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Electrical Conductivity and Viscosity of 1-Hexyl-3-methylimidazolium Bis(trifluorosulfonyl)imide, [C_6mim] [$(CF_3SO_2)_2N$] (CAS-RN# 382150-50-7)

F. J. V. Santos · C. A. Nieto de Castro ·
P. J. F. Mota · A. P. C. Ribeiro

Abstract The electrical conductivity and viscosity of 1-hexyl-3-methylimidazolium bis(trifluorosulfonyl)imide, [C_6mim][$(CF_3SO_2)_2N$], were measured at atmospheric pressure, between 270 K and 350 K, for samples with an amount of water not exceeding 200 ppm, as part of International Union of Pure and Applied Chemistry Project 2002-005-1-100. Water content was monitored before and after measurements, by coulometric Karl–Fisher titration. Special care was taken with ionic liquid manipulation in view of the measurement uncertainty budget. The uncertainties of the electrical conductivity measurements and of the viscosity measurements are estimated to be better than 2.0 % and 0.5 %, respectively. Results were compared with data from other authors, and all data were correlated as a function of temperature.

Keywords 1-Hexyl-3-methylimidazolium bis(trifluorosulfonyl)imide · Electrical conductivity · Ionic liquids · Transport properties · Viscosity

1 Introduction

The impact of ionic liquids in the chemical industry and the development of new materials have triggered a worldwide interest about the physical properties of these low-temperature molten salts (melting points below 100 °C) [1], now-a-days more commonly referred to as room temperature ionic liquids (RTILs). Their use in catal-

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F. J. V. Santos · C. A. Nieto de Castro (✉)
Centro de Ciências Moleculares e Materiais and Departamento de Química e Bioquímica,
Faculdade de Ciências da Universidade de Lisboa, Campo Grande, 1749-016, Lisboa, Portugal
e-mail: cacastro@fc.ul.pt

P. J. F. Mota · A. P. C. Ribeiro
Centro de Ciências Moleculares e Materiais,
Faculdade de Ciências da Universidade de Lisboa, Campo Grande, 1749-016, Lisboa, Portugal

ysis, as both catalyst and catalyst support as well as green solvents, became quite common in the last decade, as they have a wide range of polarities and can dissolve either organic or inorganic materials [2]. The unique properties of RTILs derive from the interactions of the ions, mostly coulombic with very important hydrogen bonding, and from the liquid structures caused by these forces, and the variety of the cations (usually large and asymmetric organic ion) and anions (organic or inorganic) allowing the design of ionic liquids for a given application.

As presented previously, the accurate measurement of the physical properties of these liquids is rather difficult, the available data being scarce and sometimes of questionable accuracy [3]. Furthermore, the increasing number of new RTILs and subsequent need for property data, makes it impossible to measure all the properties of all the liquids necessary for the design of new processes and plants in a reasonable time frame, a fact that triggers two complementary approaches: the establishment of reference materials, internationally accepted, and the development of predictive schemes for the different properties, based on the properties of parent ionic liquids. Furthermore, a recent study on the effect of the uncertainty of the thermophysical properties of ionic liquids in the design parameters of heat transfer equipment showing capital and operational costs can be significant [4].

As part of an International Union of Pure and Applied Chemistry (IUPAC) project [5] we started measurements of the viscosity, electrical conductivity, and electrical permittivity of 1-hexyl-3-methylimidazolium bis(trifluorosulfonyl)imide, [C_6mim][$(CF_3SO_2)_2N$], around room temperature. In this article we report the electrical conductivity, performed with a two parallel-plate conductivity cell and an impedance analyzer, and the viscosity measurements performed with Ostwald glass capillary viscometers. The liquid was received in our laboratory from NIST, as part of a batch synthesized by Dr. Mark Muldoon, in Professor Joan Brennecke's laboratories at the University of Notre Dame, as described previously [6], with a quoted water content of 10 ppm to 20 ppm (mass). Special care was taken in the manipulation of the liquid and in the uncertainty budget of the measurements. Because this liquid is highly hygroscopic, the water content, before and after the property measurements, was determined by coulometric Karl–Fisher titration of aliquots collected from the measuring cells for viscosity and electrical conductivity.

