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Activity Coefficients at Infinite Dilution of Polar Solutes in 1-Butyl-3-methylimidazolium Trifluoromethanesulfonate Using Gas–Liquid Chromatography

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Activity coefficients at infinite dilution γ_i^∞ have been measured for 17 polar solutes (linear and branched C_1 to C_5 alcohols, acetonitrile, acetone, 1,4-dioxane, tetrahydrofuran, ethyl acetate, and chloromethanes) in the ionic liquid 1-butyl-3-methylimidazolium trifluoromethanesulfonate ([BMIM][CF_3SO_3]) by the gas–liquid chromatographic method with the ionic liquid as the stationary phase. The measurements were carried out in the temperature range of (303.15 to 363.15) K. The partial molar excess enthalpies at infinite dilution $H_i^{E,\infty}$ of the solutes in the ionic liquid were also derived from the temperature dependence of the γ_i^∞ values.

Introduction

This work is a continuation of our studies on the determination of activity coefficients at infinite dilution γ_i^∞ of organic solutes in hydrophilic ionic liquids (ILs) by the gas–liquid chromatographic (GLC) method.[1–4] Activity coefficients at infinite dilution γ_i^∞ of a solute *i* can be used to quantify the volatility of the solute as well as to provide information on the intermolecular energy between solvent and solute.[5] The use of a steady-state GLC technique for the determination of γ_i^∞ has been employed by many researchers and has proved quite reliable.[6] ILs as an ideal stationary phase are in particular reliable for the determination of γ_i^∞ by this method because of their negligible vapor pressures and thermal stability phase. Heintz et al.[7–15] and Letcher et al.[16–24] measured γ_i^∞ of various solutes in a number of ILs.

Experimental Section

The solutes including linear and branched C_1 to C_5 alcohols (methanol, ethanol, 1-propanol, 1-butanol, 1-pentanol, 2-propanol, 2-methylpropanol, 2-butanol, 3-methylbutanol), acetonitrile, acetone, 1,4-dioxane, tetrahydrofuran, ethyl acetate, and chloromethanes (dichloromethane, trichloromethane, and tetrachloromethane) were purchased from Beijing Chemical Reagents Company. Their mass fraction purities were above 99 %. The ionic liquid 1-butyl-3-methylimidazolium trifluoromethanesulfonate ([BMIM][CF_3SO_3]) was purchased from Hangzhou Chemer Chemical Co., Ltd. Its mass fraction purity was above 99 % according to specifications, with the following certified amounts of impurities: $w\ (Cl^-) < 5 \cdot 10^{-4}$. The mass fraction of water was checked to be less than 10^{-3} by Karl Fischer titration. The chemical shifts for the ^1H NMR spectrum (parts per million, acetone-d_3) appeared as follows: δ 9.102 [s, 1H, H(2)], 7.786 [s, 1H, H(4)], 7.724 [s, 1H, H(5)], 4.369 [t, 2H, NCH_2], 4.046 [s, 3H, NCH_3], 1.950 [m, 2H, $NCH{-}CH_2$], 1.403 [m, 2H, $NCH_2CH_2{-}CH_2$], and 0.959 [t, 3H, CH_3]. These results agreed well with literature data.[25] Impurity peaks were not observed in the ^1H NMR.

Table 1. Relative Uncertainties in the Measured and Derived Quantities

parameters	relative uncertainty
$(t_r - t_G)$/min	± 3.3 %
U_0/mL·min^{-1}	± 0.5 %
p_i/kPa	± 0.6 %
P_0/kPa	± 0.02 %
J	± 1 %
p_i^0/kPa	± 0.01 % to ± 0.25 %
n_3/mol	± 0.5 %
γ_i^∞	± 4 %

Table 2. Experimental Activity Coefficients at Infinite Dilution γ_i^∞ for Various Solutes in the Ionic Liquid 1-Butyl-3-methylimidazolium Trifluoromethanesulfonate as the Stationary Phase at Temperatures of (303.15 to 363.15) K

