Viscosity of Molten Rare Earth Metal Trichlorides
II. Cerium Subgroup

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The kinematic viscosity of molten $LaCl_3$, $CeCl_3$, $PrCl_3$, $NdCl_3$, and $SmCl_3$ was measured directly by using a capillary viscometer made of quartz and a transparent electric furnace. Viscosity of molten $PmCl_3$ and $EuCl_3$ was estimated using common tendencies in the lanthanides row. The dynamic viscosity was computed by using the most reliable density data taken from literature. The viscosity of molten lanthanides trichlorides at the same temperature changes slightly within a cerium lanthanides subgroup. Molten $EuCl_3$ perhaps has abnormally low viscosity.

Key words: Viscosity; Molten Salts; Rare Earths; Chlorides.

1. Introduction

The viscosity of molten rare earth chlorides ($LnCl_3$) and their binary mixtures with alkali chlorides are poorly known to date. The data available in literature are very fragmentary and contradictory [1].

Viscosity is a very important property of liquids both from practical and theoretical points of view. The use of molten chloride salts has been proposed in the field of nuclear fuel cycle based on pyrochemistry [2] and the transmutation process of minor actinides with proton accelerator [3]. Physical and chemical properties of molten salts must be known for the development of those technologies. From the other hand, knowledge of viscosity gives substantial information on the structure of liquids [4].

The present paper is a continuation of our first publication [5], devoted to systematic study of viscosity of molten $LnCl_3$.

Viscosity measurements at high temperatures (above 700 – 900 K) are a difficult experimental field. In the case of molten rare earth halides the most important and complicated problem is the preparation of oxichlorides-free salts and keeping melt under study free from contacts with oxidants or moisture.

In this work particular attention was paid to the preparation of anhydrous salts. In order to exclude any contact of $LnCl_3$ with atmosphere during the measurements, completely closed viscometers were used.

The kinematic viscosities of molten $LaCl_3$, $CeCl_3$, $PrCl_3$, $NdCl_3$, and $SmCl_3$ were studied by using a capillary viscometer. Temperature range of measurements was from the melting point to typically 1170 – 1190 K. The viscosity of molten $PmCl_3$ and $EuCl_3$ was estimated using common tendencies in the lanthanides row. The dynamic (η) and molar viscosity (η_M) were calculated from the measured kinematic viscosity (ν) and the density (d) data recommended in [6, 7].

2. Experimental

2.1. Apparatus

A capillary viscometer entirely made of quartz was used in the present study. It consists of an outside tube sealed hermetically and a capillary, 0.4 mm inner diameter, placed inside. Upwardly, the capillary was connected with timing bulb approximately 3 – 4 ml capacity. An outlet of special shape was attached to the capillary at the bottom in order to compensate surface tension forces, connected with meniscus. For more details, see [5, 8].

2.2. Determination of the Viscometer Constants

Before each measurement the viscometer was calibrated and the constants C_1 and C_2 in the following equation were precisely determined [8]:

$$\nu = C_1 t - C_2/t. \tag{1}$$

In general, C_2/t is small as compared with $C_1 t$. Usually C_2/t does not exceed 2% of $C_1 t$, but in rare cases can reach 5 – 10 %. The choice of a suitable calibration liquid is rather important for reliable determination of the C_1 and C_2 constants. Distilled water is the best choice because the viscosity and density of water are most precisely known compared with those for all other liquids. Water covers a wide range of kinematic viscosity which is nearly the same as that of the melts under investigation. The main argument against water is essential (several hundred degrees) difference between the calibration and the working temperature. But because of the very small quartz temperature expansion coefficient ($5.8 \cdot 10^{-7}$ K^{-1} [9]) the error due to thermal quartz expansion is negligible (less than 0.15%).

For determination of the viscometer constants, the flow time of distilled water was measured in a water thermostat over the temperature range 275 – 338 K with increments of about 10 K. A typical result is shown in Figure 1. The constants are calculated with the least-squared method using (2) for viscosity of water:

$$\lg(\nu) = -5.613291 + \frac{4325.4932}{T} - \frac{1354943.57}{T^2} + \frac{166934200}{T^3}. \tag{2}$$

The resulting C_1 and C_2 values are shown in Figure 1 too. Equation (2) is an approximation of distilled water viscosity data from [9].