2 Experimental

Electrical conductivity (also referred to as electrolytic conductivity) is a measure of a material's ability to conduct an electric current, usually used as an indicator of the material's purity. The state of the art of its measurement has been described in Refs. [3] and [7]. Measurements of the electrical conductivity of [C_6mim][$(CF_3SO_2)_2N$] have been carefully performed in our laboratory, between 298 K and 343 K, with a two-platinum-electrode commercial cell (Philips PW9510) calibrated with three OIML[1]

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[1] OIML—International Organization for Legal Metrology (http://www.oiml.org/).

conductivity standard KCl solutions of 0.01 D[2], 0.1 D, and 1 D from Radiometer Analytical, France, traceable to NIST, giving a calibration constant $K = 74.765\,\mathrm{m}^{-1}$. The capacitance and conductance were measured as a function of frequency between 20 Hz and 300 kHz using an impedance analyzer (Wayne Kerr Precision Component Analyser 6425), and the f_∞ values of capacitance and conductance C_∞ and G_∞ were obtained by plotting those values for frequencies between 600 Hz and 20 kHz as a function of $f^{-1/2}$ and extrapolating to zero. The capacitance and conductance values for extreme frequency values, upper and lower values, were discarded because some distortion that might be of instrumental origin was detected, and because of polarization phenomena that can occur at the surface of the cell electrodes for low frequencies; only values measured at frequencies greater than 600 Hz were used. To avoid this error, different measuring techniques can be applied, namely, the use of a higher frequency, the use of four-electrode measurements as in platinum resistance thermometry (voltage and current leads), or making inductive or capacitive non-conductive coupling between the electrodes and the measuring instrument [3]. The existing practice established to deal with the more common ions, mostly inorganic and usually small, is suitable to deal with most high-temperature ionic liquids (HTILs), but no real experience exists in dealing with the very large ions present in low-temperature ionic liquids (LTILs). Nevertheless, the large size of the ions in LTILs and the expected low ionic mobilities might allow the use of lower measurement frequencies without the occurrence of polarization effects. High accuracy work will usually involve doing measurements at several frequencies and calculating the conductance from

$$G_\infty \lim_{\frac{1}{\sqrt{f}}\to 0} G(f) \tag{1}$$

The conductivity cell was placed inside a glass vessel, the conductivity vessel shown in Fig. 1, specifically designed to provide both the containment of the sample, its protection from moisture by a constant flow of dry nitrogen over its surface, and the support of the conductivity cell above the stirring bar. Special attention was taken in the design to minimize the amount of required sample volume. This conductivity vessel was mounted over a submersible magnetic stirring device, Rank Brothers Ltd, and the ensemble was afterwards suspended inside the thermostatic bath.

Both the electrical conductivity and the viscosity measurement apparatus use a Julabo V18 viscometer thermostatic bath with a Julabo ME temperature controller (temperature settable to 0.01 °C and stable to 0.005 °C) and an auxiliary thermostatic bath (cold circulation) from B. Braun (Model Thermomix BU + Frigomix B, stable to 0.1 °C). The temperature was measured using a platinum resistance thermometer (four-wire connected) read by a Prema Model 5017 digital multimeter (0.00001 Ω resolution) connected to a personal computer through an IEEE-488 interface. All parts of the temperature measuring system were properly calibrated, ensuring a global uncertainty for temperature of 0.01 K. A data acquisition program follows the thermostatic bath temperature evolution, temperature/time pairs being recorded at roughly 1 s inter-

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[2] D = demal, an old concentration unit for electrical conductivity standard solutions that some manufacturers still use. 1 D = 1 mol/(kg of solution).

