Activity Coefficients at Infinite Dilution Measurements for Organic Solutes and Water in the Ionic Liquid 1-Hexyl-3-methylimidazolium Thiocyanate

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Activity coefficients at infinite dilution (γ_{13}^{∞}) for 34 solutes: alkanes, alkenes, alkynes, cycloalkanes, aromatic hydrocarbons, alcohols, thiophene, ethers, acetone, and water in the ionic liquid 1-hexyl-3-methylimidazolium thiocyanate ([hmim][SCN]) were determined by gas−liquid chromatography over temperature range from (298.15 to 368.15) K. The partial molar excess enthalpies at infinite dilution values ($\Delta H_1^{E,\infty}$) were calculated from the experimental γ_{13}^{∞} values, obtained over the temperature range. The selectivities for aliphatic/aromatic hydrocarbon separation problems were calculated from the γ_{13}^{∞} and compared to the literature values for other ionic liquids with a thiocyanate-based anion or a −C≡N group in an anion or a cation, *N*-methyl-2-pyrrolidinone (NMP) and sulfolane.

Introduction

Ionic liquids (ILs) have become a subject of intensive studies in recent years. Because of their unique properties such as a wide liquid range, stability at high temperatures, and especially negligible vapor pressure, ionic liquids were considered as good replacements for the conventional volatile, often flammable, and toxic organic solvents in chemical industry. In separation processes the properties of the solvent are important, namely, selectivity and capacity,[1] which can be directly calculated from activity coefficients at infinite dilution (γ_{13}^{∞}) for different separation problems. It was found in our previous works that ionic liquids based on a thiocyanate-based anion, namely, 1-ethyl-3-methylimidazolium thiocyanate ([emim][SCN])[2] and 1-butyl-3-methylimidazolium thiocyanate ([bmim][SCN]),[3] show very high selectivities in aliphatic/aromatic hydrocarbon separation problems, with relatively low capacities. It is generally known that an increase of the capacity is observed for the longer alkyl chain on the imidazolium ring of the IL. Therefore, the ionic liquid 1-hexyl-3-methylimidazolium thiocyanate ([hmim][SCN]) was a new subject for our measurements.

Experimental Section

Materials and Methods. 1-Hexyl-3-methylimidazolium bromide was synthesized from 1-methylimidazolium and 1-bromohexane and next 1-hexyl-3-methylimidazolium thiocyanate from 1-hexyl-3-methylimidazolium bromide and sodium thiocyanate as was described in our previous work.[4] The product was dried in vacuum for 24 h at 300 K. The proper analysis, using NMR, differential scanning calorimetry (DSC), and elemental analysis, identified the obtained IL. The thermophysical properties were described earlier.[4] All solvents were purchased from Aldrich or Fluka, have the purity > 0.99 mass fraction, and were used without any further purification because the gas−liquid chromatographic (GLC) technique separated any impurities on the column.

The experiments were performed using a PerkinElmer Clarus 500 gas chromatograph equipped with a thermal conductivity detector (TCD). The data were collected and processed using TotalChrom Workstation software. The column preparation and the packing method used in this work have been described previously.[5,6] Glass columns of 1 m length and 4 mm internal diameter were used. Chromosorb W HP 80-100 mesh was used as the solid support and was supplied by Supelco. Coating the solid support material with the ionic liquid was performed by dispersing a certain portion of Chromosorb in a solution of the ionic liquid in methanol followed by the evaporation of the solvent using a rotating evaporator. The masses of the stationary phase and of the solid support were weighed with a precision of ± 0.0001 g. The solvent column packing varied from (0.458 to 0.524) mass fraction of the ionic liquid, large enough to prevent any residual adsorption of solute onto the column packing. For each temperature the measurements were repeated by using two different columns with different mass fraction packing. Care was taken to ensure that the methanol had completely evaporated from the IL-coated solid before making up the column. Before experiment each column was conditioned by blowing carrier gas at a high flow rate (about 2.0 $\text{cm}^3 \cdot \text{s}^{-1}$) at the high temperature (about 373 K) through about 8 h.

The outlet pressure P_o was kept at atmospheric pressure. The pressure drop ($P_i - P_o$) was varied between (55 and 80) kPa depending on the flow rate of carrier gas. The pressure measurement was made by means of a pressure gauge installed on the gas chromatograph with an uncertainty of ± 0.1 kPa. The atmospheric pressure was measured using a digital barometer with an uncertainty of ± 0.01 kPa.

