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Experimental Vapor Pressures of 1-Alkyl-3-methylimidazolium Bis(trifluoromethylsulfonyl)imides and a Correlation Scheme for Estimation of Vaporization Enthalpies of Ionic Liquids

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Vapor pressures for a series of 1-*n*-alkyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide (alkyl = ethyl, butyl, hexyl, and octyl) ionic liquids (ILs) were measured by the integral effusion Knudsen method. Thermodynamic parameters of vaporization for ILs were calculated from these data. The absence of decomposition of ILs during the vaporization process was proved by IR spectroscopy. Enthalpies of vaporization of ILs were correlated with molar volumes and surface tensions of the compounds.

Introduction

It is common knowledge that ionic liquids (ILs) have extremely low vapor pressures (for $[C_4mim][PF_6] \sim 10^{-10}$ Pa at 298 K).[1] In ref 2 Rebelo et al. estimated the NBP temperatures for ILs with a common anion. They also noted that 1-alkyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imides ($[C_nmim][NTf_2]$) could probably have measurable vapor pressures. In our experiments it was proved that the balance of the thermal stability and volatility for $[C_nmim][NTf_2]$ indeed allowed measurements of vapor pressures in a temperature interval large enough for a proper evaluation of thermodynamic parameters such as vaporization $\Delta_l^g H_m^o$ and $\Delta_l^g S_m^o$. In this work the results for vapor pressures for 1-*n*-alkyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide ILs (alkyl = ethyl, butyl, hexyl, and octyl) are reported as a function of temperature.

Earlier the authors of ref 3 analyzed possibilities of correlations based on molecular liquids for predicting $\Delta_l^g H_m^o$ of ILs. It was shown that these correlation schemes could only give very crude estimates. In this work a promising correlation for evaluation of $\Delta_l^g H_m^o$ from surface tension and molar volume of ILs has been developed. Estimated values of enthalpies of vaporization for a series of ILs are presented.

Experimental Section

The samples of $[C_2mim][NTf_2]$ and $[C_8mim][NTf_2]$ were kindly provided by Prof. Ya. S. Vygodskii and $[C_6mim][NTf_2]$ by Dr. M. Muldoon. The commercial sample of $[C_4mim][NTf_2]$ was used. The mole fraction purities of the samples were determined by the fractional melting method in the adiabatic calorimeter and were found to be 0.986 for $[C_2mim][NTf_2]$, 0.986 for $[C_4mim][NTf_2]$, 0.996 for $[C_6mim][NTf_2]$, and 0.996 for $[C_8mim][NTf_2]$. Before starting with effusion measurements the samples were exposed to a vacuum of 10^{-3} Pa for several days at temperatures increasing stepwise from 323 to 443 K. While being kept at the highest temperature the mass loss rate decreased by a few orders. This loss of mass was apparently due to slow removal of a volatile impurity such as 1-methylimidazole. The sample was kept at this temperature in a vacuum until the mass loss rate became constant.

The vapor pressure for $[C_nmim][NTf_2]$ was measured by the integral effusion Knudsen method. The apparatus[4] was equipped with a thermostating block for experiments at 320−530 K. The temperature was controlled within ±0.01 K and was measured with a platinum resistance thermometer placed in the block. The inner surface of the block made of copper was coated with high-temperature silicon. This compound is chemically inert toward ILs and thermally stable to 560 K. The silicon coating had been exposed to a vacuum at 523 K until no condensate was formed on a coldfinger filled with the liquid nitrogen. This coating prevents contacts of the studied compounds in the gas phase with copper parts of the block and their catalytic decomposition. Residual pressure in the system was $<10^{-3}$ Pa.

In the measurements a cylindrical cell with a height of 10.0 mm and an internal diameter of 10.0 mm was used. It was covered by a plate with an orifice of diameter (d) 3.10 ± 0.05 mm and thickness (l) 2.1 ± 0.05 mm. All the parts were made of stainless steel. A Teflon ring was used for sealing. At each fixed temperature, the cell was maintained at a constant temperature for about an hour prior to starting the effusion experiment.

