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Thermal Conductivities of [bmim][PF_6], [hmim][PF_6], and [omim][PF_6] from 294 to 335 K at Pressures up to 20 MPa

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Abstract Thermal conductivities are reported for a series of 1-alkyl-3-methylimidazolium hexafluorophosphates having butyl, hexyl, and octyl groups, which are expressed by [bmim][PF_6], [hmim][PF_6], and [omim][PF_6], respectively. The experimental method used was a transient short-hot-wire method. Since only a small amount of sample liquid is required, this method was found to be effective for the thermal-conductivity measurements of ionic liquids (ILs). The experimental temperatures ranged from 294 to 335 K at pressures up to 20 MPa. The values of the thermal conductivities of ILs at normal pressure are similar to those of benzene. It was found that an effect of the length of the alkyl chain on the thermal conductivities in ILs is negligible. From the data for the thermal conductivity and viscosity at 293.15 K and 0.1 MPa of ILs and normal alkanes, a simple correlation was developed based on the Mohanty theory. From comparisons between the thermal conductivities of ILs and those of organic liquids (*n*-hexane, benzene, and methanol), the temperature and pressure dependences of the thermal conductivity of ILs are relatively weak.

Keywords 1-butyl-3-methylimidazolium hexafluorophosphate · 1-hexyl-3-methylimidazolium hexafluorophosphate · Ionic liquids · 1-octyl-3-methylimidazolium hexafluorophosphate · Short-hot-wire method · Thermal conductivity

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1 Introduction

Room-temperature ionic liquids (ILs) have recently received recognition as green solvents for synthetic chemistry [1–4]. Since ILs have many unique advantages over traditional organic solvents, researchers have been investigating their properties to develop new environmentally friendly chemical processes [5–8]. One of the most promising characteristics of ILs is their thermal stability at high temperatures. ILs offer a wide liquid range to temperatures as high as 573 K, and thus ILs have the potential to use as heat-transfer fluids. For optimum molecular design of ILs as heat-transfer fluids, thermophysical properties such as density, viscosity, and thermal conductivity are essential. A lack of fundamental thermophysical properties, however, can impede progress in this area. So far, thermophysical properties have been measured by many research groups, who have reported volumetric, thermal, and transport properties [9–19]. Viscosities have been measured for many ILs, however, discrepancies among the published data are significant. Only two reports exist on the thermal conductivity of ILs, with those being for 1-ethyl-3-methylimidazolium tetrafluoroborate ([emim][BF_4]), 1-butyl-3-methylimidazolium tetrafluoroborate ([bmim][BF_4]), and 1,2-dimethyl-3-propylimidazolium bis(trifluorosulfonyl)imide ([dmpi]Im) by Van Valkenburg et al. [18] and for [bmim][BF_4] by our group [19]. As pointed out in our previous study, the thermal conductivity values of [bmim][BF_4] reported by Van Valkenburg et al. are about 8% larger than those of our data. This difference may be due to the experimental method as discussed later. Further experimental studies have been required to explain such differences and perhaps some of this analysis will lead to better techniques for IL measurements.

The transient hot-wire method with long wires is the most promising method to determine the thermal conductivity of liquids. Unfortunately, this method requires a relatively large volume of high-purity liquid samples, which is usually unavailable for ILs due to complicated procedures for their synthesis and purification. Fujii and Zhang [20–22] developed a transient hot-wire method with much shorter wires than the conventional ones. Their method is called the transient short-hot-wire method since the length of the hot wire is usually 10 mm, which is about 10 times shorter than those used for the transient hot-wire method. With their method, the amount of liquid sample can be reduced, which is one of the key reasons for using the method. On the other hand, the length-to-diameter aspect ratio of the hot wire used in this method is not sufficiently large to satisfy the physical model of one-dimensional unsteady heat conduction in the radial direction, and therefore, the method is based on two-dimensional numerical solutions of unsteady heat conduction from a short hot wire with the same length-to-diameter ratio and boundary conditions such as those in the actual experiments.

