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EFFECT OF COMPOSITION ON THE DENSITY OF MULTI-COMPONENT MOLTEN NITRATE SALTS

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Abstract

The density of molten nitrate salts was measured to determine the effects of the constituents on the density of multi-component mixtures. The molten salts consisted of various proportions of the nitrates of potassium, sodium, lithium and calcium. Density measurements were performed using an Archimedean method and the results were compared to data reported in the literature for the individual constituent salts or simple combinations, such as the binary Solar Salt mixture of NaNO3 and KNO3. The addition of calcium nitrate generally increased density, relative to potassium nitrate or sodium nitrate, while lithium nitrate decreased density. The temperature dependence of density is described by a linear equation regardless of composition. The molar volume, and thereby, density of multi-component mixtures can be calculated as a function of temperature using a linear additivity rule based on the properties of the individual constituents.

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Contents

Figures

Tables

EFFECT OF COMPOSITION ON THE DENSITY OF MULTI-COMPONENT MOLTEN NITRATE SALTS

Introduction

Multi-component molten salt mixtures formulated from the nitrates of sodium, potassium, lithium and calcium can 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] Physical property data concerning these multi-component nitrate molten salts are needed to properly design such systems and their components. Density is needed for system sizing calculations as well as being a constituent of many dimensionless groups used for heat transfer and fluid flow calculations, e.g., Prandtl number, Reynolds number, etc. Density data as a function of temperature is useful for assessing stability of thermoclines in such types of storage tanks. Molten salt density at the liquidus temperature can be used to determine the volume change associated with solidification and, thus, potential stresses in components subjected to freezing.

This report describes the results of experimental measurements of the density of multi-component molten nitrate salt mixtures. The effect of the constituent nitrate salts on density was determined over the temperature range of 150°C to 500°C. Such information can assist in formulating molten salts when other fluid properties are optimized, as well as for engineering design parameters. The density characteristics of the molten salt mixtures studied here may also provide fundamental information for relating the physical properties and the structure of these liquids.

Measurement Method

The density of molten salts was determined using an apparatus based on the Archimedean principle of buoyancy of immersed bodies. In the Archimedean technique, the density of a liquid is calculated from the buoyancy force exerted on a sinker of known mass and volume immersed in the liquid. The immersed sinker is suspended by a wire attached to a balance, which allows the buoyancy force to be determined by a differential measurement between the immersed mass and the baseline state of the sinker in air at ambient temperature.

The density, ρ, of a liquid sample is calculated according to Equation 1 below,

$$\rho = -(M_i - M_0) / V_s \{1 + 3C(T - T_0)\} \qquad (1)$$

where M_i and M_0 are the masses of the sinker immersed and at ambient temperature (T_0), respectively, V_s is the volume of the sinker at the ambient temperature, and C is linear thermal expansion coefficient of the sinker material. Equation 1 accounts for the correction of V_s due to expansion of the sinker at the temperature of the molten salt, T, in Celsius units.

The apparatus was configured to enable density measurements from 150°C to at least 500°C and consisted of a balance, crucible furnace and sinkers machined from titanium. A photograph of the apparatus is shown in Figure 1a. A Mettler Toledo Model PB1502 balance was used to weigh the sinkers. This balance has a hook below the electromagnetic transducer which allows masses to be suspended below the balance. The furnace was a Thermo Scientific Lindberg Blue M Model 1200 5-inch diameter crucible type with detached controller. The molten salts were contained in a stainless steel crucible fabricated from a 2.75-inch Conflat® nipple with one end welded closed, as shown in Figure 1b. The crucible was fastened to a supporting plate with stainless steel all-thread, which allowed its position to be adjusted in relation to the level of molten salt in the crucible and the distance below the balance. The assembled positioning fixture and crucible are shown in Figure 1b. The fixture was supported by the circular flange at the top, which rested on the insulation at the top of the furnace. Figure 1d shows one of the sinkers immersed in the molten salt crucible. Thermal insulation was placed below the balance platform to avoid heating the balance during the measurement periods and an air draft shield was placed to prevent air convection from impinging upon the top-loading balance pan and destabilizing readings.

