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Physicochemical Characterization of 1-Butyl-3-methylimidazolium and 1-Butyl-1-methylpyrrolidinium Bis(trifluoromethylsulfonyl)imide

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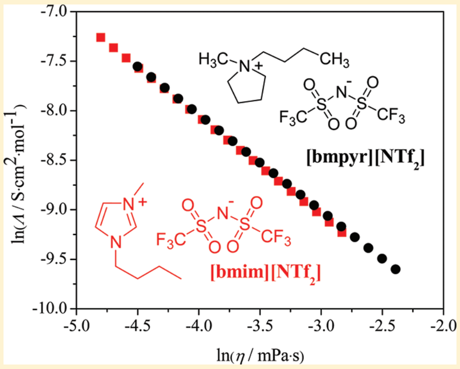
Supporting Information

ABSTRACT: Densities of two selected ionic liquids (ILs), 1-butyl-3-methylimidazolium *bis*(trifluoro-methylsulfonyl)imide, [bmim][NTf_2], and 1-butyl-1-methylpyrrolidinium *bis*(trifluoromethyl-sulfonyl)imide, [bmpyr][NTf_2], were determined in the temperature range from (293.15 to 353.15) K. From the experimental density values, the thermal expansion coefficient was calculated. The specific conductivity was measured in the range from (303.15 to 353.15) K. Our experimental results were compared with available literature data. Also, viscosity of these ionic liquids was measured from room temperature up to 353.15 K. Using the obtained data, the temperature effect on the viscosity was analyzed. Since there are limited literature data on physicochemical properties of ionic liquids, data obtained in this study can provide valuable contributions for better understanding of ILs behavior.



INTRODUCTION

The role of molten salts as solvents is now increasingly being replaced by the largest class of ionic compounds in chemistry, namely ionic liquids (ILs), due to their unique properties such as low melting point, noncorrosivity, and "non-attractivity" for the dissolved substance. On the other hand, the physicochemical properties of ionic liquids are similar to that of conventional molten salts, but the practical aspects related to their application, preservation, and handling are significantly different. Ionic liquids are often classed as green solvents due to their nonvolatility and nontoxicity. In the early publications, ionic liquids were considered as good nonaqueous polar solvents for electrochemical and spectroscopic investigation of transition metal complexes.[1−3] This led to the synthesis of air and moisture stable ionic liquids.[4] Combining the various numbers of cations and anions a large number of ionic liquids of different properties can be obtained. This can provide significant flexibility in the configuration of a specific IL with tailored properties, but also lack and literature discrepancy of some basic physicochemical properties. In this paper, the density, specific conductivity, viscosity and thermal stability of two ionic liquids with the same anion were investigated, [bmim][NTf_2] and [bmpyr][NTf_2].

EXPERIMENTAL SECTION

Chemicals. Ionic liquids [bmim][NTf_2] and [bmpyr][NTf_2] (Merck, 99% mass fraction purity) were used after determination of the water content and purification. The water content in investigated ILs was determined using a 831 Karl Fischer coulometer showing (0.1156 ± 0.0003) % water in [bmim][NTf_2] and (0.05621 ± 0.0002) % in [bmpyr][NTf_2].

In order to purify the samples, the ILs obtained commercially were treated for 15 h at 323.15 K under vacuum. They were then conditioned under a nitrogen atmosphere, which was selected because of its low solubility in the investigated ionic liquids.[5] After drying, the water content was observed to be less than 10^{-4} % in both ionic liquids.

Density. The vibrating tube densimeter, Rudolph Research Analytical DDM 2911, was used for density measurements. The accuracy and precision of the densimeter were ± 0.00001 $g\cdot cm^{-3}$. The instrument was automatically thermostatted (Peltier-type) within ± 0.01 K and was calibrated at atmospheric pressure before each series of measurements. The calibration was performed using ambient air and bidistilled ultra pure water. A Denver Instruments analytical balance weighing up to an accuracy of ± 0.00001 g was used. Each experimental density value is the average of at least three measurements at temperatures from (293.15 to 353.15) K. Repeated experimental measurements showed reproducibility within 0.01 %, and an average value was shown in this paper. The error of measured density was less than 7×10^{-5} $g\cdot cm^{-3}$ for [bmim][NTf_2] and 8×10^{-5} $g\cdot cm^{-3}$ in the case of [bmpyr][NTf_2].