In this report, we constructed a new experimental apparatus for thermal-conductivity measurements and determined the thermal conductivity of [bmim][PF_6] (1-butyl-3-methylimidazolium hexafluorophosphate), [hmim][PF_6] (1-hexyl-3-methylimidazolium hexafluorophosphate), and [omim][PF_6] (1-octyl-3-methylimidazolium hexafluorophosphate) from 294 to 335 K at pressures up to 20 MPa. We modified the physical model for the numerical simulation of Fijii and Zhang [20] by taking into consideration the heat leak from the hot wire to the lead wire.

2 Experimental

The transient short-hot-wire method was developed from the transient hot-wire technique and was based on two-dimensional numerical solutions of unsteady heat conduction from a short wire with the same length-to-diameter ratio and boundary conditions as those used in the actual hot-wire experiments. While the procedures and measurement system for thermal-conductivity measurements used in this study were almost identical to those reported by Fujii and Zhang [20–22], we developed an experimental apparatus for the thermal-conductivity measurements of liquids under pressures. Since the details of the experimental apparatus and procedures were described in our previous study [19], we show here only essential parts of the apparatus. The thermal conductivity cell used in this study is shown in Fig. 1, which was made from SUS304 and had a volume of about 30 cm^3. The short platinum wire was about 50 μm in diameter and about 10 mm in length. The dimensional values were determined by calibration using water and toluene as a reference fluid. The short platinum wire was welded at both ends to platinum lead wires of 1.5 mm in diameter which were supported with a Teflon circular plate and connected with voltage and current platinum lead wires of 0.5 mm in diameter. The Teflon plate was supported with a stainless-steel rod. The platinum hot wire was annealed at 700 K for 3 h, and the temperature coefficient of its electrical resistance was determined through a calibration for temperatures from 273 to 353 K.

The data acquisition procedure was the same as that of Fujii et al. [20]. The hot wire was heated by supplying a current of 80–200 mA. The current and voltage of the hot wire were measured 50 times per second. These measurements were carried out automatically using a sequential program and GP-IB controlled by a personal computer. A schematic diagram of the experimental apparatus is shown in Fig. 2. The thermal conductivity cell was immersed in an thermostatted oil bath. The pressure was regulated with the use of a hand pump and silicon oil as a pressure transmission fluid. The piston in the bottom part of the cell was used to transmit pressure and to separate the ionic liquid sample from the silicon oil. The temperature of the bath was measured with a quartz thermometer (Tokyo Denpa Model DMT-600B) to within an uncertainty of ±0.01 K and the pressure was measured with a Bourdon pressure gauge calibrated against a dead-weight gauge. The uncertainty of the pressure measurement was estimated to be within ±0.1 MPa. The densities of IL samples were measured with a glass piezometer method reported by Kumagai et al. [23].

Figure 3 shows the measuring system. It was composed of a dc power supply (Advantest, Model R6243; Tokyo), standard resistance (Yokogawa, 2792 Standard Resistor; Tokyo) and dummy resistance (Yokogawa, 2793 Decade Resistance Box; Tokyo), and a voltage and current measuring and control system, which consisted of a digital multimeter (Keithley, 2002 Multimeter, Cleveland), personal computer (PC), and PI/O controller (Advantest, Scanner R7210; Tokyo). The current and voltage of the short hot wire were measured 50 times per second. The measurements were carried out automatically using a sequential program and a GP-IB interface controlled by a PC.