The vapor pressure values were calculated from the results of effusion experiments by the equation

$$p = \left(1 + \frac{kS_{\text{orif}}}{\alpha S_{\text{samp}}}\right)\frac{\Delta m}{kS_{\text{orif}}\tau}\sqrt{\frac{2\pi RT}{M}} \qquad (1)$$

where Δm is the mass loss during an elapsed time τ, S_{orif} is the area of the orifice, k is the transmission coefficient (k = 0.596), M is the molar mass of the vapor, S_{samp} is the surface area of the liquid assumed to be equal to the cross-section area of the cell, and α is the vaporization coefficient. As a rule, $\alpha = 1$ holds for liquids. The vapor was assumed to consist

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of ionic pairs [$C_n$mim][NTf_2]. The uncertainty in temperature was estimated to be 0.02 K, and that in the pressure was better than 10%.

IR spectra of [C_2mim][NTf_2] in KBr pellets were recorded with a Bruker Vertex 70 spectrometer with a resolution of 4 cm^{-1}.

The surface tensions of the substances were determined by the pendant-drop method. A commercial tensiometer (Krüss GmbH, Germany) has been used. An axially symmetrical pendant drop is formed by a steel needle in a homogeneous gravitation field. In the hydrodynamical equilibrium the curvature of the drop is described by eq 2:[5]

$$\frac{d\Phi}{dS} = 2R + \frac{z\Delta\rho g}{\sigma} - \frac{\sin\Phi}{x} \qquad (2)$$

where Φ is the angle of the local surface to the horizontal x-axis perpendicular to the z-axis of the pendant drop, R is the radius of curvature at the drop apex, z is the axial coordinate of the described point to the drop apex, x is the distance of the local point of the liquid surface from the horizontal x-axis of the drop, S is the arc length to the point from the drop apex, $\Delta\rho$ is the difference of the densities of both phases, g is the gravitational acceleration, and σ the surface tension.

Equation 2 is the basic differential equation that has been used to determine σ from the total drop shape digitally registered by a CCD camera. The numerical solution of eq 2 provides the surface tension. The procedure has been performed with the appropriate software (Krüss GmbH, Germany). The surface tension σ and the densities were measured at normal pressure at 1 bar. The densities were obtained by using a vibrating tube densimeter DMA 602 (by Anton Paar, Austria) with the temperature stability of ±0.05 K. Air and bidistilled water were used for the calibration of the densimeter. The densities of these two substances are known from the literature.[6] The samples were degassed with an ultrasound bath.

Results

Stability of ILs in Effusion Experiments. ILs inside the effusion cell remained colorless. The IR spectrum of [C_2mim][NTf_2] from the effusion cell after all the experiments listed in Table 1 is presented in Figure 1. This spectrum is identical with that of pure [C_2mim][NTf_2] without vacuum treatment.

The vapor phase condensed on the coldfinger was visually controlled. In most experiments the condensate was colorless. In the case when the condensate was slightly colored the experimental vapor pressures did not deviate substantially from the smoothing line. Appearance of the color was not systematic, and the reason for this phenomenon is unknown. The IR spectrum of [C_2mim][NTf_2] condensed on the coldfinger after a special effusion experiment at 474 K is shown in Figure 1. The only essential difference in the spectra is observed at the two absorbance bands at 2925 and 2855 cm^{-1}. These bands correspond to C−H stretching vibrations in an alkyl chain. The presence of these bands in the IR spectrum is due to condensation of vacuum oil from the oil diffusion pump on the coldfinger and is not connected with decomposition of [C_2mim][NTf_2]. In order to confirm the stability of the ionic liquid in the experimental conditions of vapor pressure measurements, the sample of [C_2mim][NTf_2] has been withdrawn from the cell and analyzed using the GC−MS technique. No traces of a possible decomposition have been found in this probe as well as in the probe of condensate collected at $T = 474$ K above the effusion cell.

