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Viscosity of Multi-component Molten Nitrate Salts—Liquidus to 200°C

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Abstract

The viscosity of molten salts comprising ternary and quaternary mixtures of the nitrates of sodium, potassium, lithium and calcium was determined experimentally. Viscosity was measured over the temperature range from near the relatively low liquidus temperatures of the individual mixtures to 200°C. Molten salt mixtures that do not contain calcium nitrate exhibited relatively low viscosity and an Arrhenius temperature dependence. Molten salt mixtures that contained calcium nitrate were relatively more viscous and viscosity increased as the proportion of calcium nitrate increased. The temperature dependence of viscosity of molten salts containing calcium nitrate displayed curvature, rather than linearity, when plotted in Arrhenius format. Viscosity data for these mixtures were correlated by the Vogel-Fulcher-Tammann-Hesse equation.

Acknowledgements

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Introduction

Multi-component molten salt mixtures formulated from the nitrates of sodium, potassium, lithium and calcium display liquidus temperatures below 100°C and, as such, have potential applications as heat transfer fluids or thermal energy storage media for parabolic trough solar thermal power systems.[1,2] Parabolic trough concentrating solar power plant technology is well suited to provide central electrical power generation capacity on a scale of 100 MW or greater.[3] By integrating thermal energy storage into such systems, solar energy collected during the daylight hours can be used to generate electricity during the peak demand periods, which are often during the late afternoon in the summer and the early evening in the winter. Recent developments concerning the economic and environmental issues affecting electrical power generation have generated substantial interest in near-term investment in large-scale parabolic trough technology.[4,5]

Physical property data concerning these multi-component nitrate molten salts are not available but are required to properly design parabolic trough systems and components. A particular need is for information regarding the viscosity of molten salts as this property strongly affects pumping costs, among other aspects of system performance. This report describes experimental measurements of the viscosity of multi-component nitrate salt mixtures. The effect of the various constituent salts on viscosity was studied in order to provide a basis for formulating mixtures to optimize fluid properties, among which viscosity is an important determinant of system efficiency. The viscosity behavior of the molten salt mixtures studied here may also provide fundamental information for interpreting viscosity in terms of the structure of these liquids.

Experimental Methods

The molten salt mixtures were prepared from reagent-grade $NaNO_3$, KNO_3, $LiNO_3$ and $Ca(NO_3)_2$-tetrahydrate (Sigma-Aldrich Co., St. Louis, MO) without further purification. The nitrate mixtures were melted by weighing prescribed amounts of the individual salts into high-purity alumina crucibles that were heated in furnaces open to the atmosphere. The temperature was initially limited to approximately 150°C to allow the water of hydration of the calcium nitrate constituent to evolve slowly. After visual indications of vapor evolution ceased, the melts were heated to 350°C to 400°C, periodically stirred, and maintained at an elevated temperature for at least one overnight period. The melts were then cooled by pouring into a stainless steel crucible quenched in an ice bath. The solidified salt was recovered, roughly ground to reduce the particle size for subsequent loading into the viscometer tube, and stored in sealed containers under desiccation by molecular sieve. The compositions of the molten salt mixtures prepared for viscosity measurements are listed in Table 1.

The viscosity of molten salt mixtures were measured using a Brookfield DV-II+ viscometer (Brookfield Engineering, Middleboro, MA). The molten salt mixtures were contained in a stainless steel crucible that was heated in a furnace maintained at constant temperature by a Brookfield Thermosel controller. A Brookfield 21S stainless steel spindle was appropriate for the range of viscosity of the molten salt mixtures. All measurements were performed at a single rotational speed, 100 rpm, because molten nitrates are Newtonian fluids. The temperature was increased sequentially in 10°C increments from a starting point about 10°C–20°C above the liquidus of each mixture to a maximum between 200°C to 220°C, then successively decreased to the initial temperature to demonstrate repeatability of the measurements. The molten salt was maintained at each temperature sufficiently long for a steady state to be attained and the viscosity measurement to indicate a constant value. The viscometer configuration was limited to 20 cP at the minimum of measured values with a resolution of 0.5 cP over the entire range. Regardless, the accuracy of viscosity measurements was sufficient to compare the effect of the constituents of the molten salt mixtures on viscosity. The calibration of the viscometer was periodically verified by measuring a reference fluid, Brookfield Viscosity Standard Fluid 100, which had a certified viscosity of 99.2 cP at 25°C. No variation in the calibration of the instrument was observed during the course of the measurements.