Specific Conductivity. The electrical conductivity measurements were carried out in a Pyrex cell with platinum electrodes in the temperature range (303.15 to 353.15) K on a conductivity meter Jenco 3107. The conductivity cell was thermostatted, and the temperature of the samples was controlled with a laboratory thermostat within ± 0.01 K and

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measured by a digital thermometer with an absolute uncertainty of ± 0.05 K. Temperature and conductivity data acquisitions were made by a personal computer connected to a conductivity meter. The experimental cell was calibrated with standard 0.1000 mol·dm^{-3} KCl solution, and the resulting cell constant, K_{cell}, was 1.0353 cm^{-1}. This value was checked at regular time intervals to control any possible evolution. Specific conductivity, κ, was obtained by multiplying experimental electrical conductivity mean values with the cell constant value. The estimated uncertainty for electrical conductivity was ± 0.5 %.

Viscosity. Measurements were carried out using a Brookfield Viscosimeter DV II+ Pro connected to the thermostat (the temperature was kept constant to ± 0.01 K with an absolute uncertainty of ± 0.05 K) and filled with about 8 cm^3 of tested ionic liquid. The spindle type (S18) was immersed and rate per minute (RPM) was set in order to obtain a suitable torque. To measure the viscosity of [bmim][NTf$_2$] the selected speed was 50 rpm, and in the case of [bmpyr][NTf$_2$] 30 rpm. Workload of the device in the measured temperature range from (298.15 to 353.15) K was from 16.0 % to 83.9 % for [bmim][NTf$_2$] and from 13.2 % to 76.7 % in the case of [bmpyr][NTf$_2$]. The viscosity data at the corresponding temperatures were recorded automatically on a computer connected to the device, and then processed with Origin 8.0 software. The uncertainty in the experimental measurements has been found to be ± 0.01 mPa·s. The exposure of the sample to ambient air during the viscosity measurements was the largest source of error.

Experimental results of density, electrical conductivity and viscosity for both studied ILs at different temperatures are provided as Supporting Information of this paper.

■ RESULTS AND DISCUSSION

Density. The experimental density values obtained for [bmim][NTf$_2$] and [bmpyr][NTf$_2$] in the temperature range from (293.15 to 353.15) K are presented in (Tables 1 and 2) together with recent literature data.[6−17] It can be noted that our experimental values obtained for [bmpyr][NTf$_2$] are in very good agreement with the results from Pereiro et al.[13] The differences between data in literature are due to a different instrumentation, water content and purity of investigated ILs. The density dependence on temperature is a linear function in this temperature range (Figure 1). Equation 1 is used to express this dependence:

Table 1. Experimental and Literature Data of Density (d/g·cm^{-3}) at Different Temperatures (T/K) for [bmim][Tf$_2$N]

T / K	present work	literature
293.15	1.43927	1.43889;[6] 1.44051;[6] 1.4425;[7] 1.44142[8]
298.15	1.43430	1.43410;[6] 1.43573;[6] 1.4377;[7] 1.43664;[8] 1.4366;[9] 1.440;[10] 1.436[11]
303.15	1.42940	1.42931;[6] 1.43094;[6] 1.4329;[7] 1.43186[8]
308.15	1.42457	1.42454;[6] 1.42617;[6] 1.4280;[7] 1.4270[9]
313.15	1.41961	1.41978;[6] 1.42140;[6] 1.4232;[7] 1.42234;[8] 1.4247 (313.45 K)[12]
318.15	1.41467	1.41504;[6] 1.41666;[6] 1.4185;[7] 1.4174[9]
323.15	1.40965	1.41031;[6] 1.41194;[6] 1.4137;[7] 1.41287;[8] 1.4142 (323.35 K)[12]
328.15	1.40464	1.40560;[6] 1.40723;[6] 1.4090;[7] 1.4079[9]
333.15	1.39960	1.40092;[6] 1.40255;[6] 1.4043;[7] 1.40348;[8] 1.4054 (333.75 K)[12]
338.15	1.39443	1.3996[7]
343.15	1.38916	1.3949;[7] 1.39415[8]
348.15	1.38385	1.3903;[7]
353.15	1.37853	1.3856;[7] 1.38488[8]