TABLE 1: The Results of Vapor Pressure Measurements for [$C_n$mim][NTf_2]

T/K	Δm/mg	τ/s	p/Pa
[C_2mim][NTf_2] ($M = 0.39172$ kg·mol^{-1})			
441.70	1.18	10800	0.0062
455.21	4.38	15147	0.0167
455.22	4.11	14404	0.0165
464.97	4.13	7565	0.0319
464.98	4.07	7250	0.0329
474.46	3.44	3600	0.0565
484.16	7.32	4005	0.1091
[C_4mim][NTf_2] ($M = 0.41936$ kg·mol^{-1})			
437.84	2.57	39690	0.0036
447.61	2.77	21600	0.0071
457.66	4.17	18780	0.0125
467.52	7.46	18120	0.0233
477.68	13.09	14400	0.0521
487.54	5.63	3600	0.0906
497.53	9.97	3660	0.159
507.54	9.70	1950	0.294
517.45	15.96	1850	0.515
[C_6mim][NTf_2] ($M = 0.44741$ kg·mol^{-1})			
445.79	1.80	14400	0.0067
445.80	1.79	14400	0.0067
445.81	1.82	14460	0.0067
445.81	0.28	2130	0.0070
455.46	3.77	14520	0.0141
464.94	5.26	10800	0.0267
474.46	6.97	7320	0.0527
484.12	5.98	3605	0.0927
493.67	10.95	3600	0.1716
[C_8mim][NTf_2] ($M = 0.47546$ kg·mol^{-1})			
455.46	2.15	14540	0.0078
455.49	2.15	14520	0.0078
464.95	4.41	14400	0.0163
474.44	8.63	14400	0.0322
484.11	8.48	7215	0.0637
493.78	8.01	3735	0.1174
498.19	10.88	3880	0.1542

Further vacuum exposition of [C_2mim][NTf_2] led to complete evaporation of IL without formation of any nonvolatile residue in the effusion cell. So, one may conclude that evaporation of the investigated ILs was not caused by their decomposition.

Vapor Pressure. The results of vapor pressure measurements are shown in Table 1 and in Figure 2. The results for [C_4mim][NTf_2] above $T = 457$ K have already been reported in our preliminary communication.[7] In that paper the k coefficient was corrected with respect to the anisotropy of the effusing gas. However, in those measurements the mean-free path of molecules was much longer than the size of the cell, and the ionic pairs did not collide inside the cell. For this reason the correction for the anisotropy should not be made. The recalculated vapor pressures for [C_4mim][NTf_2] above $T = 457$ K are given in Table 1.

The temperature dependences of vapor pressures for [$C_n$mim][NTf_2] were fitted by a linear equation:

$$\ln(p/\mathrm{Pa}) = A - B/(T/\mathrm{K}) \qquad (3)$$

The enthalpies of vaporization at middle temperatures of effusion experiments $\langle T \rangle$ (Table 2) were calculated as $\Delta_l^g H_m^o = BR$. The entropies of vaporization were calculated as

$$\Delta_l^g S_m^o(\langle T \rangle) = \frac{\Delta_l^g H_m^o(\langle T \rangle)}{\langle T \rangle} + R \ln\left(\frac{p(\langle T \rangle)}{p^o}\right) \qquad (4)$$

where $p^o = 10^5$ Pa. All the parameters of eq 3 and 4 are presented in Table 2.