The carrier gas was helium. The flow rate of carrier gas was 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_{13}^{∞} determinations were made. The flow rates were corrected for water vapor pressure. Solute injections ranged from (0.01 to 0.3) μL and were considered to be at infinite dilution on the column.

Experiments were carried out at different temperatures (in steps of 10 K) between (298.15 and 368.15) K. The temperature of the column was maintained constant to within ± 0.02 K. At

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Table 1. Average Experimental Activity Coefficients at Infinite Dilution γ_{13}^{∞} for the Solutes in Ionic Liquid [hmim][SCN] at Different Temperatures and Excess Molar Enthalpies at Infinite Dilution $\Delta H_1^{E,\infty}$ for Investigated Ionic Liquid

	T/K								$\Delta H_1^{E,\infty}$
solute	298.15	308.15	318.15	328.15	338.15	348.15	358.15	368.15	$\mathrm{kJ \cdot mol^{-1}}$
n-heptane	81.4	76.7	72.1	68.3	64.5	61.7	58.6	56.0	4.9
n-octane	95.7	90.2	85.7	81.6	77.8	74.6	71.6	68.7	4.3
n-nonane	120	113	108	103	98.0	93.9	90.2	87.1	4.2
n-decane	162	152	144	136	130	125	120	115	4.4
cyclopentane	23.2	21.4	19.8	18.5	17.3	16.3	15.4	14.5	6.1
cyclohexane	28.2	26.0	24.2	22.7	21.3	20.0	18.9	17.9	5.9
cycloheptane	25.8	24.5	23.3	22.2	21.3	20.3	19.6	18.8	4.1
cyclooctane	30.1	28.6	27.2	26.0	24.8	23.8	22.8	22.0	4.1
pent-1-ene	28.0	26.1	24.6	23.1	21.9	20.8	19.8	18.9	5.1
hex-1-ene	28.7	27.4	26.2	25.3	24.4	23.5	22.7	22.1	3.4
hept-1-ene	34.2	33.0	32.1	31.2	30.3	29.6	29.0	28.3	2.4
oct-1-ene	42.1	41.0	39.9	39.0	38.1	37.3	36.5	35.7	2.1
pent-1-yne	4.04	4.14	4.24	4.34	4.43	4.52	4.60	4.70	−2.0
hex-1-yne	4.97	5.14	5.30	5.47	5.61	5.76	5.91	6.04	−2.5
hept-1-yne	6.69	6.93	7.14	7.33	7.55	7.74	7.94	8.11	−2.5
oct-1-yne	9.15	9.38	9.65	9.89	10.1	10.3	10.6	10.8	−2.1
benzene	1.91	1.95	1.99	2.02	2.05	2.08	2.11	2.14	−1.4
toluene	2.79	2.85	2.91	2.97	3.03	3.08	3.14	3.18	−1.7
ethylbenzene		4.39	4.45	4.50	4.56	4.61	4.66	4.70	−1.1
o-xylene		3.60	3.68	3.75	3.83	3.89	3.96	4.03	−1.8
m-xylene		4.44	4.51	4.58	4.65	4.72	4.77	4.83	−1.3
p-xylene		4.26	4.34	4.42	4.51	4.58	4.65	4.72	−1.6
methanol	0.532	0.522	0.514	0.505	0.497	0.490	0.483	0.477	1.4
ethanol	0.844	0.819	0.799	0.778	0.760	0.742	0.726	0.712	2.2
propan-1-ol				0.927	0.906	0.889	0.872	0.857	2.0
butan-1-ol				1.16	1.13	1.11	1.09	1.07	2.0
water				0.564	0.557	0.549	0.542	0.536	1.3
thiophene	1.24	1.28	1.31	1.34	1.37	1.40	1.43	1.45	−2.0
THF	1.60	1.63	1.65	1.68	1.70	1.73	1.75	1.77	−1.4
MTBE	8.78	8.78	8.75	8.73	8.72	8.72	8.72	8.71	0.11
diethyl ether	8.67	8.54	8.39	8.29	8.17	8.08	7.98	7.89	1.2
di-*n*-propyl ether	18.9	18.5	18.2	17.9	17.6	17.3	17.1	16.8	1.5
di-*n*-butyl ether	33.9	32.9	32.1	31.3	30.6	29.9	29.3	28.7	2.2
acetone	1.25	1.26	1.27	1.28	1.29	1.30	1.31	1.31	−0.70