Table 1. Compositions of multi-component molten nitrate salt mixtures for which viscosity was determined. The liquidus temperatures of ternary mixtures were obtained from Reference 6.

Mixture Label	$NaNO_3$	KNO_3	$Ca(NO_3)_2$	$LiNO_3$	Liquidus Temp.
	mol%	mol%	mol%	mol%	°C
Lithium-Sodium-Potassium Nitrate Mixtures					
LiNaK-1	18	45	–	37	120*
LiNaK-2	33	34	–	33	170
LiNaK-3	18	52	–	30	140
Calcium-Sodium-Potassium Nitrate Mixtures					
CaNaK-1	21	54	25	–	133*
Calcium-Lithium-Potassium Nitrate Mixtures					
CaLiK-1	–	57	12	31	117*
Quaternary Nitrate Mixtures					
QA-5Ca	17	43	5	35	<110
QA-10Ca	16	41	10	33	<110
QB-6Ca	31	32	6	31	<140
QB-10Ca	30	30	10	30	<140
QC-10Ca	16	47	10	27	<130
QC-15Ca	15	45	15	25	<120
QD	20	38	12	30	<100

* Eutectic mixtures.

Results and Discussion

Viscosity of molten lithium-sodium-potassium nitrate mixtures

The viscosity of several ternary molten salt mixtures composed of lithium-sodium-potassium (LiNaK) nitrates was measured at temperatures from about 200°C to near the liquidus temperature of each mixture. The mixtures studied were the eutectic, the equimolar and a mixture having a liquidus temperature intermediate between those two. The compositions and liquidus temperatures of these mixtures are given in Table 1.

The viscosity measurements are shown in Figure 1 using the Arrhenius format in which viscosity (log scale) is plotted vs. the inverse of the absolute temperature. The data points for the eutectic mixture (LiNaK-1) are represented by the open cross symbols, the equimolar mixture (LiNaK-2) by the filled diamonds, and the mixture melting at 140°C (LiNaK-3) by filled circles. The viscosity of these mixtures varied from 35 cP, for the eutectic mixture near the eutectic temperature, to about 6 cP at 220°C. Viscosity displayed negligible dependence on the composition of the mixtures within the range studied. No data concerning the viscosity of ternary Li-Na-K nitrate salt mixtures appear to have been published which would permit a comparison with the data reported here.

The temperature dependence of viscosity was correlated using the Arrhenius equation shown in Equation 1, in which η is the viscosity, A_0 is a constant, E_{visc} is the activation energy, R is the universal gas law constant, and T is the absolute temperature. Regression analysis of the viscosity data with the Arrhenius equation was performed using Kaleidagraph (Synergy Software, Reading, PA, version 4.04). The values of the activation energies are collected in Table 2 along with the least-squares correlation coefficient (R) for each mixture. The activation energy for viscosity of the ternary mixtures was approximately 6 kcal/mol. Table 2 compares the activation energies for the ternary Li-Na-K nitrate salt mixtures with those of the individual constituents based on data reported in the literature. The latter values range between 4 and 5 kcal/mol. The latter values were determined at temperatures above 300°C because the corresponding salts are not molten until considerably higher temperatures than the ternary mixtures.