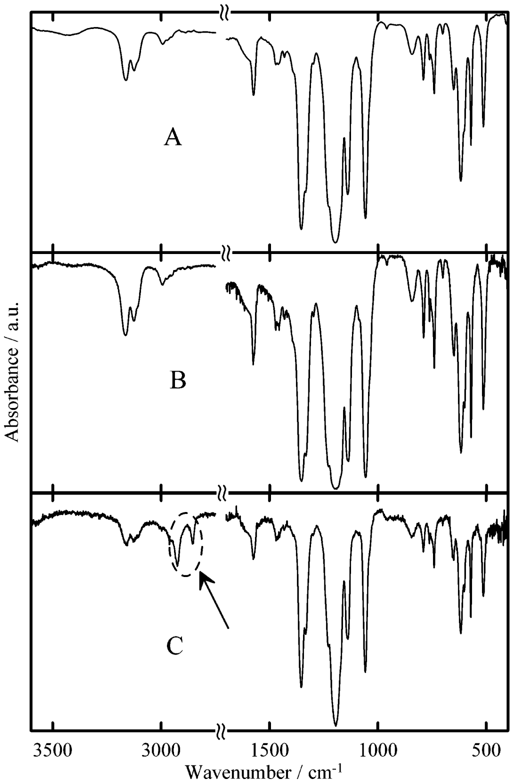

Figure 1. IR spectra for [C$_2$mim][NTf$_2$] samples: (a) initial sample, (b) from the effusion cell, and (c) condensed on the coldfinger.

The values of $\Delta_{\mathrm{l}}^{\mathrm{g}}S_{\mathrm{m}}^{\mathrm{o}}(\langle T\rangle)$ for investigated samples are close to the entropy of vaporization $\Delta_{\mathrm{l}}^{\mathrm{g}}S_{\mathrm{m}}^{\mathrm{o}}(500\ \mathrm{K}) = 131\ \mathrm{J{\cdot}K^{-1}{\cdot}mol^{-1}}$ for [C$_4$mim][PF$_6$] calculated from the results of calorimetric measurements and statistical thermodynamic calculations in refs 1 and 8. In the calculations the vapor of [C$_4$mim][PF$_6$] was also taken to consist of ionic pairs. The proximity of the experimental and calculated $\Delta_{\mathrm{l}}^{\mathrm{g}}S_{\mathrm{m}}^{\mathrm{o}}$ values indicates that this assumption about composition of vapor is acceptable.

To adjust the values of enthalpy of vaporization for [C$_n$mim][NTf$_2$] to 298.15 K one needs to evaluate the heat capacity difference between the liquid and gas phases. In our previous work[8] this heat-capacity jump was determined for [C$_4$mim][PF$_6$] from calorimetric data and statistical thermodynamic calculations to be $\Delta_{\mathrm{l}}^{\mathrm{g}}C_P(298\ \mathrm{K}) = -105\ \mathrm{J{\cdot}K^{-1}{\cdot}mol^{-1}}$. In this work the value $-\Delta_{\mathrm{l}}^{\mathrm{g}}C_P = 100\ \mathrm{J{\cdot}K^{-1}{\cdot}mol^{-1}}$ was applied for all the studied ILs.

Surface Tension and Density. The experimental results of the density and the surface tension of [C$_8$mim][NTf$_2$] and [C$_{14}$mim][NTf$_2$] are presented in Table 3.