	T/K						
solutes (*i*)	303.15	313.15	323.15	333.15	343.15	353.15	363.15
Alcohols							
methanol[a]		0.675	0.665	0.654	0.645	0.630	0.615
ethanol	1.142	1.085	1.027	0.972	0.954	0.926	0.892
1-propanol	1.475	1.401	1.349	1.289	1.234	1.181	1.125
1-butanol[a]		1.854	1.776	1.709	1.570	1.510	1.453
1-pentanol[b]			2.341	2.157	2.065	1.965	1.887
2-propanol	1.445	1.371	1.308	1.260	1.212	1.162	1.123
2-methylpropanol	1.843	1.743	1.636	1.507	1.383	1.292	1.199
2-butanol	1.967	1.815	1.690	1.603	1.554	1.488	1.425
3-methylbutanol[b]			2.005	1.897	1.806	1.754	1.635
Solvents							
acetonitrile	0.784	0.771	0.755	0.741	0.730	0.717	0.701
acetone	0.864	0.887	0.902	0.919	0.946	0.971	1.010
1, 4-dioxane	1.103	1.132	1.176	1.201	1.237	1.269	1.293
tetrahydrofuran	1.238	1.319	1.383	1.450	1.494	1.549	1.604
ethyl acetate	1.889	1.938	1.962	2.015	2.068	2.106	2.143
Chloromethanes							
dichloromethane	0.932	1.023	1.077	1.103	1.132	1.174	1.200
trichloromethane	0.827	0.865	0.924	0.980	1.042	1.101	1.162
tetrachloromethane	3.653	3.772	3.906	3.987	4.085	4.172	4.266

[a] Values are measured over the temperature range (313.15 to 363.15) K.
[b] Values are measured over the temperature range (323.15 to 363.15) K.

Before use, the IL was subjected to vacuum evaporation at (323 to 333) K over 24 h to remove possible traces of solvents and moisture. The GLC apparatus, column preparation, packing

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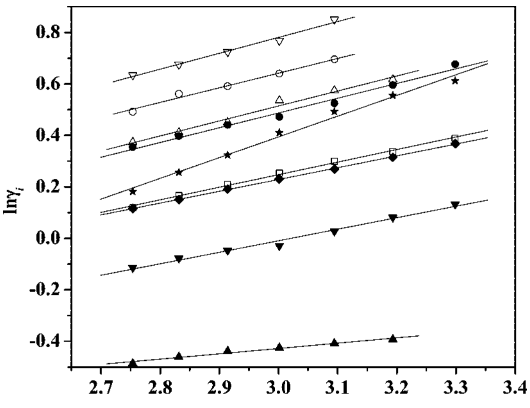

Figure 1. Plot of ln γ_i^∞ for the solutes i vs $1/T$ and linear fit: ▲, methanol; ▼, ethanol; ◆, 2-propanol; □, 1-propanol; ★, 2-methylpropanol; △, 1-butanol; ▽, 1-pentanol; ●, 2-butanol; ○, 3-methylbutanol; — linear fit.

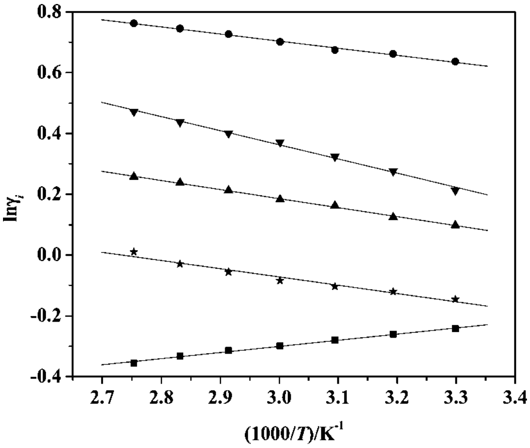

Figure 2. Plot of ln γ_i^∞ for the solutes i vs $1/T$ and linear fit: ■, acetonitrile; ★, acetone; ▲, 1,4-dioxane; ▼, tetrahydrofuran; ●, ethylacetate; — linear fit.

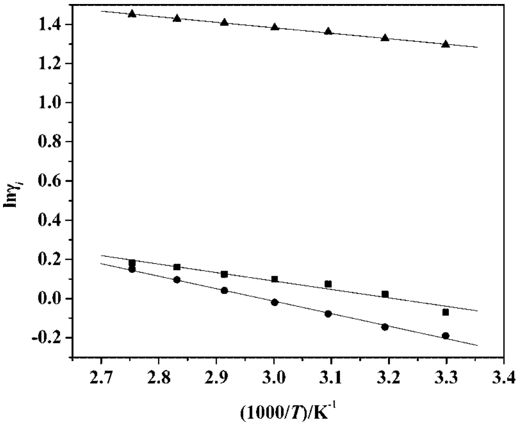

Figure 3. Plot of ln γ_i^∞ for the solutes i vs $1/T$ and linear fit: ■, dichloromethane; ●, trichloromethane; ▲, tetrachloromethane; — linear fit.

