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Activity Coefficients at Infinite Dilution of Organic Solutes in 1-Decyl-3-methylimidazolium Tetrafluoroborate Using Gas−Liquid Chromatography

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ABSTRACT: Activity coefficients at infinite dilution of pentane, hexane, heptane, octane, nonane, cyclohexane, methylcyclohexane, 2,2,4-trimethylpentane, cyclohexene, styrene, benzene, toluene, ethylbenzene, *o*-xylene, *m*-xylene, and *p*-xylene in 1-decyl-3-methylimidazolium tetrafluoroborate have been determined using gas−liquid chromatography over a temperature range of (303.15 to 363.15) K with the ionic liquid as the stationary phase. The partial molar excess enthalpies at infinite dilution were also determined for the solutes from the temperature dependence of the experimental activity values.

INTRODUCTION

This work is a continuation of a study on the activity coefficients at infinite dilution ($\gamma_{i,3}^{\infty}$) of various solutes (i) by the gas−liquid chromatographic method (GLC) for ionic liquids: 1-decyl-3-methylimidazolium tetrafluoroborate. Activity coefficients at infinite dilution of a solute $\gamma_{i,3}^{\infty}$ can be used to quantify the volatility of the solute and to provide information on the intermolecular interaction between solvent and solute. Values of $\gamma_{i,3}^{\infty}$ are important for the selection of solvents for extraction and extractive distillation and the reliable design of thermal separation processes when the traces of impurities have to be removed. Reliable information about the separation factor at infinite dilution is also required to avoid the oversizing of distillation columns or stripping processes.

Ionic liquids have been used in extraction of polluted materials from burning oil or aqueous systems. Imidazolim based ionic liquids have been used to remove sulfur[1−3] and dibenzothiophene[4] from fuels, phenols,[5] amino acids,[6] ethanol,[7] THF,[8] and metal ions[9] from water. They also were used to separate organic solutes such as ethyl acetate and ethanol.[10] It is necessary to estimate the effect of changing alkyl chain when using thermodynamic theory such as LSER and UNIFAC to make the prediction. The more experimental data, the more precise the correlated parameters are. The experiment on ionic liquid 1-decyl-3-methylimidazolium tetrafluoroborate is use with the aim of enhancing the chemical engineering data.

EXPERIMENTAL SECTION

The ionic liquid 1-decyl-3-methylimidazolium tetrafluoroborate ([C$_{10}$mim][BF$_4$]) was purchased from Shanghai Cheng Jie Chemical Co., Ltd. with a purity greater than 99% according to manufacturer's specifications, and impurities [w (Cl$^-$) < 5 × 10^{-4}, w (H$_2$O) < 5 × 10^{-4}]. The solutes provided by Beijing Chemical Reagents Company were analytical reagents with a purity of 99 % and were used without further purification. Dry helium was used as the carrier gas, and Chromosorb W/AW-DMCS 100/120 mesh was used as solid support for the ionic liquid in the GC column. The chemical shifts for the ^{1}H NMR spectrum (parts per million, C_3D_6O) appear as follows: [C$_{10}$mim-[BF$_4$], δ: 9.06 (s, 1H, CH−N), 7.73−7.67 (d, 2H, CH═CH−N), 4.15−4.12 (t, 2H, CH$_2$Me), 3.85 (s, 3H, CH$_3$N), 1.82−1.75 (t, 2H, CH$_2$N), 1.25−1.22 (m, 14H, (CH$_2$)$_7$), 0.86−0.84 (t, 3H, CH$_2$-CH$_3$).

The GLC apparatus, column preparation, packing method, experimental process, and the method of checking the stability of the experimental conditions in this work are the same as those described previously.[11−13] The amount of stationary phase (ionic liquid) was 1.5164 mmol. No measurements were taken for styrene and ethylbenzene at 303.15 K because of their long retention times.

The $\gamma_{i,3}^{\infty}$ values were obtained by the equation proposed by Cruickshank et al[14] and Everett.[15] The way of deducing $\gamma_{i,3}^{\infty}$ from the experiments was given in our previous work.[11−13]

The data for calculating the correction terms have been obtained in the following way. For all solutes, values of P_i^0 were calculated from the Antoine equation, with Antoine constants given by Boublik et al.[16] Molar volumes of solutes V_i^0 were estimated using experimental values of their densities; partial molar volumes of solute at infinite dilution V_i^{∞} have been assumed to be equal to V_i^0. Values of B_{11} and B_{12} have been estimated according to the equations suitable for nonpolar liquids by Tsonopolous' method[17] with an uncertainty of < ± 10 cm^3·mol^{-1}. The critical parameters needed for the calculations were available from ref 17. The mixed critical properties P_{cij}, T_{cij}, V_{cij}, and Z_{cij} and mixed acentric factor ω_{ij} were calculated by using equations given in refs 14 and 15. The outlet pressure P_o was kept equal to the atmospheric pressure.

