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Activity Coefficients at Infinite Dilution of Alkanes, Alkenes, and Alkyl Benzenes in 1-Ethyl-3-methylimidazolium Diethylphosphate Using Gas–Liquid Chromatography

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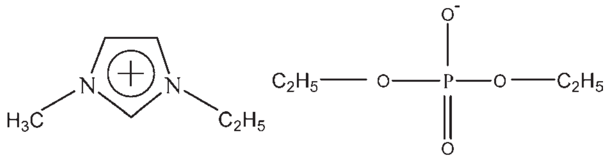
ABSTRACT: The activity coefficients at infinite dilution, γ_i^∞, for 16 organic solutes: alkanes, alkenes, and alkyl benzenes in the ionic liquid 1-ethyl-3-methylimidazolium diethylphosphate ([EMIM][DEP]) have been measured by the gas–liquid chromatographic method. The measurements were carried out in the temperature range of (303.15 to 343.15) K. The partial molar excess enthalpies at infinite dilution, $H_i^{\mathrm{E},\infty}$, of the solutes in [EMIM][DEP] were also derived from the temperature dependence of the γ_i^∞ values.

INTRODUCTION

Ionic liquids (ILs) have been receiving increasing interest as environmentally friendly solvents in recent years because of their unique physical and chemical properties.[1–3] For ILs to be used effectively as solvents, it is essential to know their interaction with different solutes. The activity coefficient at infinite dilution, γ_i^∞, which describes the degree of nonideality for species i in a mixture, gives a quantitative measure of interactions between unlike molecules in the absence of solute–solute interactions. Values of γ_i^∞ also provide information on the intermolecular energy between ILs and organic solutes and can be used to quantify the selectivity and solvent power of ILs. This work continues our studies on the determination of γ_i^∞ of various solutes (i) in ILs by the gas–liquid chromatographic method (GLC).[4–7] In this paper, γ_i^∞ have been measured for 16 organic solutes: alkanes (hexane, heptane, octane, nonane, decane, cyclohexane, methylcyclohexane, and 2,2,4-trimethylpentane), alkenes (cyclohexene and styrene), and alkyl benzenes (benzene, toluene, *o*-xylene, *m*-xylene, and *p*-xylene) in 1-ethyl-3-methylimidazolium diethylphosphate ([EMIM][DEP], CAS Registry No. 848641-69-0) by the GLC method in the temperature range of (303.15 to 343.15) K. A few thermodynamic and thermophysical properties[8] and liquid–liquid equilibria (LLE)[9–11] for [EMIM][DEP] are available in the literature. ILs with an imidazolium cation and a phosphatic anion have been scarcely studied.



1-Ethyl-3-methylimidazolium diethylphosphate

EXPERIMENTAL SECTION

The IL [EMIM][DEP] was purchased from Shanghai Chengjie Chemical Co., Ltd. Its mass fraction purity was greater than 0.99 according to manufacturer's specifications, with the following certified mass fraction of impurities: $w(\mathrm{Cl}^-) < 5 \cdot 10^{-4}$, water $< 10^{-3}$. The chemical shift for ^{1}H NMR spectrum (parts per million, D_2O) appear as follows: δ 8.574 [s, 1H, H(2)], 7.343 [s, 1H, H(4)], 7.275 [s, 1H, H(5)], 4.083 [m, 2H, NCH_2], 3.759 [m, 2H, OCH_2], 3.759 [m, 2H, OCH_2], 3.706 [s, 3H, NCH_3], 1.343 [t, 3H, CH_3], 1.091 [t, 3H, CH_3], and 1.091 [t, 3H, CH_3]. Impurity peaks were not observed in the ^{1}H NMR spectra. The organic solutes, alkanes, alkenes, and alkyl benzenes, were purchased from Beijing Chemical Reagents Company. Their mass fraction purities were greater than 0.99. The solutes were used without further purification. 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 water mass fraction analyzed by Karl Fischer analysis was less than $4 \cdot 10^{-4}$.

