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Activity Coefficients at Infinite Dilution of Alkanes, Alkenes, and Alkyl Benzenes 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 organic solutes—alkanes, alkenes, and alkyl benzenes—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 from (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

Ionic liquids (ILs) are environmentally friendly solvents with no detectable vapor pressure and good dissolubility for many organic, inorganic, and polymeric substances. The selection of solvents for extraction and extractive distillation in solvent-enhanced separation processes of organic liquid mixtures can be achieved through the examination of the activity coefficients at infinite dilution, γ_i^∞, of organic solutes. Values of γ_i^∞ are important in the selection of solvents in the reliable design of thermal separation processes when the last traces of impurities have to be removed. The use of a steady-state gas−liquid chromatographic technique for the determination of γ_i^∞ has been employed by many researchers and has proved quite reliable.[1] Ionic liquids are in particular reliable for the determination of γ_i^∞ by this method because of their negligible vapor pressures, making ionic liquids an ideal stationary phase.

Using the gas−liquid chromatograph with the ILs as the stationary phase, Heintz et al.[2−10] and Letcher et al.[11−19] measured γ_i^∞ of various solutes in a number of ILs. Recently, our group has focused attention on the physical properties of hydrophilic ionic liquids, such as the physical properties and the γ_i^∞ of various solutes in 1-butyl-3-methylimidazolium tetrafluoroborate ([BMIM][BF_4]) and 1-propyl-2,3-dimethylimidazolium tetrafluoroborate ([PDMIM][BF_4]).[20−23] 1-Butyl-3-methylimidazolium trifluoromethanesulfonate ionic liquid [BMIM]-[CF_3SO_3] is one of the air- and water-stable hydrophilic ionic liquids and is also thermally stable.

This paper reports activity coefficient γ_i^∞ data of 17 organic solutes—alkanes (pentane, hexane, heptane, octane, nonane, decane, cyclohexane, methylcyclohexane, 2,2,4-trimethylpentane), alkenes (cyclohexene, styrene), and alkyl benzenes (benzene, toluene, ethylbenzene, *o*-xylene, *m*-xylene, *p*-xylene)—in the ionic liquid [BMIM][CF_3SO_3] from (303.15 to 363.15) K. The partial molar excess enthalpies at infinite dilution, $H_i^{E,\infty}$, were also derived for the solutes from the temperature dependency of γ_i^∞. For feasibility assessment of the ionic liquid in extractive distillation, the selectivity, S_{ij}^∞, of the ionic liquid for the separation of different mixtures, i.e., alkane from aromatic compounds and three isomeric xylenes, was also determined.

Experimental Section

Materials. 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: [X^-] < 500 ppm. The mass fraction of water was checked to be less than 10^{-3} by Karl Fischer titration. The chemical shifts for the ^{1}H 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, $NCH_2{-}CH_2$], 1.403 [m, 2H, $NCH_2CH_2{-}CH_2$], and 0.959 [t, 3H, CH_3]. These results agreed well with literature data.[24] Impurity peaks were not observed in the ^{1}H NMR.

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 hydrocarbons provided by Beijing Chemical Reagents Company were analytical reagents. The solutes were used without further purification. Chromosorb PAW-DMDCS of 100/120 mesh size was used as the solid support for the ionic liquid in the GLC column. Before use, the Chromosorb was subjected to vacuum treatment with heating to remove traces of adsorbed moisture. The Chromosorb and the empty GLC column were purchased from Varian Inc.

Apparatus and Procedure. Coating the solid support with the ionic liquid was performed by dispersing a known mass amount of the support (Chromosorb) in a solution of the ionic liquid in dichloromethane followed by evaporation of the solvent in a rotating evaporator. The Chromosorb was weighed on an electronic balance of precision of ± 0.1 mg before and after the coating process. The experiments were performed on a Varian Chrompack CP-3800 GC equipped with a thermal conductivity detector. Dry nitrogen was used as the carrier gas, and the GC column used was 98 cm in length and 0.40 cm in inner diameter. The mass of the stationary phase (ionic liquid) was 3.9960 g with a precision ± 0.0003 g. According to the literature,[4,8] the mass fraction of the ionic liquid coating was

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about (27 to 60) % of the support material. In this work, the mass fraction was about 60 % of the support material. The column was filled uniformly with the help of an ultrasound vibrator.