Retention time, dead time, column temperature, flow rate, input and output pressure, and the mass of the stationary phase

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Table 1. Experimental Activity Coefficients at Infinite Dilution of Various Polar Solutes in the Ionic Liquid 1-Decyl-3-methylimidazolium Tetrafluoroborate as the Stationary Phase at Temperatures of (303.15 to 363.15) K

solutes	T/K = 303.15	T/K = 313.15	T/K = 323.15	T/K = 333.15	T/K = 343.15	T/K = 353.15	T/K = 363.15
pentane	6.88	6.72	6.53	6.29	6.12	6.01	5.93
hexane	7.36	7.16	7.03	6.94	6.86	6.81	6.68
heptane	8.01	7.97	7.95	7.93	7.91	7.89	7.88
octane	9.30	9.26	9.21	9.20	9.17	9.16	9.13
nonane	10.72	10.71	10.69	10.68	10.67	10.65	10.63
cyclohexane	4.42	4.43	4.43	4.47	4.50	4.56	4.69
methylcyclohexane	5.19	5.23	5.24	5.28	5.32	5.36	5.39
2,2,4-trimethylpentane	9.13	9.11	9.10	9.08	9.07	9.07	9.06
cyclohexene	3.03	3.01	2.99	2.97	2.96	2.96	2.96
styrene[a]		1.25	1.22	1.20	1.18	1.16	1.14
benzene	1.15	1.14	1.11	1.10	1.08	1.07	1.06
toluene	1.52	1.49	1.47	1.43	1.41	1.40	1.35
ethylbenzene[a]		2.02	2.01	1.99	1.98	1.97	1.96
o-xylene	1.79	1.77	1.75	1.73	1.72	1.72	1.71
m-xylene	2.03	2.02	1.97	1.95	1.94	1.92	1.91
p-xylene	1.99	1.96	1.93	1.92	1.91	1.88	1.86

[a] Values are measured over the temperature range (313.15 to 363.15) K.

Table 2. Coefficients of eq 1, a and b, Calculated (at 298.15 K) Activity Coefficients at Infinite Dilution ($\gamma_{i,298.15\mathrm{K}}^{\infty}$) Using eq 1, Values of $H_i^{E,\infty}$ Derived from eq 1, and Standard Deviation σ

	r_i[a]	δ_i[a]		b		$H_i^{E,\infty}$	
solutes i	$\mathrm{cm^3 \cdot mol^{-1}}$	$(\mathrm{J \cdot cm^{-3}})^{0.5}$	a	K	$\gamma_{i,298.15\mathrm{K}}^{\infty}$	$\mathrm{kJ \cdot mol^{-1}}$	σ[b]
pentane	58.03	13.858	0.975	289.7	7.01	2.409	0.0053
hexane	68.26	14.988	1.447	164.5	7.38	1.368	0.0044
heptane	78.49	15.208	1.983	29.4	8.02	0.244	0.0007
octane	88.72	15.360	2.122	32.3	9.30	0.269	0.0010
nonane	98.95	15.527	2.323	15.0	10.73	0.125	0.0004
cyclohexane	61.4	16.7	1.795	−96.5	4.36	−0.802	0.0092
methylcyclohexane	70.46	16.27	1.875	−69.6	5.16	−0.579	0.0018
2,2,4-trimethylpentane	88.69	14.051	2.165	13.9	9.13	0.116	0.0005
cyclohexene	57.04	17.611	0.956	45.5	3.03	0.378	0.0027
styrene	66.25	19.127	−0.425	202.5	1.29	1.684	0.0014
benzene	48.4	18.706	−0.378	157.3	1.16	1.308	0.0036
toluene	59.51	18.346	−0.254	204.8	1.54	1.703	0.0061
ethylbenzene	69.74	18.043	0.473	72.2	2.04	0.600	0.0010
o-xylene	70.66	18.453	0.301	84.3	1.79	0.701	0.0035
m-xylene	70.66	18.09	0.317	118.7	2.04	0.987	0.0043
p-xylene	70.66	17.838	0.301	116.7	2.00	0.970	0.0031

[a] Values are obtained from ref 25. [b] $\sigma = [\sum(\gamma_{\mathrm{cal}} - \gamma_{\mathrm{exp}})^2/n]^{1/2}$.

all have experimental errors. The uncertainty of the flow rate U_0 is estimated to be ± 0.1 mL/min at column temperature. The uncertainties of the input pressure P_i and the output pressure P_0 are estimated to be ± 0.6 kPa and ± 0.03 kPa, respectively. The uncertainty of determining n_3 was estimated to be ± 0.1 mg. The effect caused by the cross virial coefficient is 2 % to 5 %, considering this correction, according to the error propagation law, $\gamma_{i,3}^{\infty}$ is estimated to have a relative uncertainty within ± 5.6 %.

