

Viscosities at pressures up to 20 MPa and thermal conductivities at 0.1 MPa of 1-alkyl-3-methylimidazolium bis[(trifluoromethyl)sulfonyl]amides

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The density, viscosity and thermal conductivity values are reported for a series of 1-alkyl-3-methylimidazolium bis[(trifluoromethyl)sulfonyl] amide compounds, where the alkyl group is butyl ([BMIM][NTf₂]), hexyl ([HMIM][NTf₂]), or octyl ([OMIM][NTf₂]). The densities were measured with a vibrating tube densimeter over the temperature range 293–353 K at pressures up to 20 MPa. The estimated combined standard uncertainty was 0.1%. The viscosities were measured using a rolling-ball viscometer over the temperature range 293–353 K, at pressures up to 20 MPa. The combined standard uncertainty in the viscosity was estimated to be 1.6%. The experimental values were correlated with a hybrid Vogel–Fulcher–Tammann/Tait equation. The thermal conductivities were measured using the transient short hot-wire method over the temperature range 294–354 K, at 0.1 MPa. The combined standard uncertainty in the thermal conductivity was estimated to be 2.2%.

Keywords: Density, Ionic liquid, Reference ionic liquid, Thermal conductivity, Viscosity, Transient short hot-wire method, High pressure

1 INTRODUCTION

The bis[(trifluoromethyl)sulfonyl]amide anion is stable in water, and is difficult to hydrolyze, unlike PF₆ and BF₄ anions. Ionic liquids (ILs) containing

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this anion are therefore easy to handle. 1-Hexyl-3-methylimidazolium bis[(trifluoromethyl)sulfonyl]amide ([HMIM][NTf₂]) is used as the reference IL, and the recommended values of its basic physical properties such as density have been determined by the International Union of Pure and Applied Chemistry (IUPAC) [1,2]. However, recommended values of its thermal conductivity are not available.

The thermal conductivity of [HMIM][NTf₂] has been reported by Ge *et al.*, Fröba *et al.*, and Tenney *et al.* [3–5]. The thermal conductivity of 1-butyl-3-methylimidazolium bis[(trifluoromethyl)sulfonyl]amide ([BMIM][NTf₂]) has been reported by Ge *et al.* and Chen *et al.* [3,6]. The thermal conductivity of 1-octyl-3-methylimidazolium bis[(trifluoromethyl)sulfonyl]amide ([OMIM][NTf₂]) has been reported by Ge *et al.* [3].

The density is related to the viscosity, and equations for calculating the density at 0.1 MPa and at high pressures have been proposed. However, although an equation for calculating the viscosity at 0.1 MPa has been proposed, there is no standard equation for calculating viscosities at high pressures. A few groups have measured the viscosities of [BMIM][NTf₂] [7,8] and [HMIM][NTf₂] [9,10] at high pressures, but high-pressure viscosity data have not been reported for [OMIM][NTf₂].

In this study, we measured the densities, viscosities, and thermal conductivities of [BMIM][NTf₂], [HMIM][NTf₂], and [OMIM][NTf₂]. The densities were measured using a vibrating-tube densimeter over the temperature range 293–353 K, at pressures ≤ 20 MPa. The viscosities were measured using a rolling-ball viscometer over the temperature range 293–353 K, at pressures ≤ 20 MPa. The transient short hot-wire method was used to measure the thermal conductivities over the temperature range 294–354 K at 0.1 MPa.

2 EXPERIMENTAL

2.1 Materials

[BMIM][NTf₂] and [OMIM][NTf₂] were prepared using the following procedure. The starting materials were 1-methylimidazole (Sigma-Aldrich, St. Louis, MO, USA, 99%), 1-chlorobutane (Sigma-Aldrich, St. Louis, MO, USA, 99%), 1-chlorooctane (Sigma-Aldrich, St. Louis, MO, USA, 99%), and lithium bis(trifluoromethylsulfonyl)imide (Wako Pure Chemical Industries, Japan, 97%). First, 1-methylimidazole (1.0 mol) was mixed with a small excess of 1-chloroalkane (1.15 mol) at 343 K for more than 72 h under nitrogen. The resulting viscous yellow–brown liquid was washed several times with ethyl acetate, and then the remaining ethyl acetate was removed by heating the solution at 343 K for more than 24 h under vacuum. The chloride anion was exchanged for the bis(trifluoromethylsulfonyl)imide anion as follows: 1-alkyl-3-methylimidazolium chloride was dissolved in ion-exchanged water and lithium bis(trifluoromethylsulfonyl)imide was added. The reaction was conducted

for 72 h at room temperature. The upper aqueous phase was decanted, and the lower IL phase was washed more than 10 times with deionized water. The IL was dried under vacuum at 343 K for 24 h. Activated charcoal was added to the IL and the mixture was stirred at room temperature for 24 h. The activated charcoal was removed by filtration through a neutral alumina column. The IL was heated under vacuum at 343 K for 48 h to remove excess water.