$$\eta = A_0 \exp (E_{visc}/RT) \tag{1}$$

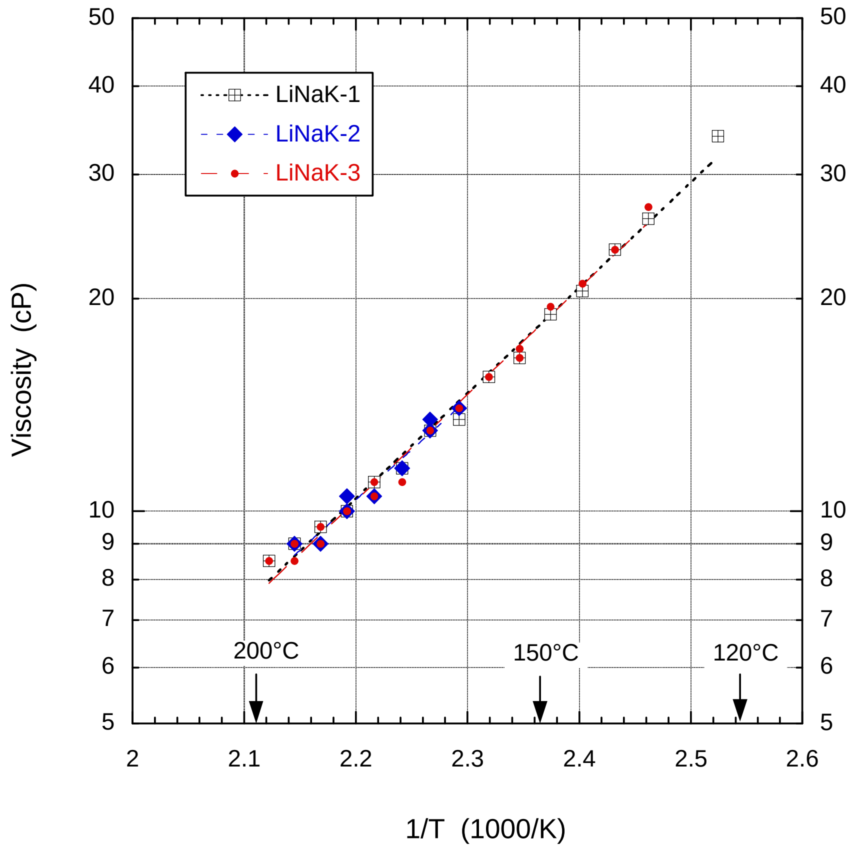

Figure 1. The viscosity of several molten lithium-sodium-potassium nitrate mixtures vs. temperature plotted in Arrhenius format.

Table 2. Activation energies for viscosity of ternary molten nitrate salt mixtures composed of lithium, sodium and potassium nitrates. The literature values for the individual nitrates and the equimolar mixture of sodium nitrate and potassium nitrate are shown for comparison.

$NaNO_3$	KNO_3	$LiNO_3$	E_{visc}	R
mol%	**mol%**	**mol%**	**kcal**	
18	45	37	6.83	0.996
33	34	33	6.00	0.990
18	52	30	6.39	0.994
50	50	–	4.6 [7], (a)	–
50	50	–	3.2 [7], (b)	–
100	–	–	3.89 [8]	–
–	100	–	4.97 [9]	–
–	–	100	4.39 [10]	–
–	–	100	4.58 [11]	–

(a) Temperatures above 385°C.

(b) Temperatures below 385°C.

Viscosity of ternary nitrate eutectics containing calcium nitrate

The viscosity data for the eutectic mixtures of calcium-sodium-potassium nitrate (CaNaK-1, filled diamonds) and calcium-lithium-potassium nitrate (CaLiK-1, filled circles) are plotted in Figure 2, using the conventional semi-log Arrhenius format. The compositions of these nitrate salt mixtures are given in Table 1. The viscosity of molten salt mixtures with calcium nitrate was considerably greater than any of the Li-Na-K-nitrate melts described in the preceding section. Whereas the latter molten salts displayed viscosities of about 7 cP at 200°C, the calcium nitrate-containing mixtures were 15 cP (CaLiK-1) and 26 cP (CaNaK-1), respectively. The data in Figure 2 also show that the relatively large proportion of $Ca(NO_3)_2$ in the Ca-Na-K-nitrate eutectic (25 mol%) resulted in a molten salt that was more viscous than the Ca-Li-K-nitrate eutectic (12 mol%) by a factor of two to three. At temperatures approaching the liquidus of these eutectics, the viscosity of mixture CaNaK-1 exceeded 100 cP while that of mixture CaLiK-1 exceeded 50 cP.