a given temperature, each experiment was repeated two to four times to check the reproducibility. Retention times were generally reproducible within (0.001 to 0.01) min depending on the temperature and the individual solute. At each temperature value of the dead time, t_G, identical to the retention time of a nonretainable component, was measured. While our GC was equipped with a TCD detector, air was used as a nonretainable component. The estimated overall error in γ_{13}^{∞} was less than 3 %, taking into account the possible errors in determining the column loading, the retention times, and solute vapor pressure. The GLC technique was tested for the hexane in hexadecane system at 298.15 K, and the results compared very favorably with the literature values.[7]

Theoretical Basis. The equation developed by Everett[8] and Cruickshank et al.[9] was used in this work to calculate the γ_{13}^{∞} of solutes in the ionic liquid

$$\ln \gamma_{13}^{\infty} = \ln\left(\frac{n_3RT}{V_NP_1^*}\right) - \frac{P_1^*(B_{11} - V_1^*)}{RT} + \frac{P_oJ_2^3(2B_{12} - V_1^{\infty})}{RT} \tag{1}$$

The 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} (where 2 refers to the carrier gas, helium) 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[10] equation for alkanes and the Tsonopolous[11] equation for the rest of the solvents. Using the Hudson and McCoubrey combining rules,[12,13] critical parameters for mixtures were calculated from the critical properties of the pure component.

The pressure correction term J_2^3 is given by

$$J_2^3 = \frac{2}{3}\frac{(P_i/P_o)^3 - 1}{(P_i/P_o)^2 - 1} \tag{2}$$

The net retention volume of the solute V_N is given by

$$V_N = J_2^3U_o(t_R - t_G) \tag{3}$$

where t_R and t_G are the retention times for the solute and an unretained gas, respectively, and U_o is the column outlet flow rate.

The vapor pressure values were calculated using an equation and constants taken from the literature.[14−16] Critical data used to calculate B_{11} and B_{12} were obtained from literature.[17,18]

Results and Discussion

Table 1 lists the average γ_{13}^{∞} values for the varying amounts of solvent on the column packing for the investigated ionic liquid over the temperature range from (298.15 to 368.15) K and partial molar excess enthalpies at infinite dilution $\Delta H_1^{E,\infty}$ determined from the Gibbs−Helmholtz equation:

$$\frac{\partial \ln \gamma_i^{\infty}}{\partial(1/T)} = \frac{\Delta H_1^{E,\infty}}{R} \tag{4}$$

The $\Delta H_1^{E,\infty}$ values are negative for alk-1-ynes, aromatic hydrocarbons, thiophene, tetrahydrofuran (THF), and acetone.

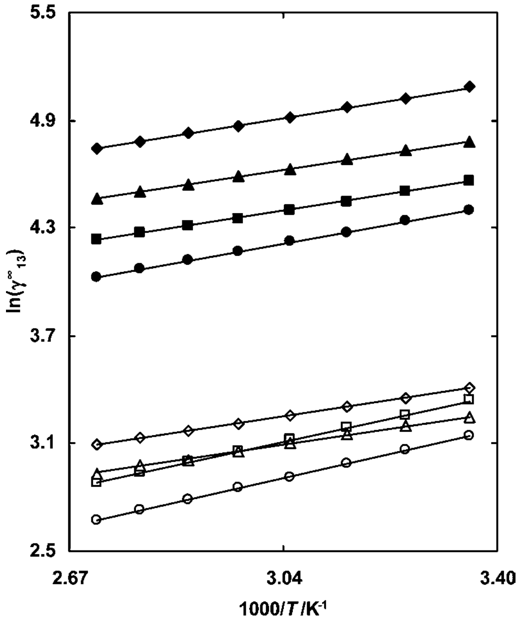

Figure 1. Plot of ln(γ_{13}^{∞}) for the ionic liquid [hmim][SCN] versus 1/*T* for the solutes. ●, *n*-heptane; ■, *n*-octane; ▲, *n*-nonane; ♦, *n*-decane; ○, cyclopentane; □, cyclohexane; △, cycloheptane; ◇, cyclooctane. The solid line represents the linear correlation.