[HMIM][NTf₂] was prepared using the procedure reported by Widegren and Magee [11], which is the IUPAC sample synthesis method.

The product purities were confirmed by CHN elemental analysis. The products were degassed and dried under vacuum for 24 h at 343.15 K before analysis. After analysis, the water content of each IL sample was determined by coulometric Karl-Fischer titration. The water contents of the ILs were lower than 100 ppm. The chloride concentrations were measured using a chloride-selective electrode, and were less than the detection limit (18×10^{-6}). The results are listed in Table 1.

2.2 Measurements

The viscosities were measured using a rolling-ball viscometer; this has been described in detail elsewhere [12,13]. The glass tube was approximately 10 cm in length with an internal diameter of 7.15 mm (± 0.01 mm), and the diameter of the stainless-steel ball was 7.00 ± 0.01 mm. The sample, which was degassed and dried under vacuum for 24 h at 343 K, was introduced into the viscometer. The temperature was measured with a quartz thermometer (DMT-610, Tokyo Denpa, Co., Japan). The pressure was measured using a Bourdon-tube pressure gauge, which was periodically calibrated against a dead-weight gauge. The standard uncertainty in the temperature was 10 mK. The standard uncertainties in the pressure at 0.1 MPa and 5.0 MPa $\leq P \leq 20.0$ MPa were 4 kPa and 0.1 MPa, respectively. The combined standard uncertainties of the reported viscosity values were estimated to be 1.5%. The

TABLE 1
Purities of synthesized ionic liquids

<i>Element</i>	[BMIM][NTf ₂]		[HMIM][NTf ₂]		[OMIM][NTf ₂]	
	<i>Theoretical</i>	<i>Measured</i>	<i>Theoretical</i>	<i>Measured</i>	<i>Theoretical</i>	<i>Measured</i>
	(mass%)		(mass%)		(mass%)	
C	28.64	28.41	32.21	32.06	35.36	35.12
H	3.60	3.39	4.28	4.11	4.88	4.73
N	10.02	9.98	9.39	9.32	8.84	8.62
Water (ppm)	91		84		79	
[Cl ⁻] (ppm)	-*		-*		-*	

*Detection limit <18 ppm.

densities of the samples were determined using a vibrating-tube densimeter (DMA512P, Anton Paar, Graz, Austria); The temperature was measured with a quartz thermometer (DMT-610, Tokyo Denpa, Co., Japan). The pressure was measured using a Bourdon-tube pressure gauge, which was periodically calibrated against a dead-weight gauge. The standard uncertainty in the temperature was 10 mK. The standard uncertainties in the pressure at 0.1 MPa and $5.0 \text{ MPa} \leq P \leq 20.0 \text{ MPa}$ were 4 kPa and 0.1 MPa, respectively. The estimated combined standard uncertainty was 0.1%.

The thermal conductivities were determined using the transient short hot-wire method [14]; this has been described in detail elsewhere [15,16]. A platinum wire of diameter about 50 μm and length about 13 mm was used as the short wire. The temperature had increased by less than 2 K after 1 s. The temperature was measured with a quartz thermometer (DMT-610, Tokyo Denpa, Co., Japan). The standard uncertainty in the temperature was 10 mK. The combined standard uncertainty in the thermal conductivity data was estimated to be within 2.2%.

3 RESULTS AND DISCUSSION

The experimental density and viscosity data for [BMIM][NTf₂], [[HMIM][NTf₂], and [OMIM][NTf₂] are presented in Tables 2, 3, and 4, respectively.

TABLE 2
Density and viscosity of [BMIM][NTf₂]

T (K)	P (MPa)	ρ (kg m ⁻³)	η (mPa s)
293.15	0.1	1441.7	62.0
	5.0	1445.4	66.2
	10.0	1448.6	73.0
	15.0	1451.7	75.8
	20.0	1455.6	80.1
298.15	0.1	1437.1	50.5
313.15	0.1	1423.3	27.8
	5.0	1427.0	29.2
	10.0	1430.0	31.3
	15.0	1433.8	33.3
	20.0	1436.9	34.5
333.15	0.1	1405.1	15.1
	5.0	1409.0	16.0
	10.0	1411.9	16.7
	15.0	1415.7	17.5
	20.0	1419.4	18.5
353.15	0.1	1386.1	9.54
	5.0	1390.0	9.94
	10.0	1393.6	10.4
	15.0	1397.9	10.7
	20.0	1401.6	11.2