Table 3. Coefficients *a* and *b* of Equation 4, γ_i^∞ at 298.15 K Calculated Using Equation 4, Values of $H_i^{E,\infty}$ Derived from Equation 4, and Standard Deviation *σ*

solute (i)	a	b/K	$\gamma^\infty_{i,298.15\mathrm{K}}$	$H_i^{E,\infty}$/kJ·mol^{-1}	σ
		Alcohols			
methanol	−1.0469	206.23	0.70	1.71	0.01
ethanol	−1.3530	447.93	1.16	3.72	0.01
1-propanol	−1.2180	488.42	1.52	4.06	0.01
1-butanol	−1.2325	582.37	2.06	4.84	0.01
1-pentanol	−1.0724	617.84	2.72	5.14	0.01
2-propanol	−1.1479	458.84	1.48	3.81	0.003
2-methylpropanol	−2.0243	806.24	1.97	6.70	0.02
2-butanol	−1.2324	573.25	1.99	4.77	0.01
3-methylbutanol	−1.0679	570.04	2.33	4.74	0.01
		Solvents			
acetonitrile	−0.9076	202.36	0.80	1.68	0.003
acetone	0.7435	−271.87	0.85	−2.26	0.01
1,4-dioxane	1.0769	−297.04	1.08	−2.47	0.003
tetrahydrofuran	1.7584	−465.23	1.22	−3.87	0.01
ethylacetate	1.4075	−234.55	1.86	−1.95	0.004
		Chloromethanes			
dichloromethane	1.3831	−431.13	0.94	−3.58	0.02
trichloromethane	1.9015	−638.23	0.79	−5.31	0.01
tetrachloromethane	2.2246	−280.61	3.61	−2.33	0.003

Table 4. Activity Coefficients at Infinite Dilution γ_i^∞ at 298.15 K for Methanol in Ionic Liquid Solvents

solvents	$\gamma^\infty_{i,298.15\mathrm{K}}$	ref
[BMIM][N(CF_3SO_2)$_2$]	1.3	13
[BMIM][$OcOSO_3$]	0.386	22
[BMIM][$MDEGSO_4$]	0.345	23
[BMIM][BF_4]	0.86[a]	2
[BMIM][CF_3SO_3]	0.70[a]	this work

[a] Extrapolated value.

method, experimental process, and the method of checking the stability of the experimental conditions in this work are the same as those described previously.[1–4] According to the literature,[9,13] the mass fraction of the ionic liquid coating was about (27 to 60) % of the support material. In this work, the mass fraction was about 60 % of the support material, and dichloromethane was used as solvent to coat the ionic liquid onto the solid support. Dry nitrogen was used as the carrier gas. The volume of the samples injected into the GC probes was about (0.1 to 3) μL, and the peaks were found to be symmetrical, independent of the carrier gas flow rate. The experiments were carried out from (303.15 to 363.15) K. At a given temperature, each operation was repeated at least three times to check the reproducibility. To check the stability of the experimental conditions, such as the possible elution of the stationary phase by the carrier gas stream, the measurements of retention time were repeated systematically every (6 to 8) h for hexane and benzene. No change of the retention time was observed during 80 h of continuous operation.

The γ_i^∞ values were obtained by the equation proposed by Cruickshank et al.[26] and Everett.[27]

$$\ln \gamma_{i,3}^\infty = \ln\left(\frac{n_3RT}{V_\mathrm{N}p_i^0}\right) - \frac{B_{11} - V_i^0}{RT}p_i^0 + \frac{2B_{12} - V_i^\infty}{RT}Jp_0 \quad (1)$$

$$V_\mathrm{N} = JU_0(t_\mathrm{r} - t_\mathrm{G})\frac{T_\mathrm{col}}{T_\mathrm{f}}\left[1 - \frac{p_w^0}{p_0}\right] \quad (2)$$

In eq 1, the $\gamma_{i,3}^\infty$ is the activity coefficient of solute i at infinite dilution in the stationary phase (3). The data needed for the equations have been obtained in the following way. Molar volumes of solutes V_i^0 were estimated using their experimental densities, and partial molar volumes of solute at infinite dilution V_i^∞ have been assumed to be equal to V_i^0. Values of p_i^0 for all solutes were calculated from the Antoine equation, with Antoine coefficients given by Boublik et al.[28] B_{11} is the second virial coefficient of the solute (1), and B_{12} is the cross second virial coefficient of the solute (1) with the carrier gas (2). B_{11} and B_{12} have been estimated according to the equations suitable for polar liquids by Tsonopolous's method.[29] Critical parameters needed for the calculations were available from the literature.[29]

Table 5. Selectivities, S_{ij}^{∞}, at Infinite Dilution for Hexane (*i*)[a]/Polar Solute (*j*) Separation at 298.15 K

linear alcohol (*j*)	S_{ij}^{∞}	branched alcohol (*j*)	S_{ij}^{∞}	solvents (*j*)	S_{ij}^{∞}	chloromethanes (*j*)	S_{ij}^{∞}
methanol	55.92	2-propanol	26.51	acetonitrile	49.28	dichloromethane	41.74
ethanol	33.76	2-methylpropanol	19.86	acetone	46.39	trichloromethane	49.79
1-propanol	25.75	2-butanol	19.66	1,4-dioxane	36.16	tetrachloromethane	10.86
1-butanol	19.07	3-methylbutanol	16.85	tetrahydrofuran	32.16		
1-pentanol	14.42			ethylacetate	21.07		

[a] The $\gamma_{i,298.15\text{K}}^{\infty}$ value of hexane was from ref 4.