RESULTS AND DISCUSSION

The values of $\gamma_{i,3}^{\infty}$ of solutes in $[\mathrm{C_{10}mim}][\mathrm{BF_4}]$ obtained over a temperature range of (303.15 to 363.15) K were listed in Table 1.

The results of $\gamma_{i,3}^{\infty}$ were correlated with temperature by the following equation:

$$\ln \gamma_{i,3}^{\infty} = a + \frac{b}{(T/K)} \qquad (1)$$

According to the Gibbs−Helmholtz equation, the partial molar excess enthalpy at infinite dilution, $H_i^{E,\infty}$, can be obtained from eq 1. The coefficients a and b, $\gamma_{i,3}^{\infty}$ at the standard condition (T = 298.15 K) calculated using eq 1, the standard deviations σ are calculated according to $\sigma = [\Sigma(\gamma_{\mathrm{cal}} - \gamma_{\mathrm{exp}})^2/n]^{1/2}$, where n is the number of experimental data, and values of $H_i^{E,\infty}$ derived from eq 1 are listed in Table 2. The plots of measured $\ln \gamma_{i,3}^{\infty}$ vs $1/T$

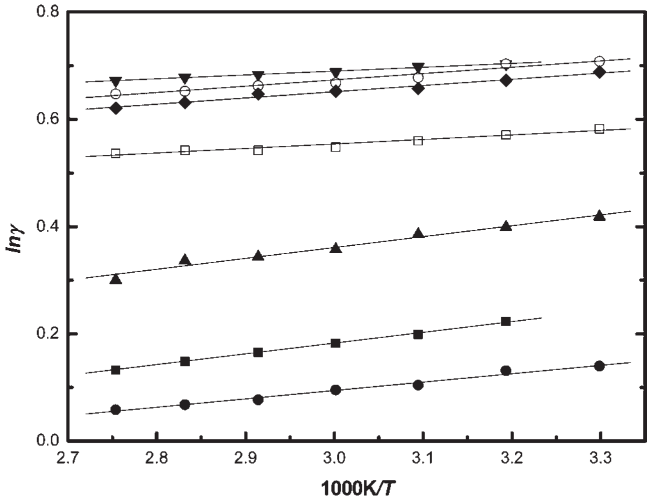

Figure 1. Plot of $\ln \gamma_i^\infty$ vs $1/T$ for the solutes: ■, styrene; ●, benzene; ▲, toluene; ▼, ethylbenzene; ♦, p-xylene; □, o-xylene; ○, *m*-xylene; —, linear correlation.

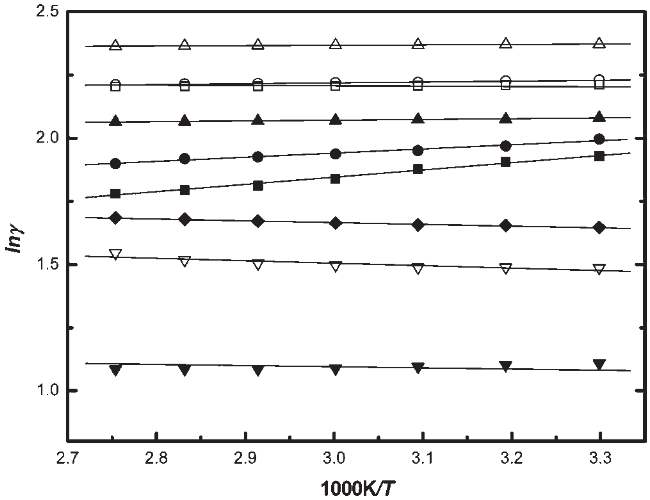

Figure 2. Plot of $\ln \gamma_i^\infty$ vs $1/T$ for the solutes: ■, pentane; ●, hexane; ▲, heptane; ○, octane; Δ, nonane; ▽, cyclohexane; ♦, methylcyclohexane; □, 2,2,4-trimethylpentane; ▼, cyclohexene; —, linear correlation.