TABLE 3
Density and viscosity of [HMIM][NTf₂]

<i>T</i> (K)	<i>P</i> (MPa)	ρ (kg m ⁻³)	η (mPa s)
293.15	0.1	1375.1	89.5
	5.0	1378.7	96.1
	10.0	1382.4	101
	15.0	1386.0	110
	20.0	1388.9	117
298.15	0.1	1370.7	70.2
313.15	0.1	1358.0	37.4
	5.0	1361.6	39.2
	10.0	1365.1	42.2
	15.0	1368.7	43.7
333.15	20.0	1372.3	47.3
	0.1	1340.1	19.2
	5.0	1344.2	20.2
	10.0	1347.6	21.2
	15.0	1351.1	22.0
353.15	20.0	1355.3	22.8
	0.1	1323.4	11.2
	5.0	1327.2	11.7
	10.0	1330.6	12.0
	15.0	1334.0	12.4
	20.0	1337.4	12.9

TABLE 4
Density and viscosity of [OMIM][NTf₂]

<i>T</i> (K)	<i>P</i> (MPa)	ρ (kg m ⁻³)	η (mPa s)
293.15	0.1	1324.4	122
	5.0	1327.9	127
	10.0	1330.7	139
	15.0	1334.1	149
	20.0	1337.6	160
298.15	0.1	1320.4	94.7
313.15	0.1	1307.8	47.7
	5.0	1311.1	51.1
	10.0	1314.4	52.4
	15.0	1317.8	56.6
	20.0	1320.5	59.3
333.15	0.1	1289.9	23.0
	5.0	1293.4	24.1
	10.0	1297.3	25.2
	15.0	1300.6	26.6
	20.0	1304.5	27.8
353.15	0.1	1273.5	13.0
	5.0	1277.4	13.7
	10.0	1280.6	14.3
	15.0	1284.4	14.9
	20.0	1288.2	15.6

Figure 1 shows deviations of the literature density data at 0.1 MPa for [BMIM][NTf₂] [17–20] from a quadratic equation fitted by Kanakubo and Harris [21]. Our results are in good agreement with those values calculated by that equation. Figure 2 shows deviations of the literature density data at 0.1 MPa for [HMIM][NTf₂] [1,10,11,22] from recommended values by IUPAC [2]. Our results are in good agreement with recommended values by

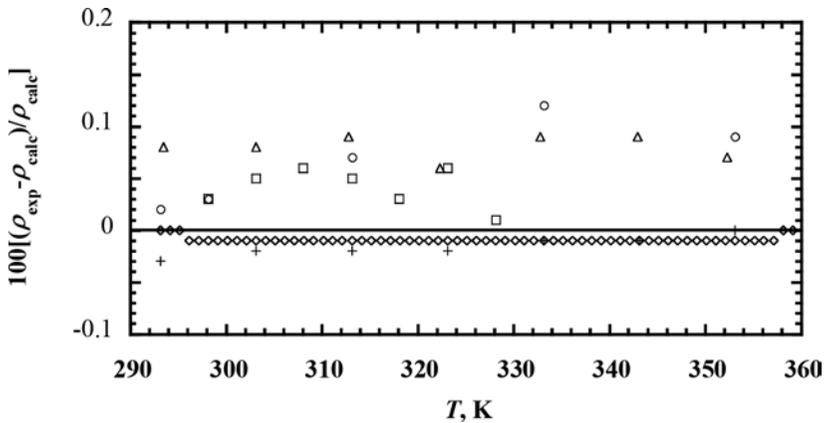


FIGURE 1

Deviations for fit of experimental atmospheric pressure and literature densities for [BMIM][NTf₂] to a quadratic equation fitted by Kanakubo and Harris [21] as function of temperature: ○, this work; □, Azevedo et al. [17]; △, Jacquemin et al. [18]; ◇, Castro et al. [19]; +, Hiraga et al. [20].

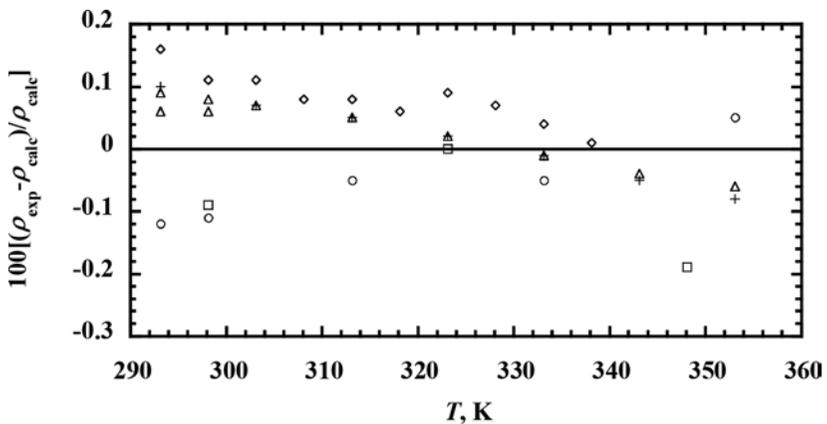


FIGURE 2

Deviations for fit of experimental atmospheric pressure and literature viscosities for [HMIM][NTf₂] to recommended values by IUPAC [2] as function of temperature: ○, this work; □, Kandil et al. [10]; △, Widegren and Magee [11]; ◇, Esperanca et al. [22]; +, Driver and Seddon [1].