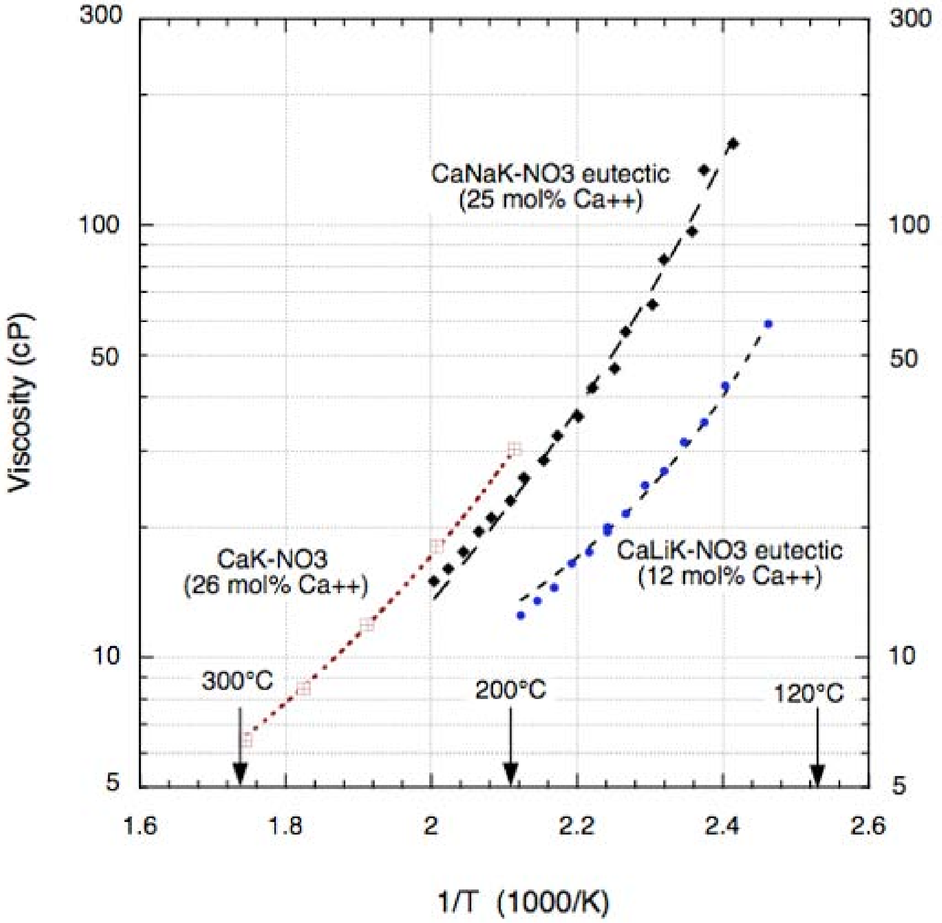

Figure 2. The viscosity of molten Ca-Na-K nitrate and Ca-Li-K nitrate eutectic mixtures vs. temperature plotted in Arrhenius format. The compositions of each mixture are given in Table 1. The data for the binary Ca-K nitrate mixture are from Ref. 12.

The published data for viscosity of molten nitrate salts containing calcium nitrate is quite limited. However, the viscosity of these ternary molten salt mixtures can be compared to data reported by Rhodes for a mixture of calcium nitrate (26 mol%) and potassium nitrate.[12] The minimum temperature of Rhodes' data was limited to about 200°C due to solidification. These data are also plotted in Figure 2 and are represented by the open square symbols. The mixture CaNaK-1, containing 25 mol% calcium nitrate, displays comparable viscosity and a similar trend with temperature compared to the binary mixture in the temperature region in which they overlap.

It is apparent from Figure 2 that viscosity of these calcium-nitrate-containing molten salts does not depend linearly on inverse absolute temperature in this semi-log format, i.e., following the Arrhenius equation, but displays curvature. The temperature dependence of the viscosity can be described by the Vogel-Fulcher-Tammann-Hesse equation, hereafter referred to as the VFTH equation.[13-15] This equation was originally developed to describe the viscosity of supercooled or glassy materials but subsequently has been applied successfully to a broad spectrum of the physical properties of liquids.[13] The typical form of the VFTH equation is shown in Equation 2, where A, B and T_0 are parameters determined by the best fit to the data. These parameters do not necessarily have a physical relationship to the molecular composition of the liquid system in most cases.

$$\log_{10} \eta = A + B/(T-T_0) \tag{2}$$

Regression analysis of the data using the VFTH equation was performed using the software application Kaleidagraph as noted above with an allowable error criterion of 0.1%. The lines in Figure 2 indicate the close correlation achieved by the VFTH equation for these sets of data. The parameters obtained by regression analysis for the two eutectic salt mixtures are given in Table 3.

Table 3. VFTH equation parameters for viscosity correlations of ternary molten nitrate salt mixtures containing calcium nitrate. The units of parameters B and T_0 are Kelvin.