The experiments were performed on a Varian Chrompack CP-3800 GC equipped with a thermal conductivity detector. The column preparation and the packing method used in this work have been described previously.[4] The GC column used was 100 cm in length and 0.40 cm in inner diameter. Chromosorb W, acid-washed and silanized, was used as the solid support. Coating the solid support with [EMIM][DEP] was performed by dispersing a known mass amount of the Chromosorb in a solution of [EMIM][DEP] in ethanol followed by evaporation of the solvent in a rotating evaporator. The Chromosorb was weighed on an electronic balance of precision of ± 0.0001 g before and after the coating process. The amount of the stationary phase ([EMIM][DEP]) was 2.096 g with a precision ± 0.0001 g. The mass fraction of [EMIM][DEP] was about 40 % of the support material, large enough to prevent any residual absorption of solute onto the column packing.[12,13] The column was filled uniformly with the help of an ultrasound vibrator and finally heated under nitrogen for 8 h at the column temperature 120 °C. Dry nitrogen was used as the carrier gas. The flow rate of carrier gas was

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determined using a calibrated soap bubble flowmeter which was placed at the outlet after the detector. The flow rate was set for a series of runs and was allowed to stabilize for at least 15 min before any γ_i^∞ determinations were made. The volume of the samples injected into the GC probes was about (0.05 to 0.5) μL, and the peaks were found to be symmetrical, independent of the carrier gas flow rate. The experiments were carried out over a temperature range from (303.15 to 343.15) 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 as the nonretainable pure component under the assumption that the effect of the solubility of methane in the IL was negligible. The measured dead time in the temperature range has a deviation of ± 0.01 min.

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.

Table 1. Relative Uncertainties in the Measured and Derived Quantities

parameters	relative uncertainty
$(t_r - t_G)$/min	± 2.7 %
U_0/mL·min^{-1}	± 0.5 %
p_i/kPa	± 0.6 %
p_o/kPa	± 0.02 %
J	± 1 %
p_i^0/kPa	± 0.01 % to ± 0.25 %
n_3/mol	± 0.5 %
γ_i^∞	± 5 %

The γ_i^∞ values were obtained by the equation proposed by Cruickshank et al.[14] and Everett.[15]

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

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, and V_N is the standardized retention volume obtained by eq 2,

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

where t_r denotes the retention time, t_G the dead time, U_0 the flow rate of the carrier gas, T_{col} the column temperature, T_f the flow meter temperature, p_w^0 the saturation vapor pressure of water at T_f, and p_o the pressure at the column outlet.

The second and third terms in eq 1 are correction terms arise 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 stationary phase (3) at infinite dilution.

For all solutes, values of p_i^0 were calculated from the Antoine equation, with Antoine coefficients given by Boublik et al.[16] Molar volumes of solutes V_i^0 were estimated using their experimental densities;[17] 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}

Table 2. Experimental Activity Coefficients at Infinite Dilution γ_i^∞ for Various Solutes in the IL 1-Ethyl-3-methylimidazolium Diethylphosphate at Temperatures of (303.15 to 343.15) K

	γ_i^∞				
solute (i)	T/K = 303.15	T/K = 313.15	T/K = 323.15	T/K = 333.15	T/K = 343.15
			Alkanes		
pentane	46.84	43.53	40.88	38.62	36.26
hexane	77.98	71.39	67.85	62.97	58.28
heptane	125.1	115.9	105.2	97.04	88.45
octane	177.4	160.9	142.2	125.8	109.5
nonane	256.9	234.5	218.4	203.7	190.8
decane	401.8	365.3	337.8	310.1	291.2
cyclohexane	38.64	35.34	32.85	30.17	28.96
methylcyclohexane	55.98	52.39	50.05	47.37	44.87
2,2,4-trimethylpentane	161.0	146.9	128.7	107.9	93.67
			Alkenes		
cyclohexene	17.89	16.07	14.25	12.79	11.23
styrene	5.002	4.986	4.962	4.938	4.911
			Alkyl Benzenes		
benzene	2.516	2.479	2.433	2.397	2.361
toluene	4.734	4.571	4.403	4.298	4.105
o-xylene	6.524	6.389	6.256	6.138	6.046
m-xylene	7.169	7.105	7.057	6.969	6.912
p-xylene	6.835	6.698	6.583	6.404	6.287