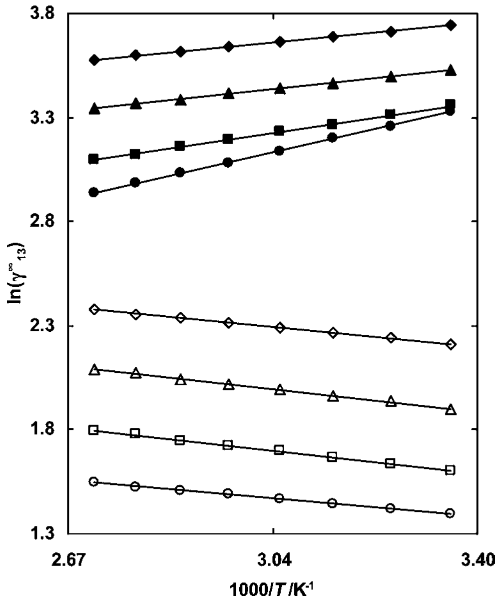

Figure 2. Plot of ln(γ_{13}^{∞}) for the ionic liquid [hmim][SCN] versus 1/*T* for the solutes. ●, pent-1-ene; ■, hex-1-ene; ▲, hept-1-ene; ♦, oct-1-ene; ○, pent-1-yne; □, hex-1-yne; △, hept-1-yne; ◇, oct-1-yne. The solid line represents the linear correlation.

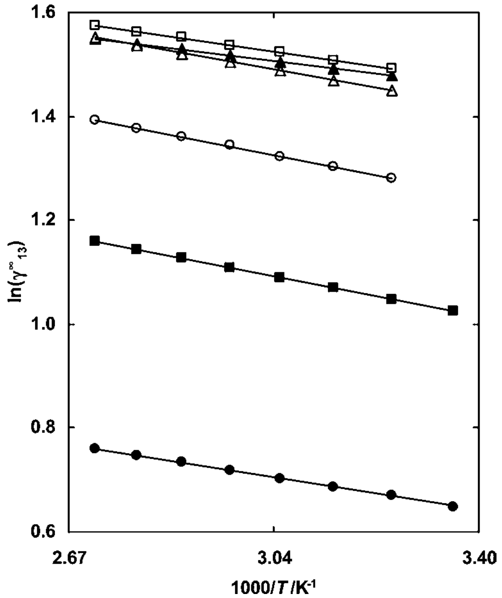

Figure 3. Plot of ln(γ_{13}^{∞}) for the ionic liquid [hmim][SCN] versus 1/*T* for the solutes. ●, benzene; ■, toluene; ▲, ethylbenzene; ○, *o*-xylene; □, *m*-xylene; △, *p*-xylene. The solid line represents the linear correlation.

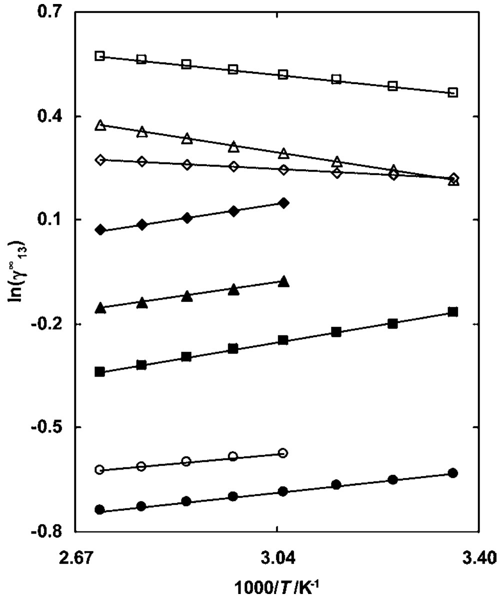

Figure 4. Plot of ln(γ_{13}^{∞}) for the ionic liquid [hmim][SCN] versus 1/*T* for the solutes. ●, methanol; ■, ethanol; ▲, propan-1-ol; ♦, butan-1-ol; ○, water; △, thiophene; □, THF; ◇, acetone. The solid line represents the linear correlation.

The negative values of the partial molar excess enthalpies at infinite dilution mean that the interactions of solute–solvent pairs are higher than those for solute–solute pairs. This behavior is caused by interaction among the polar anions of the IL, the triple bond in alk-1-ynes, and the polarizable π-electrons in aromatic compounds and thiophene. For THF and acetone, the $\Delta H_1^{E,\infty}$ values are negative for the ILs that have been investigated.

Figures 1 to 5 show the natural logarithm of the activity coefficients in the ionic liquid as a function of the inverse absolute temperature for all investigated solutes for [hmim]-[SCN] ionic liquid.

