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Viscosity of Molten Alkali Chlorides

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Precision determinations of the viscosities of the molten alkali chlorides are reported. The viscosities are described by Arrhenius equations with a standard deviation of ±0.10% or less. The equations for the viscosity in mPa s are η(LiCl) = 0.10888 exp(2295.5/T), η(NaCl) = 0.08931 exp(2841/T), η(KCl) = 0.06168· exp(3012.4/T), η(RbCl) = 0.06776 exp(2984.7/T), and η(CsCl) = 0.07136 exp(2842.8/T). The accuracy is estimated to be ±0.3% at the 68% confidence level. We have identified and eliminated bubble formation on the oscillating body as a significant error source.

Introduction

The viscosity of molten NaCl was recently redetermined (*1*) as a part of the Molten Salts Standard Program Project (*2*). The results have been recommended to the U.S. National Bureau of Standards as a set of new reference values. They revise the previous recommendation (*3*) up to 30%.

Although there are many previous reports on the viscosity of alkali chlorides, including several from this laboratory (*4–6*), it was considered of interest to use the recently developed high-precision viscometer for a redetermination of alkali chlorides other than NaCl with an accuracy approaching that for NaCl (0.2% at the 68% confidence level).

Experimental Section

Viscosity was determined either from the damping of an oscillating cylinder immersed in the melt or, with less accuracy, from the change in the time of swing. Both methods are absolute. The automated viscometer assembly, the control measurements, the experimental procedure, and the working equations were described in a previous publication (*1*), and only details on salt preparation and handling will be given here.

The alkali chlorides used were p.a.-grade chemicals from Merck (KCl and RbCl), Baker (LiCl), and Hopkin and Williams (CsCl). The alkali chlorides, with the exception of LiCl, were dried under vacuum at 400–500 °C and then melted in a nitrogen atmosphere and allowed to crystallize at a slowly decreasing temperature. Clear crystals were selected and used for measurements. Anhydrous LiCl was prepared by treatment with HCl gas as described by Grjotheim et al. (*7*), followed by repeated recrystallizations: twice in the first series and thrice in the second measuring series.

The purified salts were weighed out (drybox, <2 ppm H_2O) in the platinum crucible to be used in the viscometer. The crucible was transported to the viscometer in a closed vessel and placed in the cold N_2-filled viscometer furnace. The viscometer was rapidly closed and evacuated to 10^{-3} mbar. The furnace was heated slowly to 350–400 °C and kept under 10^{-4} mbar at that temperature for ~0.5 h. Highly purified N_2 was then admitted, and the furnace heated to the operating temperature.

After temperature stabilization, the period and damping in nitrogen were measured and checked against their respective temperature functions, obtained in auxiliary experiments. The crucible with the molten alkali chloride was then raised to immerse the oscillating cylinder 15 ± 1 mm beneath the liquid surface, and the viscosity measurements were started 2 h later. Four experiments were carried out at each temperature, with at least 2 h allowed for temperature equilibration between the series. The temperature stability during the four experiments (30 min) was 0.1 °C or better.

The temperatures were first varied in a semirandom fashion, but this procedure was later abandoned (see Discussion). The upper experimental temperature was usually restricted by a chosen limit of 5 mbar for the vapor pressure. Evaporation losses were always less than 1.5%.

The number of periods measured was increased from 24 (*1*) to 40 in order to improve the precision.

Two experiments were performed for each salt. The second experiment was carried out after a lapse of at least 1 week and after a different chloride had been determined in the meantime. Although salt preparation was not considered critical except for LiCl, the salts for the two experiments were prepared independently of each other. A series of control measurements in water was carried out before the alkali chloride experiments.

Results

The viscosity data are given in the order measured in Table I. The data were fitted to our usual model equation (*1*)

$$\eta = A \exp\{B(1/T - 1/T_m)\} \tag{1}$$

where T_m is a mean temperature introduced to improve orthogonality in A and B. The parameters A, B, and T_m with standard deviations are given in Table II for each experiment and for the two combined experiments for each salt. The viscosities are depicted in Figure 1, where filled and open symbols represent the two independent experiments for each salt.