Table 3. Coefficients a and b of eq 1, γ_i^∞ at 298.15 K Calculated Using eq 1, Values of $H_i^{E,\infty}$ Derived from eq 1, and Standard Deviation σ

solute (i)	a	b/K	γ_i^∞ 298.15K	$H_i^{E,\infty}$ kJ·mol^{-1}	σ
			Alkanes		
pentane	1.6764	657.52	48.51	5.47	0.003
hexane	1.9286	735.80	81.17	6.12	0.010
heptane	1.8538	904.78	132.7	7.52	0.010
octane	1.0545	1256.2	194.0	10.4	0.021
nonane	3.0156	766.49	266.8	6.37	0.005
decane	3.2172	841.43	419.6	7.00	0.005
cyclohexane	1.1200	766.60	40.09	6.37	0.011
methylcyclohexane	2.1596	565.11	57.68	4.70	0.005
2,2,4-trimethylpentane	0.3584	1442.2	180.5	12.0	0.035
			Alkenes		
cyclohexene	−1.0768	1204.3	19.34	10.0	0.014
styrene	1.4523	48.080	5.021	0.400	0.001
			Alkyl Benzenes		
benzene	0.3720	167.25	2.542	1.39	0.001
toluene	0.3688	360.14	4.839	2.99	0.006
o-xylene	1.2145	200.28	6.595	1.67	0.001
m-xylene	1.6547	95.850	7.215	0.797	0.002
p-xylene	1.1981	220.18	6.935	1.83	0.004

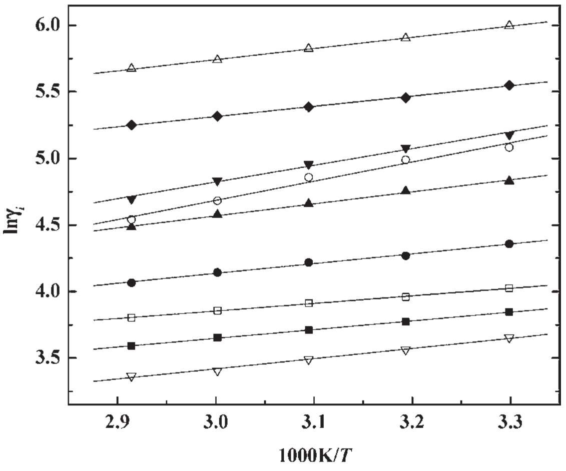

Figure 1. Plot of ln γ_i^∞ vs $1/T$ for the solutes together with a linear correlation of the data: ■, pentane; ●, hexane; ▲, heptane; ▼, octane; ◆, nonane; △, decane; ▽, cyclohexane; □, methylcyclohexane; ○, 2,2,4-trimethylpentane. —, linear fit.

have been estimated according to the equations suitable for nonpolar liquids by Tsonopolous's method.[18] Critical parameters needed for the calculations were available from the literature.[18] The cross critical properties P_{cij}, T_{cij}, V_{cij}, Z_{cij}, and acentric factor ω_{ij} were calculated by using equations given in the literature.[18,19]

The pressure correction term J is given by[20]

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

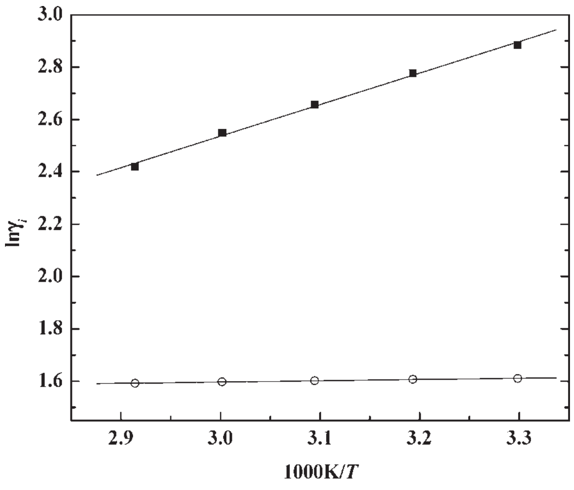

Figure 2. Plot of ln γ_i^∞ vs $1/T$ for the solutes together with a linear correlation of the data: ■, cyclohexene; ○, styrene. —, linear correlation.

where p_i and p_o are the inlet and outlet pressures of the GC column, respectively. The inlet column pressure p_i was determined by inner manometer. Outlet pressure p_o was kept equal to atmospheric pressure. According to the error propagation law, γ_i^∞ is estimated to have an uncertainty within ± 5 %. The uncertainties in the measured and derived quantities are listed in Table 1.

■ RESULTS AND DISCUSSION

Experimental results of γ_i^∞ for 16 solutes in [EMIM][DEP] at different temperatures are presented in Table 2. The γ_i^∞ values for the linear n-alkanes increase with increasing chain length. The branching of the alkane skeleton (e.g., cyclohexane, methylcyclohexane, or 2,2,4-trimethylpentane) reduces the values of γ_i^∞ in comparison with the corresponding linear alkanes: hexane, heptane, and octane. The 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^∞ are distinctly lower in comparison with those of the alkanes, and the values of γ_i^∞ increase with increasing size of the alkyl group. The smallest values indicate the stronger interactions between solvent and solute.

