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Surface Tension and Density of 1-Methyl-3-hexylimidazolium Chloroindium

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The ionic liquid (IL) [C_6mim][$InCl_4$] (1-methyl-3-hexylimidazolium chloroindium) was prepared by directly mixing $InCl_3$ and 1-methyl-3-hexylimidazolium chloride with a molar ratio of 1:1 under dry argon. The density and surface tension of the IL were determined in the temperature range of (283.15 to 338.15) K. The ionic volume and surface entropy of the IL were estimated by extrapolation, respectively. In terms of Glasser's theory, the standard molar entropy and lattice energy of the IL were estimated, respectively. Using Kabo's method and Rebelo's method, the molar enthalpy of vaporization of the IL, $\Delta_l^g H_m^0$ (298 K), and $\Delta_l^g H_m^0$ (T_b) at a hypothetical normal boiling point, $T_b = 787$ K, were estimated, respectively. According to the interstice model, the thermal expansion coefficient of IL [C_6mim][$InCl_4$] was estimated.

1. Introduction

Ionic liquids (ILs) are a class of compounds that are being extensively studied because of their unusual properties.[1−5] In particular, these liquids have very low vapor pressures. Thus, they have great potential as "green" solvents for industrial processes. This potential is being explored vigorously as recent publications hint at their use in many typical chemical reactions.[6,7] To expand our knowledge of IL chemistry and because of our interest in the chemistry of indium,[8,9] we decided to determine whether similar salts could be prepared by replacing $AlCl_3$ with $InCl_3$. There is a limited number of reports of ionic liquids based on metals other than aluminum.[10−12]

This paper reports that the ionic liquid [C_6mim][$InCl_4$] (1-methyl-3-hexylimidazolium chloroindium) was prepared. In terms of Glasser's theory,[13] the volumetric and surface properties were discussed. Using Kabo's method[14] and Rebelo's method,[15] the molar enthalpy of vaporization, $\Delta_l^g H_m^0$ (298 K), and $\Delta_l^g H_m^0$ (T_b) at a hypothetical normal boiling point, T_b, were estimated, respectively. An interstice model was applied to predict the thermal expansion coefficient of IL [C_6mim][$InCl_4$], α, and the value was compared with the experimental one.

2. Experimental

Chemicals. Anhydrous $InCl_3$ (purity is 99.99 %) was obtained from Aldrich. It was opened in the glovebox filled with dry argon and used without further purification. 1-Methylimidazole, AR grade reagent, was obtained from ACROS and was vacuum distilled prior to use. Chlorohexane, AR grade reagent, was obtained from Beijing Chemicals Co. and was distilled before use. Ethyl acetate and acetonitrile were distilled and then stored over molecular sieves in tightly sealed glass bottles, respectively.

Preparation of Ionic Liquids. According to ref 16, 1-methyl-3-hexylimidazolium chloride ([C_6mim][Cl]) was synthesized. The product is a slightly yellow liquid. The yield is approximately 80 %. Analysis of [C_6mim][Cl] by ^{1}H NMR resulted in a spectrum that is in good agreement with the literature.[16]

$InCl_3$ was added slowly with stirring to a small glass vial containing [C_6mim][Cl] (mole ratio $InCl_3$/[C_6mim][Cl] = 1:1) in a glovebox filled with dry argon, and then the slightly yellow and transparent ionic liquid compound [C_6mim][$InCl_4$] was obtained. Analysis of the product by ^{1}H NMR gave a spectrum identical to that for [C_6mim][Cl]. The thermal decomposition temperature, $T_d = 726$ K, for the IL was determined by thermogravimetric analysis using a TA Instruments (SDT) model Q600 thermogravimetric analyzer. The Raman spectrum of the product was measured on a Renishaw Microscopic Confocal Raman spectrometer (RM2000). Laser excitation was provided by a semiconductor laser (785 nm) and was passed through a line filter and a cylindrical lens and was focused onto the NMR tube containing the sample. The laser power at the sample was approximately 0.90 mW. Scattered light was collected at 90° from the excitation beam. The light was dispersed via an 1800 line/mm grating and detected via a liquid nitrogen cooled charge-coupled device (CCD). The Raman spectrum of the product appears as the special peaks of $InCl_4^-$ at 322.0 cm^{-1} and is in good agreement with that of ref 8.