IUPAC. Figure 3 shows deviations of the literature density data at 0.1 MPa for [OMIM][NTf₂] [23–26] from a quadratic equation fitted by Jacquemin et al. [18]. Our results are in good agreement with those values calculated by that equation.

The viscosities at 0.1 MPa were correlated with the Vogel–Fulcher–Tammann (VFT) equation (Eq. 1) [27–29].

$$\eta = \eta' \exp[B/(T - T_0)] \quad (1)$$

where η' , B , and T_0 are adjustable parameters. The values of these parameters were determined from the present experimental results using a least-squares method, and are listed in Table 5. The values obtained using the VFT equation and the experimental values for [BMIM][NTf₂], [HMIM][NTf₂], and [OMIM][NTf₂] correlated within $\pm 1.3\%$, $\pm 0.5\%$, and $\pm 0.6\%$, respectively.

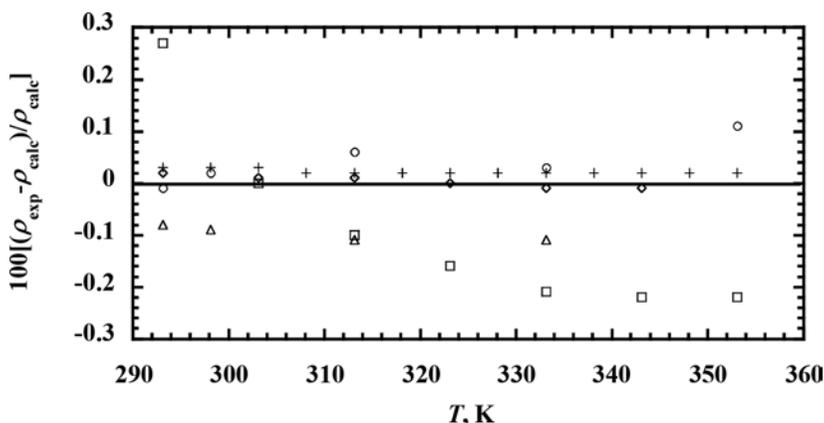


FIGURE 3

Deviations for fit of experimental atmospheric pressure and literature densities for [OMIM][NTf₂] to a quadratic equation fitted by Jacquemin et al. [18] as function of temperature: \circ , this work; \square , Gardas et al. [23]; Δ , Tariq et al. [24]; \diamond , Montalbán et al. [25]; $+$, Santos et al. [26].

TABLE 5
VFT equation parameters

	η' (mPa s)	B (K)	T_0 (K)
[BMIM][NTf ₂]	0.1977	711.5	169.5
[HMIM][NTf ₂]	0.1173	876.8	161.0
[OMIM][NTf ₂]	0.0775	1008.3	156.2

Figure 4 shows deviations of the literature viscosity data at 0.1 MPa for [BMIM][NTf₂] [7,8,20,30–36] from Eq. (1). Our results are in excellent agreement with those reported by Tariq *et al.* [32]. The results reported by Harris *et al.* [7], Atilhan *et al.* [8], Vranes *et al.* [33], Hiraga *et al.* [20], and Malek and Ijardar [34] are in good agreement with our results. The results reported by Jacquemin *et al.* [30] agree with ours, except for one point.

Figure 5 shows deviations of the literature viscosity data at 0.1 MPa for [HMIM][NTf₂] [2,9–11,32,36–44] from Eq. (1). Our results are in excellent agreement with those reported by Widegren and Magee [11], Tariq *et al.* [32], Nazet *et al.* [39], Iguchi *et al.* [41], and IUPAC [2]. The results reported by Seoane *et al.* [40], Diogo *et al.* [42], Santos *et al.* [43], and Calado *et al.* [44] are in good agreement with our results.

Figure 6 shows deviations of the literature viscosity data at 0.1 MPa for [OMIM][NTf₂] from Eq. (1). The results reported by Tariq *et al.* [32] are in good agreement with our results. The results reported by Tokuda *et al.* [36] agree with our results within the experimental uncertainty.