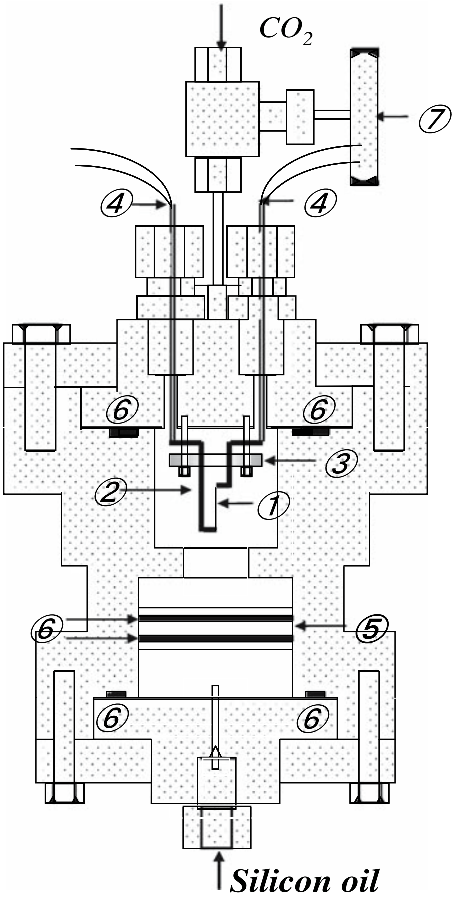

Fig. 1 Short-hot-wire cell: ① Platinum wire (50 μm ϕ), ② Platinum lead terminal (1.5 mm ϕ), ③ Teflon plate, ④ Voltage and current terminal (0.5 mm ϕ), ⑤ Piston, ⑥ o-ring, ⑦ Valve

3 Numerical Analysis

The numerical analysis was performed based on the model proposed by Fujii and Zhang [20]. In the Fujii–Zhang model, the following three assumptions were adopted: (a) the heat generation rate per unit volume and time is uniform and constant; (b) the temperature of the lead wires is kept at its initial temperature during the heating process; and (c) the temperature distribution in the vessel is symmetric. In this study, we applied assumptions (a) and (c), but abandoned assumption (b) and tried to simulate the heat conduction from the hot wire to the lead wire. So we included the lead wire in the physical model as shown in Fig. 4. The liquid sample was filled into a cylindrical vessel of radius r_0, and a short hot wire of radius r_i and length $2l$ was located on the center axis and was supported by a lead wire at each end. The hatched region in Fig. 4 is taken as the solution domain. The two-dimensional heat-conduction equations for

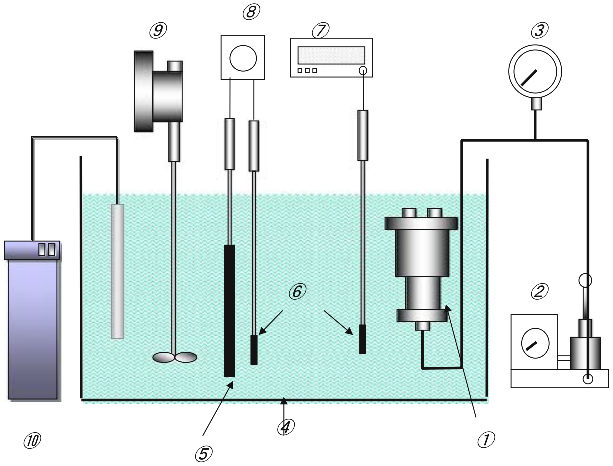

Fig. 2 Schematic diagram of experimental apparatus: ①Short hot-wire cell, ②Pressure pump, ③Pressure gauge, ④Oil bath, ⑤Heater, ⑥Thermocouple, ⑦Quartz thermometer, ⑧Temperature controller, ⑨Stirrer, ⑩ Cooler

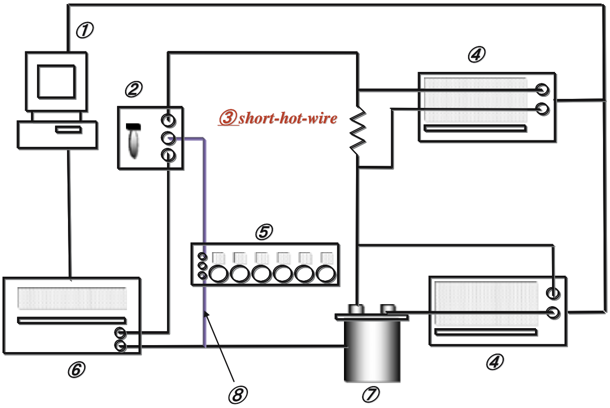

Fig. 3 Schematic diagram of measuring system: ①Personal computer, ②Relay switch, ③Short hot wire, ④Digital multimeter, ⑤Dummy resistance, ⑥DC power supply, ⑦Standard resistance, ⑧Dummy circuit

the hot wire, liquid layer, and lead wire were expressed as follows:

$$\frac{\partial \theta_s}{\partial Fo} = \frac{1}{C}\left(\frac{\partial^2 \theta_s}{\partial R^2} + \frac{1}{R}\frac{\partial \theta_s}{\partial R} + \frac{\partial^2 \theta_s}{\partial Z^2}\right) + \frac{D}{C} \quad \text{(hot wire)} \tag{1}$$

$$\frac{\partial \theta_f}{\partial Fo} = \frac{\partial^2 \theta_f}{\partial R^2} + \frac{1}{R}\frac{\partial \theta_f}{\partial R} + \frac{\partial^2 \theta_f}{\partial Z^2} \qquad \text{(liquid layer)} \tag{2}$$

$$\frac{\partial \theta_l}{\partial Fo} = \frac{1}{C}\left(\frac{\partial^2 \theta_l}{\partial R^2} + \frac{1}{R}\frac{\partial \theta_l}{\partial R} + \frac{\partial^2 \theta_l}{\partial Z^2}\right) \quad \text{(lead wire)} \tag{3}$$

where θ, R, and Z are the dimensionless temperature, radial and longitudinal coordinates, respectively, and subscripts s, f, and l refer to the hot wire, liquid layer, and lead wire, respectively. θ, R, Z, and the Fourier number Fo are defined as follows:

$$\theta = \lambda \frac{T - T_0}{q_v r_i^2} \tag{4}$$

$$Fo = \frac{\alpha t}{r_i^2} \tag{5}$$

$$R = \frac{r}{r_i} \tag{6}$$

$$Z = \frac{z}{r_i} \tag{7}$$

where q_v is the heat flux per unit volume of the short hot wire, t is the heating time, and T and T_0 are the temperatures at time t and the initial temperature, respectively. C and D are the ratios of the thermal diffusivity α and thermal conductivity λ, respectively, to those of the wire as defined by

$$C = \frac{\alpha}{\alpha_s}, \quad D = \frac{\lambda}{\lambda_s} \tag{8}$$

where subscripts s indicates the short hot wire. Equations (2)–(4) were solved numerically by a finite difference method with an implicit subsequent substitution scheme. The details for the numerical analysis of these equations were described in our previous study [19].

4 Determination of Thermal Conductivity

From the measurements with the short hot wire, the measured temperature rise of the wire can be approximated by the following equation:

$$T_v = a \ln t + b \tag{9}$$

where T_v is the volume-averaged wire temperature rise based on T_0, and a and b are coefficients. The values of a and b were determined by least squares from a plot of $\ln t$ vs. T_v.

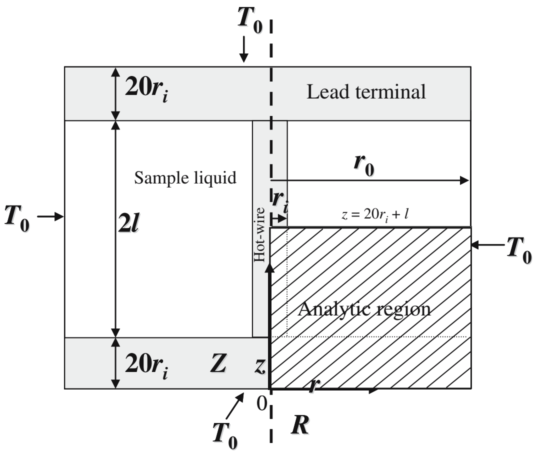

Fig. 4 Physical model and coordinate system for simulation

From the numerical analysis, the relationship between the dimensionless temperature of the wire and the Fourier number was obtained. Generally, the dimensionless temperature increased linearly from $Fo = 5$ to 130 for the three ILs studied in this work, with the real time corresponding to times from 0.04 to 1 s. The numerical results in this time range were approximated by the following equation:

$$\theta_\mathrm{v} = A \ln Fo + B \tag{10}$$

where A and B are coefficients determined by the least-squares method.