Table 1. Values of Density, ρ, and Surface Tension, γ, of [C_6mim][$InCl_4$] at 283.15 to 338.15 K

T/K	283.15	288.15	293.15	298.15	303.15	308.15
ρ/(g·cm^{-3})	1.49197	1.48706	1.48189	1.47692	1.4718	1.46729
γ/(mJ·m^{-2})	42.0	41.6	41.5	41.1	40.7	40.3
T/K	313.15	318.15	323.15	328.15	333.15	338.15
ρ/(g·cm^{-3})	1.46265	1.45799	1.45336	1.44869	1.44385	1.43923
γ/(mJ·m^{-2})	40.2	39.9	39.5	39.4	39.1	38.9

Measurement of Density and Surface Tension. The density of the sample was measured with an Anton Paar DMA 4500 oscillating U-tube densitometer, provided with automatic viscosity correction under dry argon from (283.15 to 338.15) K. The temperature in the cell was regulated to ± 0.01 K with a solid-state thermostat. The apparatus was calibrated once a day with dry air and double-distilled with freshly degassed water.

Using the tensiometer of the forced bubble method (DP-AW type produced by Sang Li Electronic Co.), we measured the surface tension of water at (283.15 to 338.15) K, which was in good agreement with that in the literature[17] within an experimental error of ± 0.1 mJ·m^{-2}. The surface tension of ionic liquid [C_6mim][$InCl_4$] was measured by the same method under dry argon in the same temperature range.

3. Results and Discussion

The values of density and surface tension of ionic liquid [C_6mim][$InCl_4$] are listed in Table 1, respectively. Each value in Table 1 is the average of three determinations.

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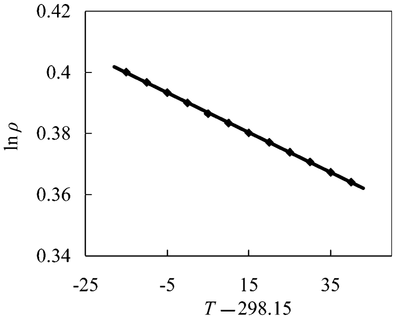

Figure 1. Plot of ln ρ vs ($T - 298.15$).

Estimation of Volumetric Properties for the IL. The experimental values of ln[ρ/(g·cm^{-3})] against T/K − 298.15 were fitted by the method of the least-squares and a linear equation

$$\ln[\rho/(\mathrm{g{\cdot}cm^{-3}})] = a - b(T/\mathrm{K} - 298.15) \qquad (1)$$

where $a = 0.39009$ and $b = 6.52{\cdot}10^{-4}$ was obtained by the fitting; the correlation coefficient is 0.9999; and the standard deviation $s = 1.7{\cdot}10^{-4}$. Figure 1 is a plot of ln[ρ/(g·cm^{-3})] vs (T/K − 298.15). The coefficient of thermal expansion of $C_6MIInCl_4$, α, is defined by the following equation

$$\alpha \equiv (1/V)(\partial V/\partial T)_p = -\{\partial \ln[\rho/(\mathrm{g{\cdot}cm^{-3}})]/\partial T\}_p \qquad (2)$$

Then $\alpha = 6.52{\cdot}10^{-4}\ \mathrm{K^{-1}}$ is the empirical parameter b of eq 1. In eq 2, V is the molar volume of ionic liquid [C_6mim][$InCl_4$].