The retention time t_r of each injected component was recorded by GC. The volume of the samples injected into the GC probes was about (0.1 to 2) μL. The experiments were carried out over a temperature range from (303.15 to 363.15) K. The temperature of the GC column was maintained constant within ± 0.05 K. At a given temperature, each operation was repeated at least three times to check the reproducibility. The deviation of the retention time of the three measurements was within ± 0.05 min. The value of the dead time t_G was determined with methane[25] as the nonretainable pure component under the assumption that the effect of the solubility of methane in the ionic liquid was negligible. The measured dead time in the temperature range had a deviation of ± 0.01 min. Absolute values of ($t_r - t_G$) varied between (1 and 400) min depending on the individual solute and temperature.

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 values of γ_i^∞ for a volatile solute (i) in an involatile solvent (3) were obtained by the equation proposed by Cruickshank et al.[26] and Everett.[27]

$$\ln \gamma_{i,3}^\infty = \ln\left(\frac{n_3RT}{V_N P_i^0}\right) - \frac{B_{11} - V_i^0}{RT}P_i^0 + \frac{2B_{12} - V_i^\infty}{RT}JP_0 \qquad (1)$$

$$V_N = JU_0(t_r - t_G)\frac{T_{col}}{T_f}\left[1 - \frac{P_w^0}{P_0}\right] \qquad (2)$$

In eq 1, $\gamma_{i,3}^\infty$ is the activity coefficient of solute i at infinite dilution in the stationary phase (3); P_i^0 is the vapor pressure of the pure liquid solute i; n_3 is the number of moles of the stationary phase component on the column; V_N is the standardized retention volume obtained by eq 2, where t_r is the retention time; t_G is the dead time; U_0 is the flow rate of the carrier gas, measured by a soap bubble flow meter; T_{col} is the column temperature; T_f is the flow meter temperature; P_w^0 is the saturation vapor pressure of water at T_f; and P_0 is the pressure at the column outlet.

The second and third terms in eq 1 are correction terms arising from the nonideality of the mobile gaseous phase. B_{11} is the second virial coefficient of the solute; B_{12} is the cross second virial coefficient of the solute (1) with the carrier gas (2); V_i^0 is the liquid molar volume of pure solute; and V_i^∞ is the partial molar volume of the solute in the ionic liquid at infinite dilution.

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 coefficients given by Boublik et al.[28] Molar volumes of solutes V_i^0 were estimated using their experimental densities; partial molar volumes of solute at infinite dilution V_i^∞ have been assumed to be equal to V_i^0. B_{11} and B_{12} have been estimated according to the equations suitable for nonpolar liquids by Tsonopolous's method.[29] Critical parameters needed for the calculations were available from the literature.[29] The cross critical properties P_{cij}, T_{cij}, V_{cij}, and Z_{cij} and acentric factor ω_{ij} were calculated by using equations given in the literature.[29,30]

Table 1. Relative Uncertainties in the Measured and Derived Quantities

parameters	relative uncertainty
$t_r - t_G$	± 0.033
U_0	± 0.005
P_i	± 0.006
P_o	± 0.0002
J	± 0.01
P_i^0	± 0.0001 to ± 0.0025
n_3	± 0.005
γ_i^∞	± 0.04

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.

The outlet pressure P_0 was kept equal to the atmospheric pressure. The specified pressure drop ($P_i - P_0$) was varied between (20.3 and 101.3) kPa, providing suitable retention time with sharp peaks. The pressure drop was recorded by GC automatically measured using a membrane manometer with an uncertainty of ± 0.2 kPa.