The experimental viscosity data at high pressures were fitted with the Tait equation for viscosity [45], as follows:

$$\ln(\eta_p/\eta_0) = E \ln[(D + P)/(D + 0.1)] \quad (2)$$

where η_p and η_0 are the viscosities at pressure P and 0.1 MPa, respectively; and E and D are adjustable parameters. Table 6 shows the values of the parameters determined from the present data. The E values were constant,

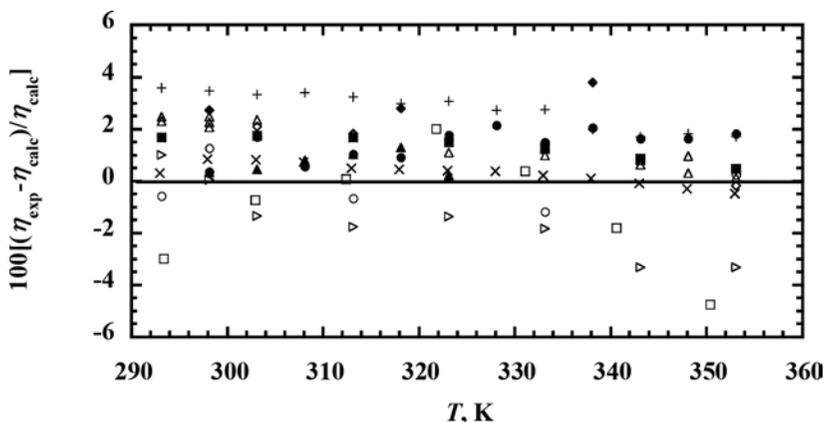


FIGURE 4

Deviations for fit of experimental atmospheric pressure and literature viscosities for [BMIM][NTf₂] to Eq. (1) as function of temperature: ○, this work; □, Jacquemin *et al.* [30]; △, Harris *et al.* [7]; ◇, Atilhan *et al.* [8]; +, Salgado *et al.* [31]; ×, Tariq *et al.* [32]; ●, Vranes *et al.* [33]; ■, Hiraga *et al.* [20]; ▲, Malek and Ijardar [34]; ◆, Salinas *et al.* [35]; and ▷, Tokuda *et al.* [36].

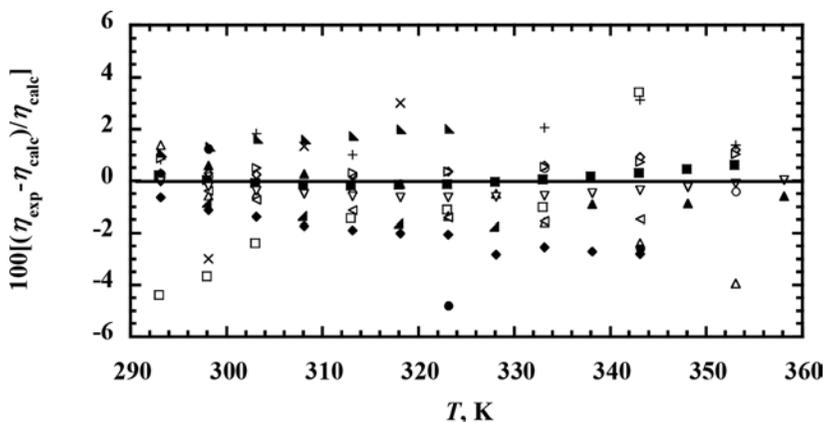


FIGURE 5
 Deviations for fit of experimental atmospheric pressure and literature viscosities for [HMIM][NTf₂] to Eq. (1) as function of temperature: ○, this work; □, Crosthwaite et al. [37]; △, Tokuda et al. [36]; ◇, Widegren and Magee [11]; +, Kandil et al. [10]; ×, Muhammad et al. [38]; ●, Aho-seini and Scurto [9]; ■, Tariq et al. [32]; ▲, Nazet et al. [39]; ◆, Seoane et al. [40]; ▷, Iguchi et al. [41]; ▴, Diogo et al. [42]; ◁, Santos et al. [43]; ▴, Calado et al. [44]; and ▽, IUPAC [2].