From the experimental density, the molecular volume, V_m, of [C_6mim][$InCl_4$] was calculated using the following equation

$$V_m = M/(N\rho) \qquad (3)$$

where M is the molar mass (423.9 g·mol^{-1}); N is Avogadro's constant; and V_m = 0.4768 nm^3 for [C_6mim][$InCl_4$], V_m = 0.4507 nm^3 for [C_5mim][$InCl_4$],[10] V_m = 0.4225 nm^3 for [C_4mim][$InCl_4$],[12] and V_m = 0.3725 nm^3 for [C_2mim][$InCl_4$],[18] at 298.15 K. The data of ionic liquid based on indium are listed in Table 2. From Table 2, the mean contribution to molecular volume per methylene (−CH_2−) group is 0.0261 nm^3. This agrees well with methylene contributions of 0.0280 nm^3 from n-alcohols, 0.0272 nm^3 from n-amines, and 0.0267 nm^3 from n-paraffins.[13]

As may be seen in Figure 2, there is a linear relationship between molecular volume, V_m/nm^3, and molar mass of positive ions, M_+/(g·mol^{-1}). Then the linear regression of V_m against M_+ was carried out. The correlation coefficient was 0.9997, and the standard deviation was 0.0011 for the linear regression. The intercept of the linear regression may be approximately regarded as the volume of anion $InCl_4^-$, that is (0.1642 ± 0.001) nm^3, so that the volume, V_+, and the radii, r_+, of the positive ions were estimated and are listed in Table 2. In comparison with the value of the volume of $AlCl_4^-$ reported previously,[10] which is 0.1390 nm^3, the volume of negative ion $InCl_4^-$ is much larger.

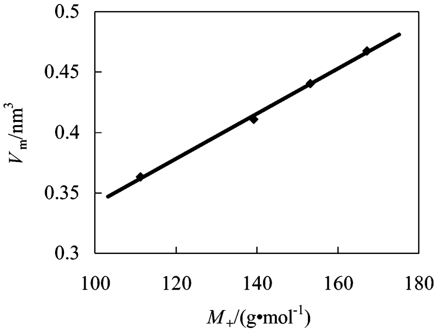

Figure 2. Plot of molecular volume, V_m, of the ILs as a function of molar mass of the cation, M_+.

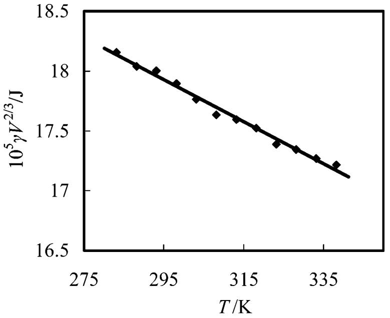

Figure 3. Product of γ and $V^{2/3}$ obtained from the experiment as a function of temperature, T.

According to Glasser's theory,[13] the standard molar entropy for the ionic liquid is given by

$$S^0(298)/(\mathrm{J{\cdot}K{\cdot}mol^{-1}}) = 1246.5(V_m/\mathrm{nm^3}) + 29.5 \qquad (4)$$

so that $S^0(298)/(\mathrm{J{\cdot}K{\cdot}mol^{-1}})$ = 623.8 for [C_6mim][$InCl_4$] and $S^0(298)/(\mathrm{J{\cdot}K{\cdot}mol^{-1}})$ = 493.9 for [C_2mim][$InCl_4$]. This implies that the entropy contribution per methylene group to the standard entropy is 32.5 J·K^{-1}·mol^{-1}. This value is in excellent agreement with the value of 32.2 J·K^{-1}·mol^{-1} from earlier literature for an extended group of organic compounds.[19] The value is closer than the value of 33.9 J·K^{-1}·mol^{-1} from [$C_n$mim][BF_4].[13]

Estimation of Surface Properties for the IL. The surface tension, γ, has been related to the temperature by the Eötvös equation:[20]

$$\gamma V^{2/3} = k(T_c - T) \qquad (5)$$

where V is the molar volume of the liquid; T_c is critical temperature; and k is an empirical constant. The linear regression of product of γ and $V^{2/3}$ obtained from this experiment against absolute temperature T was made, and a straight line was obtained (see Figure 3). From the slope of the straight line and the intercept, the values of $k = 1.763{\cdot}10^{-7}$ J·K^{-1} and T_c = 1312 K were obtained, respectively. For the majority of organic liquids, k is about $2.1{\cdot}10^{-7}$ J·K^{-1}, but for fused salts, k is small, $k \approx 0.4{\cdot}10^{-7}$ J·K^{-1} for fused NaCl.[20] However, the value may be regarded as an important measure for the polarity of liquid.

