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Determination of Activity Coefficients at Infinite Dilution of Solutes in the Ionic Liquid 1-Hexyl-3-methylimidazolium Tetrafluoroborate Using Gas−Liquid Chromatography at the Temperatures 298.15 K and 323.15 K

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The activity coefficients at infinite dilution, γ_{13}^{∞}, for both polar and nonpolar solutes in an ionic liquid, 1-hexyl-3-methylimidazolium tetrafluoroborate ($[HMIM^+][BF_4^-]$), have been determined by gas−liquid chromatography at $T = (298.15$ K and 323.15 K). This work is part of our research focus on ionic liquids. The selectivity values have been calculated at $T = 298.15$ K, and the results indicate that the ionic liquid, $[HMIM^+][BF_4^-]$, should be a good solvent for separation of benzene and alkanes. The partial molar excess enthalpy values at infinite dilution have also been determined at $T = 298.15$ K and have been discussed in terms of intermolecular interactions. The results have been discussed in terms of measurements of γ_{13}^{∞}, using other ionic liquids, taken from the recent literature.

Introduction

Ionic liquids have a very much greater affinity for polar and polarizable compounds than they do for nonpolar compounds, and as a result, they may be considered as possible replacement solvents in the extractive distillation of hydrocarbon mixtures. Ionic liquids have the added advantage of extremely low vapor pressures and high boiling points, which allows for ease of recycling.[1]

In this work activity coefficients at infinite dilution, γ_{13}^{∞}, have been determined for alkanes, alkenes, alkynes, cycloalkanes, benzene, and methanol in the ionic liquid 1-hexyl-3-methylimidazolium tetrafluoroborate ($[HMIM^+][BF_4^-]$) using gas−liquid chromatography at $T = (298.15$ K or 323.15 K).

The selectivity value,[2] S^{∞}, of hexane with respect to benzene was calculated and used to determine the potential of the ionic solvent for extractive distillation in the separation of aromatic compounds from aliphatic compounds. The partial molar excess enthalpies at infinite dilution, $\Delta H_1^{E,\infty}$, were also calculated from the γ_{13}^{∞} values obtained at the two temperatures. These values give some indication of the interaction between the hydrocarbons and the ionic liquid.

The activity coefficients and selectivity values determined in this work have been discussed in terms of recent results, using other ionic liquids, taken from the literature.

Experimental Section

Chemicals. The solvent $[HMIM^+][BF_4^-]$ was supplied by Fluka and was reported to have a purity of 0.98 mass fraction. The ionic liquid was further purified by subjecting the liquid to a very low vapor pressure of about 5×10^{-4} Pa for approximately 30 min, resulting in a loss of material, equivalent to 0.02 mass fraction. This process caused the sample to foam for a few seconds. This we believe was water boiling off, as the water concentration in the resultant ionic liquid sample was found to be lower than 0.01 mass %, as determined from a Karl Fisher titration. No attempt was made to analyze any other impurities in the ionic liquid. The solutes (purchased from Aldrich) were used without further purification because the GLC technique separated any impurities.

Procedure. The gas−liquid chromatography method is a well established and accurate method used to obtain the γ_{13}^{∞} values[3,4] and was used in this work. The GLC apparatus, the column preparation, and the packing method used in this work have been described previously.[5,6] Stainless steel columns of length 1.0 m and 0.47 mm diameter were used together with Chromosorb WHP (80/100 mesh) as the solid support. The latter was supplied by Supelco. A thermal conductivity detector was used, and the carrier gas was dry helium. The carrier gas flow rate was determined using a calibrated soap bubble flow meter which was placed at the outlet of 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_{13}^{∞} determinations were made. The flow rates were corrected for water vapor pressure and ranged from (0.1 to 0.5) $\mu m^3 s^{-1}$. The column temperature was controlled in a water bath using a Tronac controller fitted with a low heat capacity intermittent heater. The water bath was fitted with a vigorous stirrer to ensure a uniform temperature. A temperature control of better than 0.01 K was achieved. A glass-walled manometer (i.d. 8 mm so as to reduce the capillary effect) was placed before the inlet of the column to determine the inlet column pressure. The outlet pressure was measured with a normal Fortin barometer. The solvent column packing varied from (25 to 30) mass % of $[HMIM^+][BF_4^-]$, large enough to prevent any residual absorption onto the column packing. Sample injections ranged from (0.1 to 1 μL) and were considered small enough to ensure the condition of infinite dilution of