Table 2. Experimental and Literature Data of Density (d/g·cm^{-3}) at Different Temperatures (T/K) for [bmpyr][Tf$_2$N]

T / K	Present work	Literature
293.15	1.39900	1.39902;[13] 1.38701;[14] 1.4109[15]
298.15	1.39457	1.39459;[13] 1.38202;[14] 1.394;[16] 1.3931[17]
303.15	1.39002	1.39020;[13] 1.37802;[14] 1.3930;[15] 1.390[16]
308.15	1.38548	1.38580;[13] 1.37403;[14] 1.388[16]
313.15	1.38093	1.38142;[13] 1.37003;[14] 1.3816;[15] 1.386[16]
318.15	1.37628	1.37704;[13] 1.36503;[14] 1.383[16]
323.15	1.37164	1.37269;[13] 1.3731;[15] 1.381[16]
328.15	1.36690	1.36835;[13] 1.379[16]
333.15	1.36220	1.36402;[13] 1.3637;[15] 1.377[16]
338.15	1.35735	1.35971;[13] 1.373[16]
343.15	1.35244	1.35541;[13] 1.3550;[15] 1.370[16]
348.15	1.34748	1.366[16]
353.15	1.34248	1.3464[15]

$$d/\mathrm{g{\cdot}cm^{-3}} = a + b(T/\mathrm{K}) \tag{1}$$

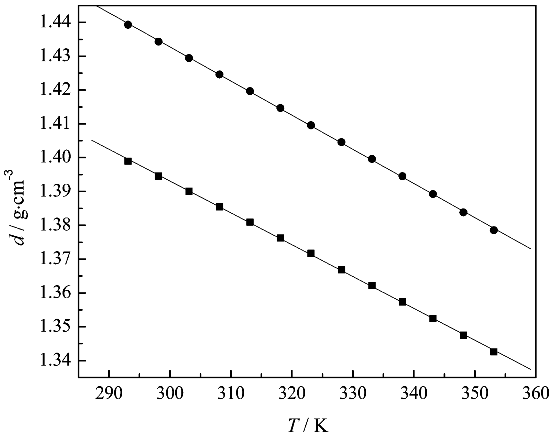

Figure 1. Experimental density dependence on temperature T: ■, [bmim][NTf$_2$]; ●, [bmpyr][NTf$_2$].

The parameters a and b of eq 1 together with the standard deviations (σ) and correlation coefficients (R) are given in Table 3. The deviations were calculated by applying the following expression:

$$\sigma = \left[\sum_{1}^{m} (d_{\mathrm{exp}} - d_{\mathrm{calc}})^2/(p - q)\right]^{1/2} \tag{2}$$

Table 3. Coefficients of eq 1: d/g·cm^{-3} = a + b(T/K) for both ILs

	a / g·cm^{-3}	$-b{\cdot}10^4$ / g·cm^{-3}·K^{-1}	R	$\sigma\cdot10^4$ / g·cm^{-3}
[bmim][NTf$_2$]	1.67531	9.408	0.9998	3.37
[bmpyr][NTf$_2$]	1.73539	10.10	0.9999	3.11

where p denotes the number of experimental points and q is the number of coefficients in eq 1. It can be observed from Figure 1, that with increasing temperature the density decreased. Also, at the same temperature, [bmim][NTf$_2$] has a higher density compared to [bmpyr][NTf$_2$].

The coefficients of thermal expansion, α_P, of [bmim][NTf$_2$] and [bmpyr][NTf$_2$] as a function of temperature at atmospheric pressure are estimated using eq 3 and presented in Table 4

$$\alpha_P = -\frac{1}{d}\left(\frac{\partial d}{\partial T}\right)_{P,m} \tag{3}$$

Table 4. Coefficients of Equation $\alpha_P/K^{-1} = m + n(T/K)$ for both ILs

	$m \cdot 10^4$	$n \cdot 10^7$		$\sigma \cdot 10^7$
	K^{-1}	K^{-2}	R	K^{-1}
[bmpyr][NTf$_2$]	5.1313	4.784	0.9997	2.58
[bmim][NTf$_2$]	5.7322	5.057	0.9996	2.96

The thermal expansion coefficients for both ILs are presented in Figure 2 as a good linear function of temperature.