Fig. 1 Electrical conductivity vessel with conductivity cell and magnetic stirring bar

vals. Those files are afterwards processed to evaluate the mean temperature value and standard deviation for each viscosity run.

Glass capillary viscometers from Cannon (Ostwald type, C size, #71) were modified (arm length adjustment) to be properly fitted to a ViscoClock unit from Schott–Gerätte to measure flow times to ±0.01s, and were calibrated by a step-up procedure based on water satisfying ISO3696:1995(E) [8] requisites for grade 1, using kinematic viscosity values from ISO/TR3666:1998(E) [9].

The required amount of ionic liquid was carefully drawn from the transport/storage Schlenk vessel using glass syringes, stainless steel needles, and Teflon tubing, always under a protective blanket of dry nitrogen, and introduced either into the viscometer or the conductivity vessel. Operation of the viscometer was also made under a protective blanket of dry nitrogen, fluid movement being made by applying pressure with dry nitrogen. The water content in the ionic liquid was determined by coulometric titration, by the Karl–Fisher method, using a Metrohm 831 KF coulometer. To avoid extensive electrical calibration of the instrument's electrical charge and timely measurements, we have used a standard reference material, Hydranal 100 ppm (mass) Water Standard (NIST SRM 2890 traceable) just before measurements. As the amount of water present in the samples was very small (less than 200 ppm), and the amount of the ionic liquid available was small, the error in the determination of water can be as large as 30 %, depending very much on the amount of sample used. The small amount of available sample imposed severe constraints on the water content determination, so, samples much smaller than recommended by current standards for this technique were used:

typically just one determination with a 1 mL sample was used to measure the water content before property measurements, and 3 to 4 determinations with 1 mL to 1.5 mL were used for the water content determination after property measurements.

Viscosity measurements were made first, and the water content was measured to be (119.3 ± 30) ppm (mass) just after the sample was put into the viscometer and (196.4 ± 3) ppm (mass) after the viscosity measurements were made, two days later. The increased water content was justified because the calibrated viscometers could not be dried in an oven prior to the measurements and the adsorbed water film was minimized just by extended flushing with dry nitrogen. In the case of electrical conductivity measurements, the conductivity vessel was dried in an oven and always kept under dry nitrogen before introduction of the sample. The conductivity cell was flushed with absolute ethanol and dried under a dry nitrogen flow. As a result of these procedures and of the cell size being much smaller, the water content of the sample inside the cell, before and after the measurements (also a two-day interval), was (21.1 ± 3) ppm and (40.7 ± 3) ppm, respectively, much smaller values and much smaller variation than those for the viscosity measurements.

3 Results and Discussion

The kinematic viscosity of $[C_6mim][(CF_3SO_2)_2N]$ was measured using Ostwald glass capillary viscometers to minimize the surface tension effects on viscosity. Measurements were made under conditions (long flow time) that avoid the need to introduce Hagenbach corrections. Dynamic viscosities were calculated using densities obtained from the quadratic equation presented by Widegren et al. [6,10] with an estimated uncertainty better than 0.3 %. Experimental and calculated data are presented in Table 1, along with the corresponding uncertainties. Table 1 also presents the values of the viscosity corrected for "dry" or water-free conditions, η_{wf}, calculated using the procedure described by Widegreen et al. [10], whereby the effect of a given mass fraction of water present, w, could be estimated, assuming that it is temperature independent. Also present is the relative uncertainty of each variable, calculated from the root-mean-square deviations of the different contributions. The total relative uncertainty in viscosity, at a 95 % confidence level ($k = 2$), taking already into account the uncertainty of 0.5 % in density, was calculated using the standard formula:

$$s_X^2 = \sum_{\mathrm{i}} \left(\frac{\partial X}{\partial s_{\mathrm{i}}} \right)^2 s_{\mathrm{i}}^2 \tag{2}$$

with $s_{\mathrm{i}} = 2u_{\mathrm{i}}$, and $X = \eta$ (or κ in Table 3), and estimated to be 2.1 %. We use the function developed by Widegreen et al. [10], given by

$$\eta_{\mathrm{wf}} = \eta(1 + 47w) \tag{3}$$

It can be seen that the maximum correction in the viscosity is 0.56 % at the lowest temperature (first measurements, $w = 119.3$ ppm) and 0.93 % at the highest temperature (last measurements, $w = 196.4$ ppm), an effect about twice the uncertainty of the