By direct transformation of the equations given for the combined experiments in Table II the following simpler equations are obtained (1 mPa s = 10^{-3} N s m^{-2} = 1 cP):

$$\text{LiCl} \qquad \eta(\text{mPa s}) = 0.10888 \exp(2295.5/T) \tag{2}$$

$$\text{NaCl } (1) \qquad \eta(\text{mPa s}) = 0.08931 \exp(2641/T) \tag{3}$$

$$\text{KCl} \qquad \eta(\text{mPa s}) = 0.06168 \exp(3012.4/T) \tag{4}$$

$$\text{RbCl} \qquad \eta(\text{mPa s}) = 0.06776 \exp(2984.7/T) \tag{5}$$

$$\text{CsCl} \qquad \eta(\text{mPa s}) = 0.07136 \exp(2842.8/T) \tag{6}$$

The control measurements in water gave the result 0.8912 ± 0.0001 mPa s at 24.948 °C. The ICPS 74 (*8*) recommendation at this temperature is 0.8913 mPa s.

The following density equations of van Artsdalen and Yaffe, recommended by Janz et al. (*3*), were chosen for computation of the viscosity:

$$\text{LiCl} \qquad \rho(\text{g/cm}^3) = 1.8842 - (0.4328 \times 10^{-3})T \tag{7}$$

$$\text{KCl} \qquad \rho(\text{g/cm}^3) = 2.1359 - (0.5831 \times 10^{-3})T \tag{8}$$

$$\text{RbCl} \qquad \rho(\text{g/cm}^3) = 3.1210 - (0.8832 \times 10^{-3})T \tag{9}$$

$$\text{CsCl} \qquad \rho(\text{g/cm}^3) = 3.7692 - (1.065 \times 10^{-3})T \tag{10}$$

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Table I. Viscosities of LiCl, KCl, RbCl, and CsCl