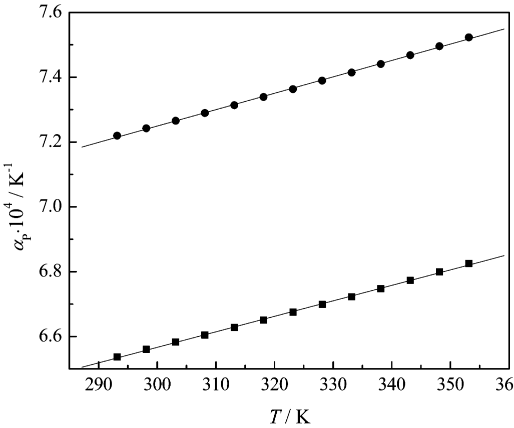

Figure 2. Temperature dependence of thermal expansion coefficients: ●, [bmim][NTf$_2$]; ■, [bmpyr][NTf$_2$].

Higher values of α_P in the case of [bmim][NTf$_2$] indicate weaker interactions in this ionic liquid in comparison with [bmpyr][NTf$_2$].

Specific Conductivity. Literature data for the specific conductivity of ionic liquids are limited and there are some discrepancies between published results for the same ionic liquid. It is expected that ionic liquids have higher values of conductivity than electrolyte solutions and organic solvents. However, in this case values are only comparable with the conductivity of the electrolyte solution and organic solvents and are less than the conductivity of aqueous solutions of electrolytes. Lower conductivity is attributed to the reduced mobility of ion species, as a consequence of the size of cations and anions, stronger ion−ion interactions and extremely high viscosity of these systems. Figure 3 shows the specific conductivity dependence on temperature for the two investigated ionic liquids. It can be seen from Figure 3 that the specific conductivity increases as a function of temperature. It can also be noted that [bmim][NTf$_2$] ionic liquid at the same temperature has a higher conductivity compared to [bmpyr][NTf$_2$]. The higher conductivity of [bmim][NTf$_2$] is a direct consequence of its lower viscosity and will be discussed later.

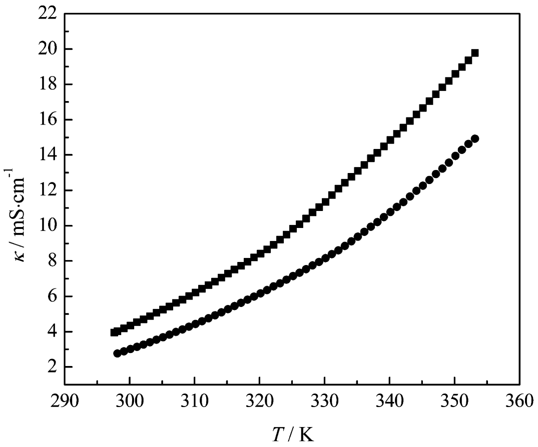

Figure 3. Specific conductivity vs temperature: ■, [bmim][NTf$_2$]; ●, [bmpyr][NTf$_2$].

Comparing our conductivity data for [bmim][NTf$_2$] with available literature values, it can be calculated that the maximum deviation from Wideren et al.[18] is 4.5 % at 298.15 K and 3.7 % at 323.15 K, respectively, whereas in the case of [bmpyr][NTf$_2$], it ranges between 15 and 25 % with the reference data of Zarrougui et al., depending on selected temperature.[14]

The addition of molecular solvent increases the specific conductivity of [bmim][NTf$_2$] and [bmpyr][NTf$_2$] mixed with propylene carbonate (PC) at 303.15 and 353.15 K. In Figure 4 the specific conductivity obtained for the mixtures with different [bmim][NTf$_2$] mole fraction at two temperatures is presented. A maxima of the specific conductivity curve at the mole fraction x(IL) = 0.2 at both temperatures can be observed. This can be explained with the solvation process and dipole-ion interactions in the propylene carbonate rich region, which increase mobility of the ions and thus, specific conductivity in the mixture. The same behavior is observed for [bmpyr][NTf$_2$] binary mixtures with PC at 303.15 and 353.15 K (Figure 5).

