185
	benzene	1.8360	−2963.3	0.0040
	toluene	1.8365	−2903.0	0.0078
	o-xylene	−2.0829	−957.4	0.0027
	m-xylene	−1.2812	−1179.8	0.0056
	p-xylene	−1.5924	−1246.8	0.0066

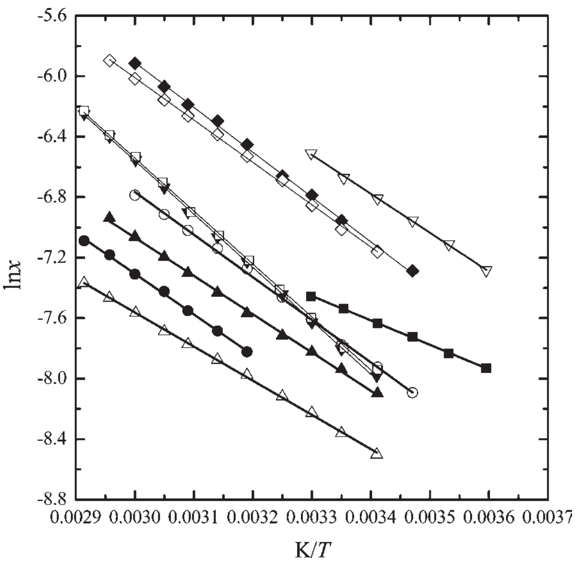

Figure 1. Mole fraction solubilities of two ILs in alkanes versus temperature: experimental data: ■, $[\mathrm{HMIM}][\mathrm{NO_3}]$ + *n*-pentane; ○, $[\mathrm{HMIM}][\mathrm{NO_3}]$ + *n*-hexane; ▲, $[\mathrm{HMIM}][\mathrm{NO_3}]$ + cyclohexane; ●, $[\mathrm{HMIM}][\mathrm{NO_3}]$ + *n*-heptane; △, $[\mathrm{HMIM}][\mathrm{NO_3}]$ + *n*-octane; ▽, $[\mathrm{OMIM}][\mathrm{NO_3}]$ + *n*-pentane; ◆, $[\mathrm{OMIM}][\mathrm{NO_3}]$ + *n*-hexane; ◇, $[\mathrm{OMIM}][\mathrm{NO_3}]$ + cyclohexane; ▼, $[\mathrm{OMIM}][\mathrm{NO_3}]$ + *n*-heptane; □, $[\mathrm{OMIM}][\mathrm{NO_3}]$ + *n*-octane; —, solubility curve calculated values according to eq 2.

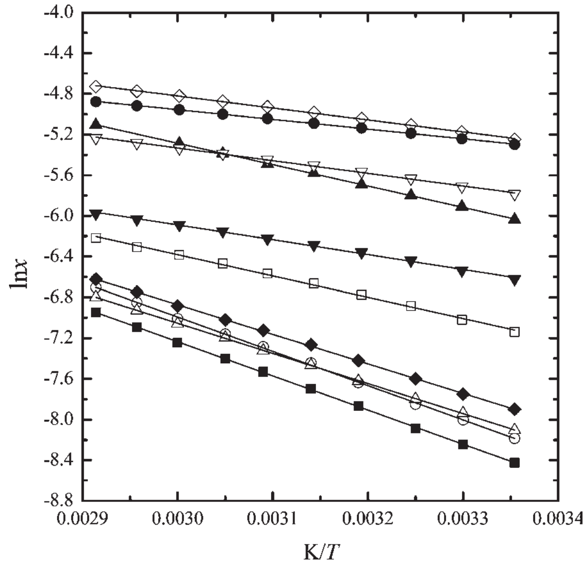

Figure 2. Mole fraction solubilities of two ILs in aromatic solvents versus temperature: experimental data: ■, [HMIM][NO_3] + benzene; ○, [HMIM][NO_3] + toluene; ▲, [HMIM][NO_3] + *m*-xylene; □, [HMIM][NO_3] + *p*-xylene; ▼, [HMIM][NO_3] + *o*-xylene; △, [OMIM][NO_3] + benzene; ◆, [OMIM][NO_3] + toluene; ◇, [OMIM][NO_3] + *m*-xylene; ▽, [OMIM][NO_3] + *p*-xylene; ●, [OMIM][NO_3] + *o*-xylene; —, solubility curve calculated values according to eq 2.

solubility as well. The solubilities of [OMIM][NO_3] in alkanes are higher than that of [HMIM][NO_3], which confirms the expectation from the theory of "similarity and intermiscibility".

The ion−dipole type interaction between the solvent and the solute gets stronger with the increase of the polarity of the solvent, which can be explained as one of the reasons that these two ILs are partially soluble in the weak-polar solvents selected, whereas they are completely miscible with many strong polar solvents, such as water and alcohols (methanol, ethanol, etc.) as well as organic acids (formic acid, acetic acid, etc.).

In all cases, the calculated solubilities coincided with the experimental data, which verified the feasibility of correlating the solubility data of these two ILs in the 10 selected solvents by eq 2. Despite of their partial mole fraction solubility in the solvents investigated, the obtained solubility data would be useful for its purification. Also, the miscibility of these two ILs with many strong polar ones (water, alcohols, and organic acids) may have potential important implications in the design of separation processes.

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