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Table 1. Critical Constants V_c and T_c (Obtained from Refs 12 and 13) and Ionization Energies I (Obtained from Ref 12) of the Solutes and the Carrier Gas Used in the Calculation of the Virial Coefficients

solute	V_c $cm^3\ mol^{-1}$	T_c K	I $kJ\ mol^{-1}$
pentane	304	496.6	998.6
hexane	370	507.6	977.4
heptane	425	540.6	957.1
octane	492	568.7	947.4
hex-1-ene	354	504.0	910.8
hept-1-ene	413	537.2	910.8
oct-1-ene	464	566.7	909.9
hex-1-yne	332	516.2	960.0
hept-1-yne	387	547.2	960.0
oct-1-yne	442	574.2	951.3
cyclopentane	260	511.6	1014.1
cyclohexane	309	553.6	951.3
cycloheptane	353	604.2	962.0
benzene	256	562.1	892.1
methanol	118	512.6	1046.9
helium	57	5.25	2374.0

the solute on the column.

The GLC technique was tested for the system hexane in hexadecane at 298.15 K, and the results were within 2% of literature values.[2]

Theory

The equation developed by Everett[7] and Cruickshank[8]

$$\ln \gamma_{13}^{\infty} = \ln\left(\frac{n_3RT}{V_N P_1^*}\right) - \left(\frac{(B_{11}-V_1^*)P_1^*}{RT}\right) + \left(\frac{(2B_{12}-V_1^{\infty})P_oJ_2^3}{RT}\right) \quad (1)$$

was used in this work to calculate the γ_{13}^{∞} values of solutes in an involatile solvent, [HMIM$^+$][BF$_4^-$], from gas−liquid chromatography. In this work the subscript 1 refers to a solute, 2 to the carrier gas, and 3 to the solvent [HMIM$^+$]-[BF$_4^-$]. V_N denotes the net retention volume of the solute, P_o the outlet pressure, $P_oJ_2^3$ the mean column pressure, n_3 the number of moles of solvent on the column packing, T the column temperature, P_1^* the saturated vapor pressure of the solute at temperature T, B_{11} the second virial coefficient of pure solute, V_1^* the molar volume of the solute, V_1^{∞} the partial molar volume of the solute at infinite dilution in the solvent, and B_{12} the mixed second virial coefficient of the solute and the carrier gas. The values of B_{11} and B_{12} were calculated using the McGlashan and Potter[9] equation:

$$B/V_c = 0.430 - 0.886(T_c/T) - 0.694(T_c/T)^2 - 0.0375(n-1)(T_c/T)^{4.5} \quad (2)$$

where n refers to the number of carbon atoms. Using the Lorentz[4] and Hudson and McCoubrey[10,11] combining rules, $V_{c,12}$ and $T_{c,12}$ were calculated from the critical properties of the pure component. The vapor pressure values were calculated using the Antoine equation from constants obtained from the literature.[12,13] Critical data[12,13] used to calculate B_{11} and B_{12}, and ionization energies[12] used in the calculation of $T_{c,12}$, are given in Table 1. The values of P_1^*, V_1^*, B_{11}, and B_{12} used in the calculation of γ_{13}^{∞} are given in Table 2.

Table 2. Vapor Pressure P_1^*, Molar Volumes V_1^*, and Virial Coefficients B_{11} and B_{12} Used in the Calculation of γ_{13}^{∞} at the Temperatures T = (298.15 K and 323.15 K)