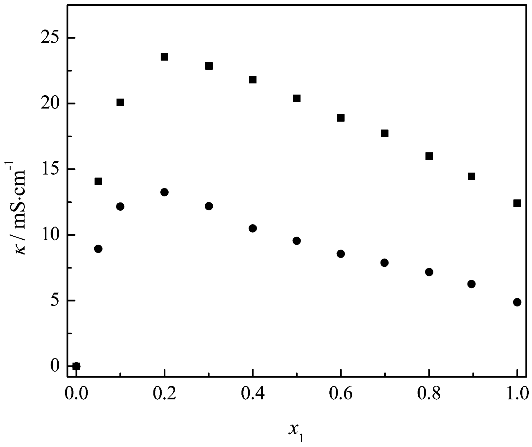

Figure 4. Specific conductivity variation in the mixture {[bmim][NTf$_2$] (1) + PC (2)} at ●, 303.15; ■, 333.15 K.

Viscosity. One of the disadvantages of ionic liquids is their high viscosity. Viscosity was determined for both ionic liquids at (298.15, 303.15, 308.15, 313.15, 318.15, 323.15, 328.15,

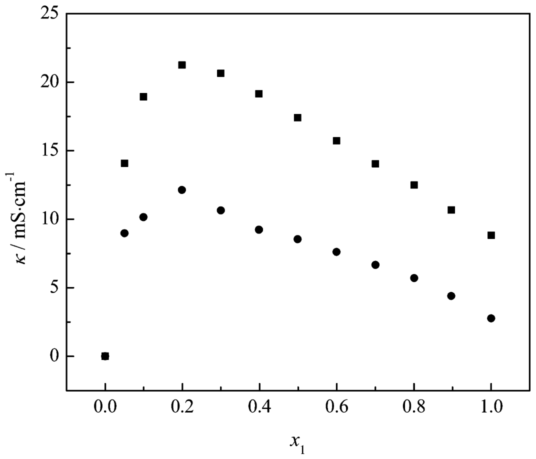

Figure 5. Specific conductivity variation in the mixture {[bmpyr][NTf_2] (1) + PC (2)} at ●, 303.15; ■, 333.15 K.

333.15, 338.15, 343.15, 348.15, and 353.15) K. Both Arrhenius's (4) and Vogel−Tamman−Fulcher (5) equations can be applied to describe the temperature dependence of viscosity of ionic liquids with asymmetric cations

$$\ln \eta = \ln \eta_\infty + E_\eta/RT \tag{4}$$

$$\eta = A \exp(B/(T - T_0)) \tag{5}$$

The correlated parameters determined from the experimental data and the relative deviations of eqs 4 and 5 are presented in Tables 5 and 6.

Table 5. Coefficients of Arrhenius eq 4 $\ln \eta = \ln \eta_\infty + E_\eta/RT$ for Both ILs

	$\ln \eta_\infty \cdot 10^4$	E_η		σ
	mPa·s	kJ·mol^{-1}	R	mPa·s
[bmim][NTf_2]	13.0430	26.049	0.9987	0.028
[bmpyr][NTf_2]	9.6238	27.847	0.9987	0.031

Table 6. Coefficients of Vogel−Tamman−Fulcher eq 5: $\eta = A \exp(B/(T-T_0))$ for Both ILs

	A	B	T_0		σ
	mPa·s	K	K	R	mPa·s
[bmim][NTf_2]	0.203	775.638	167.487	0.99997	0.011
[bmpyr][NTf_2]	0.191	731.645	166.820	0.99996	0.015

The activation energy for viscous flow (E_η) and the viscosity at infinite temperature (η_∞) in eq 4 were calculated from the slope and intercept of the Arrhenius plot, respectively. The activation energy E_η is the barrier which must be overcome, in order for the ions to move and the value of E_η may be correlated with ILs structure. The larger E_η corresponds to a lower ionic activity, due to ion size or stronger ion−ion interactions in ILs. Hence, the ions in the ionic liquid will be the more ordered if parameter E_η is larger. Conversely, the parameter η_∞ is representative of the structural contribution of ions to viscosity. At infinite temperature, the ionic interactions in IL are no longer effective and the viscous flow is governed only by the structure and geometry of the ions.[19]

The parameters of the Vogel−Tamman−Fulcher equation (5) A, B, and T_0 were obtained with a good regression indicated by the correlation coefficient R. The reference temperature T_0 is related to the glass transition temperature, as the charge is transported in the amorphous phase of the system. At this temperature a solid-like material undergoes phase transition to a liquid-like system and can also be ascribed to the ideal vitreous transition temperature, at which segments of the system start to move.[20] Obviously, the dependence of viscosity on temperature can be fitted by both equations with high precision.