Table 2. Volumetric Properties of Ionic Liquids Based on Indium (V_m/nm^3) at 298.15 K

ionic liquid	M / g·mol^{-1}	M_+ / g·mol^{-1}	ρ / g·cm^{-3}	V_m / nm^3	V_+ / nm^3	r_+ / nm	S^0 / J·K^{-1}·mol^{-1}
[C_2mim][$InCl_4$][a]	367.8	111.2	1.6392	0.3725	0.2083	0.368	493.9
[C_4mim][$InCl_4$][b]	395.9	139.2	1.5557	0.4225	0.2583	0.395	556.1
[C_5mim][$InCl_4$][c]	409.9	153.2	1.5108	0.4507	0.2865	0.409	591.3
[C_6mim][$InCl_4$][d]	423.9	167.2	1.47692	0.4768	0.3126	0.421	623.8

[a] Ref 18. [b] Ref 12. [c] Ref 10. [d] This work.

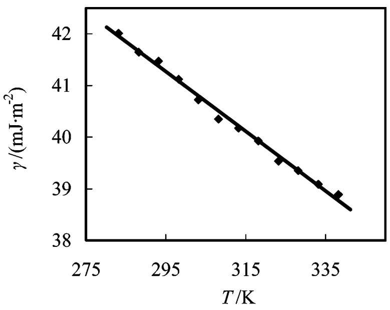

Figure 4. Surface tension, γ, obtained at different temperatures as a function of T.

The value of $k = 1.763 \cdot 10^{-7}$ J·K^{-1} implies that the polarity of [C$_6$mim][InCl$_4$] is less than fused salt and closer to an organic liquid.

The values of γ obtained at different temperature have been fitted against T by least-squares to a linear equation (see Figure 4). The correlation coefficient is 0.997, and the standard deviation is 0.08 mJ·m^{-2} for the fitting. From the slope of the fitted line, the surface excess entropy, S_a, could be obtained, that is, $S_a = -(\partial\gamma/\partial T)_p = 57.9 \cdot 10^{-3}$ mJ·K^{-1}·m^{-2}. In addition, the surface excess energy likewise may be obtained from the surface tension measured in this work: $E_a = \gamma - T(\partial\gamma/\partial T)_p = 58.4$ mJ·m^{-2}. In comparison with fused salts, for example, $E_a = 146$ mJ·m^{-2} (for fused NaNO$_3$), and the value of E_a for [C$_6$mim][InCl$_4$] is much lower and is close to an organic liquid, for example, 67 mJ·m^{-2} (for benzene) and 51.1 mJ·m^{-2} (for octane).[20] This fact shows that interaction energy between ions in [C$_6$mim][InCl$_4$] is less than that in fused salts. By Glasser's theory,[13] the lattice energy, U_{POT}, may be estimated using the following equation

$$U_{POT}/\text{kJ·mol}^{-1} = 1981.2[\rho/M]^{1/3} + 103.8 \qquad (6)$$

so that $U_{POT} = 404.1$ kJ·mol^{-1} for [C$_6$mim][InCl$_4$] was obtained, and the value is much less than that of fused salts, for example, $U_{POT} = 613$ kJ·mol^{-1} for fused CsI[17] which is the lowest crystal energy among alkali-halides. The low crystal energy is the underlying reason for forming an ionic liquid at room temperature.