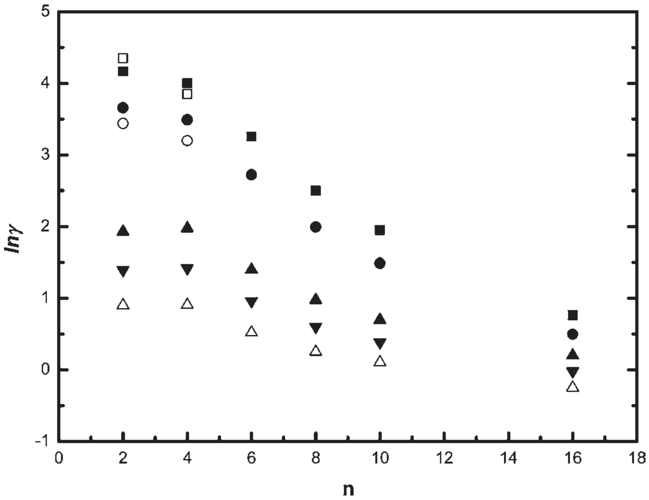

Figure 3. ln γ_i^∞ of hexane, cyclohexane, benzene, toluene, and ethylbenzene in $C_n$mim BF_4 ionic liquids at 343.15 K. ■, hexane, this work and refs 14−18; □, hexane, refs 11 and 12; ●, cyclohexane, this work and refs 14−18; ○, cyclohexane, refs 11 and 12; Δ, benzene, this work and refs 14−18; ▼, toluene, this work and refs 14−18; ▲, ethylbenzene, this work and refs 14−18.

Table 3. Selectivities S_{12}^∞ for Separation Problem: Hexane (1) + Benzene (2) and Cyclohexane (1) + Benzene (2) for 1-Alkyl-3-methylimidazolium Tetrafluoroborate ([$C_n$mim][BF_4], n = 2,4,6,8,10,16) and Selected ILs at 343.15K

	S_{12}		
ILs	hexane (1) + benzene (2)	cyclohexane (1) + benzene (2)	refs
[C_2mim][BF_4]	38.35	15.45	11
[C_4mim][BF_4]	25.70	13.41	12
[C_6mim][BF_4]	15.51	9.05	13
[C_8mim][BF_4]	9.46	5.71	13
[C_{10}mim][BF_4]	6.33	3.99	this work
[C_{16}mim][BF_4]	2.76	2.12	18
[1,3bmPY][CF_3SO_3]	15.63	8.44	21
[C_2OHmim][Tf_2N]	13.85	10.36	22
[C_2OC_1mim][Tf_2N]	13.73	8.45	22

values are given in Figures 1 and 2. The $\gamma_{i,3}^\infty$ values for styrene and ethylbenzene at 303.15 K were not measured since the retention times were far beyond the scope allowed by GC.

Figure 3 shows the activity coefficients at infinite dilution of hexane, cyclohexane, benzene, toluene, and ethylbenzene in $C_n$mim BF_4 (n = 2, 4, 6, 8, 10, 16) ionic liquids at T = 343.15K.[11−13,18−20] It can be seen that the γ^∞ decrease with the increasing of carbon chain length on the cations, and the difference of γ^∞ between different solute in ILs are also decrease with the increasing of carbon chain. The difference can be used to calculate the selectivity. Selectivity at infinite dilution ($S_{12}^\infty=\gamma_1^\infty/\gamma_2^\infty$) is used to describe the capacity of entrainer for a given separation problem. Table 3 shows selectivities for separation problem: hexane (1)/ benzene (2) and cyclohexane (1)/ benzene (2) for different imidazolium ionic liquids, 1-alkyl-3-methylimidazolium tetrafluoroborate ([$C_n$mim][BF_4], n = 2, 4, 6, 8, 10, 16) and selected new measured Ils.[21,22] The highest values of selectivity for the two separation system were obtained for 1-ethyl-3-methylimidazolium tetrafluoroborate.[11] The value of S_{12}^∞ decrease with the increasing of the carbon chain length on the cations.