Table 1 Viscosity and density of [C_6mim][$(CF_3SO_2)_2$N] for the temperature range of 298 K to 343 K

$T \pm s_T$(K)	$\nu \pm s_\nu$ ($mm^2 \cdot s^{-1}$)	$\rho \pm s_\rho$($kg \cdot m^{-3}$)	$\eta \pm s_\eta$($mPa \cdot s$)	η_{wf}($mPa \cdot s$)
298.149 ± 0.002	50.62 ± 0.02	1371.65 ± 2.74	69.43 ± 0.17	69.82
303.151 ± 0.002	40.39 ± 0.05	1367.17 ± 2.73	55.23 ± 0.18	55.57
313.156 ± 0.002	26.98 ± 0.04	1358.21 ± 2.72	36.64 ± 0.12	36.89
323.149 ± 0.003	18.97 ± 0.01	1349.28 ± 2.70	25.60 ± 0.06	25.79
333.153 ± 0.005	13.92 ± 0.01	1340.35 ± 2.68	18.66 ± 0.05	18.81
343.150 ± 0.005	10.59 ± 0.01	1331.43 ± 2.66	14.10 ± 0.04	14.23

experimental data [7]. This result justifies that, for accurate viscosity measurements, it is necessary to worry about water content in the samples and how they are handled before and during measurements.

Several authors have measured the viscosity of [C_6mim][$(CF_3SO_2)_2$N], namely, Widegren et al. [6], using a Stabinger viscometer (a special type of rotational coaxial cylinder viscometer) with an estimated expanded uncertainty (including the contributions from temperature and density) of 2 % for temperatures above 293 K and 5 % for temperatures below 293 K, Crosthwaite et al. [11] using a cone-and-plate rotational rheometer with an estimated uncertainty of 2 % and temperature uncertainty of 0.25 K, Tokuda et al. [12] used a cone-plate viscometer under nitrogen atmosphere but without any claims of uncertainty or detailed information about measurement errors, and Kandill et al. [13] used a vibrating wire viscometer, a wire diameter of about 0.15 mm, with an estimated uncertainty of 2 %. All the data sets, except that of Tokuda et al. [12], were made on samples of the same batch of [C_6mim][$(CF_3SO_2)_2$N] synthesized by Muldoon [6], taking great care to prevent contamination with water, because it is well established that all physical properties for [C_6mim][$(CF_3SO_2)_2$N] are strongly dependent on the water content [6,10]. Tokuda et al. [12] used an independently synthesized sample, without control of the water content.

In order to correlate the three data sets, a fit was made of as simple a nature as possible using all the available data. An Arrhenius-type correlation was excluded by the observation of a pronounced curvature in the plot, but the inclusion of a quadratic term was demonstrated to be sufficient, as presented in Eq. 1, where $\eta_0 = 1\ \mathrm{mPa \cdot s}$:

$$\ln \frac{\eta}{\eta_0} = \frac{a_2}{T^2} + \frac{a_1}{T} + a_0 \tag{4}$$

Several weighting schemes for the fit were tried but no significant improvements on the correlation were detected, so values presented in Table 2 are the best non-weighted values for coefficients a_i of Eq. 1. Figure 2 presents the deviations of all the data sets from the fitted, non-weighed, correlation described by Eq. 4. The sets identified by Kandil (A) and Kandil (B) are the two samples measured by Kandil et al. [13], sample A with water content between 43 ppm and 410 ppm and sample B between 7 ppm and 117 ppm.