T K	P_1^* kPa	V_1^* $cm^3\ mol^{-1}$	B_{11} $cm^3\ mol^{-1}$	B_{12} $cm^3\ mol^{-1}$
		Pentane		
298.15	68.34	116.11	−1356	45.7
323.15	159.22	120.88	−1096	47.1
		Hexane		
298.15	20.18	131.60	−1905	52.9
323.15	54.07	136.26	−1519	54.5
		Heptane		
298.15	6.09	147.40	−2864	58.3
323.15	18.88	152.21	−2236	60.0
		Octane		
298.15	1.86	163.50	−4224	65.4
323.15	6.71	168.38	−3257	67.3
		Hex-1-ene		
298.15	24.80	125.90	−2074	51.9
323.15	64.72	130.53	−1425	53.5
		Hept-1-ene		
298.15	7.52	141.8	−2728	57.3
323.15	22.60	146.4	−2138	59.5
		Oct-1-ene		
298.15	2.32	157.9	−4002	63.0
323.15	8.07	162.6	−3087	65.4
		Hex-1-yne		
323.15	48.82	119.7	−1427	53.5
		Hept-1-yne		
323.15	17.53	138.4	−2192	56.2
		Oct-1-yne		
323.15	6.28	159.1	−3016	62.8
		Cyclopentane		
298.15	42.29	94.7	−1257	40.3
323.15	103.76	98.0	−1013	41.7
	41.7			
		Cyclohexane		
298.15	13.01	108.8	−2058	45.4
323.15	36.25	112.0	−1622	47.0
		Cycloheptane		
323.15	10.38	129.6	−2295	51.4
		Benzene		
298.15	13.06	89.4	−1786	39.6
323.15	37.04	92.2	−1405	41.0
		Methanol		
298.15	16.66	40.74	−371	23.3
323.15	55.53	41.99	−321	24.4

Results and Discussion

Table 3 lists the γ_{13}^{∞} values at T = (298.15 K or 323.15 K) for the different moles of solvent on the column packing. The results were determined for different columns, each operated by two different researchers. There is always some doubt concerning the determination of activity coefficients of polar solutes using GLC because of the possibility of adsorption onto the solid support. However, in this work the methanol peaks were found to be symmetrical and also independent of flow rate, indicating no significant absorption. This is due, no doubt, to the high column packing which masks the solid support form the polar solute.[14] It was not possible to obtain γ_{13}^{∞} values for all the solutes at T = 298.15 K, as some solutes had very low vapor pressures and their retention times were very large, resulting in very broad peaks. These results were not recorded. The nonideality of the vapor phase was taken into account in calculating the activity coefficients for each of the solutes. This effect amounted to less than 4% in the worst case.

Table 4 lists the average γ_{13}^{∞} values at T = (298.15 K or 323.15 K). The γ_{13}^{∞} values obtained for methanol, benzene, and the alkynes are much lower than the values for the alkanes and cycloalkanes. The former group are reasonably

Table 3. Moles of Solvent n_3 on Each Column and Activity Coefficient at Infinite Dilution γ_{13}^{∞} Values for Pentane, Hexane, Heptane, Octane, Cyclopentane, Cyclohexane, Cycloheptane, Cyclooctane, Hex-1-ene, Hept-1-ene, Hex-1-yne, Hept-1-yne, Octy-1-yne, Benzene, or Methanol in the Ionic Liquid [HMIM$^+$][BF$_4^-$] at the Temperatures 298.15 K and 313.15 K

solute	n_3 mmol	γ_{13}^{∞}	n_3 mmol	γ_{13}^{∞}
	$T = 298.15$ K			
pentane	3.400	14.8	2.951	14.2
hexane	3.400	22.6	3.262	21.7
heptane	3.400	31.4	2.951	31.6
octane	3.400	43.3	3.262	43.4
cyclopentane	3.400	8.8	2.951	8.3
cyclohexane	3.400	13.0	2.951	12.8
hex-1-ene	3.400	10.9	2.951	10.7
hept-1-ene	3.400	15.2	2.951	15.4
oct-1-ene	3.400	21.8	3.262	21.3
benzene	3.400	0.95	2.951	0.96
methanol	3.400	0.78	3.262	0.75
	$T = 323.15$ K			
pentane	3.400	12.9	2.951	12.3
hexane	3.400	18.3	2.951	18.0
heptane	3.400	26.3	3.262	24.9
octane	3.400	32.5	3.262	31.5
cyclopentane	3.400	7.3	3.262	7.3
cyclohexane	3.400	10.4	3.262	10.1
cycloheptane	3.400	11.9	3.951	11.9
hex-1-ene	2.123	8.1	2.951	8.0
hept-1-ene	2.123	11.1	2.951	12.5
oct-1-ene	3.400	18.3	3.262	17.8
hex-1-yne	3.400	2.3	3.262	2.3
hept-1-yne	3.400	3.4	3.262	3.3
oct-1-yne	3.400	4.8	3.262	4.7
benzene	3.400	0.94	2.951	0.95
methanol	3.400	0.62	3.262	0.60

strongly associated with the ionic liquid, since they are polar or have delocalized electrons. 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 the methanol is no doubt strongly attracted to the anion. The benzene molecule, being an aromatic compound, has delocalized π electrons that can associate with the ionic liquid by strong van der Waals interactions. These are probably weaker than the methanol−ionic liquid interactions.