Comparing the corresponding coefficients of Eqs. (9) and (10), the thermal conductivity and thermal diffusivity are expressed as follows:

$$\lambda = q_\mathrm{v} r^2 \frac{A}{a} = \frac{VI}{2\pi l}\frac{A}{a} \tag{11}$$

$$\alpha = r^2 \exp\left(\frac{b}{a} - \frac{B}{A}\right) \tag{12}$$

where V and I are the voltage and current supplied to the wire and l and r are the length and diameter of the wire. The values of l and r were determined from calibration with the reference liquids. In this study, we used water and toluene as the reference liquids, and experimental data were quoted from Ramires et al. [24] and Watanabe and Seong [25], respectively. The values of A and B changed in relation to the aspect ratio and the thermal conductivity and thermal diffusivity of the liquid sample. Therefore, an iterative process was required to evaluate the thermal conductivity and thermal diffusivity. The initial values of the thermal conductivity and thermal diffusivity for the numerical analysis were determined from the equations used in the transient hot-wire method and were generally about 10% larger than those of the converged values. Usually 3–5 iterations were required to obtain convergence. Since reproducibility of the determination of values of a and A in Eqs. (9) and (10) from experiments and simulations was very good, the values of the thermal conductivity should be consistent. On the other hand, both b and B were very sensitive to the initial few experimental data points. So reliability of the values of the thermal diffusivity are probably not so good compared with the thermal-conductivity values. The uncertainty of the present thermal-conductivity data is estimated to be within 2.0%.

5 Synthesis of Ionic Liquids

[bmim][PF_6], [hmim][PF_6], and [omim][PF_6] were synthesized according to literature procedures [26,27]. The starting materials were 1-methylimidazorium, heafluorophosphoric acid, and 1-chlorobutane, 1-chlorohexane, or 1-chlorooctane. All chemicals were reagent grade and purchased from Aldrich Chemical Co. First, 1-alkyl-3-methylimidazolium chloride was prepared by mixing equal molar amounts of 1-methylimdazole and 1-chloroalkane at 343 K for more than 72 h. The resulting viscous liquid was washed several times with ethyl acetate, and then the remaining ethyl acetate was removed by heating to 343 K under vacuum. To exchange the chloride anion for the hexafluorophosphate anion, a stirred mixture of 1-alkyl-3-methylimidazolium chloride and water was cooled to 273 K, and heafluorophosphoric acid was added very slowly. After stirring the biphasic mixture for 24 h, the upper aqueous phase was decanted, and the lower ionic liquid phase was washed more than 10 times with distilled and de-ionized water. The ionic liquid was dried under vacuum at 343 K for 24 h.

Activated charcoal was added to the ionic liquid and stirred at room temperature for 24 h. Then, activated charcoal was removed by filtration through a neutral alumina column. The ionic liquid was heated under vacuum at 343 K for 48 h to remove any excess water. It was confirmed that the residual chloride was very minor in any of the ionic liquids by the elemental titration analysis. The water content in the ionic liquids was checked by a Karl–Fisher analysis after measurement and was always within a range of 70–90 ppm.

6 Results and Discussion

First, the characteristics of the short-hot-wire probe were examined using water and toluene as reference liquids [24,25]. The temperature trends for these liquids were compared with corresponding numerical results, and the evaluated thermal conductivities were compared with reference values. Then, the effective hot-wire length and diameter, and the resistance ratio of the lead terminals and the entire probe were determined. This calibration was performed at 294.2 K and 0.1 MPa.

The accuracy and reliability of our thermal conductivity measurements based on toluene and n-octane are shown in Table 1. For toluene, the present results agreed well with those of Nagasaka and Nagashima [28], Kashiwagi et al. [29], Assael et al. [30], and Watanabe and Seong [25], while the data of Ogiwara et al. [31] and Shulga et al. [32] deviated from the present results by about 2–4%. For n-octane, the present results agreed well with those of Watanabe and Seong [25].