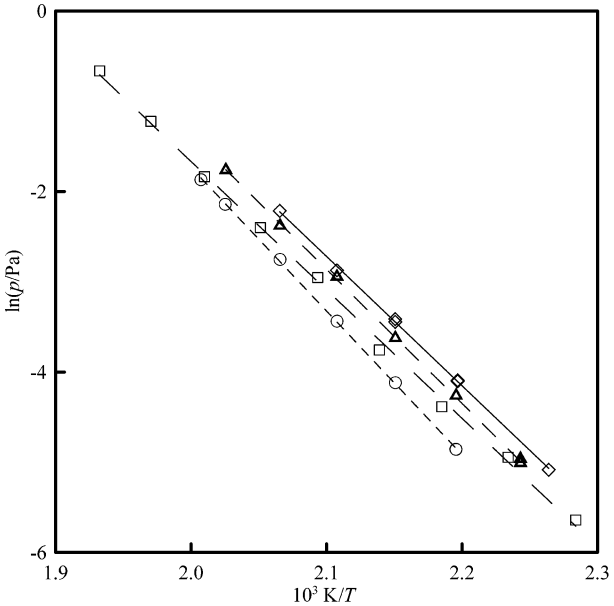

Figure 2. Temperature dependence of the vapor pressure for [C$_n$mim][NTf$_2$], where rhombs are the data for [C$_2$mim][NTf$_2$], squares are the data for [C$_4$mim][NTf$_2$], triangles are the data for [C$_6$mim][NTf$_2$], and circles are the data for [C$_8$mim][NTf$_2$].

Discussion

The trend of $\Delta_{\mathrm{l}}^{\mathrm{g}}H_{\mathrm{m}}^{\mathrm{o}}(298\ \mathrm{K})$ in the series of [C$_n$mim][NTf$_2$] is shown in Table 2. All the values lie between 135 and 151 kJ·mol^{-1}. These values are of the same order of magnitude as $\Delta_{\mathrm{l}}^{\mathrm{g}}H_{\mathrm{m}}^{\mathrm{o}} = 161\ \mathrm{kJ{\cdot}mol^{-1}}$ calculated for [C$_4$mim][PF$_6$] from the molecular-dynamic simulations reported in ref 9.

Rebelo et al.[2] conceived that $\Delta_{\mathrm{l}}^{\mathrm{g}}H_{\mathrm{m}}^{\mathrm{o}}$ for ILs should be close to 300 kJ·mol^{-1} without fixing the temperature to which the values refer. According to Rebelo's estimate of $\Delta_{\mathrm{l}}^{\mathrm{g}}H_{\mathrm{m}}^{\mathrm{o}}$ and T_{b} the values of $\Delta_{\mathrm{l}}^{\mathrm{g}}S_{\mathrm{m}}^{\mathrm{o}}(T_{\mathrm{b}})$ will be from 450 to 600 J·K^{-1}·mol^{-1}. This estimate is not consistent with $\Delta_{\mathrm{l}}^{\mathrm{g}}H_{\mathrm{m}}^{\mathrm{o}}$ and $\Delta_{\mathrm{l}}^{\mathrm{g}}S_{\mathrm{m}}^{\mathrm{o}}$ values obtained in this work and the results of $\Delta_{\mathrm{l}}^{\mathrm{g}}S_{\mathrm{m}}^{\mathrm{o}}$ calculations for [C$_4$mim][PF$_6$][1,8] with calorimetric $\Delta_0^{298.15}S_{\mathrm{m}}^{\mathrm{o}}(\mathrm{liq})$ and $\Delta_0^{298.15}S_{\mathrm{m}}^{\mathrm{o}}(\mathrm{g})$ calculated by statistical thermodynamics.

Alternatively, the $\Delta_{\mathrm{l}}^{\mathrm{g}}H_{\mathrm{m}}^{\mathrm{o}}$ for ILs can be estimated at T_{b} by using the normal boiling temperatures estimated by Rebelo and a Trouton constant (≈100 J·mol^{-1}·K^{-1}). In this case $\Delta_{\mathrm{l}}^{\mathrm{g}}H_{\mathrm{m}}^{\mathrm{o}}$ for ILs will be from 50 to 70 kJ·mol^{-1}. But such values of enthalpy of vaporization are typical for molecular liquids.