Figure 6 shows viscosity variation of both ionic liquids with temperature according to eq 5. It can be seen that the viscosity decreases with an increase in temperature and that [bmim][NTf_2] has an overall lower viscosity. There is an excellent agreement between our experimental results with the viscosity data obtained by Jacquemin et al.[21] and Kenneth et al.[8] for [bmim][NTf_2] and Pereiro et al,[13] for [bmpyr][NTf_2]. Our experimental values differ from the corresponding literature data not more than 1.6 %.

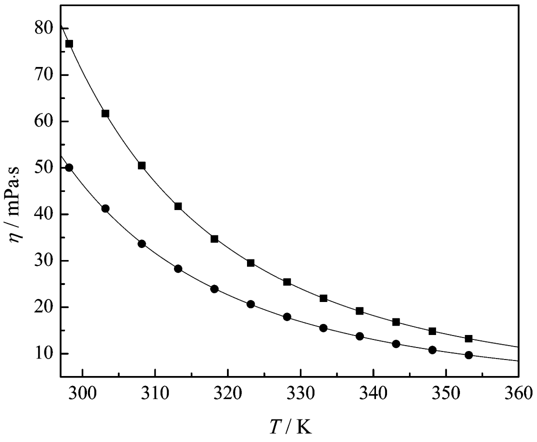

Figure 6. Variation of viscosity with temperature for ●, [bmim][NTf_2]; ■, [bmpyr][NTf_2].

Obtained conductivity and viscosity data were also analyzed using the fractional Walden rule.[22] The molar conductivities calculations were used to draw the Walden plot (Figure 7) and a linear data fitting according to eq 7 where M is the molar mass of selected IL

$$\Lambda = \kappa \frac{M}{d} \tag{6}$$

$$\Lambda \eta^\alpha = \text{const.} \tag{7}$$

Figure 7 shows an excellent linear correlation between neat ILs conductivities and viscosities with an average slope of α = (0.9984 ± 0.0085) for [bmim][NTf_2] and α = (0.9710 ± 0.0075) for [bmpyr][NTf_2]. The obtained fit parameters allow calculation of ILs viscosity from conductivity data which is more obtainable experimentally than viscosity. The deviation of measured viscosities from calculated viscosities obtained from corresponding conductivities is below 1 %, which demonstrates the fractional Walden rule and the Walden plot as quite useful tools for these purposes.

Using our viscosity data, the activation parameters ΔG^*, ΔH^*, and ΔS^* were determined using eq 8 derived by Eyring[23]