2.3. Chemicals

Anhydrous lanthanum, cerium, and praseodymium trichlorides were obtained by dehydration of $LaCl_3 \cdot 7H_2O$, $CeCl_3 \cdot 7H_2O$, and $PrCl_3 \cdot 7H_2O$, respectively. Anhydrous $NdCl_3$ and $SmCl_3$ were synthesized from the corresponding oxides. The initial salt and oxides were Soekawa Chemicals (Japan) 99.9% materials. The oxides were dissolved in concentrated HCl with subsequent crystallization of the hydrates $LnCl_3 \cdot 6H_2O$:

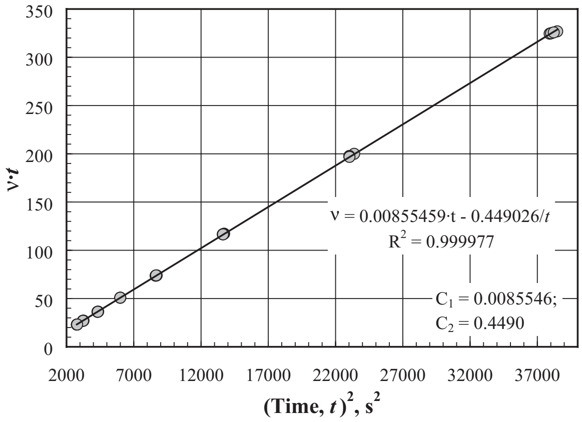

Fig. 1. Calibration of the viscometer with distilled water. Cell 53-A. ν: kinematic viscosity of water; t: time in s.

$$Ln_2O_3 + 6HCl_{acid} = 2LnCl_3 + 3H_2O. \tag{3}$$

Dehydration of crystalline hydrates was carried out by heating in a flow of dry N_2 up to 130 – 180 °C (depends on the element). The resulting product was $LnCl_3 \cdot (1-2)H_2O$. Then the N_2 flow was replaced with a flow of dry $HCl_{(gas)}$, and the salt was further heated up to melting. It is very important to carry out the dehydration slowly to prevent the formation of oxichlorides. Finally, the rare earth chlorides were distilled under vacuum (∼ 1 Pa) and subsequently were handled under inert atmosphere with a water content of 1 – 2 ppm.

No insoluble matter was found in the course of dissolving the chlorides in distilled water. By the solubility tests [5, 10] the quality of each lot of $LnCl_3$ was controlled. About 0.5 g of the salt was dissolved in 3 – 4 cm^3 distilled water. The solution is absolutely transparent or has a weak opalescence when oxichlorides are absent. In several cases, especially for the heavy lanthanides, hydrolysis occurs during the dissolution with hydroxide formation. In this case, 1 – 2 drops of concentrated HNO_3 were enough to suppress hydrolysis. If the solution still remained opaque after 2 drops of HNO_3, the content of oxichlorides was considered to be too high and the product was used as a row material. For more details, see [5].

3. Results and Discussion

The experimental kinematic viscosity values of molten $LaCl_3$, $CeCl_3$, $PrCl_3$, and $NdCl_3$ are shown in Figure 2. In all cases viscosities decrease with temperature increases. The viscosities of molten cerium, praseodymium, neodymium, and samarium chlorides are very close to each other. For example maximum difference between them does not exceed 2.2% at 800 °C and 5.3% at 900 °C. The kinematic viscosity of lanthanide chlorides as a function of temperature is adequately approximated by the equation

$$\ln(\nu) = \ln(\nu_0) + \frac{E_A}{R \cdot T}, \tag{4}$$

where ν is the kinematic viscosity, and E_A is the activation energy of viscous flow.

The coefficients ν_0 and E_A are summarized in Table 1.

The dependence of kinematic viscosities on reverse radius of lanthanide cation at 1073 and 1173 K is shown in Figure 3. In such coordinates, the tendency of viscosity variation in a row of lanthanides is clearer.

The dynamic viscosity (η) was calculated according to (5):

$$\eta = \nu \cdot d, \tag{5}$$

where d is the density of the melt.

For computing the dynamic viscosity, the densities of molten $LnCl_3$ recommended in [6, 7] were used. The densities published in [6, 7] are a complete and self-consistent set of data and therefore the data are preferable. The results are summarized in Table 2 and are juxtaposed with literature data in Figures 4 – 6. For molten $CeCl_3$, $NdCl_3$, and $SmCl_3$ such comparison was made earlier [5]. The coefficients in dynamic viscosity equations for molten $CeCl_3$, $NdCl_3$, and $SmCl_3$ given in Table 2 slightly differ from the data presented in paper [5] as the more reliable density data were used.