The viscosity values reported here agree with the other four data sets [6,11–14] within ±3 %, especially in the temperature range from 290 K to 370 K. The agreement

Table 2 Best-fit values of coefficients a_i of Eq. 4

a_0	3.2058 ± 0.28
a_1 (K)	$-3.5586 \times 10^3 \pm 1.8 \times 10^2$
a_2 (K^2)	$1.15496 \times 10^6 \pm 2.9 \times 10^4$

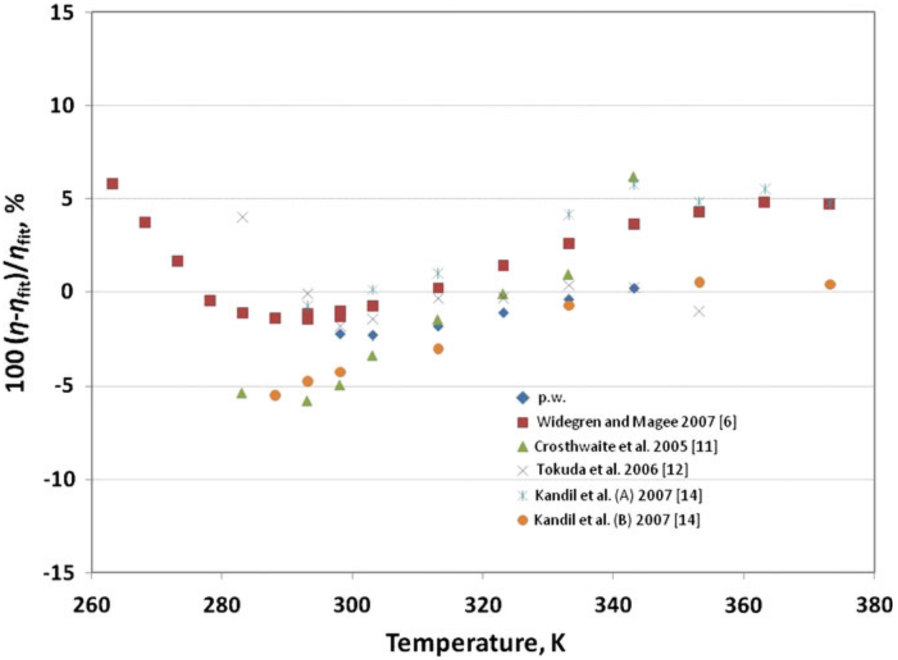

Fig. 2 Deviations of available experimental viscosity values from quadratic modification of Arrhenius fit

is even better (±2 %) in the range from 295 K to 330 K. However, as the amount of absorbed water along the experimental measurements is different in the several data sets, we might expect that the agreement can be better, if the amount of water is taken into account using Eq. 2, not only for our measurements but also with the data from authors that have registered its water content. If we compare our water-free values with those of Widegren et al. [6] (10 ppm), we have an agreement of 0.66 % for Run 1 measurements and 0.38 % for Run 2 measurements, well within the mutual uncertainty of the measurements.

The electrical conductivity of [C_6mim] [$(CF_3SO_2)_2N$] was measured using the smallest voltage that provided the maximum reading resolution of the impedance analyzer, 30 mV, for 17 frequencies between 20 Hz and 300 kHz. For each frequency 10 measurements were made after allowing some time for frequency stabilization of the impedance analyzer internal generator. In order to check the success of the humidity control procedure, measurements were made from 298 K to 333 K and then from 278 K to 298 K; the agreement between the G_∞ values of the two data sets at 298 K was 0.02 % without taking into account the difference in water content between the two data sets.