temp, °C	viscosity, mPa s	10^{-4}SD, mPa s	temp, °C	viscosity, mPa s	10^{-4}SD, mPa s
KCl (First Series)					
837.9	0.9279	4	901.5	0.8020	3
	0.9274	4		0.8020	4
	0.9275	4		0.8012	3
	0.9278	4		0.8018	4
881.1	0.8387	5	860.5	0.8813	4
	0.8383	4		0.8804	4
	0.8386	3		0.8801	4
	0.8384	3		0.8800	3
800.2	1.0234	4	781.0	1.0739	5
	1.0231	4		1.0742	4
	1.0233	4		1.0742	4
	1.0226	4		1.0743	4
821.9	0.9671	3			
	0.9668	4			
	0.9671	4			
	0.9670	4			
RbCl (First Series)					
759.7	1.2218	4	723.0	1.3576	10
	1.2216	5		1.3580	11
	1.2217	4		1.3564	7
	1.2210	4		1.3566	8
741.5	1.2832	5	839.4	0.9927	3
	1.2833	5		0.9923	3
	1.2837	4		0.9925	4
	1.2834	3			
820.4	1.0387	4			
	1.0397	4			
	1.0392	4			
	1.0399	5			
CsCl (First Series)					
674.9	1.4315	4	700.0	1.3257	3
	1.4321	4		1.3256	5
	1.4325	4		1.3254	4
	1.4323	4		1.3255	4
650.8	1.5488	5	724.6	1.2331	3
	1.5481	4		1.2332	4
	1.5489	4		1.2338	4
	1.5481	5		1.2336	4
RbCl (Second Series)					
749.6	1.2537	4	810.0	1.0654	4
	1.2530	4		1.0647	3
	1.2534	3		1.0648	3
	1.2532	4		1.0649	3
730.9	1.3229	3	830.1	1.0133	4
	1.3231	3		1.0135	3
	1.3227	3		1.0136	4
	1.3235	4		1.0132	4
769.5	1.1856	4	848.9	0.9686	3
	1.1847	3		0.9686	4
	1.1853	4		0.9684	4
	1.1851	4		0.9683	4
779.3	1.1549	2	860.7	0.9420	3
	1.1557	3		0.9422	3
	1.1547	3		0.9420	3
	1.1554	3		0.9422	3
788.7	1.1270	4	799.0	1.0949	4
	1.1274	4		1.0947	3
	1.1271	3		1.0950	3
	1.1273	3			
LiCl (First Series)					
658.5	1.2795	4	779.8	0.9635	5
	1.2790	4		0.9634	5
	1.2788	4		0.9632	5
	1.2786	5		0.9633	3
621.8	1.4130	5	818.7	0.8918	3
	1.4149	6		0.8919	3
	1.4149	5		0.8918	4
	1.4150	6		0.8914	4
	1.4139	5	857.9	0.8276	2
700.6	1.1511	4		0.8274	4
	1.1510	3		0.8273	3
	1.1513	4		0.8273	4
	1.1508	3			
738.8	1.0530	4			
	1.0537	4			
	1.0534	5			
	1.0533	5			
KCl (Second Series)					
788.1	1.0528	4	849.4	0.9017	4
	1.0532	4		0.9028	4
	1.0527	5		0.9030	3
	1.0529	5		0.9030	4
811.3	0.9916	4	869.8	0.8600	4
	0.9911	5		0.8593	2
	0.9916	5		0.8605	3
	0.9915	5		0.8601	3
829.1	0.9478	3	889.0	0.8234	3
	0.9480	4		0.8235	3
	0.9477	4		0.8237	3
	0.9481	4		0.8232	3
CsCl (Second Series)					
661.3	1.4941	6	787.1	1.0425	5
	1.4949	4		1.0424	3
	1.4939	4		1.0421	4
	1.4949	4		1.0416	3
688.3	1.3706	6	813.3	0.9778	4
	1.3713	4		0.9779	4
	1.3708	5		0.9781	3
	1.3714	4		0.9780	4
711.0	1.2810	3	749.7	1.1487	2
	1.2815	3		1.1486	3
	1.2811	4		1.1487	3
	1.2802	5		1.1482	2
736.3	1.1919	3	774.1	1.0764	3
	1.1911	4		1.0764	3
	1.1920	4		1.0771	3
	1.1919	4		1.0765	3
761.5	1.1128	3	799.5	1.0105	2
	1.1134	4		1.0108	3
	1.1132	4		1.0108	4
	1.1137	6		1.0108	4
LiCl (Second Series)					
638.2	1.3512	5	757.8	1.0093	4
	1.3515	5		1.0094	4
	1.3512	5		1.0090	3
	1.3511	4		1.0091	5
679.2	1.2135	4	800.4	0.9236	4
	1.2132	3		0.9235	3
	1.2132	6		0.9231	5
	1.2138	5		0.9234	3
720.2	1.0990	4	840.8	0.8529	4
	1.0996	4		0.8537	4
	1.0986	4		0.8533	4
	1.0990	4		0.8533	4

The viscosities reported may be converted to other density bases by using eq 11. Viscosities so converted will be correct

$$\Delta\eta(\%) = -0.8\Delta\rho(\%) \qquad (11)$$

within 0.1% relative to uncorrected viscosities for densities revised up to ±2% from the expressions above.

Discussion

A thorough error analysis of the instrumental ensemble has been given previously (*1*, *9*, *10*). It concluded with a best-case accuracy of 0.11% standard deviation, the major contributions being the uncertainties in the cylinder dimensions and the net damping constant. There is an additional error source arising from the sensitivity of the viscous flow to temperature, which