Our viscosity data on $LaCl_3$ are the lowest ones (see Figure 3). At 1173 K the difference with data [11, 12] is approximately 6.3% and ≈ 8.4% with data [13].

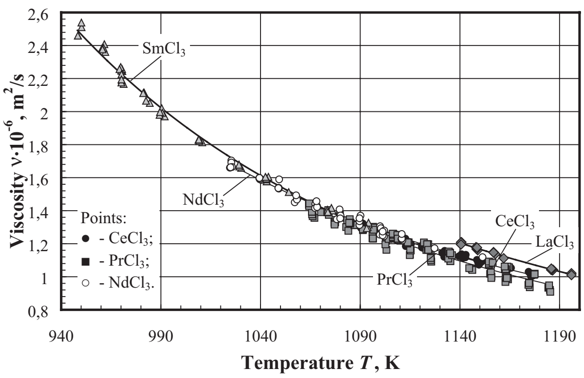

Fig. 2. Kinematic viscosities of molten rare earth chlorides of cerium subgroup.

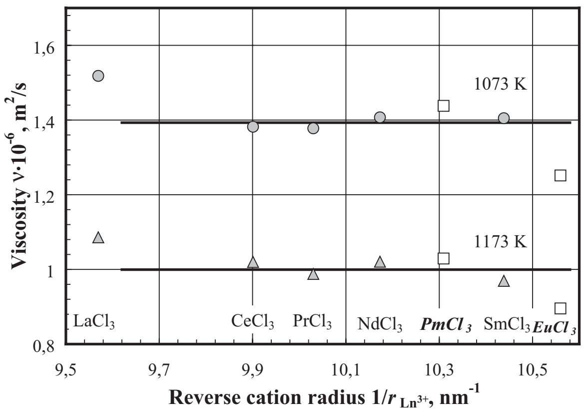

Fig. 3. Kinematic viscosities of molten $LnCl_3$ vs. $1/r_{Ln^{3+}}$.

Table 1. Kinematic viscosity ($\ln(\nu) = \ln(\nu_0) + E_A/RT$) of molten rare earth chlorides. Total error does not exceed ± 3%, with the exception of $EuCl_3$, for which error estimation is 5 – 6 %. $R = 8.31441$ J/(K · mol), T [K].

Salt	$\ln(\nu_0)$	E_A, [J/mol]	$\nu \cdot 10^{-6}$, [m^2/s] 1073 K	1173 K	ΔT, [K]
$LaCl_3$	−3.5115	35051	1.517	1.086	1140 – 1196
$CeCl_3$ [5]	−3.2396	31788	1.381	1.019	1090 – 1177
$PrCl_3$	−3.5051	34087	1.371	0.990	1064 – 1185
$NdCl_3$ [5]	−3.4252	33605	1.406	1.020	1025 – 1151
$PmCl_3$	−3.572	35000	1.438	1.029	–
$SmCl_3$ [5]	−4.0118	38823	1.404	0.969	948 – 1094
$EuCl_3$	−3.704	35000	1.25	0.895	–

Table 2. Dynamic viscosity ($\ln(\eta) = \ln(\eta_0) + E_A/RT$) of molten rare earth chlorides. Total error does not exceed ±4%, except of $EuCl_3$. $R = 8.31441$ J/(K · mol), T [K].

Salt	$\ln(\eta_0)$	E_A, [J/mol]	η, [mPa · s] 1073 K	1173 K	ΔT, [K]
$LaCl_3$	−2.6050	37455	4.918	3.438	1140 – 1196
$CeCl_3$	−2.3488	34420	4.521	3.254	1090 – 1177
$PrCl_3$	−2.6029	36637	4.496	3.168	1064 – 1185
$NdCl_3$	−2.4788	35875	4.673	3.317	1025 – 1151
$PmCl_3$	−2.6366	37389	4.729	3.309	–
$SmCl_3$	−3.0131	40837	4.776	3.233	948 – 1094
$EuCl_3$	−2.6954	36972	4.256	2.989	–