Table 3 Electrical conductivity of $[C_6mim][(CF_3SO_2)_2N]$ for the temperature range of 298 K to 343 K

$T \pm s_T$(K)	$\kappa \pm s_\kappa$ ($S \cdot m^{-1}$)	κ_{wf} ($S \cdot m^{-1}$)
298.150 ± 0.002	0.2174 ± 0.0043	0.2172
303.150 ± 0.002	0.2682 ± 0.0054	0.2680
308.150 ± 0.002	0.3260 ± 0.0065	0.3257
313.150 ± 0.002	0.3910 ± 0.0078	0.3905
323.150 ± 0.003	0.5430 ± 0.0109	0.5423
333.150 ± 0.005	0.7256 ± 0.0145	0.7246
278.150 ± 0.002	0.0783 ± 0.0016	0.0782
283.150 ± 0.002	0.1045 ± 0.0021	0.1043
288.150 ± 0.002	0.1359 ± 0.0027	0.1357
293.150 ± 0.002	0.1735 ± 0.0035	0.1732
298.150 ± 0.002	0.2176 ± 0.0044	0.2172

The electrical conductivity cell constant was obtained by the same procedure as the G_∞ values for the standard solution of 0.01 D, the one with conductance values of the same magnitude as the fluid under measurements, although the fit was a quadratic one as a function of $f^{-1/2}$.

Experimental and calculated data are presented in Table 3, along with the corresponding uncertainties. Table 3 also presents the values of the electrical conductivity corrected for "dry" or water-free conditions, $\kappa_{\infty \mathrm{wf}}$, calculated using the procedure described by Widegreen et al. [10], assuming a linear increase in water content from the first data point to the last one:

$$\kappa_\infty = \kappa_{\infty \mathrm{wf}} (1 + 46w) \tag{5}$$

The electrical conductivity of $[C_6mim]$ $[(CF_3SO_2)_2N]$ was measured by Widegren et al. [13] and Widegren and Magee [6], using a commercial borosilicate glass conductivity cell with two platinum black electrodes modified to provide insulation from atmospheric moisture by nitrogen blankets at both ends and sealing o-rings. The electrical conductivity was measured by an LCR meter driven at 1 V_{rms} for frequencies between 0.8 kHz and 5 kHz calibrated with commercial standard KCl solutions, with an estimated expanded uncertainty (including the contributions from temperature) of 2 %, the water content being 10 ppm at all times. Kandill et al. [14] used a similar type of conductivity cell also measured with an LCR meter for six frequencies between 0.5 kHz and 10 kHz at 1 V_{rms}, with an estimated uncertainty of 2.0 %, the water content varying from 90 ppm at the beginning to 180 ppm at the end. Fitchett et al. [15] have estimated the electrical conductivity at 295.1 K with an estimated uncertainty of 8 %, and finally Tokuda et al. [12] used a similar type of conductivity cell to the ones previously described to carry out measurements with an AC impedance meter driven at 10 mV_{rms} for frequencies between 5 Hz and 13 MHz calibrated with one standard KCl solution but without any reference to measurement uncertainty, the water content being measured just on the synthesized sample: less than 10 ppm [16]. The 2005 data set of

Table 4 Best-fit values of coefficients a_i of Eq. 6

$a_0 (\mathrm{S \cdot m^{-1}})$	9.3599 ± 0.11
$a_1 (\mathrm{S \cdot m^{-1} \cdot K^{-1}})$	$-0.071208 \pm 7.0 \times 10^{-4}$
$a_2 (\mathrm{S \cdot m^{-1} \cdot K^{-2}})$	$1.3598 \times 10^{-4} \pm 1.1 \times 10^{-6}$

Tokuda et al. [17] available in ILThermo (http://ilthermo.boulder.nist.gov/ILThermo/mainmenu.uix) as values generated from a fit provided by the author was not used, because the experimental data for that fit were published as supporting information of Tokuda et al. [12]. All the data sets, except that of Tokuda et al. [12], were made on samples of the same batch of [C_6mim] [$(CF_3SO_2)_2N$] synthesized by Muldoon [6], taking great care to prevent contamination with water, because it is well established that all physical properties for [C_6mim] [$(CF_3SO_2)_2N$] are strongly dependent on the water content [6,10]. Tokuda et al. [12] used an independently synthesized sample, without control of the water content before and after conductivity measurements.