Titanium was chosen as the sinker material because its density, 4.056 gm/cm^3 at 25°C, is about twice that expected for the molten salt mixtures.[3] Thus, a significant buoyancy force will result when the sinkers are immersed. The sinkers are shown in Figure 1c and weighed from 10 to 20 grams. Titanium is resistant to corrosion by molten nitrate salts and the thin tarnish film that developed after repeated immersions did not change the mass or volume so as to measurably affect the density determinations. The coefficient of thermal expansion of titanium is 8.6 x 10^{-6} m/m/K at 25°C.[4] The sinkers were suspended by 0.008 in. diameter stainless steel wire. The correction for the immersed wire was less than 0.01% of any measurement.

Surface tension forces on the wire connecting the sinker to the balance may also require a correction to the above equation for some liquids. However, this correction was negligible in the present study because the suspending wire had a very small diameter. As a refinement of the basic method, the Archimedean ‘double-bob’ technique employs two sinkers of known mass and volume that are lowered sequentially into the liquid whose density is to be determined.[6] The sinkers are connected by the same type of wire as the suspension wire, resulting in forces due to surface tension being cancelled in the calculations of density. The double-bob method has a further advantage in that it yields two density measurements per temperature. We used both the double-bob and single sinker techniques and obtained the same results.

The correct functioning of the apparatus and measurement technique was confirmed by measuring the density of deionized water at ambient temperature (20°C). A value of 0.997 gm/cm^3 was obtained compared to the published value of 0.9982.[5]