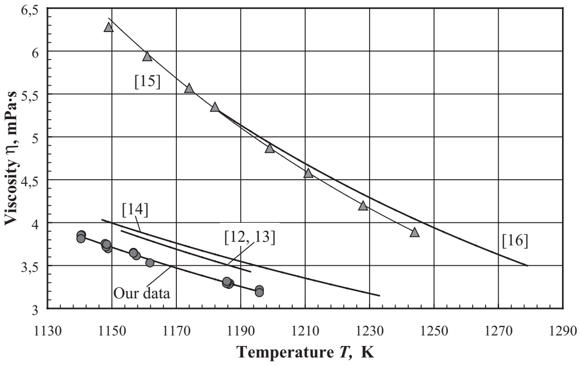

Fig. 4. Dynamic viscosity of molten $LaCl_3$. All data available.

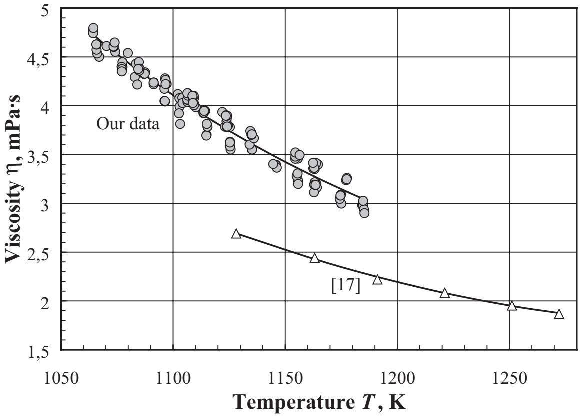

Fig. 5. Dynamic viscosity of molten $PrCl_3$. All data available.

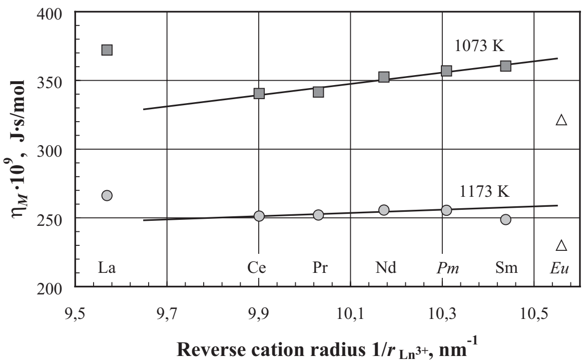

Fig. 6. Molar viscosity of molten $LnCl_3$ of cerium subgroup.

One of the possible reasons of the discrepancy is the postmelting effect, which is considered below. More than 60% deviation from [14, 15] is undoubtedly the consequence of inadequate $LaCl_3$ preparation. The presence of fine powder of insoluble LaOCl increases total viscosity.

The somewhat lower viscosity of $PrCl_3$ according to Cho and Kuroda [16] is probably a consequence of some defect of his measuring procedure.

Similar to molar conductivity the molar viscosity (η_M) is a property normalized to the same number (1 mole) of particles [4, 5, 17 – 19]. In this sense the molar viscosity is the most correct characteristic for comparison of different substances.

Molar viscosity is the energy which dissipates a flow of one mole of liquid running with unit velocity gradient owing to internal friction forces.

It was calculated according to

$$\eta_M = \eta \cdot V_m, \tag{6}$$

where V_m is the molar volume.

Since $\eta = \nu \cdot d$ and $V_m = M/d$, another expression is obtained for molar viscosity:

$$\eta_M = \nu \cdot M, \tag{7}$$

where M is the molar mass.

Until now there is no conventionally accepted unit for η_M. According to (7) the unit is

$$[\eta_M] = \frac{m^2}{s} \cdot \frac{kg}{mol} = \frac{kg \cdot m^2}{s \cdot mol}. \tag{8}$$

Since $J = kg \cdot m^2/c^2$

$$[\eta_M] = J \cdot s/mol. \tag{9}$$

In the present paper, second version (9) is used.

Molar viscosities of investigated $LnCl_3$ are summarized in Table 3 and are plotted as η_M vs. $1/r_{Ln^{3+}}$ in Figure 6. Generally the viscosity slightly increases within the cerium subgroup; however, the viscosity values for $LaCl_3$ and $EuCl_3$ lie out of the main tendency.