In order to correlate the available data sets, a fit was made using all data measured for the IUPAC sample [5], of as simple a nature as possible:

$$\kappa = a_2 T^2 + a_1 T + a_0 \tag{6}$$

the coefficients a_i being provided in Table 4, while the residuals of the fit are plotted on Fig. 3, with an RMS deviation of 0.8 %. It is quite obvious from Fig. 3 that all the measurements made using the IUPAC sample are in close agreement within their mutual uncertainties for all temperatures, in a much better case than for viscosity. Although mentioned on the legend of Fig. 3, the data point of Fitchett et al. [15] is not visible because it deviates from the fit by 38 %, while most of the data of Tokuda et al. [12] matches quite well the present fit, except for the lowest temperature data points (263.15 K and 273.15 K) which have deviations of 42 % to 44 % and 6 % to 8 %, respectively, and as such cannot be seen on the graph's scale. There is a noticeable tendency of these two data sets to be generally on the positive side of the residuals, meaning that there is probably more water in the samples than the values measured prior to performing the actual measurements. In spite of these minor problems, there is generally good agreement between all the data sets, IUPAC sample and others, for temperatures between 280 K and 380 K within their mutual uncertainties.

4 Conclusions

The electrical conductivity and viscosity of [C_6mim][$(CF_3SO_2)_2N$] have been measured between 298.15 K and 333.15 K (343.15 K for viscosity), at atmospheric pressures, with uncertainties better than 2.0 % and 0.5 %, respectively. These data were found to agree within their mutual uncertainties, with the data obtained by other authors, especially those that followed the strategy of IUPAC project 2002-005-1-100. From these results we recommend that if we want to obtain results for these properties with a credible uncertainty, it is fundamental to determine, *before* and *after* the measurements, the type and amount of impurities present and in the case of

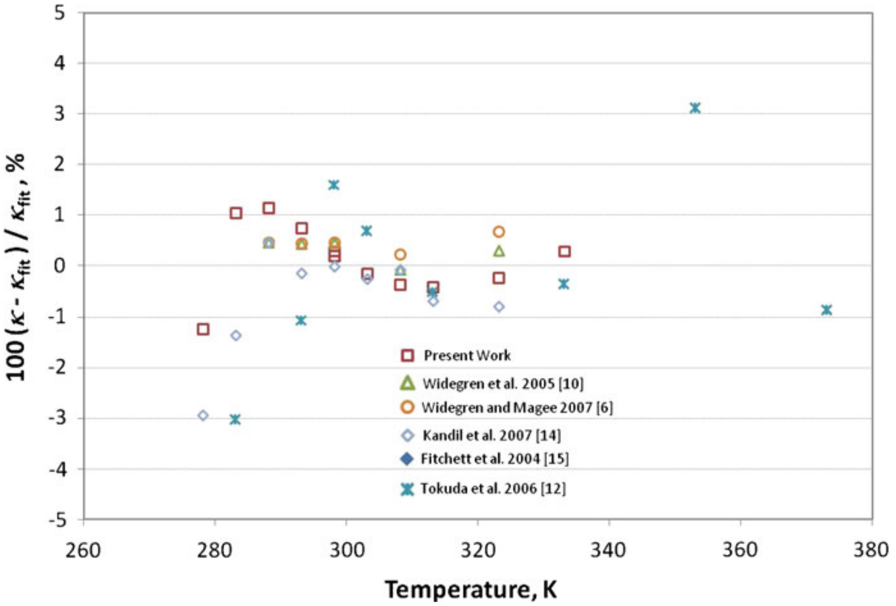

Fig. 3 Deviations of available experimental electrical conductivity values from Eq. 6

a hydrophobic RTIL the amount of water present, making the measurements under properly controlled conditions (under a flush of dry nitrogen in our laboratory).

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