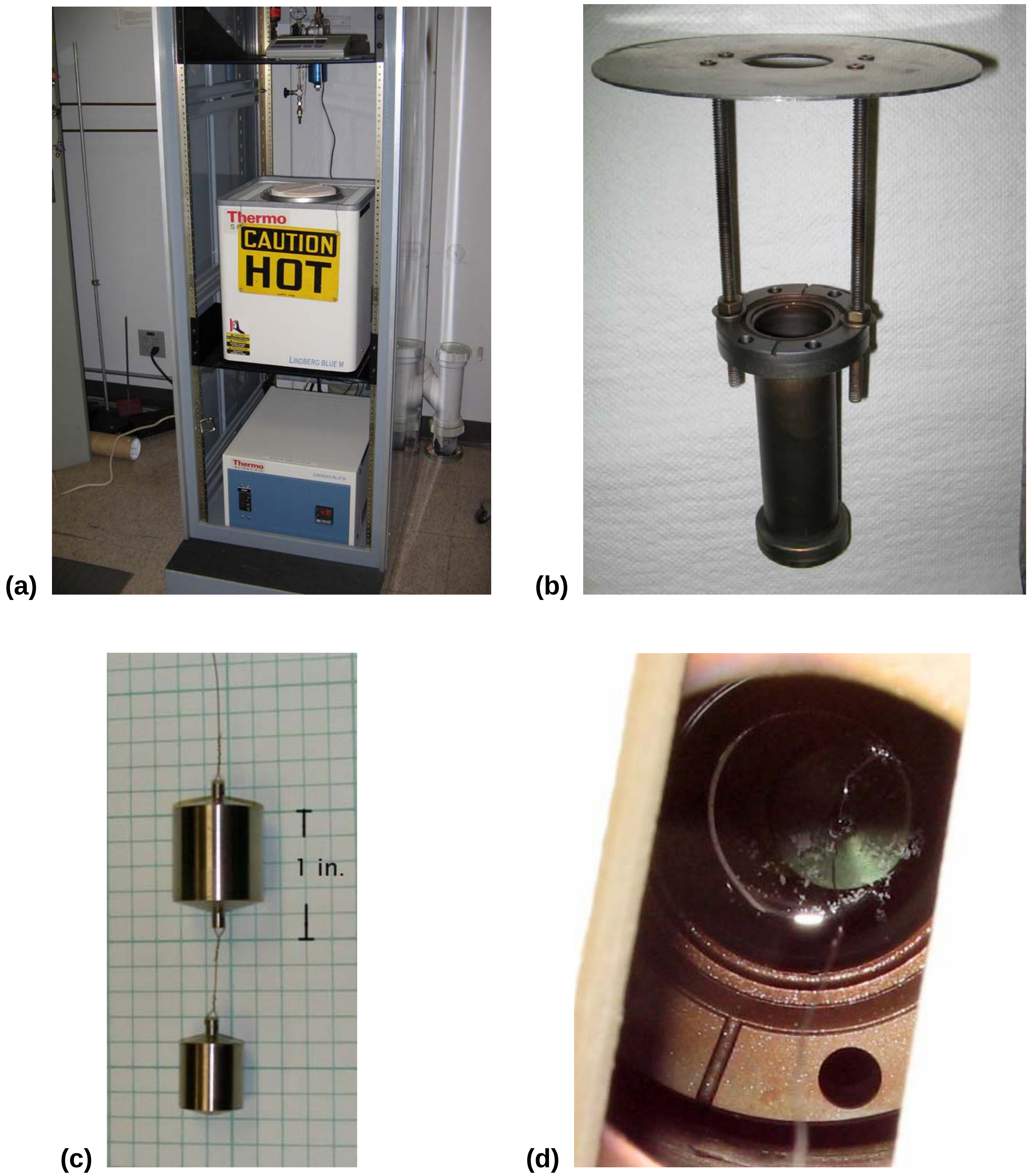

Figure 1. Photographs of apparatus for measuring density of molten salts.
a) Overall view of apparatus showing balance and furnace
b) Fixture to control positioning of molten salt cell in furnace
c) Titanium sinkers, “double-bob” configuration
d) Photograph of sinker immersed in molten salt cell.

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 poured into the density cell, allowed to solidify, and stored in sealed desiccated containers for later use.

The general procedure was to install the density cell fixture in the furnace, attain a steady-state cell temperature, then pre-heat the sinker above the molten salt to minimize the temperature change upon immersion. At least several minutes were allowed for the immersed sinker to achieve constant temperature and thus buoyant mass, as indicated by a constant balance reading. The temperature of the molten salt was measured immediately after removing the sinker using a calibrated Type K thermocouple. The sinkers were rinsed to remove the salt residue after each measurement and dried before the next immersion. The temperature of the furnace was increased in approximately 50°C increments to obtain density measurements over a range of temperature from somewhat above the liquidus temperatures of individual mixtures to 500°C.

The factors that affect precision of the measurements are the precision of the balance, initial measurements of the sinker masses and the mass of immersed support wires. These errors are all quite small. The balance had a precision of 0.01 gm and was calibrated prior to measurements. Thus, the maximum error introduced when measuring buoyant masses of 10 gm was 0.1%. The mass of the immersed wire was less than 0.004 gm, resulting in a maximum error of 0.04%, which would tend to cause systematic overages of this amount. The initial sinker mass was determined by a calibrated analytical balance to an accuracy of 0.0001 gm and thereby contributed insignificantly to errors. Similarly, the buoyancy correction due to weighing the sinkers in air was negligible.

Results and Discussion

Density of Quaternary Nitrate Mixtures

The densities of several quaternary nitrate salt mixtures were measured from about 150°C to 500°C. The compositions of these mixtures are specified in Table 1 and approximate the composition of quaternary mixtures having low liquidus temperatures, although the calcium nitrate was varied systematically to observe the effect on density. The density data are plotted in Figure 2, where density is given as gm/cm^3 and temperature in degrees Celsius. The individual mixtures are identified according to the mol-percent (mol%) of calcium nitrate to simplify discussion, e.g., Q-15Ca refers to the mixture given by the first line in Table 1, which contains 15 mol% calcium nitrate. The density of these molten salts were clustered around 2.00 gm/cm^3 at 150°C and 1.75 gm/cm^3 at the high temperature end. The density of this group of molten salts generally increased as the proportion of calcium nitrate increased over the range of 5 to 15 mol%. This result is consistent with literature reports for binary mixtures of Ca-K-NO_3.[10]

Table 1. Temperature dependence of the density of molten quaternary nitrate salts and the ternary Li-Na-K-nitrate eutectic. The coefficients 'A' and 'B' are defined in Eqn. 2.