The estimated error in γ_{13}^{∞} is of the order of (3 to 4)% as a result of the limitations on determining the carrier gas flow rate, the solute retention time, and the amount of solvent on the column.[15]

The selectivity, S^{∞},[2] values are given in Table 5 together with the literature values for solvents that are used in industry for the separation of aromatic and aliphatic compounds. This property has been calculated from the ratio of activity coefficients at infinite dilution, and it is given by the equation $S^{\infty} = \gamma_{i3}^{\infty}/\gamma_{j3}^{\infty}$, where i refers to hexane and j to benzene. The selectivity, S^{∞}, for [HMIM$^+$]-[BF$_4^-$] at $T = 298.15$ K is large, second (23.1) only to the value for sulfolane (30.5) in Table 5. This result highlights the important role the ionic liquid, [HMIM$^+$][BF$_4^-$], can play in separating organic liquids, and aromatic and aliphatic compounds in particular, by an extractive distillation process. The large disparity in interactions between the ionic liquid and benzene and between the ionic liquid and hexane (reflected in the large S^{∞} value) indicates that the ionic liquid, [HMIM$^+$][BF$_4^-$], is also likely to be a good solvent, in solvent extractive processes for separating aromatic compounds from aliphatic compounds.

Table 4. Experimental Activity Coefficient at Infinite Dilution γ_{13}^{∞} Values and Partial Molar Excess Enthalpies at Infinite Dilution for the Solutes in the Ionic Liquid [HMIM$^+$][BF$_4^-$] at the Temperatures 298.15 K and 323.15 K

solute	T 298.15 K	323.15 K	$\Delta H_1^{\mathrm{E},\infty}$ J mol^{-1}
pentane	14.5	12.6	4500
hexane	22.1	18.2	6200
heptane	31.2	25.6	6300
octane	43.4	32.0	9800
cyclopentane	8.6	7.3	5300
cyclohexane	12.9	10.3	7200
cycloheptane		11.9	
hex-1-ene	10.9	8.2	6500
hept-1-ene	15.2	11.6	6000
oct-1-ene	21.8	18.1	6000
hex-1-yne		2.3	
hept-1-yne		3.4	
oct-1-yne		4.8	
benzene	0.96	0.95	300
methanol	0.77	0.61	7500

Table 5. Selectivity Values S^{∞} at Infinite Dilution of Various Solvents for the Hexane (i)−Benzene (j) Separation: $S^{\infty} = \gamma_{i3}^{\infty}/\gamma_{j3}^{\infty}$, As Defined by Tiegs[3] at 298.15 K

solvent	S^{∞}
sulfolane[a]	30.5
dimethyl sulfoxide[a]	22.7
diethylene glycol[a]	15.4
N-methyl-2-pyrrolidinone[a] (NMP)	12.5
acetonitrile[a]	9.4
dichloroacetic acid[a]	6.1
aniline[a]	11.2
[HMIM$^+$][BF$_4^-$]	23.1

[a] Reference 3.

The chemical nature of the groups that make up an ionic liquid plays an important role in determining the magnitude of the activity coefficient of the solute dissolved in an ionic liquid. To illustrate this, activity coefficient data (extrapolated to 298.2 K) for the solutes hexane and benzene, in a variety of ionic liquid solvents, have been extracted from the literature and are presented in Table 6, together with results produced in this work. Looking at the values of γ_{13}^{∞} for systems 1, 2, and 3 of Table 6, it is evident that an increase in the length of an alkyl side chain attached to the cation ring results in a decrease of γ_{13}^{∞} for both hexane and benzene. This is no doubt due to the enhanced association between the alkyl chains and the solute hydrocarbons with increasing alkyl chain length. Moreover, the ionic liquid with the largest alkyl group attached (see system 9) does have the smallest activity coefficients for hexane of all the results presented here.