Mixture	**VFTH equation parameters**			
	A	**B**	**T_0**	**R**
CaLiK-1	0.0767	164.3	36.04	0.998
CaNaK-1	-0.676	416.3	-3.62	0.992

Viscosity of quaternary molten nitrate salts

Three pairs of quaternary molten salt mixtures were prepared such that the calcium nitrate content was varied within each pair. These mixtures were selected in order to clarify the effect of calcium nitrate on the viscosity of molten salts that may have solar energy applications. The compositions of these mixtures are given in Table 1, where the designators QA, QB and QC are used for each pair and the mol% of calcium nitrate is given by the suffix. The viscosity data for these quaternary nitrate mixtures are plotted in Figure 3 using the Arrhenius format. The data are organized such that open symbols represent the lower $Ca(NO_3)_2$ content of each pair and the filled symbols, the relatively higher one. The viscosity data clearly demonstrate that increasing the calcium nitrate content of any of the baseline mixtures increases the viscosity significantly. For example, mixture QA-10Ca displays viscosity values (filled square symbols, dotted line) that are about 50% greater than those of QA-5Ca (open square symbols, dotted line) over the entire temperature range of the measurements. The viscosity of some of these mixtures exceeded 100 cP at the low end of the temperature range of the measurements, approximately 100°C.

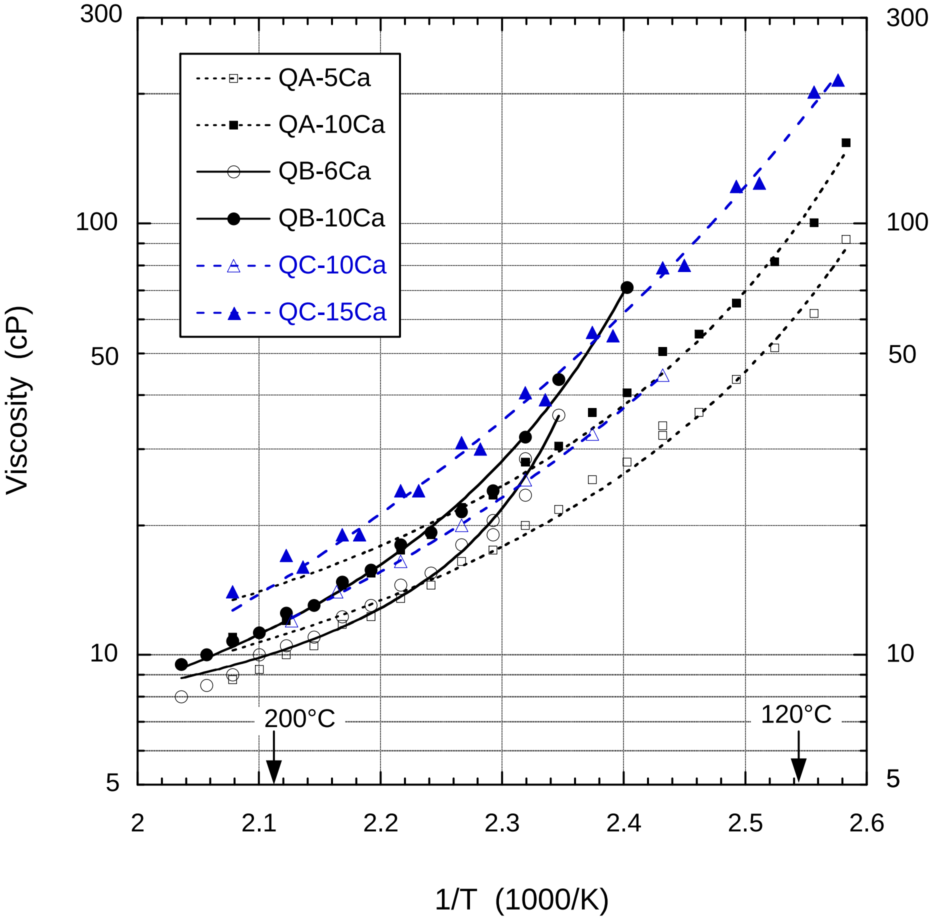

Figure 3. The effect of calcium nitrate on viscosity of several quaternary molten nitrate mixtures. Viscosity is plotted vs. temperature in Arrhenius format.