$NaNO_3$ mol%	KNO_3 mol%	$LiNO_3$ mol%	$Ca(NO_3)_2$ mol%	A gm/cm^3	B (x 10^3) gm/cm^3/°C	Regression coefficient
Quaternary Mixtures						
15	45	25	15	2.1697	0.8222	0.998
20	38	30	12	2.1295	0.7416	0.999
18	45	29	8	2.1206	0.7375	0.999
23	46	26	5	2.1102	0.7330	0.999
Ternary Mixture						
18	45	37	—	2.0777	0.7352	0.998

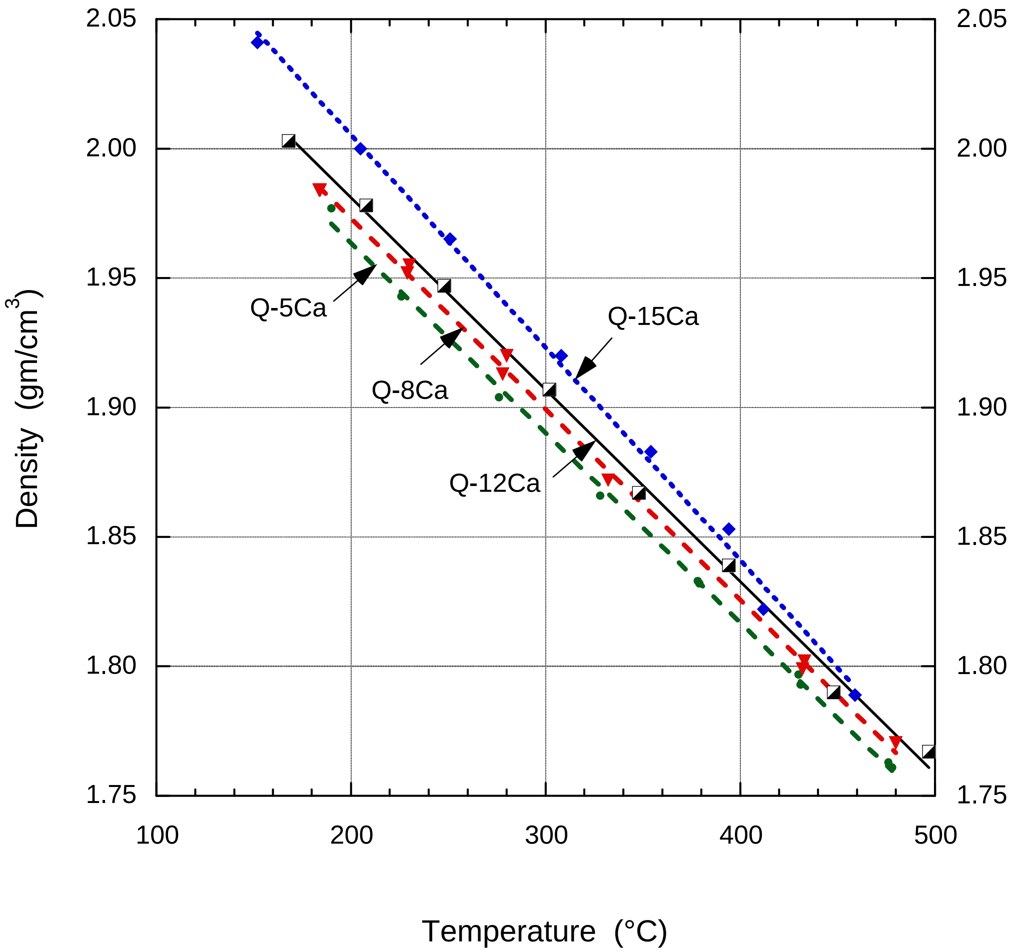

Figure 2. Density of several molten quaternary nitrate salts versus temperature. The variation in calcium nitrate content is indicated.