$LaCl_3$. The surprising thing is that the viscosity of molten $LaCl_3$ is higher than the viscosities of all chlorides from $CeCl_3$ to $SmCl_3$. The nature of this abnormality is not yet entirely known. At the same time,

Table 3. Molar viscosity ($\ln(\eta_M) = \ln(\eta_{M0}) + E_A/RT$) of molten rare earth chlorides. $R = 8.31441$ J/(K · mol), T [K].

Salt	$\ln(\eta_{M0})$	E_A, [J/mol]	$\eta_M \cdot 10^9$, [J · s/mol] 1073 K	1173 K	ΔT, [K]
$LaCl_3$	1.9908	35051	372.1	266.2	1140 – 1196
$CeCl_3$	2.2677	31788	340.4	251.3	1090 – 1177
$PrCl_3$	2.0054	34087	338.9	244.7	1064 – 1185
$NdCl_3$	2.09865	33605	352.4	255.7	1025 – 1151
$PmCl_3$	1.9549	35000	356.9	255.5	–
$SmCl_3$	1.5362	38823	360.4	248.7	948 – 1094
$EuCl_3$	1.8502	35000	321.4	230.1	–

this is not the first evidence of somewhat unusual behaviour of molten $LaCl_3$. Molar electrical conductivity of molten $LaCl_3$ is a little lower and activation energy of the electricity transport is higher as follows from the common tendency (see Figure 2 in [20], Figures 12, 14 in [21]). Lower conductivity is easy explained due to higher viscosity of this melt. In thermophysical studies [22–24, and references therein] authors point to reproducible hysteresis between enthalpies of melting and crystallization. They attribute this phenomenon to the postmelting effect with the formation of particular chain-like structure of liquid. Similar ideas have already been proposed by Savin et al. [25, 26]. According to [27–31] the average coordination number of La^{3+} ions is essentially higher (7–8) as compared with other Ln^{3+} ions (typically 6 [32, 33]). The higher is the coordination number the higher is the viscosity and the lower is the conductance.

$PmCl_3$. Promethium was not available in any form to the authors for investigation in this study. According to analysis of electronic structure of Pm^{3+} ion [34], the properties of molten $PmCl_3$ should closely fall in common tendencies in properties changes from $LaCl_3$ to $LuCl_3$. In this sense $NdCl_3$ is the closest analogue of $PmCl_3$. The estimation of viscosity of molten $PmCl_3$ made by interpolation is included in Tables 1–3 and illustrated in Figures 3 and 6.

$EuCl_3$. It is not possible to measure the viscosity of molten $EuCl_3$ in the same manner as that of other chlorides because of its thermal decomposition. The most reliable estimation of $EuCl_3$ melting point is 632 °C [34]. Appreciable decomposition begins at 400–500 °C and of course essentially increases under reduced pressure. Furthermore, molten and solid $EuCl_3$, as a rule, exhibit physical and chemical properties lying outside of general tendency [20, 35]. Electrical conductivity of molten $EuCl_3$ is higher by 20% approximately as follows from common tendency in lanthanide row. The fact was well established in papers [35, 36] and confirmed by our independent measurements [20, 37]. $EuCl_3$ is a single salt with one type of cations (Eu^{3+}) and one type of anions (Cl^-). Higher conductivity of molten $EuCl_3$ in comparison with neighbouring $SmCl_3$ and $GdCl_3$ can be the consequence of looser structure of the melt. Looser structure leads to higher mobility of ions, thus to larger conductivity and also to lower viscosity. The idea that high conductivity arises due to partial thermal decomposition of $EuCl_3$ with formation of more mobile Eu^{2+} cations is unfounded. Electrical conductance of molten $EuCl_3 - EuCl_2$ system has been measured over all composition range [20, 37]. A partial $EuCl_3$ decomposition ranging 5–10 % leads to increasing of conductivity only by 2.8–5.7 %.

To a first approximation the viscosity of molten $EuCl_3$ is taken also lower about by 20% as follows from the tendency, see Figures 3 and 6 and Tables 1–3.

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Supplementary Tables. Kinematic viscosities ν [m^2/s] of molten rare earth chlorides. Initial data.