Table II. Parameters in the Exponential Temperature Function Eq 1 for the Viscosity of Molten Alkali Chlorides

salt	series[a]	temp span, K	data points	A, mPa s	10^{-4}(SD) in A, mPa s	B, K	SD[b] in B, K	T_m, K	SD in η, %	viscosity at 1073 K, mPa s
LiCl	1	895–1131	29	1.0603	1.8	2292.7	2.1	1008.54	0.10	0.9250
	2	911–1114	24	1.0506	1.8	2300.6	2.4	1012.58	0.08	0.9244
	1 + 2	895–1131	53	1.0559	1.4	2295.5	1.6	1010.37	0.09	0.9248
NaCl[c]	1	1085–1204	42	0.8998	0.6	2646	2.2	1143.25	0.04	1.0470[d]
	2	1097–1193	36	0.8989	0.7	2632	3.2	1143.65	0.05	1.0460[d]
	1 + 2	1085–1204	78	0.8994	0.5	2641	2.0	1143.45	0.05	1.0467[d]
KCl	1	1054–1175	28	0.9227	1.9	3016.3	6.0	1113.74	0.10	1.0226
	2	1061–1162	24	0.9241	0.8	3005.1	3.2	1112.60	0.04	1.0210
	1 + 2	1054–1175	52	0.9233	1.4	3012.4	4.8	1113.21	0.10	1.0219
RbCl	1	996–1113	19	1.1746	2.6	2977.3	5.8	1046.66	0.09	1.0954
	2	1004–1134	39	1.1028	1.1	2979.6	2.8	1069.76	0.06	1.0936
	1 + 2	996–1134	58	1.1255	1.6	2984.7	3.6	1062.19	0.10	1.0941
CsCl	1	924–998	16	1.3765	0.9	2841.4	2.0	960.72	0.02	1.0101
	2	934–1086	40	1.1537	1,1	2834.8	1.8	1021.36	0.06	1.0094
	1 + 2	924–1086	56	1.2108	1.3	2842.8	2.0	1004.04	0.08	1.0094

[a] Equations 2–6 equivalent to the combined series 1 + 2 are recommended for use. [b] SD = standard deviation. [c] Data from ref 1. [d] Extrapolated.

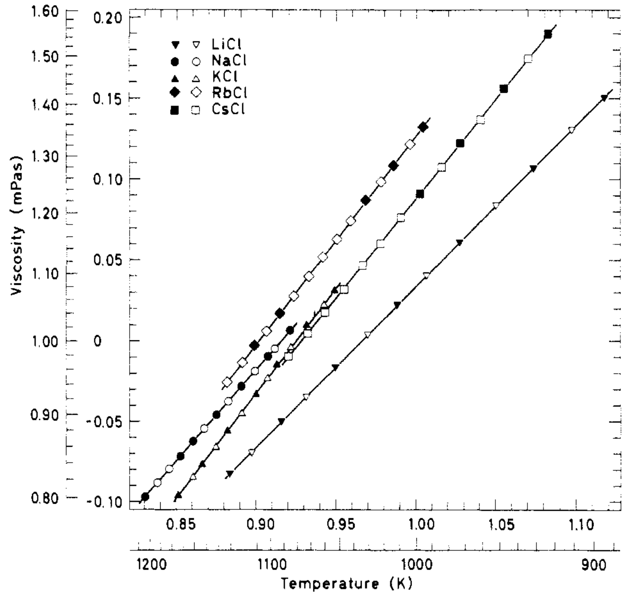

Figure 1. Arrhenius plot (log η vs. 1000/T) of the viscosity of molten alkali chlorides. Filled symbols: 1 series. Open symbols: 2 series.

will be sample dependent. We thus associated an error of 0.2% (68% confidence level) with the NaCl values obtained in the Standards Program.

The checking procedures employed by the NaCl determination (*1*) were extremely time-consuming, and some of the most rigorous exercises were relaxed in the present work. The Pt–10%Ir cylinder was not remachined despite a 5-μm unroundness that had developed before the alkali chloride experiments, and the following checks were no longer performed *before each experiment*: redetermination of the cylinder dimension, the temperature profile in the furnace, the time of swing and the damping in N_2 as functions of temperature and the moment of inertia at 25 °C for the pendulum assembly. Our accumulated experience with the viscometer indicates that no serious deterioration of the experimental accuracy will result provided suitable precautions are taken. Furthermore, the slight loss in accuracy relative to the error analysis cited above and to the Standards Program work in NaCl is partly compensated by an increased knowledge of the details of operation and performance of the viscometer.