The temperature dependence of the density of all of the quaternary mixtures followed a linear equation as shown in Equation 2. Regression analysis of the density data was performed using Kaleidagraph (Synergy Software, Reading, PA, version 4.04). The values of the coefficients A and B for each mixture are collected in Table 1. The density data fit a linear equation extremely well, as demonstrated by regression coefficients nearly equal to unity for each molten salt shown in Table 1. The maximum deviation of any datum from its corresponding equation was less than 0.2%. The effect of calcium nitrate content, to increase density, is implied by the values of the parameter, A, which increase as the mol-percent of calcium nitrate increases.

$$\rho = A - B * T \qquad (2)$$

Density of Li-Na-K-NO_3 Eutectic

The density of the ternary eutectic of lithium, sodium and potassium nitrates was measured to further compare the effects of the constituents. The composition of the eutectic is 37 mol% lithium nitrate, 18 mol% sodium nitrate and 45 mol% potassium nitrate and the liquidus temperature is 120°C.[7] The density of this mixture does not appear to have been reported in the literature. The density values are plotted in Figure 3 in the same format as Fig. 2 for data obtained over a range from 150°C to 500°C. The density of an equimolar mixture of $NaNO_3$ and KNO_3 is also plotted (solid line) in Fig. 3 for comparison, based on data reported by Nissen [8] and by James and Liu.[9] The density of the ternary eutectic is significantly less than the molten salt that does not contain lithium nitrate. For example, at 400°C, the density of the ternary eutectic was 1.78 gm/cm^3 compared to 1.84 gm/cm^3 for the binary molten salt. The results obtained for the ternary eutectic molten salt are consistent with literature reports regarding the density of binary mixtures of Li-K-nitrate, which showed that additions of $LiNO_3$ reduced the density of such molten salt mixtures.[10] The temperature dependence of the density of the ternary eutectic followed Eqn. 2, in which the values of the coefficients A and B are 2.0777 and 0.7352, respectively. The deviation of the data from the linear equation was less than 0.2% over the range of measurements as shown in Table 1.

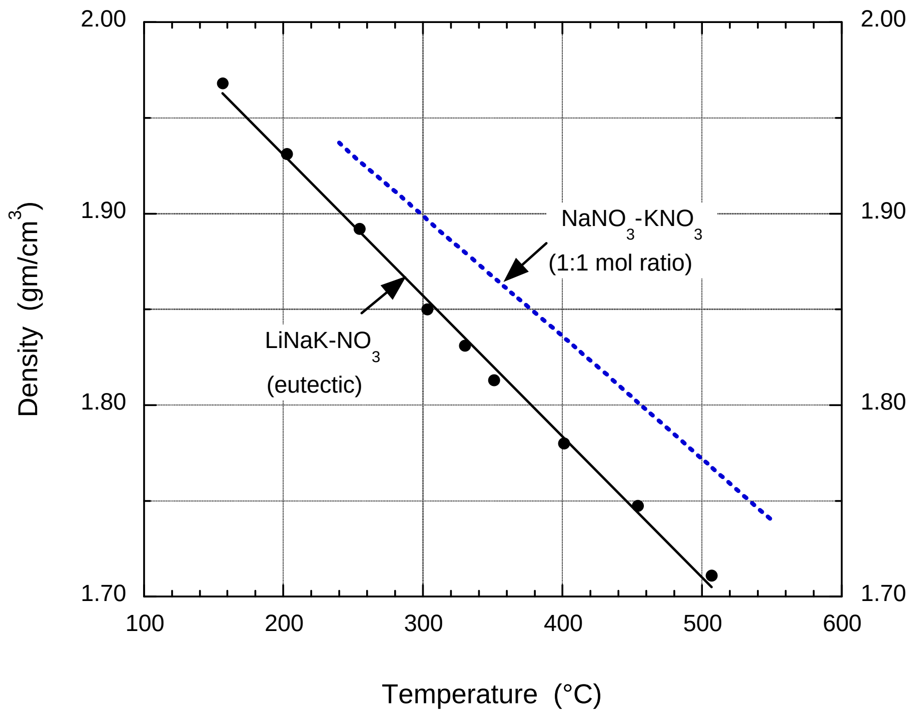

Figure 3. Density of Li-Na-K nitrate eutectic (solid line) versus temperature compared to a binary mixture of Na-K-nitrate (equimolar proportions, dotted line).