Retention time, dead time, column temperature, flow rate, input and output pressure, and the mass of the stationary phase all have experimental errors. According to the error propagation law, γ_i^∞ is estimated to have a relative uncertainty within ± 0.04. The uncertainties in the measured and derived quantities are listed in Table 1.

Results and Discussion

The values of γ_i^∞ of different solutes in [BMIM][CF$_3$SO$_3$] obtained over a temperature range of (303.15 to 363.15) K were listed 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 coefficients a and b, the correlation coefficient R^2, and the standard deviation σ as well as the values of γ_i^∞ at 298 K calculated based on the fitted equation are listed in Table 3. The γ_i^∞ values for the linear n-alkanes increase with increasing chain length. However, the branching of the alkane skeleton (e.g., cyclohexane, methylcyclohexane, or 2,2,4-trimethylpentane) reduces the value of γ_i^∞ in comparison with the corresponding linear alkanes: hexane, heptane, and octane. Introduction of the double bond in the six-membered ring (cyclohexene) causes a reduction of γ_i^∞.

For the aromatic compounds, the values of γ_i^∞ were distinctly lower in comparison with those of the alkanes and alkenes, and the value of γ_i^∞ increases with increasing size of the alkyl group.

Table 3 lists the partial molar excess enthalpies at infinite dilution values, $H_i^{E,\infty}$, which can be directly obtained from the slope of a straight line derived from eq 4, according to the Gibbs−Helmholtz equation.

$$\frac{\partial \ln \gamma_i^\infty}{\partial (1/T)} = \frac{H_i^{E,\infty}}{R} \qquad (5)$$

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

solutes (i)	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
			Alkanes				
pentane	26.47	24.90	23.89	23.19	22.22	21.19	19.87
hexane	37.76	35.74	33.93	32.09	30.34	29.11	27.62
heptane	56.69	51.63	48.52	45.97	43.28	41.15	38.84
octane	87.19	79.91	74.07	68.34	63.20	60.28	55.06
nonane	122.1	111.4	103.4	93.32	88.21	81.31	75.92
decane	897.2	709.1	576.5	482.8	409.3	348.3	305.9
cyclohexane	21.79	20.46	19.16	18.06	16.92	15.49	14.55
methylcyclohexane	30.24	28.48	26.80	25.42	24.12	22.90	21.56
2,2,4-trimethylpentane	56.95	52.92	50.88	47.92	45.64	43.90	40.84
			Alkenes				
cyclohexene	10.86	10.43	10.07	9.686	9.339	9.147	8.825
styrene	2.456	2.475	2.490	2.511	2.531	2.543	2.570
			Alkyl Benzenes				
benzene	1.812	1.825	1.831	1.841	1.858	1.873	1.897
toluene	2.723	2.749	2.777	2.804	2.833	2.853	2.875
ethylbenzene	4.615	4.571	4.542	4.520	4.501	4.490	4.467
o-xylene	3.659	3.668	3.676	3.704	3.711	3.758	3.784
m-xylene	4.717	4.658	4.577	4.472	4.413	4.331	4.066
p-xylene	4.302	4.317	4.332	4.369	4.408	4.419	4.422

Table 3. Coefficients *a* and *b* of Equation 4, Correlation Coefficient R^2, Standard Deviation σ, γ_i^∞ at 298.15 K Calculated Using Equation 4, and Values of $H_i^{\mathrm{E},\infty}$ Derived from Equation 4