Solubility Parameters. The activity coefficients at infinite dilution of solutes in a given solvent can be used to estimate the solvent solubility parameter. The activity coefficients at infinite dilution can be represented by a two-term equation which the combinatorial term can be represented by the Kikik et al.[23] modification to Flory's equation, the residual term is given by the regular solution theory

$$\ln \gamma_i^\infty = \ln \gamma_i^{\infty,\text{comb}} + \ln \gamma_i^{\infty,\text{res}} \qquad (2)$$

$$\ln \gamma_i^{\infty,\text{comb}} = \ln(r_i/r_s)^{2/3} + 1 - (r_i/r_s)^{2/3} \qquad (3)$$

$$\ln \gamma_i^{\infty,\text{res}} = (v_i/RT)(\delta_i - \delta_s)^2 \qquad (4)$$

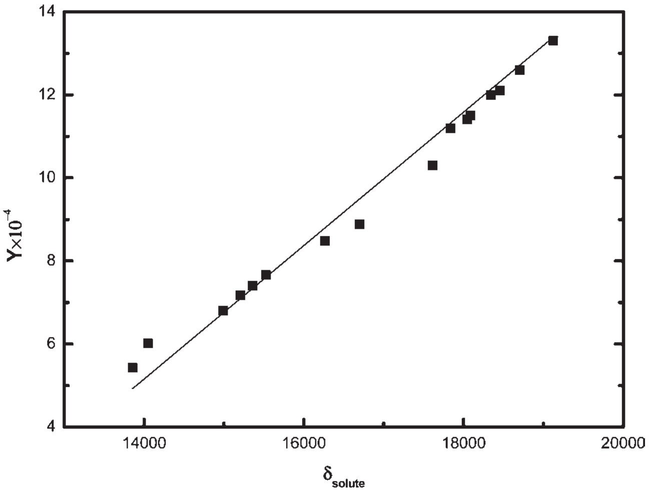

Figure 4. Residual function Y for $[C_{10}mim][BF_4]$ vs solute solubility parameters δ_{solute}. Regression line: $Y = 16.07\ \delta_{\text{solute}} - 173454$ ($R^2 = 0.9798$).

Table 4. Solubility Parameters of the Ionic Liquids

IL	$\delta \times 10^4$ $(\text{J}\cdot\text{m}^{-3})^{1/2}$	ref
$[C_2mim^+][BF_4^-]$	2.44	24
$[C_4mim^+][BF_4]$	2.43	24
$[C_6mim^+][BF_4]$	2.33	24
$[C_8mim^+][BF_4]$	2.25	24
$[C_{10}mim^+][BF_4]$	2.16	this work

where r_i is the van der Waals volumes of solute, r_s is the van der Waals volumes of solvent, v_i is the solute molar volume, and δ_i and δ_s are the solubility parameters of solute and solvent, respectively. A residual function Y can be rearranged from eq 4 according to ref 24

$$Y_i = -\frac{\ln \gamma_i^{\infty,\text{res}}}{v_i} + \frac{\delta_i^2}{RT} = \frac{2\delta_s}{RT}\delta_i - \frac{\delta_s^2}{RT} \qquad (5)$$

This equation shows that there is a linear relation between Y_i and the solute solubility parameter δ_i for a given solvent and temperature T. The value of the solvent solubility parameter δ_s can be obtained from the slope of this line. The values of $\gamma^{\infty,\text{comb}}$ were calculated by eq 3, The values of experimental γ^{∞} were obtained from this work, the value of $\ln \gamma^{\infty,\text{comb}}$ were calculated by eq 2, and the value of Y_i for each solvent were calculated by eq 5. Information on v_i, δ_i, and r_i were obtained from ref 25. The van der Waals volumes of ILs were calculated by group contribution method.[26]

The linear dependence between Y_i and δ_i obtained from the γ^{∞} data on $[C_{10}mim][BF_4]$ was displayed in Figure 4. The solubility parameter calculated by this procedure was 21588 $(\text{J}\cdot\text{m}^{-3})^{1/2}$, and it is the smallest compared with the solubility parameters for the 1-alkyl-3-methylimidazolium tetrafluoroborate ILs studied by Bottini;[24] the order is $[C_2mim][BF_4] > [C_4mim][BF_4] > [C_6mim][BF_4] > [C_8mim][BF_4] > [C_{10}mim][BF_4]$ as listed in Table 4.

The solubility parameter provides a numerical estimation of the degree of interaction between materials. A large activity coefficient of a solute in a solvent usually means that they have small mutual solubilities and weak molecular interaction. The large activity coefficient of solute in $[C_2mim][BF_4]$ means that it can escape easily when using this IL as the entrainer for the separation of aliphatics from aromatics. The solubility parameter provides another probability to analyze the selectivity. The difference between the aliphatic and that of $[C_2mim][BF_4]$ is large (their interactions are weak), and the difference between that of the aromatic and the $[C_2mim][BF_4]$ is small (their interactions are stronger). It explains the difference of selectivity between different ionic liquids, and it can provide the potential ILs when the anion of the ionic liquid is changed to make it the best entrainer, as the difference in the solubility parameter of the solute and IL is the largest.

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