The values of γ_{13}^{∞} for the series of solutes increase with an increase in the solute alkyl chain.[1−4] The highest values of γ_{13}^{∞} are for alkanes, alkenes, and cycloalkanes. This behavior was also observed for other ionic liquids based on the imidazolium cation. High values of γ_{13}^{∞} signify very small interactions between solute and solvent. The values of γ_{13}^{∞} for alkenes are lower than those for alkanes for the same number of carbon atoms. This is caused by the interaction of double bonding in alkenes with a polar ionic liquid. The cyclic structure of cycloalkanes reduces the value of γ_{13}^{∞} in comparison to the corresponding linear alkane. Alkynes and aromatic hydrocarbons have smaller values of γ_{13}^{∞} than alkanes, alkenes, and cycloalkanes. This is a result of stronger interactions between solvent and solute. It probably reveals interactions between the triple bond in alkynes and six π-delocalized electrons in aromatics with the polar cation and/or anion of ionic liquid. The smallest values of γ_{13}^{∞} are for alcohols and water, which indicates that these types of polar compounds show the highest interaction

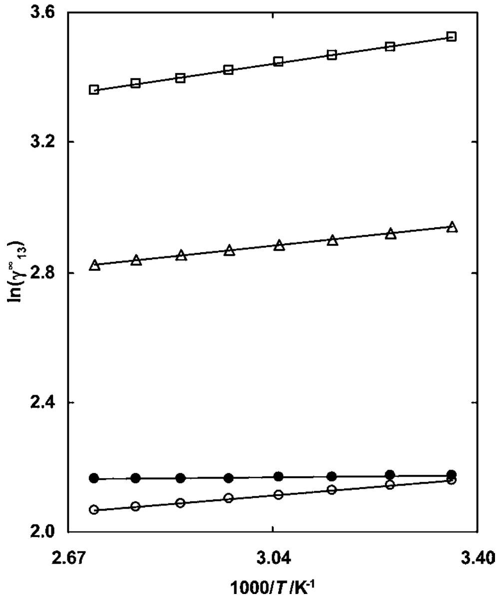

Figure 5. Plot of $\ln(\gamma_{13}^{\infty})$ for the ionic liquid [hmim][SCN] versus $1/T$ for the solutes. ○, diethyl ether; △, di-*n*-propyl ether; □, di-*n*-butyl ether; ●, methyl *tert*-butyl ether (MTBE). The solid line represents the linear correlation.

with [hmim][SCN] ionic liquid. It is a confirmation of the solubility measurements of [hmim][SCN] in water and alcohols.[4] The simple eutectic system was observed for water with complete miscibility in the liquid phase. Also, complete miscibility has been observed in the systems of [hmim][SCN] with alcohols ranging from methanol to 1-decanol at the temperature $T = 298.15$ K. On the other side the upper critical solution temperatures (UCSTs) were observed for the systems with hydrocarbons.[4] Also very small values of γ_{13}^{∞}, but higher than those of alcohols and water, were noted for thiophene, acetone, and THF. This is also caused by strong interactions of polar groups from these compounds with a polar ionic liquid. These interactions are, however, lower than those with water and alcohols. Although ethers contain oxygen in their structure, they do not interact as strongly as alcohols with [hmim][SCN]. It is caused by steric effects: the alkyl groups block oxygen from two sides. In general, the more the aliphatic character of the compound, the higher the values of γ_{13}^{∞} and the lower interactions are.

A very important factor in designing extraction processes is the knowledge about selectivity S_{12}^{∞} and capacity k_2^{∞} of the entrainers, which can be calculated directly from experimental γ_{13}^{∞} values:[1]

$$S_{ij}^{\infty} = \gamma_{i3}^{\infty}/\gamma_{j3}^{\infty} \qquad (5)$$

$$k_j^{\infty} = 1/\gamma_{j3}^{\infty} \qquad (6)$$

where 3 is an ionic liquid.