The initial control experiment in water agreed with the recommended value within 0.01%. This is in line with earlier results obtained over the last 3 yr, which have always been within 0.1% of the recommendation. The standard deviation in the temperature fit for each chloride series was 0.02–0.1%; the standard deviation for the combined series was 0.05–0.10 at 1073 K (Table II). The thermocouples used were calibrated against the melting points of superpure Ag and Sn, but temperature errors may nevertheless dominate. The impact of an estimated 1 K maximum measuring error (possibly optimistic) on chloride viscosities at 1073 K varies from 0.20% to 0.27%. In summary we consider the present results to be accurate within ±0.3% at the 68% confidence level. Equation 11 may be used to correct for density errors if better density values should appear.

The viscosities from our second generation viscometer (*6*) compare at 1073 K with the present values as follows: LiCl, -4.4%; NaCl, +1.5%; KCl, -0.8%; RbCl, -0.1%; CsCl, +3.4%. A total of 21 other works on the viscosity of alkali chlorides was cited and compared in ref *6*. Since then we have become aware of some additional references, but the general picture of both low accuracy and high scatter is maintained. It is difficult to judge the quality of the investigations by comparing reported data since close agreement among erroneous results occurs (*10*). Deviations of more than 10% can probably only be accounted for by poor experimentation, but we have in the course of this work identified an important source of error that can explain discrepancies up to this approximate limit and still escape detection fairly easily, i.e., gas bubbles adhering to the oscillating body. We earlier (*10*) suspected this effect but had not been able to confirm it with reasonable certainty until the present series of experiments in several pure salts.

The viscosities of KCl and RbCl were determined at alternating decreasing and increasing temperature intervals. It was then observed that results tended to be erroneous when the melt had been cooled from a high temperature. An example of the effect is shown in Figure 2, which is a good illustration of the advantage of computing both "period" and "damping" viscosities in spotting irregular behavior. In the upper printout example the period viscosity is 125% higher than the damping viscosity, while all other goodness parameters appeared to be normal. Moving the crucible up and down and repeating the measurements is seen to cause the two types of computed viscosities to converge and finally attain constant values. The final damping viscosity is 9.4% below the initial value. The period viscosity is still 1.2% too high, but this deviation is not significant considering the uncertainty in the period measurements (*1*). The "load corrected period" in the printout is the measured period corrected for the effect of the negative axial load caused by the buoyancy of the oscillating cylinder in the melt.

```
VISCOSITY FROM PERIOD  CP    3.3498    0.0026
VISCOSITY FROM DAMPING CP    1.4849    0.0005
```

a

```
VISCOSITY FROM PERIOD  CP    2.2125    0.0022
VISCOSITY FROM DAMPING CP    1.4278    0.0005
```

b

```
VISCOSITY FROM PERIOD  CP    1.8358    0.0032
VISCOSITY FROM DAMPING CP    1.4039    0.0008
```

c

```
VISCOSITY FROM PERIOD  CP    1.3760    0.0021
VISCOSITY FROM DAMPING CP    1.3588    0.0008
```

d

```
SYSTEM                2  RUN NUMBER          48
DATE             190979  PERIODS INCL     5 -  28
TEMPERATURE C    723.00  CYL RADIUS CM   0.92927
CYL HEIGHT CM   1.90264  ROD RADIUS CM     0.070
ROD IMMERSION CM    1.5  PERIOD GAS MS   1676.92
DAMPING GAS    0.001184  LOAD CO*E5 CM2/S2  1.10
INERTIA GCM2     80.039  DENSITY G/CM3   2.24119

                                MEAN     ST.DEV      LEFT      RIGHT
PERIOD T                  MS  1684.86    0.007
OSCILLATION ZERO          MM                      - 0.906      0.906
MEAN AMP, MIN L, MAX R MM       317                    197        436
DAMPING CONSTANT   D*10000   338.18      0.09      338.12     338.23
STANDARD DEVIATION *10000                            0.13       0.14
CYL GAS DAMPING    D*10000     1.74
ROD DAMPING        D*10000     0.20
TOTAL DAMP CORR    D*10000    10.29
LOAD CORRECTED PERIOD  MS   1684.97
VISCOSITY FROM PERIOD  CP    1.3727    0.0024
VISCOSITY FROM DAMPING CP    1.3564    0.0007
```