Table 1 Experimental results for toluene and *n*-octane

T (K)	P (MPa)	λ ($\mathrm{W \cdot m^{-1} \cdot K^{-1}}$)
Toluene		
294.2	0.1	0.132
294.2	10	0.136
294.2	20	0.139
314.1	0.1	0.127
314.1	10	0.131
314.1	20	0.135
334.4	0.1	0.122
334.4	10	0.126
334.4	20	0.129
n-Octane		
294.5	0.1	0.126
314.7	0.1	0.121
334.8	0.1	0.115

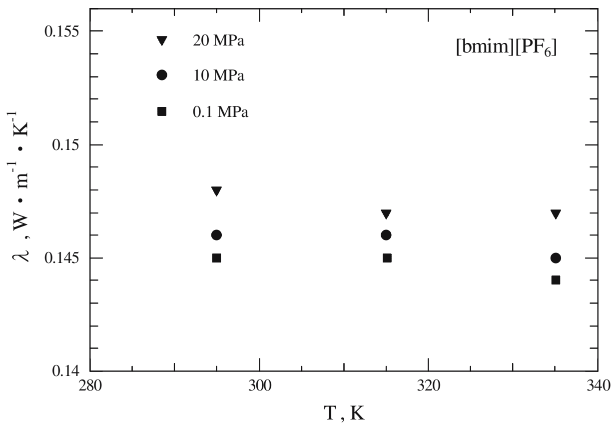

Fig. 5 Thermal conductivity of [bmim][PF_6]

Since ILs are highly electrically conducting liquids, one should consider the effect of current leakage. Based on the Turnbull equation [33], we estimated the leakage current through the ILs which was found to be negligible for the present experimental conditions. Numerical analysis was performed based on the estimated leakage current, but it was found that they had negligible influence on the thermal conductivity. We also checked the effect of leakage current based on the graphical analysis proposed by Takeuchi et al. [34]. Since the ratio of the diameter of the hot wire and its length was in the range of 2.4×10^{-3} to 3.6×10^{-3} and this ratio was much larger than that for the standard transient hot-wire method, it was found that the effect of leakage current was negligible.

The experimental thermal conductivities of [bmim][PF_6], [hmim][PF_6], and [omim][PF_6] are listed in Table 2 and shown in Figs. 5–7. The measured densities of the ILs are also presented in Table 2. The values of the thermal conductivity of these ILs at 294 K and at 0.1 MPa are close to those of benzene (0.1424

Table 2 Experimental thermal conductivities for ILs

T (K)	P (MPa)	ρ(kg.m^{-3})	λ (W · m^{-1}· K^{-1})
	[bmim][PF$_6$]		
294.9	0.1	1369	0.145
294.9	10	1373	0.146
294.9	20	1378	0.148
315.0	0.1	1352	0.145
315.0	10	1357	0.146
315.0	20	1362	0.147
335.1	0.1	1336	0.144
335.1	10	1341	0.145
335.1	20	1347	0.147
	[hmim]PF$_6$]		
294.1	0.1	1294	0.145
294.1	10	1300	0.147
294.1	20	1306	0.148
315.1	0.1	1278	0.145
315.1	10	1284	0.146
315.1	20	1290	0.148
335.2	0.1	1262	0.144
335.2	10	1268	0.146
335.2	20	1274	0.147
	[omim][PF$_6$]		
295.1	0.1	1236	0.145
295.1	10	1243	0.147
295.1	20	1249	0.147
315.1	0.1	1222	0.145
315.1	10	1228	0.146
315.1	20	1234	0.147
335.2	0.1	1207	0.144
335.2	10	1213	0.146
335.2	20	1219	0.147

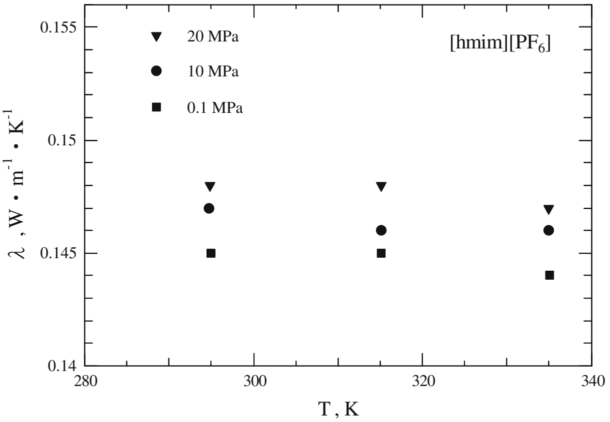

Fig. 6 Thermal conductivity of [hmim][PF$_6$]