Figure 6 shows the influence of the structure of ionic liquids based on the $[\text{hmim}]^+$ cation or containing a $-\text{C}{\equiv}\text{N}$ group on S_{12}^{∞} and k_2^{∞} for aliphatic/aromatic separation problem at $T = 323.15$ K in contrast to *N*-methyl-2-pyrrolidinone (NMP) and sulfolane. The presentation in Figure 6 is not complete for the one IL because of the incomplete literature data. For ionic liquids based on the $[\text{hmim}]^+$ cation, the highest values of selectivities are for the investigated [hmim][SCN] ionic liquid (35.0 for *n*-heptane/benzene, 23.8 for *n*-heptane/toluene, and 11.7 for cyclohexane/benzene), but the capacities are the lowest in comparison with NMP, other ILs, or sulfolane ($k_2^{\infty} = 0.50$ for benzene and $k_2^{\infty} = 0.34$ for toluene). The order of selectivity is the following: NMP[19] < $[\text{hmim}][\text{NTf}_2]$[20] < $[\text{hmim}][\text{CF}_3\text{SO}_3]$[21] < $[\text{hmim}][\text{PF}_6]$[22] < sulfolane[23] < $[\text{hmim}][\text{BF}_4]$[24] < [hmim][SCN]. For ionic liquids based on the $[\text{SCN}]^-$ anion, the selectivities increase with a decrease in the alkyl chain length on the imidazolium cation. The capacities behave in the opposite order. The highest value of selectivity is for [emim][SCN],[2] but the highest value of capacity is for [hmim][SCN]. High values of selectivities also reveal other ionic liquids with the $-\text{C}{\equiv}\text{N}$ group in an anion or cation structure.[25,26]

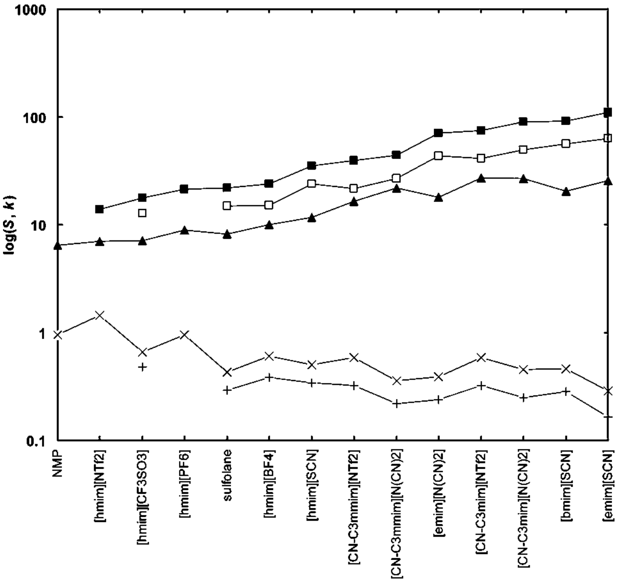

Figure 6. Structure influence on *S* and *k* for the aliphatic/aromatic separation problem for ionic liquids based on the $[\text{hmim}]^+$ cation or containing a $-\text{C}{\equiv}\text{N}$ group in the structure at $T = 323.15$ K. —■—, *S* *n*-heptane/benzene; —□—, *S* *n*-heptane/toluene; —▲—, *S* cyclohexane/benzene; —X—, *k* benzene; —+—, *k* toluene. NMP;[19] $[\text{hmim}][\text{NTf}_2]$;[20] $[\text{hmim}][\text{CF}_3\text{SO}_3]$;[21] $[\text{hmim}][\text{PF}_6]$;[22] sulfolane;[23] $[\text{hmim}][\text{BF}_4]$;[24] [hmim][SCN] (this work). $[\text{CN-C}_3\text{mmim}][\text{NTf}_2]$;[25] $[\text{CN-C}_3\text{mmim}][\text{N(CN)}_2]$;[25] $[\text{emim}][\text{N(CN)}_2]$;[26] $[\text{CN-C}_3\text{mim}][\text{NTf}_2]$;[25] $[\text{CN-C}_3\text{mim}][\text{N(CN)}_2]$;[25] [bmim][SCN];[3] [emim][SCN].[2]

Conclusions

Activity coefficients at infinite dilution for various solutes in the ionic liquid 1-hexyl-3-methylimidazolium thiocyanate were measured by gas−liquid chromatography at temperatures from (298.15 to 368.15) K.

It was found that the investigated [hmim][SCN] ionic liquid shows much higher selectivity at infinite dilution than the generally used organic solvents such as NMP[16] or sulfolane[20] and other ionic liquids based on the $[\text{hmim}]^+$ cation in the separation process of aliphatic/aromatic hydrocarbons. The capacity is also satisfactory and is higher than that for sulfolane.

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