It is apparent from Figure 3 that the viscosity of quaternary molten nitrate salts does not depend linearly on inverse absolute temperature in this semi-log format but displays curvature. Such temperature dependence of viscosity can be described by the VFTH equation, as discussed in the preceding section. Data regression using the VFTH equation was performed as noted above and the VFTH equation parameters determined for the six quaternary salt mixtures are given in Table 4. The linear least squares regression coefficients, R in Table 4, indicate that good correlations were obtained for all mixtures. Similarly, the coincidence of data points and calculated lines in Figure 3 indicate the close correlation achieved by the VFTH equation for these sets of data.

Table 4. VFTH equation parameters for viscosity correlations of quaternary molten nitrate mixtures.

Mixture Label	A	B (K)	T_0 (K)	R	B/T_0
QA-5Ca	0.4914	75.96	61.64	0.994	1.2
QA-10Ca	0.5644	81.37	63.24	0.994	1.3
QB-6Ca	0.7084	21.51	127.6	0.990	0.17
QB-10Ca	0.7175	28.31	122.3	0.997	0.23
QC-10Ca	0.1958	138.2	42.39	0.999	3.3
QC-15Ca	-0.383	315.0	-1.79	0.994	-175.
QD	0.3460	392.3	29.97	0.999	13.1

The parameters of the VFTH equations for multi-component molten nitrate salts can be characterized according to several criteria which may provide additional confidence in the correlations; for example, should the viscosity data be extrapolated to higher temperatures than the range of measurements.

The parameter, T_0, is a characteristic temperature at which the viscosity increases without limit. T_0 corresponds to the glass transition temperature for those materials that display such second-order phase transitions, such as silicate and borate glasses.[17] Molten binary nitrate salts that contain appreciable amounts of calcium nitrate tend to supercool significantly and display a metastable glassy phase.[18] The glass transition behavior of quaternary molten nitrate salts was not determined; however, we observed that such melts tended to supercool and form very viscous, metastable glassy liquids that eventually solidified. Binary molten salts composed of KNO_3 and $Ca(NO_3)_2$ require at least 30 mol% calcium nitrate to form a glassy phase according to Angell.[19] Thus, the quaternary molten salts studied here may not form true glasses because the proportion of calcium nitrate may be insufficient. Regardless, the value of T_0 should be less than the glass transition temperature of a given material and certainly less than the liquidus temperature of non-eutectic mixtures, such as the quaternary nitrate salts studied here. Table 4 shows that the T_0 values (note that the units are Kelvin) are very low for all of the quaternary molten salt mixtures and thus these VFTH parameters are consistent with this criterion.

Another consistency criterion for VFTH parameters concerns the ratio B/T_0, which has been termed the "D" parameter, and is a metric of the strength of the glassy network formed by a given material.[13] A threshold value of D = 30 has been suggested as a lower limit for strong network-forming glasses, such as silica or borates.[20] In contrast, binary molten nitrate salts containing potassium nitrate and calcium nitrate, as discussed by Xu and Angell [21] would be categorized as "fragile" glasses. Table 4 shows that the D parameters for all of the quaternary molten salt mixtures are significantly less than 30 and thus the VFTH parameters are consistent with the experimental observations of metastable glassy-appearing liquids noted above.

Understanding the effect of molten salt composition on viscosity or the structure of the molten salt is beyond the scope of this report. However, we can observe that the behavior of these molten nitrate salt mixtures will likely depend, to some extent, upon the dissimilar characteristics of the ions that comprise the melt. Although the cations are spherical, the nitrate anion is a relatively large planar structure with three-pointed star symmetry.[16] That anion shape can affect viscosity due to the rotational restrictions that are inherent in its geometry.[7] The presence of a significant molar fraction of divalent calcium cations also affects the structure of the liquid phase as two nitrate anions are introduced into the liquid for each calcium cation, thereby upsetting the cation-anion balance from a purely space-filling aspect. These additional anions may also experience restricted rotation during fluid shear and increase the viscosity. However, as mentioned previously, the molten nitrate mixtures remain Newtonian fluids.

Effects of sodium nitrate and sodium chloride on viscosity

The effect of sodium nitrate on viscosity of multi-component molten salts was studied in one instance. A molten salt mixture was prepared based on the Ca-Li-K-nitrate eutectic in which 20 mol% of the KNO_3 was replaced by an equal amount of $NaNO_3$. The resulting composition is given in Table 1 by the entry designated QD. The viscosity measurements for these two mixtures are compared in the Arrhenius plot in Figure 4. The viscosity of the molten salt containing $NaNO_3$, represented by the filled circles, was somewhat higher than that of the Ca-Li-K-nitrate eutectic (shown by the square symbols) over the temperature range studied. The increase was approximately 10% to 20%, a much smaller effect than the increase caused by comparable additions of calcium nitrate. The VFTH equation parameters for mixture QD are given in Table 4.