$$\eta = \frac{hN_A}{V} \exp\left(\frac{\Delta G^*}{RT}\right) \tag{8}$$

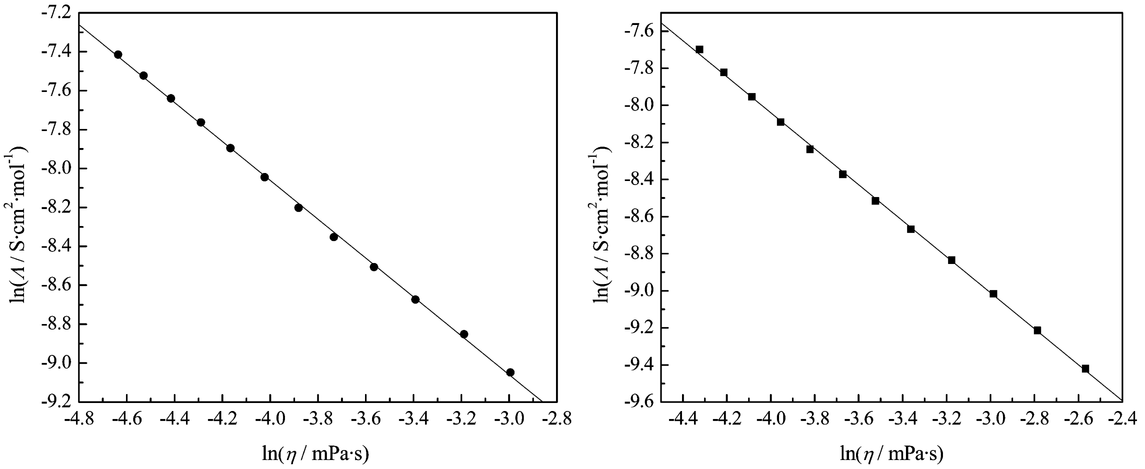

Figure 7. Logarithmic plot of molar conductivity, Λ, dependence on viscosity, η: ●, [bmim][NTf$_2$]; ■, [bmpyr][NTf$_2$].

where η is the viscosity of IL, h, N_A, and V are Planck's constant, Avogadro's number and molar volume, respectively. When $\ln(\eta V/hN_A)$ is plotted against $1/T$, the slope is equal to $\Delta H^*/R$ and the intercept is equal to $-\Delta S^*/R$. Using a least-squares fit method, the activation parameters ΔH^* and ΔS^* were obtained for both ILs and presented in Table 7. Using the equation

$$\Delta G^* = \Delta H^* - T\Delta S^* \tag{9}$$

the molar Gibbs free energy for the activation of viscous flow was calculated and presented in Table 8. Higher values of ΔG^* for [bmpyr][NTf$_2$] indicate stronger interactions in this liquid relative to imidazolium based IL. Since the dependence of $\ln(\eta V/hN_A)$ is a linear function of $1/T$, it can be assumed that ΔH^* is practically an independent function in this temperature interval. Conversely, the negative contribution of ΔS^* in the case of [bmim][NTf$_2$] is possibly the result of synchronization between movements necessary for a successful unit act of flow.[24]

Table 7. Activation Parameters ΔH^* and ΔS^* for Both Investigated ILs

	ΔH^*	ΔS^*
	kJ·mol^{-1}	J·mol^{-1}·K^{-1}
[bmim][NTf$_2$]	25.420	−1.7215
[bmpyr][NTf$_2$]	27.243	0.5969

Table 8. Molar Gibbs Free Energy for Both ILs at Different Temperatures

T	ΔG^*	
	kJ·mol^{-1}	
K	[bmim][NTf$_2$]	[bmpyr][NTf$_2$]
293.15	25.925	27.068
298.15	25.933	27.065
303.15	25.942	27.062
308.15	25.950	27.059
313.15	25.959	27.056
318.15	25.968	27.053
323.15	25.976	27.050
328.15	25.985	27.047
333.15	25.994	27.044
338.15	26.002	27.041
343.15	26.011	27.038
348.15	26.019	27.035
353.15	26.028	27.032

■ CONCLUSION

This paper is a part of our systematic investigation of pure ionic liquids and their mixtures with molecular liquids. Experimental density, specific conductivity and dynamic viscosity data for two ionic liquids with a common anion, [bmim][NTf$_2$] and [bmpyr][NTf$_2$] are presented as a function of the temperature at atmospheric pressure. A comparison of the properties between our measured ILs and other with different origin is described. The densities were correlated as a function of temperature and then the thermal expansion coefficient is easily derived from fitting parameters. Both Arrhenius and Vogel–Tamman–Fulcher equations were used to fit temperature dependence of viscosity with high precision. Experimental conductivity and viscosity of selected ILs were analyzed using the Walden plot. Walden rule in the case of [bmim][NTf$_2$] and [bmpyr][NTf$_2$] allows accurate calculation of their viscosities from the conductivity measurements.

■ ASSOCIATED CONTENT

Ⓢ Supporting Information

Experimental density data for [BMIM][NTf$_2$] and [BMPy]-[NTf$_2$] as a function of temperature (Table S1). Experimental conductivity data and specific conductivity for [BMIM][NTf$_2$] and [BMPy][NTf$_2$] as a function of temperature (Table S2). This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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