Estimation of Vaporization Enthalpies for the IL. Kabo and colleagues[14] put forward an empirical equation for estimation of the enthalpy of vaporization, $\Delta_l^g H_m^0$ (298 K), of ionic liquids

$$\Delta_l^g H_m^0\,(298\text{ K}) = A(\gamma V^{2/3} N^{1/3}) + B \qquad (7)$$

where N is Avogadro's constant, and A and B are empirical parameters, with their values being $A = 0.01121$ and $B = 2.4$ kJ·mol^{-1}, respectively. The molar enthalpy of vaporization for ionic liquid [C$_6$mim][InCl$_4$] calculated from eq 7 was found to be 171.8 kJ·mol^{-1} at 298 K.

Rebelo et al.[15] put forward a method of estimating the hypothetical temperature of the normal boiling point (NBP) of an ionic liquid, T_b, in terms of critical temperature, T_c, obtained from the Eötvös equation. They thought that the relationship between T_b and T_c is $T_b \approx 0.6T_c$ for an ionic liquid; hence, T_b of ionic liquid [C$_6$mim][InCl$_4$] is 787 K approximately. The molar enthalpy of vaporization for ionic liquid [C$_6$mim][InCl$_4$] at NBP, $\Delta_l^g H_m^0$ (T_b), can be estimated by a Trouton constant ($\approx$90 J·mol^{-1}·K^{-1}), In this case, $\Delta_l^g H_m^0$ (787 K) = 78.7 kJ·mol^{-1} at NBP. The difference between $\Delta_l^g H_m^0$ (T_b) estimated in terms of Rebelo's method and $\Delta_l^g H_m^0$ (298 K) estimated in terms of Kabo's method is due to the heat capacity difference between the liquid and gas phases at different temperatures.

Table 3. Estimated Values for the Vaporization Enthalpy and Normal Boiling Point of Ionic Liquids

	γ	10^6V	$\Delta_l^g H_m$	T_c	T_b	$\Delta_l^g H_m(T_b)$
ionic liquid	mJ·m^{-2}	m^3·mol^{-1}	kJ·mol^{-1}	K	K	kJ·mol^{-1}
[C$_5$mim][InCl$_4$][a]	43.4	271.3	174.7	1368	821	73.9
[C$_6$mim][InCl$_4$][b]	41.1	287.0	171.8	1312	787	70.8
[C$_5$mim][GaCl$_4$][a]	43.1	265.2	170.7	1398	839	75.5
[C$_4$mim][AlCl$_4$][c]	45.6	248.8	173.2	1492	895	80.6
[C$_5$mim][AlCl$_4$][a]	42.6	265.4	169.0	1341	805	72.4

[a] Ref 10. [b] This work. [c] Ref 21.

Supposing a linear change of $\Delta_l^g H_m^0$ with temperature in the range between 298 K and T_b, the hypothetical vapor pressures, p, of the IL [C$_6$mim][InCl$_4$] may be estimated by the Clapeyron−Clausius equation. The values of p are 101.325 kPa at 787 K, 18.53 kPa at 698 K, 895 Pa at 598 K, 5.9 Pa at 498 K, $1 \cdot 10^{-3}$ Pa at 398 K, and $7 \cdot 10^{-11}$ Pa at 298 K. The result is in agreement with our experience: very low vapor pressure before decomposition of the IL and negligible vapor pressure at ambient temperature.

Applying Kabo's method and Rebelo's method to our previous work, the results are listed in Table 3. From the values in Table 3, $\Delta_l^g H_m^0$ ([C$_5$mim][InCl$_4$]) is larger than $\Delta_l^g H_m^0$ ([C$_6$mim][InCl$_4$]), and $\Delta_l^g H_m^0$ ([C$_4$mim][AlCl$_4$]) is larger than $\Delta_l^g H_m^0$ ([C$_5$mim][AlCl$_4$]). This implies that the estimated enthalpy of vaporization of ILs decreases with length of aliphatic chains in the 1-alkyl-3-methylimidazolium cation, and it can be interpreted considering that longer side chains decrease the relative importance of Coulomb forces leading to smaller values of $\Delta_l^g H_m^0$. This is in great agreement with the values of [C$_n$mim][PF$_6$] in Table 5 of ref 14. For example, $\Delta_l^g H_m^0$ ([C$_4$mim][PF$_6$]) = 154.8 kJ·mol^{-1} > $\Delta_l^g H_m^0$ ([C$_6$mim][PF$_6$]) = 139.8 kJ·mol^{-1} and $\Delta_l^g H_m^0$ ([C$_4$mim][BF$_4$]) = 128.2 kJ·mol^{-1} > $\Delta_l^g H_m^0$ ([C$_8$mim][BF$_4$]) = 122.0 kJ·mol^{-1}.