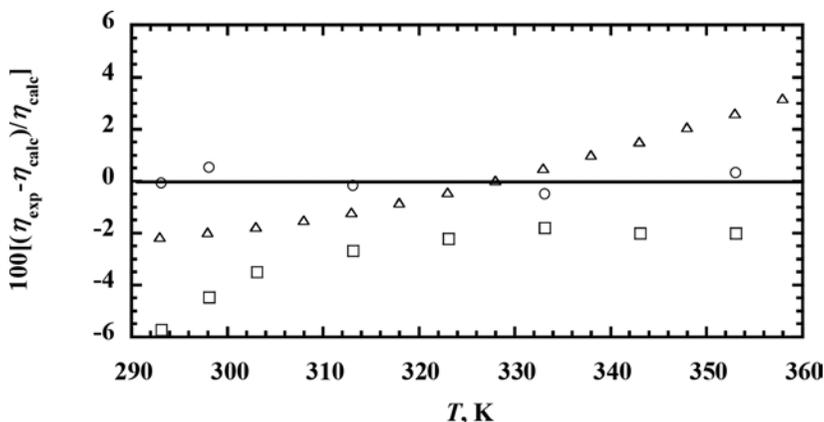


FIGURE 6
 Deviations for fit of experimental atmospheric pressure and literature viscosities for [OMIM][NTf₂] to Eq. (1) as function of temperature: ○, this work; □, Tokuda et al. [36]; and △, Tariq et al. [32].

TABLE 6
 Tait equation parameters

	D (MPa)	E (-)
[BMIM][NTf ₂]	$0.6635T/K - 136.9$	0.862
[HMIM][NTf ₂]	$0.8888T/K - 203.9$	0.898
[OMIM][NTf ₂]	$1.399T/K - 242.8$	2.28

and the D values varied linearly with temperature. If the value calculated using the VFT equation is substituted for η_0 , the viscosity at an arbitrary temperature and pressure can be interpolated. This equation correlated with the experimental values for [BMIM][NTf₂], [HMIM][NTf₂], and [OMIM][NTf₂] with maximum deviations of 2.2%, 2.5%, and 2.7%, respectively.

The viscosities of [BMIM][NTf₂] at high pressure were measured by Harris *et al.* (pressures up to 299 MPa) [7] and Atilhan *et al.* (pressures up to 73 MPa) [8]. Figure 7 shows the deviations of the literature viscosity data at 0.1 MPa for [BMIM][NTf₂] from Eq. (2). The literature values agree with those obtained using Eq. (2) at pressures less than 30 MPa. The results reported by Harris *et al.* [7] and Atilhan *et al.* [8] agree with ours.

The viscosities of [HMIM][NTf₂] at high pressure were measured by Ahosseini *et al.* (pressures up to 124 MPa) [9] and Kandil *et al.* (pressures up to 50 MPa) [10]. Figure 8 shows deviations of the literature viscosity data at 0.1 MPa for [HMIM][NTf₂] from Eq. (2). The literature values agree with those obtained using Eq. (2) at pressures under 20 MPa. The results reported by Ahosseini *et al.* [9] agree with ours. The results reported by Kandil *et al.* [10] also agree with ours, except at 333.15 K above 30 MPa, and at 353.15 K above 30 MPa.

The thermal conductivities of [BMIM][NTf₂], [HMIM][NTf₂], and [OMIM][NTf₂] are shown in Table 7. The effect of the alkyl chain length on the thermal conductivities of the 1-alkyl-3-methylimidazolium hexafluorophosphate series [46] was not significant, but for the 1-alkyl-3-methylimidazolium tetrafluoroborate series [47] and *N*-alkylpyridinium tetrafluoroborate series [48], the thermal conductivities decreased slightly with increasing alkyl chain length. In the 1-alkyl-3-methylimidazolium bis[(trifluoromethyl)

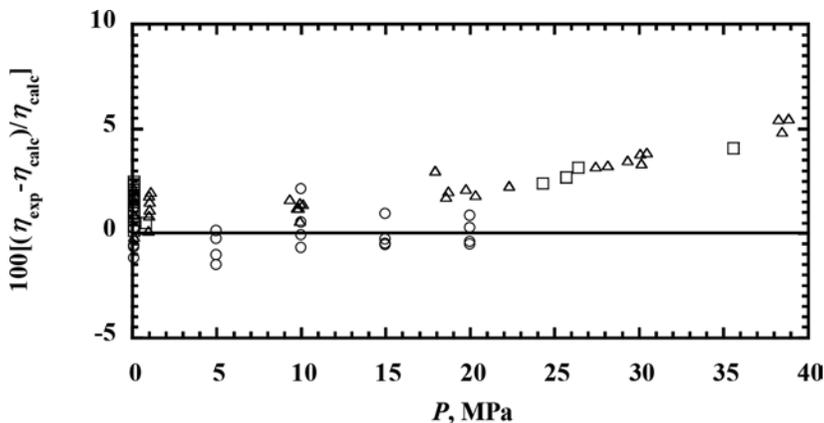


FIGURE 7

Deviations of experimental and literature data for viscosities of [BMIM][NTf₂] at high pressures from Eq. (2): \circ , this work; \square , Harris *et al.* [7]; and Δ , Atilhan *et al.* [8].