The vapor pressure values were extrapolated to 101.3 kPa. The values of T_{b} estimated from eq 3 are 907 K for [C$_2$mim][NTf$_2$], 933 K for [C$_4$mim][NTf$_2$], 885 K for [C$_6$mim][NTf$_2$], and 857 K for [C$_8$mim][NTf$_2$]. These values are essentially (280

TABLE 2: Parameters of Vaporization for [C$_n$mim][NTf$_2$]

substance	$\langle T\rangle$/K	A	$10^{-3}B$/K	$\Delta_{\mathrm{l}}^{\mathrm{g}}H_{\mathrm{m}}^{\mathrm{o}}(\langle T\rangle)$/ kJ·mol^{-1}	$\Delta_{\mathrm{l}}^{\mathrm{g}}S_{\mathrm{m}}^{\mathrm{o}}(\langle T\rangle)$/ J·K^{-1}·mol^{-1}	$\Delta_{\mathrm{l}}^{\mathrm{g}}H_{\mathrm{m}}^{\mathrm{o}}(298\ \mathrm{K})$/ kJ·mol^{-1}
[C$_2$mim][NTf$_2$]	463.0	27.29 ± 0.35	14.29 ± 0.16	118.8 ± 1.3	131.2 ± 2.9	135.3 ± 1.3
[C$_4$mim][NTf$_2$]	477.6	26.78 ± 0.42	14.22 ± 0.20	118.3 ± 1.7	126.9 ± 3.5	136.2 ± 1.7
[C$_6$mim][NTf$_2$]	461.8	28.29 ± 0.22	14.84 ± 0.10	123.4 ± 0.8	139.5 ± 1.8	139.8 ± 0.8
[C$_8$mim][NTf$_2$]	475.2	30.08 ± 0.20	15.91 ± 0.10	132.3 ± 0.8	154.4 ± 1.7	150.0 ± 0.8

TABLE 3: Experimental Results on Measurements of Density and Surface Tension of ILs

substance	T/K	ρ^{a}/kg·m^{-3}	σ/mN·m^{-1}
[C$_8$mim][NTf$_2$]	298.15 ± 0.05	1321.00 ± 0.02 (9)	30.63 ± 0.10 (50)
	308.15 ± 0.05	1311.97 ± 0.03 (9)	29.78 ± 0.13 (70)
[C$_{14}$mim][NTf$_2$]	308.15 ± 0.05	1206.40 ± 0.01 (9)	29.39 ± 0.09 (90)

[a] Number of measurements is given in parentheses.

TABLE 4: Data for Calculations of the Parameters of Eq 7 ($T = 298.15$ K)

substance	σ/mJ·m^{-2}	$10^4 V_m$/m^3·mol^{-1}	$\Delta_l^g H_m^o$/kJ·mol^{-1}
$[C_2mim][NTf_2]^a$	34.90	2.575	135.3
$[C_4mim][NTf_2]^a$	31.76	2.918	136.2
$[C_6mim][NTf_2]^a$	30.94	3.280	139.8
$[C_8mim][NTf_2]$	30.63	3.599	150.0

[a] The values of σ and V_m from ref 2.

TABLE 5: Estimated Values of Enthalpies of Vaporization of Ionic Liquids ($T = 298.15$ K)

no.	substance	$10^4 V_m$/ m^3·mol^{-1}	σ/ mJ·m^{-2}	ref	$\Delta_l^g H_m^o$/ kJ·mol^{-1}
1	$[C_2mim][NTf_2]$	2.575	34.90	2	136.1
2	$[C_4mim][NTf_2]$	2.918	31.76	2	134.6
3	$[C_6mim][NTf_2]$	3.280	30.94	2	141.6
4	$[C_8mim][NTf_2]$	3.663	30.24	this work	149.0
5	$[C_{10}mim][NTf_2]$	4.029	29.66	2	155.5
6	$[C_{14}mim][NTf_2]$	4.639^a	29.39^a	this work	169.0
7	$[C_4mim][PF_6]$	2.078	45.89	2	154.8
8	$[C_6mim][PF_6]$	2.415	37.45	2	139.8
9	$[C_8mim][PF_6]$	2.778	35.24	2	144.3
10	$[C_{10}mim][PF_6]$	3.090	30.88	2	135.9
11	$[C_{12}mim][PF_6]$	3.466	23.49	2	112.0
12	$[C_4mim][BF_4]$	1.877	40.56	2	128.2
13	$[C_8mim][BF_4]$	2.557	31.39	2	122.0
14	$[C_{12}mim][BF_4]$	3.502	27.29	2	130.7
15	$[C_8mim][Cl]$	2.308	33.8	2	122.7
16	$[Ph(CH_2)mim][NTf_2]$	3.041	40.8	12	177.0
17	$[Ph(CH_2)_2mim][NTf_2]$	3.180	42.1	12	188.0
18	$[Ph(CH_2)_3mim][NTf_2]$	3.309	43.5	12	199.3
19	$[Ph(CH_2)_3mim][PF_6]$	2.461	33.0	12	125.0