Experimental results of γ_i^∞ at different temperatures were correlated with temperature by the following equation:

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

The coefficients a and b, the standard deviation σ of the fitted equations, and the values of γ_i^∞ at 298.15 K are listed in Table 3. The plots of measured ln γ_i^∞ versus $1/T$ values and the linear fit of their data are given in Figures 1 to 3, which show a fairly good fitting quality of eq 4. According to the Gibbs−Helmholtz equation, the partial molar excess enthalpies at infinite dilution, $H_i^{E,\infty}$, can be directly obtained from the slope of a straight line derived from eq 5:

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

where R is the gas constant. The values of $H_i^{E,\infty}$ for the solutes studied are also listed in Table 3. For the linear alkanes, cyclohexene, and alkyl benzenes, $H_i^{E,\infty}$ all have positive values. The relative uncertainties of $H_i^{E,\infty}$ is about ± 10 %.

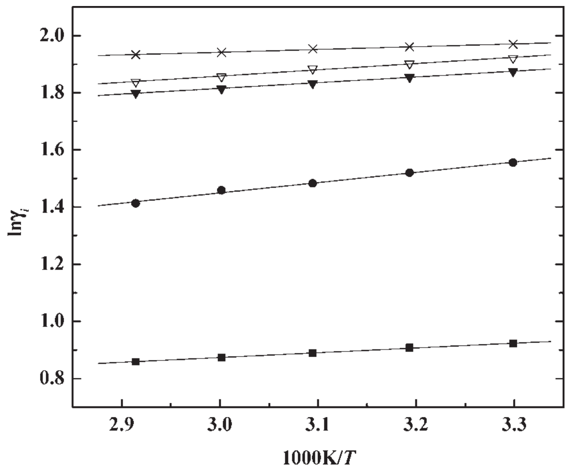

Figure 3. Plot of ln γ_i^∞ vs $1/T$ for the solutes together with a linear correlation of the data: ■, benzene; ●, toluene; ▼, *o*-xylene; ×, *m*-xylene; ▽, *p*-xylene; —, linear correlation.

Table 4. Selectivity, S_{ij}^∞, at Infinite Dilution for Different ILs with 1-Ethyl-3-methylimidazolium Cation at 298.15 K (Extrapolated)

ILs	S_{ij}^∞ hexane (i)/ benzene (j)	cyclohexane (i)/ benzene (j)	ref
$[\mathrm{EMIM}][\mathrm{BF_4}]$	49.5	38.9	7
$[\mathrm{EMIM}][\mathrm{NO_3}]$	-	29.0	12
$[\mathrm{EMIM}][\mathrm{N(CF_3SO_2)_2}]$	23.1	13.2	22
$[\mathrm{EMIM}][\mathrm{N(CF_3SO_2)_2}]$	24.5[a]	13.8[a]	23
$[\mathrm{EMIM}][\mathrm{EtSO_4}]$	40.8[a]	21.7[a]	23
[EMIM][TFA]	30.7	14.4	24
$[\mathrm{EMIM}][\mathrm{CF_3SO_3}]$	33.6	17.2	25
[EMIM][SCN]	95.4	33.2	26
[EMIM][FAP]	15.0	13.2	27
[EMIM][DCA]	18.8	46.0	28
[EMIM][DEP]	31.9	15.8	this work

[a] The values of γ_i^∞ from the dilutor technique.

The selectivity, S_{ij}^∞, is defined as $S_{ij}^\infty = \gamma_{i,\mathrm{IL}}^\infty/\gamma_{j,\mathrm{IL}}^\infty$ (where i and j refer to the solutes to be separated).[21] Table 4 lists S_{ij}^∞ at infinite dilution for ILs based on 1-ethyl-3-methylimidazolium cation for hexane (i)/benzene (j) and cyclohexane (i)/benzene (j) separation problems at 298.15 K. The results indicated that [EMIM][DEP] is not the most ideal extraction solvent for separation of hexane (i)/benzene (j) and cyclohexane (i)/benzene (j) binary systems, 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 1.09 (*m*-xylene/*o*-xylene), 1.04 (*m*-xylene/*p*-xylene), and 1.05 (*p*-xylene/*o*-xylene), respectively. The results indicated that [EMIM][DEP] was not an ideal extraction solvent for the separation of xylenes.

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