Interstice Model for Ionic Liquid. For a pure ionic liquid, a new theoretic model[2] has been put forward on the basis of the following assumptions: (1) Because of the large size and the asymmetric shape, the ions may not be closely packed, and lots of interstices between ions exist. (2) To calculate the volume, the interstice is regarded as a bubble. (3) There are $2N$ interstices for 1 mol of 1−1 ionic liquid, where N is Avogadro's constant. (4) The interstice in [C$_6$mim][InCl$_4$] can move about like an ion or another particle, and in the movement, the interstice does not vanish but can be compressed and expanded, which has an extra feature of motion of an interstice called the breathing motion.

According to the same procedure of the whole model of molten salt, the expression of calculation of interstice volume, v, was obtained on the classical statistical mechanics

$$v = 0.6791(k_b T/\gamma)^{3/2} \qquad (8)$$

where k_b is the Boltzmann constant; T is thermodynamic temperature; and γ is surface tension of the ionic liquid. According to eq 8, the values of the average volume of the interstices of ionic liquids at different temperatures are obtained. From Table 1, the surface tension of [C$_6$mim][InCl$_4$], γ, is 41.1 mJ·m^{-2}, at 298.15 K, and the average volume of the interstice, v, is $21.51 \cdot 10^{-24}$ cm^3. Then, the total volume of the interstice is $\sum v = 2Nv = 25.89$ cm^3 (formula unit)$^{-1}$. The volume fraction of interstice, $\sum v/V$, is about 9 % for ionic liquid [C$_6$mim][InCl$_4$]. This value most closely approaches those of the majority of materials which exhibit (10 to 15) % volume expansion in the process from the solid to the liquid state.

The volume of ionic liquid, V, consists of the inherent volume, V_i, and the total volume of the all interstices, $\sum \upsilon = 2N\ \upsilon$; that is

$$V = V_i + 2N\nu \tag{9}$$

If the expansion of IL volume only results from the expansion of the interstices when temperature increases, then calculation expression of α was derived from the interstice model

$$\alpha = (1/V)(\partial V/\partial T)_p = 3N\nu/VT \tag{10}$$

The value of α (calculated) = $4.54 \cdot 10^{-4}\ K^{-1}$ at 298.15 K, and that of α (experimental) = $6.52 \cdot 10^{-4}\ K^{-1}$; that is, α (calculated) was compared with the experimental one.

4. Conclusions

The density and surface tension of the IL $[C_6mim][InCl_4]$ were determined in the temperature range of (283.15 to 338.15) K. The volume of anion $InCl_4^-$, (0.1642 ± 0.001) nm^3, and the surface entropy of the IL, $S_a = 57.9 \cdot 10^{-3}\ mJ \cdot K^{-1} \cdot m^{-2}$, were estimated by extrapolation, respectively. In terms of Glasser's theory, the standard molar entropy, S^0 (298) = 623.8 $J \cdot K \cdot mol^{-1}$, and lattice energy, U_{POT} = 404.1 $kJ \cdot mol^{-1}$, of the IL were estimated, respectively. Using Kabo's method and Rebelo's method, the molar enthalpy of vaporization of the IL, $\Delta_l^g H_m^0$ (298 K), and $\Delta_l^g H_m^0$ (T_b), at a hypothetical normal boiling point, T_b = 787 K, were estimated, respectively. According to the interstice model, the thermal expansion coefficient of IL $[C_6mim][InCl_4]$ estimated was close to the experimental one.

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