Table 4. $LaCl_3$ (Exp. 23-4).

t [°C]	$\nu \cdot 10^{-6}$	t [°C]	$\nu \cdot 10^{-6}$	t [°C]	$\nu \cdot 10^{-6}$	t [°C]	$\nu \cdot 10^{-6}$	t [°C]	$\nu \cdot 10^{-6}$
913.6	1.0422	912.7	1.0496	884.6	1.1372	875.2	1.1676	867.4	1.2082
913.3	1.0384	922.8	1.02125	883.7	1.1492	874.9	1.1793	867.4	1.1952
912.8	1.0435	922.8	1.0101	883.7	1.1467	875.4	1.1768		
912.6	1.0419	888.8	1.1122	875.8	1.1609	867.6	1.2098		

Table 5. $PrCl_3$ (Exp. 23-5).

t [°C]	$\nu \cdot 10^{-6}$	t [°C]	$\nu \cdot 10^{-6}$	t [°C]	$\nu \cdot 10^{-6}$	t [°C]	$\nu \cdot 10^{-6}$	t [°C]	$\nu \cdot 10^{-6}$
804.3	1.329	810.3	1.342	841.9	1.168	871.5	1.052	901.8	0.9649
804.3	1.344	823.3	1.230	852.2	1.162	882.7	1.002	901.9	0.9361
803.9	1.329	823	1.270	852.5	1.091	882	1.017	901.2	0.9506
804	1.323	823	1.232	852.3	1.116	882.4	1.026	901.6	0.9630
804	1.316	823.2	1.232	852.3	1.101	882.6	0.9923	901.5	0.9617
793.9	1.356	830	1.163	852.2	1.113	890.8	0.9950	911.2	0.9335
793	1.366	829.8	1.217	862	1.094	891	0.9838	911.5	0.9354
792.7	1.378	829.8	1.248	862.1	1.095	889.8	0.9925	912	0.9203
792.8	1.394	829.5	1.196	861	1.110	890	1.0000	912.2	0.9085
792.6	1.394	841.8	1.133	872.8	1.050	889.7	0.9682	911.6	0.9479
809.8	1.301	841.7	1.131	872.8	1.047	889.9	0.9925		
810.9	1.278	842.2	1.159	873.1	1.041	890.4	0.9920		

Table 6. $PrCl_3$ (Exp. 23-7).

t [°C]	$\nu \cdot 10^{-6}$	t [°C]	$\nu \cdot 10^{-6}$	t [°C]	$\nu \cdot 10^{-6}$	t [°C]	$\nu \cdot 10^{-6}$	t [°C]	$\nu \cdot 10^{-6}$
806.7	1.374	829	1.256	831.1	1.229	818.2	1.287	814.2	1.313
797.2	1.391	829.7	1.243	818.2	1.287	814.4	1.317		
791.1	1.437	831	1.246	818.2	1.282	813.9	1.312		

Table 7. $PrCl_3$ (Exp. 23-13).

t [°C]	$\nu \cdot 10^{-6}$	t [°C]	$\nu \cdot 10^{-6}$	t [°C]	$\nu \cdot 10^{-6}$	t [°C]	$\nu \cdot 10^{-6}$	t [°C]	$\nu \cdot 10^{-6}$
848.8	1.208	811.8	1.322	837	1.218	850.8	1.162	881.4	1.079
833.3	1.233	811.3	1.326	836.2	1.246	851	1.197	881.4	1.092
833.4	1.239	801.1	1.375	836.3	1.228	850.4	1.197	883.3	1.085
833.2	1.241	800.4	1.390	836.1	1.253	851	1.163	891.5	1.057
833.5	1.253	800.3	1.391	836.2	1.224	861.8	1.147	890.7	1.061
833.4	1.260	800.8	1.404	835.6	1.249	862.9	1.130	890	1.042
833.2	1.260	791.2	1.428	835.5	1.253	861.3	1.153	889.4	1.044
823.3	1.302	791.3	1.444	835.7	1.231	861.8	1.141	889.8	1.046
824	1.285	841.2	1.208	851.3	1.165	862	1.143	889.3	1.062
823.6	1.291	840.4	1.201	850	1.162	882.5	1.073	904.4	1.019
823.5	1.298	840.6	1.209	850.5	1.198	882.5	1.084	904	1.012
823.5	1.285	840.5	1.202	850.7	1.180	881.3	1.072	904.3	1.015
811.6	1.348	840.7	1.208	850.8	1.189	881.9	1.087		

Note 1. Final equation for kinematic viscosity of molten $PrCl_3$ (Table 1 in the main text) was computed as an average of equations, calculated from data of Exp. 23-5, Exp. 23-7, and Exp. 23-13, at that the equation after Exp. 23-7 was taken with weight $1/2$.