[a] $T = 308.15$ K

K) higher than those estimated by Rebelo.[2] Taking into account $\Delta_l^g C_P$ will only increase this disagreement.

The demonstrated problems found in Rebelo's work show that the Eotvos−Guggenheim correlations are hardly applicable for ILs because they were obtained for molecular liquids. Furthermore, it should be noted that in ref 2 the density and surface tension at $T = 300$ K were used to calculate T_b and T_c. This produces additional uncertainty to the estimates. Therefore, a new correlation equation for $\Delta_l^g H_m^o$ of ILs should be developed.

Correlations of Enthalpy of Vaporization. The use of the Stefan equation

$$\sigma = \frac{\Delta_l^g H^o}{V^{2/3} {N_A}^{1/3}} \frac{Z_S}{Z} \tag{5}$$

for calculation of the enthalpy of vaporization on the basis of correlation equation $\Delta_l^g H_m^o = f(\sigma, V_m, M, T)$ was studied earlier,[3] where σ is the surface tension, V_m is molar volume of the compound, M is its molar mass, and Z_S, and Z are surface and bulk coordination numbers for the molecules in the liquid.

It was found that for ILs it is more appropriate to use Fowkes[10] approach in which surface tension is presented as a sum

$$\sigma = \sigma_d + \sigma_n \tag{6}$$

where σ_d is the part of the surface tension caused by dispersive interactions and σ_n is the part related to nondispersive interactions (dipole−dipole, Coulomb, etc.). In this case the correlation equation for the enthalpy of vaporization is:

$$\Delta_l^g H_m^o = A(\sigma \, {V_m}^{2/3} {N_A}^{1/3}) + B \tag{7}$$

where B is a nondispersive part. The B values are (1.9 ± 1.1) kJ·mol^{-1} for hydrocarbons, (8.7 ± 1.8) for carbonyl compounds, and (22.6 ± 1.3) for alcohols. It seems reasonable to apply eq 7 for estimation of $\Delta_l^g H_m^o$ of ILs.

The parameters of eq 7 were found from the data shown in Table 4. Enthalpies of vaporization of $[C_nmim][NTf_2]$, surface tensions, and densities of $[C_8mim][NTf_2]$ and $[C_{14}mim][NTf_2]$ were obtained in this work. The other values were extrapolated to $T = 298.15$ K with use of the data from ref 2. The parameters A and B were calculated by the least-squares method: $A = 1.121 \times 10^{-2}$, $B = 2.4$ kJ·mol^{-1}. The correlation coefficient R^2 was 0.94. The relative error in prediction of $\Delta_l^g H_m^o$ did not exceed 2%.

The enthalpy of vaporization $\Delta_l^g H_m^o$ for $[C_4mim][PF_6]$ calculated from eq 7 was found to be 154.8 kJ·mol^{-1}. This value is in satisfactory agreement with the results of molecular-dynamic simulations[9] $\Delta_l^g H_m^o$(298.15K) = 161 kJ·mol^{-1}. The calculated values of $\Delta_l^g H_m^o$(298.15K) for other ILs are presented in Table 5.

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