The cross critical properties P_{cij}, T_{cij}, V_{cij}, and Z_{cij} and acentric factor ω_{ij} were calculated by equations given in the literature.[29,30]

The factor *J* appearing in eqs 1 and 2 corrects for the influence of the pressure drop along the column given by[31]

$$J = \frac{3}{2}\frac{(p_i/p_0)^2 - 1}{(p_i/p_0)^3 - 1} \qquad (3)$$

where p_i and p_0 are the inlet and outlet pressures of the GC column, respectively.

Retention time, dead time, column temperature, flow rate, inlet and outlet pressure, and the mass of the stationary phase all have experimental errors. The errors in the γ_i^{∞} were obtained from the law of propagation of errors. The relative uncertainties in the measured and derived quantities are listed in Table 1.

Results and Discussion

Experimental results of γ_i^{∞} for 17 solutes in [BMIM][CF_3SO_3] at different temperatures were presented in Table 2. They were correlated with temperature by the following equation

$$\ln \gamma_i^{\infty} = a + \frac{b}{(T/\text{K})} \qquad (4)$$

The plots of measured ln γ_i^{∞} vs $1/T$ values and the linear fit of their data are given in Figures 1 to 3, which showed a fairly good fitting quality of eq 4. According to the Gibbs–Helmholtz equation, the values for the partial molar excess enthalpy at infinite dilution $H_i^{E,\infty}$ of solute *i* can be obtained from the slope of a straight line derived from eq 4, which were listed in Table 3 as well as the coefficients *a* and *b*, the standard deviation σ of the fitted equation, and the values of γ_i^{∞} at 298.15 K. The relative uncertainty of $H_i^{E,\infty}$ is about ± 10 %.

The γ_i^{∞} values of the linear *n*-alkanols increase with increasing chain length. Branching in the alkane skeleton slightly reduces the γ_i^{∞} values in comparison with the corresponding linear alcohol. The γ_i^{∞} values for other polar solutes was in the order of ethylacetate > tetrahydrofuran > 1,4-dioxane > acetone > acetonitrile. For dichloromethane and trichloromethane, the results are very similar but clearly lower than the γ_i^{∞} values of tetrachloromethane. This behavior indicates that more polar solutes have better solubilities in the ionic liquid because of the preferred attractive interaction of polar molecules with the charged ions of the IL, and they were prone to strongly retain in the IL stationary phase. For example, the γ_i^{∞} values for methanol and 1-butanol at 303.15 K and for 1-pentanol and 3-methylbutanol at (303.15 and 313.15) K were not determined since the retention times were so far beyond the scope allowed by GC.

For comparison of intermolecular interactions of polar solutes in different ILs, Table 4 lists the γ_i^{∞} values of methanol at $T =$ 298.15 K in the ILs with the same cation [BMIM]$^+$ and different anions.[10,17,18,21] In the case of methanol, the lone pair of electrons on the oxygen atom could interact with the ionic liquid cation, and the acidic proton of methanol is no doubt strongly attracted to the atoms in the anion. The γ_i^{∞} values obtained for [N(CF_3SO_2)$_2$]$^-$ are the largest, which are higher than that for [BF_4]$^-$ and [CF_3SO_3]$^-$. For [MDEGSO$_4$]$^-$ and [OcOSO$_3$]$^-$, the γ_i^{∞} values are distinctly much lower in comparison to that for [N(CF_3SO_2)$_2$]$^-$. This is no doubt due to the enhanced association between the anion and methanol. It means that the activity coefficients and intermolecular of polar solutes in different ILs are very much dependent on the chemical structure of the anion. However, there is the possibility of interfacial adsorption for the solutes onto the solid support, which may result in the γ_i^{∞} values having a tendency of underestimation in particular for hydrocarbons and low polarity solutes.[32]

The selectivity S_{ij}^{∞} is defined as $S_{ij}^{\infty} = \gamma_{i3}^{\infty}/\gamma_{j3}^{\infty}$[33] (where *i*, *j* refer to the solutes to be separated and 3 refers to the IL solvent). The values of S_{ij}^{∞} for alkane (*i*)/polar solute (*j*) mixtures at $T =$ 298.15 K were determined with hexane (*i*) (values of γ_i^{∞} from ref 1)/polar solutes (*j*) and were presented in Table 5. The results are larger than 10. Therefore, [BMIM][CF_3SO_3] can play an important role for the separation of polar solutes from alkanes.

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