The nature of the anion has an important effect on the magnitude of γ_{13}^{∞} for both hexane and benzene, as can be seen by comparing the activity coefficients of systems 2 with those of systems 4 and 6 and the results from system 8 with those for system 10.

The effect of adding a small, third alkyl (methyl) group to the cation ring has little effect on the activity coefficients, as can be seen by comparing results from systems 2 or 6 (they are identical systems with slightly different results, done by two independent sets of workers) with the results from system 7.

The chemical nature of the cation ring is important and can be seen by comparing γ_{13}^{∞} results with the solutes hexane and benzene in an ionic liquid made up of an imidazolium ring (system 10) with results for an ionic liquid containing a pyridinium ring (system 5).

The influence of the nature of the ionic liquid on the value of S^{∞} is also important. Again from Table 6, the

Table 6. Activity Coefficients at Infinite Dilution γ_{13}^{∞} and Selectivity Values S^{∞} for the Solutes Hexane and Benzene in Ionic Liquid Solvents

syst	solvent	solute	ref	γ_{13}^{∞}	S^{∞}
1	1-methyl-3-methylimidazolium	hexane	16	41.5	31.1
	bis(trifluoromethylsulfonyl)imide	benzene	16	1.34	
2	1-ethyl-3-methylimidazolium	hexane	16	29.0	24.5
	bis(trifluoromethylsulfonyl)imide	benzene	16	1.19	
3	1-butyl-3-methylimidazolium	hexane	16	14.65	16.7
	bis(trifluoromethylsulfonyl)imide	benzene	16	0.88	
4	1-ethyl-3-methylimidazolium	hexane	16	112.7	41.3
	ethyl sulfate	benzene	16	2.72	
5	4-methyl-*n*-butylpyridinium	hexane	17	64.86	39.8
	tetrafluoroborate	benzene	17	1.63	
6	1-ethyl-3-methylimidazolium	hexane	18	26.84	22.9
	bis(trifluoromethylsulfonyl)imide	benzene	18	1.17	
7	1-ethyl-2,3-dimethylimidazolium	hexane	18	27.7	25.3
	bis(trifluoromethylsulfonyl)imide	benzene	18	1.09	
8	1-hexyl-3-methylimidazolium	hexane	19	22.4	21.6
	hexafluorophosphate	benzene	19	1.05	
9	1-octyl-3-methylimidazolium	hexane	20	17.2	8.7
	chloride	benzene	20	1.99	
10	1-hexyl-3-methylimidazolium	hexane	this	22.1	23.1
	tetrafluoroborate	benzene	work	0.96	

largest value for S^{∞} (41.3) is found for system 4, involving an imidazolium cation with an ethyl sulfate anion, while the smallest value (8.9) is for system 9, involving an imidazolium cation and a Cl^- anion. As a result of the recent work quoted here from the literature, it does appear that we are almost at the stage of designing an ionic liquid for a particular role such as separating hydrocarbons.

Table 4 lists the partial molar excess enthalpies at infinite dilution, $\Delta H_1^{E,\infty}$ which were calculated from the Gibbs−Helmholtz equation:

$$\left[\frac{\partial \ln \gamma_i^{\infty}}{\partial(1/T)}\right] = \frac{\Delta H_1^{E,\infty}}{R} \qquad (3)$$

The $\Delta H_1^{E,\infty}$ values increase in the order benzene < alk-1-ene < cycloalkane < alkane.

The small value obtained for the benzene molecule indicates that there is some interaction between the benzene molecule and the ionic liquid to compensate for the positive enthalpy effect due to the dissociation of the benzene on mixing. This is no doubt a result of the polarizability of the benzene molecule. The alk-1-enes, in spite of their π electrons, have $\Delta H_1^{E,\infty}$ values which are similar in magnitude to those found for the alkanes and cycolalkanes. The $\Delta H_1^{E,\infty}$ value for methanol is positive and can be thought of as a sum of the negative enthalpy effect of association of the methanol molecule with the ionic liquid and the positive enthalpy effect due to the dissociation of the methanol molecules on mixing. This result highlights the magnitude of the large positive enthalpy due to the dissociation of the hydrogen bonds of methanol. In the alkane and cycloalkane series, the $\Delta H_1^{E,\infty}$ values increase with increasing carbon number.

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