The effect of sodium chloride on viscosity of multi-component molten salts was studied briefly because NaCl is the primary impurity in the least costly commodity grades of the constituents of the multi-component nitrate mixtures. A molten salt mixture was prepared by adding 5 mol% NaCl to the baseline Ca-Li-Na-K-nitrate mixture designated QC-10Ca in Table 1. Such an addition is much larger than the anticipated impurity content of 1% but was used to accentuate any effects due to chloride. The viscosity measurements for these two mixtures are compared in the Arrhenius plot in Figure 5. The viscosity of the molten salt containing NaCl, represented by the filled circles, was approximately 10% higher than that of the quaternary mixture (square symbols) over the temperature range studied. The behavior observed here agrees with the effect of chloride additions to KNO_3-$Ca(NO_3)_2$ molten salts reported by Rhodes.[18]

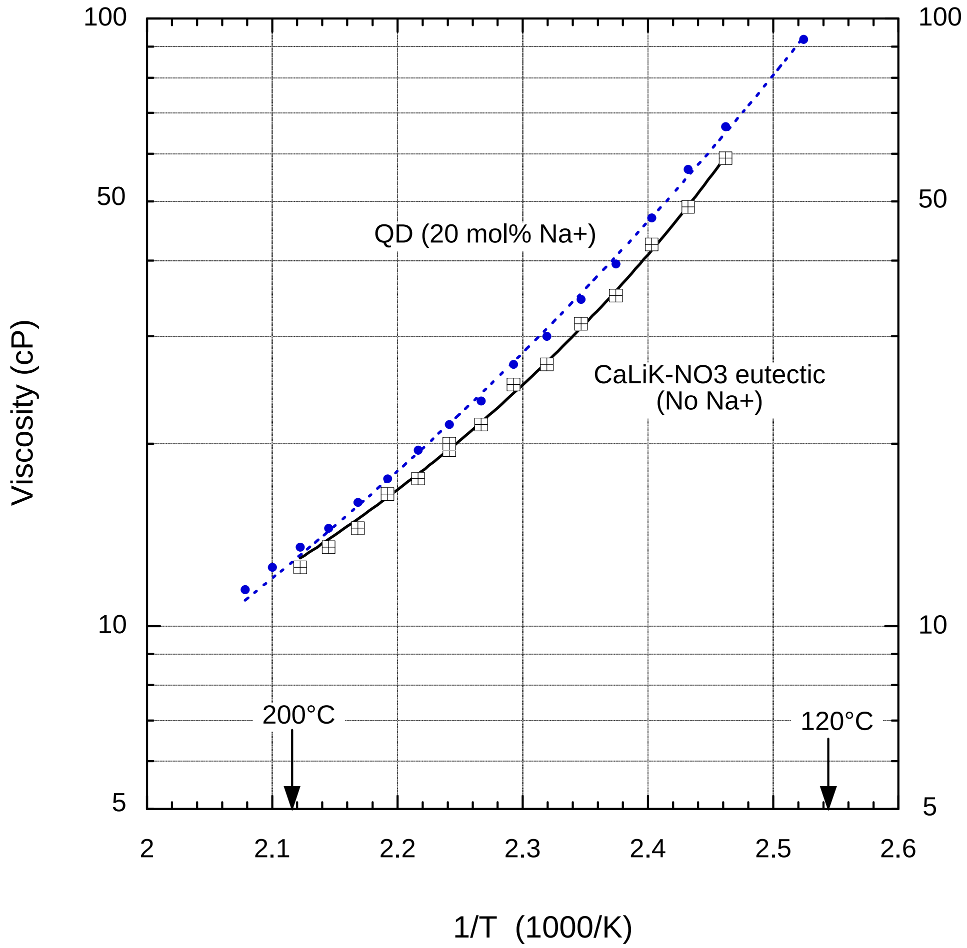

Figure 4. The effect of sodium nitrate content on viscosity of multi-component molten nitrate salts. The data compare the Ca-Li-K-nitrate eutectic and quaternary molten salt mixture QD. The compositions are given in Table 1.