e

Figure 2. Examples of printouts taken from the first measuring series in RbCl (CP = centipoise = mPa s): (a) crucible not moved after cooling from 820 to 723 °C; (b–d) repeated movements of crucible between runs; (e) a complete printout of the final, accepted results.

We feel confident that the erroneous results described were caused by a reduced gas solubility resulting in gas bubbles adhearing to the oscillating body. Such bubbles affect both the effective moment of inertia and the friction (similar effects are observed if the melt contains solid particles that adhere to the oscillating body). It is difficult to predict the magnitude or even the sign of the error in the general case, but in the present work the irregular damping viscosities were always too high. The problem was eliminated by performing the experiments with ascending temperatures only.

Experimental data for the alkali chloride melts frequently have been used to check the accuracy of values predicted from theory. Conclusions drawn from such comparisons are likely to have suffered from the presence of experimental errors, particularly in the case of NaCl. A survey would be well outside the scope of this discussion, but we have examined three of the most recent works using the present set of improved viscosities.

Young and O'Connell (*11*) applied some of the commonly encountered formalisms for transport phenomena to a number of alkali metal molten salts. They produced equations for corresponding states and developed relationships between characteristic properties for predictive use when no data are available. The viscosities that can be calculated from the given relations disagree from −11.4% to −40.7% with our experimental values, emphasizing the long-recognized qualitative nature of existing theories. For rough estimates of alkali chloride viscosities, formalisms may as well be dispensed with. According to the data in Table II, the mean value of all of the salts at the lowest common temperature is 1.02 mPa s ±2.6% standard error. In fact, the viscosity of any chloride at any temperature is 0.95 mPa s ±7% maximum error in the temperature regime where all salts are liquid and the evaporation losses negligible (~1073–1133 K). The experimental factor B, proportional to the "activation energy", also is essentially constant.

This puzzling independence of cation size has been noted by others, e.g., Reeves and Janz (*12*), who also compared the activation energies of viscous flow (E_η) and equivalent conductance (E_Λ). The ratio E_η/E_Λ for the alkali chlorides and also the alkaline earth chlorides was found to be between 4.4 and 1.9, in contrast to the group 1 nitrates and thiocyanates with a ratio close to unity (1.1–1.2). The chlorides as a group thus seemed different from the other salts, with respect to viscous flow and conductance. However, the ratios become much smaller when our viscosity data are introduced. They furthermore decrease fairly smoothly toward unity: LiCl, 2.3; NaCl, 1.8; KCl, 1.7; RbCl, 1.4; CsCl, 1.1.

The similarity in values for the activation energy for viscous flow and conductivity has been taken to indicate that the mechanisms for the two processes are also similar. Such mechanisms have been interpreted in terms of ionic association (*13*). It is doubtful whether this view can be applied to the alkali chloride melts, since it would imply a higher degree of association in CsCl than LiCl.

Cicotti et al. (*14*) have performed molecular dynamics (MD) calculations of the transport properties of molten alkali halides. The agreement between their calculated values and the present experimental results is good for RbCl, being 1.4% at 1119 K. The discrepancy is larger for NaCl, where the authors arrive at a value at 1262 K that is 14.6% too high. This is surprising since the model used does not consider the polarizability of the ions in computing the transport properties and hence should have given a better fit for NaCl than for RbCl. Still, the results are promising; the MD method seems at present to constitute the best approach to a quantitative description of the transport properties of molten salts.

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