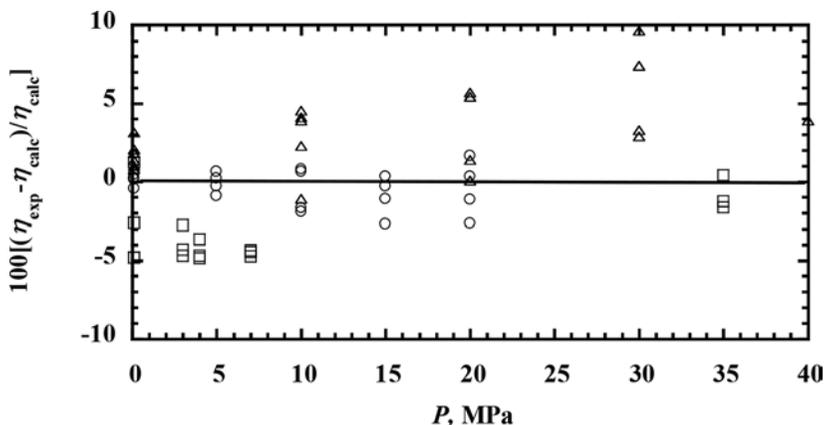


FIGURE 8
 Deviations of experimental and literature data for viscosities of [HMIM][NTf₂] at high pressures from Eq. (2): ○, this work; □, Ahosseini et al. [9]; and Δ, Kandil et al. [10].

TABLE 7
 Thermal conductivities of [BMIM][NTf₂], [HMIM][NTf₂], and [OMIM][NTf₂] at 0.1 MPa

<i>T</i> (K)	λ (W m ⁻¹ K ⁻¹)
[BMIM][NTf ₂]	
294.1	0.122
314.2	0.120
334.4	0.119
354.4	0.118
[HMIM][NTf ₂]	
294.2	0.121
314.3	0.120
334.3	0.120
354.3	0.119
[OMIM][NTf ₂]	
294.1	0.123
314.3	0.123
334.3	0.123
354.4	0.122

sulfonyl]amide series, the thermal conductivity was not significant, similar to the case for the 1-alkyl-3-methylimidazolium hexafluorophosphate series.

The experimental thermal conductivity data at 0.1 MPa can be represented by the linear equation

$$\lambda_0(T) = c_0 + c_1T \tag{3}$$

The coefficients in Eq. (3) are shown in Table 8. The values obtained using this equation correlated with the experimental values for [BMIM][NTf₂], [HMIM][NTf₂], and [OMIM][NTf₂] within $\pm 0.6\%$, $\pm 0.5\%$, and $\pm 0.6\%$, respectively. Figure 9 shows the deviations of the literature thermal conductivity data [3,6] from the data for [BMIM][NTf₂] obtained using Eq. (3). The values obtained in this study were smaller than those reported by Ge *et al.*[3] and Chen *et al.* [6]. Both groups measured the thermal conductivity using a KD2 Pro thermal property meter (Labcell Ltd., UK), using a transient hot-wire method. Ge *et al.* used a thermal probe of diameter 1.3 mm and length 60 mm. Chen *et al.* used a thermal probe of diameter 0.9 mm and length 60 mm. In principle, a thin, long metal wire should be used in the transient hot-wire method, but both groups used a thick, short metal wire. This may have caused the differences between the literature values and our experimental ones.

Figure 10 shows the deviations of the literature thermal conductivity data [3–5] from the data for [HMIM][NTf₂] obtained using Eq. (3). The

TABLE 8
Coefficients in Eq. (3)

c_0 (W m ⁻¹ K ⁻¹)		c_1 (W m ⁻¹ K ⁻²)
0.141	[BMIM][NTf ₂]	-6.47×10^{-5}
0.130	[HMIM][NTf ₂]	-3.00×10^{-5}
0.128	[OMIM][NTf ₂]	-1.49×10^{-5}

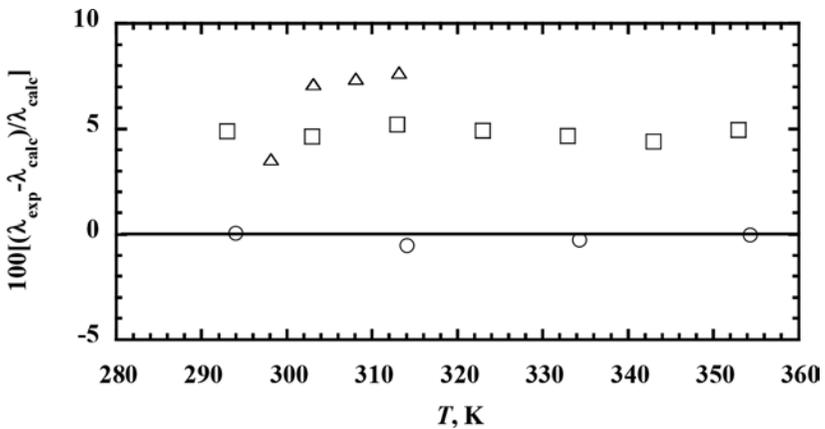


FIGURE 9
Deviations of experimental and literature data for thermal conductivity of [BMIM][NTf₂] at 0.1 MPa from Eq. (3): ○, this work; □, Ge *et al.* [3]; and △, Chen *et al.* [6].