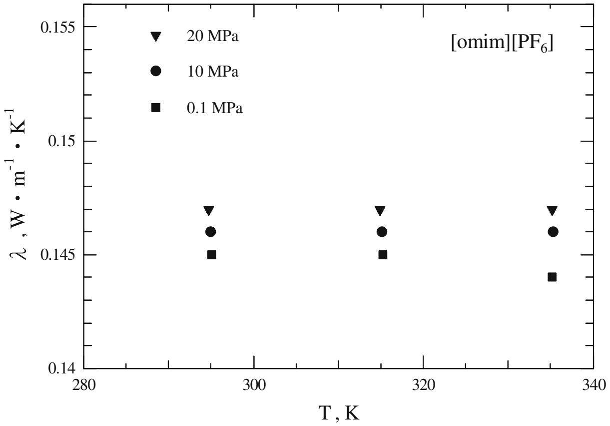

Fig. 7 Thermal conductivity of [omim][PF$_6$]

$W \cdot m^{-1} \cdot K^{-1}$ at 293.15 K and 0.1 MPa). As shown in Table 2, the values of the thermal conductivity of the three ILs were almost the same. The temperature and pressure dependences of the thermal conductivity were small. The order of the thermal conductivity of the three ILs at the same temperature and pressure conditions with respect to the chemical structure was not clear, which means that the effect of the alkyl chain length on the thermal conductivity of the ILs is not significant. This conclusion is different from that obtained for the thermal conductivity of n-alkanes [25] and for the viscosity of ILs [11].

Based on the Andrade theory for liquid viscosity and the Bridgeman theory for molecular collisions, Mohanty [35] proposed a very simple relationship between thermal conductivity and viscosity as follows:

$$M\lambda/\eta = \text{const.} \tag{13}$$

where M is the molar mass, λ is the thermal conductivity, and η is the viscosity. Figure 8 shows a plot of $M\lambda/\eta$ vs. M for the three ILs measured in this study and n-alkanes, from n-pentane to n-tetradecane, at 293.15 K and 0.1 MPa. It can be seen that Eq. (13) does not hold for ILs and n-alkanes and $M\lambda/\eta$ decreased with increasing M. Moreover, the relationship between $M\lambda/\eta$ and M for the n-alkanes was different from that for the ILs, which may be due to the relatively higher viscosity values of ILs compared with the n-alkanes having similar molar masses. (For example, at 293.15 K, λ=0.145 $W \cdot m^{-1} \cdot K^{-1}$ and η=382 mPa· s for [bmim][PF$_6$], while λ=0.146 $W \cdot m^{-1} \cdot K^{-1}$ and $\eta = 2.32$ mPa· s for n-tetradecane.) Figure 9 shows the results obtained by assuming that the molar mass of the IL is two times larger than the true value. For this case, the plots of the n-alkanes and ILs could be correlated. From least-squares fitting, the following equation was obtained:

$$\log(M\lambda/\eta) = 1.9596 - 0.004499M \tag{14}$$

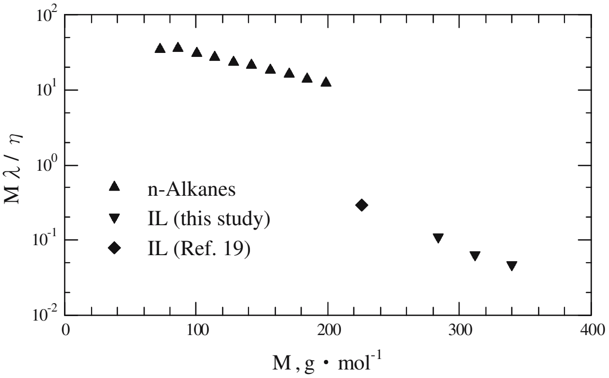

Fig. 8 $M\lambda/\eta$ vs. M plot for n-alkanes and ionic liquids

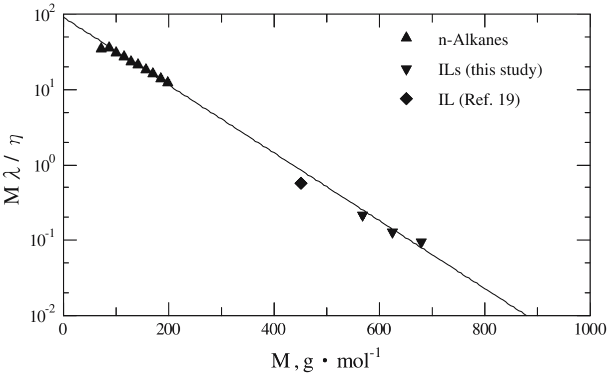

Fig. 9 $M\lambda/\eta$ vs. M plot for n-alkanes and ionic liquids under the assumption that M of the ionic liquid is twice its true value

From this plot, the thermal conductivity of ILs at 293.15 K and 0.1 MPa can be estimated from the viscosity and molar mass. It should be pointed out that Eq. (14) is not expected to give quantitative results. To develop more accurate correlations, more experimental data for the thermal conductivity for different classes of ILs are needed.

Figure 10 shows the variation of the thermal conductivity of [bmim][PF_6] with pressure. Similar trends were obtained for [hmim][PF_6] and [omim][PF_6]. For comparison, the pressure dependence of n-hexane [36], methanol [36], and benzene [36] are also plotted on the same figure. It was found that the density dependence of ILs was smaller than those for organic materials. This result may be due to the fact that ILs have much smaller compressibilities than organic liquids.

7 Conclusion

Based on the transient short-hot-wire method proposed by Fujii and Zhang, we developed a new experimental apparatus for measurement of the thermal conductivity of liquids under pressure. We also developed a mathematical model considering heat

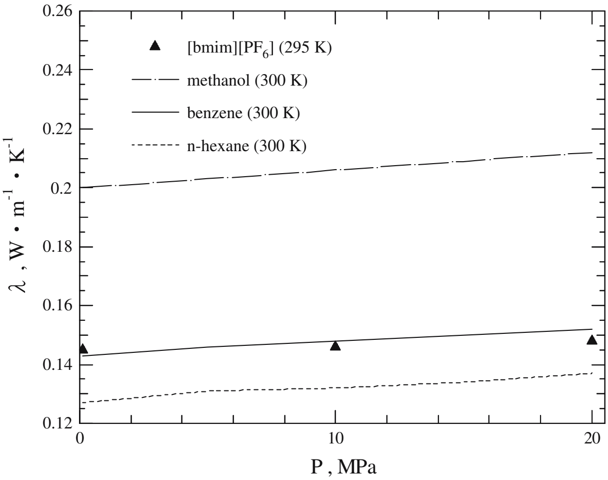

Fig. 10 Comparison of pressure dependences of the thermal conductivity for [bmim][PF_6], n-hexane, benzene, and methanol

transfer from the short hot wire to the lead wire. With the use of the experimental apparatus and the numerical model, we determined the thermal conductivities of [bmim][PF_6] (1-butyl-3-methylimidazolium hexafluorophosphate), [hmim][PF_6] (1-hexyl-3-methylimidazolium hexafluorophosphate), and [omim][PF_6] (1-octyl-3-methylimidazolium hexafluorophosphate) in the temperature range from 294 to 335 K and at pressures up to 20 MPa. The values of thermal conductivities of these three ILs were almost the same at the present experimental conditions. The effect of the alkyl side chain length on the thermal conductivity was not significant, which is different from that observed for the thermal conductivity of normal paraffins and also for the viscosities of ILs. The thermal conductivity values of the three ILs at 294 K and at 0.1 MPa were close to that of benzene. The experimental results at normal pressure were analyzed with Mohanty's theory. The simple relation between $M\lambda/\eta$ vs. M that has been used for the n-alkanes from n-pentane to n-tetradecane and the four ILs (the three ILs measured in this study and one IL in our previous study) can be obtained if the M is replaced with $2M$ for the ILs only. The temperature and density dependences of the thermal conductivities of ILs were smaller than those of organic compounds such as benzene, methanol, and n-hexane.

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