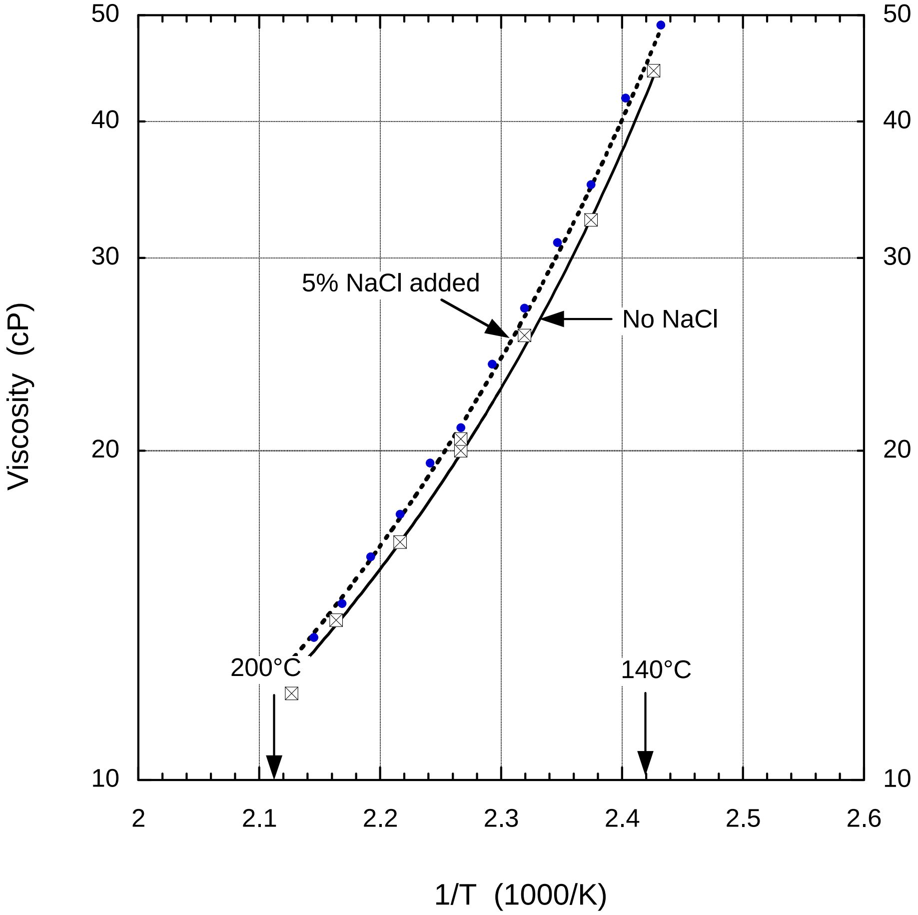

Figure 5. The effect of sodium chloride on viscosity of a quaternary molten nitrate salt. The data compare quaternary molten salt mixture QC-10Ca with and without 5 mol% NaCl.

Summary

The viscosity of several ternary and quaternary molten nitrate salt mixtures, consisting of various proportions of the nitrates of sodium, potassium, lithium and calcium, were determined experimentally at temperatures from near the liquid–solid phase transition temperatures to approximately 200°C. The addition of calcium nitrate to molten salts that were predominantly potassium nitrate and sodium nitrate significantly increased the viscosity. The viscosity of these mixtures increased proportionally to the calcium nitrate content. In contrast, the lithium nitrate content of molten salts that were predominantly potassium nitrate and sodium nitrate had relatively little effect on viscosity within the composition range studied. The temperature dependence of viscosity of mixtures composed of lithium, sodium and potassium nitrates followed the Arrhenius equation, while the addition of calcium nitrate produced a temperature dependence described by the VFTH equation.

Advanced designs of parabolic trough solar collector systems are intended to operate at temperatures up to 500°C, given suitable improvements in heat transfer fluids.[22] The quaternary molten nitrate salts described in this report may be acceptable for such applications; however, the fluid properties need to be specified over the operating temperature range of the system to enable engineering design calculations. Although the experimental apparatus used here was unable to measure viscosity above 200°C, the good fit of viscosity data versus temperature using the VFTH equation discussed above provides the basis to extrapolate viscosity of the quaternary nitrate mixtures to higher temperatures of engineering interest. Additional viscosity data for temperatures above 200°C and extrapolated viscosity values based on the VFTH equation are discussed in a subsequent report.[23]

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