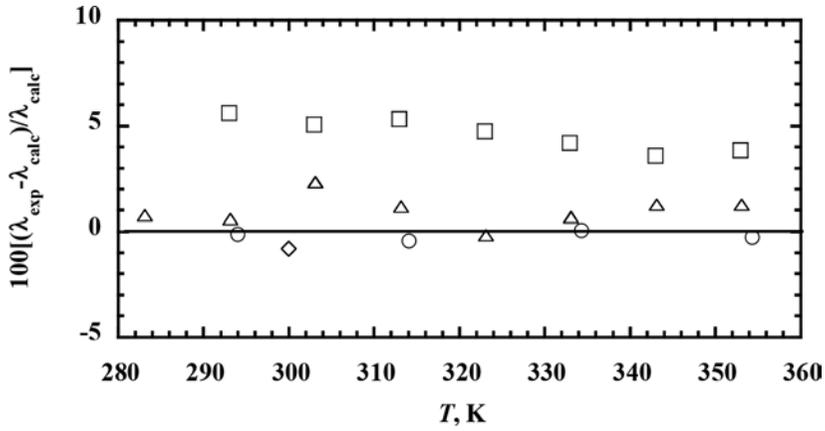


FIGURE 10
 Deviations of experimental and literature data for thermal conductivity of [HMIM][NTf₂] at 0.1 MPa from Eq. (3): ○, this work; □, Ge et al. [3]; △, Fröba et al. [4]; and ◇, Tenney et al. [5].

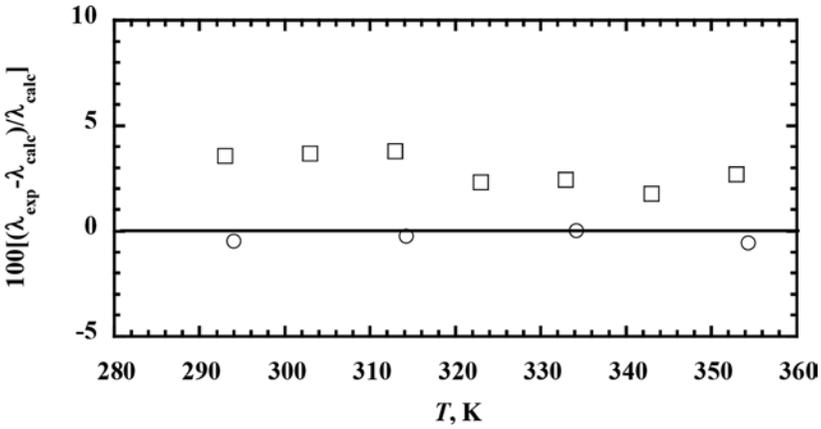


FIGURE 11
 Deviations of experimental and literature data for thermal conductivity of [OMIM][NTf₂] at 0.1 MPa from Eq. (3): ○, this work; and □, Ge et al. [3].

data reported by Fröba et al. [4] and Tenney et al. [5] are in good agreement with our data. However, the values reported by Ge et al. [3] are higher than ours.

Figure 11 shows the deviations of the literature thermal conductivity data [3] from the data for [OMIM][NTf₂] obtained using Eq. (3). The values reported by Ge et al. [3] agree with ours within the experimental uncertainty.

4 CONCLUSION

Viscosity and thermal conductivity values are reported for [BMIM][NTf₂], [HMIM][NTf₂], and [OMIM][NTf₂]. The viscosity was measured using a rolling-ball viscometer between 293.15 and 353.15 K and at pressures up to 20.0 MPa. The experimental values were correlated with a hybrid VFT–Tait equation within $\pm 2.7\%$. The thermal conductivity was determined using the transient short hot-wire method from 294 K to 335 K and 0.1 MPa. For the 1-alkyl-3-methylimidazolium bis[(trifluoromethyl)sulfonyl]amide series, the effect of the alkyl chain length on the thermal conductivity was not significant, similar to the case for the 1-alkyl-3-methylimidazolium hexafluorophosphate series. The thermal conductivities obtained for [BMIM][NTf₂] in this study were smaller than those reported in the literature.

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