solute (i)	a	b/K	R^2	σ	$\gamma^\infty_{i\ 298.15\mathrm{K}}$	$H_i^{\mathrm{E},\infty}$/kJ·mol^{-1}
			Alkane			
pentane	1.6512	492.86	0.9844	0.0133	27.2	4.1
hexane	1.7418	574.28	0.9987	0.0045	39.2	4.8
heptane	1.8106	671.56	0.9968	0.0081	58.2	5.6
octane	1.7414	827.15	0.9974	0.0090	91.4	6.9
nonane	1.9290	872.44	0.9983	0.0078	128.4	7.2
decane	0.2773	1970.32	0.9984	0.0169	978.3	16.4
cyclohexane	0.6491	742.09	0.9907	0.0155	23.1	6.2
methylcyclohexane	1.3949	611.90	0.9982	0.0055	31.4	5.1
2,2,4-trimethylpentane	2.1215	581.95	0.9930	0.0105	58.8	4.8
			Alkene			
cyclohexene	1.1353	378.83	0.9981	0.0036	11.1	3.2
styrene	1.1654	−81.279	0.9913	0.0016	2.4	−0.68
			Alkyl Benzenes			
benzene	0.8539	−79.572	0.9489	0.0040	1.8	−0.66
toluene	1.3362	−101.58	0.9988	0.0008	2.7	−0.84
ethylbenzene	1.3404	56.49	0.9788	0.0018	4.6	0.47
o-xylene	1.4962	−61.62	0.9130	0.0041	3.6	−0.51
m-xylene	0.7601	243.79	0.9007	0.0174	4.8	2.0
p-xylene	1.6484	−57.876	0.9558	0.0027	4.3	−0.48

where R is the gas constant. For the linear alkanes, $H_i^{\mathrm{E},\infty}$ was positive and increases with increasing chain length. The introduction of double bonds decreases the positive values of $H_i^{\mathrm{E},\infty}$. For molecules containing an aromatic ring, $H_i^{\mathrm{E},\infty}$ became negative; however, increasing the size of the alkyl group in the aromatic ring compensated for this effect, and in the case of ethylbenzene and *m*-xylene, $H_i^{\mathrm{E},\infty}$ was again positive. For alkanes and alkenes, the range of relative uncertainties of $H_i^{\mathrm{E},\infty}$ is within ± 0.06; nevertheless, for alkylbenzenes, the range of relative uncertainties of $H_i^{\mathrm{E},\infty}$ is about ± 0.10.

The selectivity S_{ij}^∞ is defined as $S_{ij}^\infty = \gamma_{i3}^\infty/\gamma_{j3}^\infty$.[32] It indicates suitability of a solvent for separating mixtures of components i and j by extraction. The values of selectivity S_{ij}^∞ for the separation of a hexane (i)/benzene (j) mixture at T = 298.15 K using ILs with cation [BMIM]$^+$ and different anions are taken from the literature.[10,17,18,21] The results of this work are presented in Table 4, which indicated that for a given cation [BMIM]$^+$ γ_i^∞ values for the anions [MDEGSO$_4$]$^-$ and [BF$_4$]$^-$ are much higher than those for the anion [OcOSO$_3$]$^-$, and γ_i^∞ values for [N(CF$_3$SO$_2$)$_2$]$^-$ and [CF$_3$SO$_3$]$^-$ are second. It means that the activity coefficients and intermolecular interactions of different solutes in IL are very much dependent on the chemical structure of the anion. Comparing our results with those for other ILs, [BMIM][CF$_3$SO$_3$] is not the most ideal extraction solvent for separation of the hexane and benzene binary system, but as a solvent, many factors must be thought of, such as density, viscosity, toxicity, and cost. The values of S_{ij}^∞ for three isomeric xylenes at 298.15 K were 1.33 (*m*-xylene/*o*-xylene), 1.13 (*m*-xylene/*p*-xylene), and 1.18 (*p*-xylene/*o*-xylene), respectively. The results indicated that [BMIM][CF$_3$SO$_3$] was not an ideal extraction solvent for separation of xylenes.

Table 4. Selectivities, S_{ij}^∞, at Infinite Dilution of Various Solvents for Hexane (i)/Benzene (j) Separation at T = 298.15 K

solvents	S_{ij}^∞	ref
[BMIM][N(CF$_3$SO$_2$)$_2$]	17.5[b]	8
[BMIM][OcOSO$_3$]	5.1	17
[BMIM][MDEGSO$_4$]	39.7	18
[BMIM][BF$_4$]	37.3[a]	21
[BMIM][CF$_3$SO$_3$]	21.8[a]	this work

[a] Extrapolated value. [b] Interpolated value.

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