Literature Data for Binary Nitrate Mixtures

The effect of additions of either $LiNO_3$ or $Ca(NO_3)_2$ on the density of binary molten nitrate mixtures with KNO_3 has been studied. These data are plotted in Figure 4 and compared with the density of KNO_3. The lithium nitrate content of these binary melts was 24.5 mol% and 37.5 mol% [10] and the range of calcium nitrate in the mixtures was 20 mol% and 30 mol%.[11] The effect of lithium nitrate was to decrease the density of its mixtures with KNO_3 while density was found to increase as the proportion of calcium nitrate increased. The temperature dependence of density of all of these mixtures is linear and the 'A' and 'B' parameters for each mixture, according to Equation 2, are collected in Table 2. The parameters for temperature dependence of density for the pure salts, except calcium nitrate, are also collected in Table 2 along with the corresponding literature references for the data.

It is apparent that for binary mixtures, calcium nitrate and lithium nitrate have offsetting effects on density. This was also the qualitative observation from density measurements discussed earlier concerning quaternary and ternary mixtures. The following section describes how the effects of constituents in multi-component nitrate molten salts can be described quantitatively.

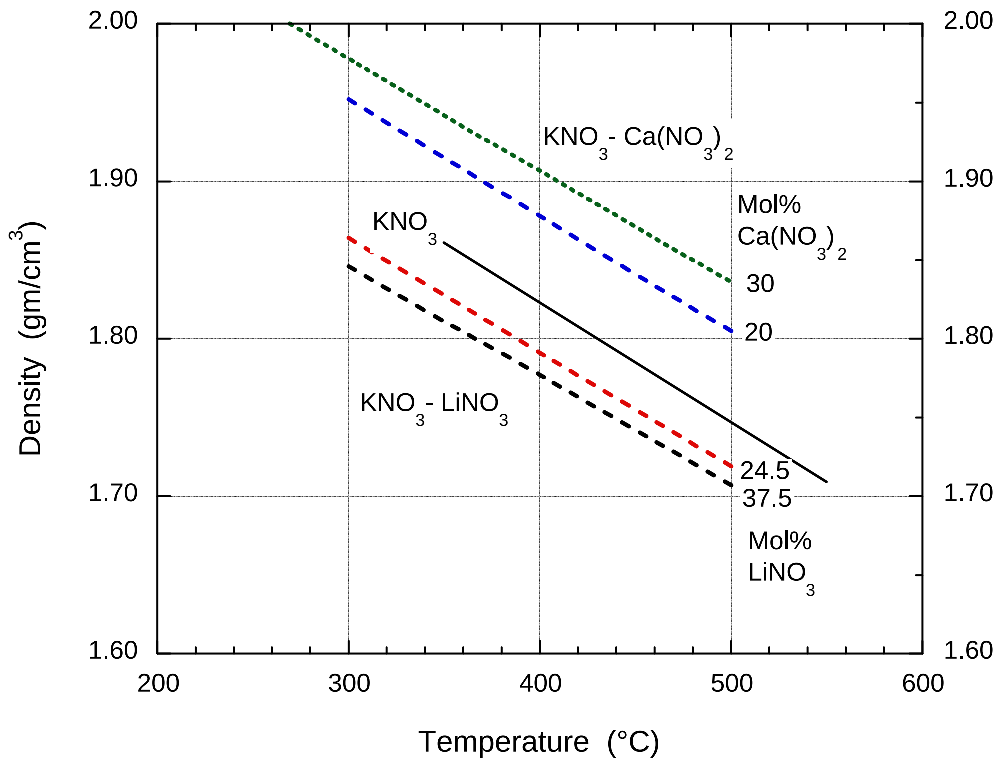

Figure 4. Literature data for the density of KNO_3 and its binary mixtures with either $LiNO_3$ or $Ca(NO_3)_2$ plotted versus temperature. The sources of data are References 12, 10 and 11, respectively.

Table 2. Summary of literature data concerning the temperature dependence of density of individual molten nitrate salts and several binary mixtures. The coefficients 'A' and 'B' are defined in Equation 2.

Salt	Composition mol%	A gm/cm^3	B (x 10^3) gm/cm^3/°C	Reference
KNO_3	100	2.1271	0.7601	12
$NaNO_3$	100	2.3339	0.7665	13
$LiNO_3$	100	1.922	0.556	10
Na-K-NO_3	equimolar	2.090	0.636	8
Na-K-NO_3	equimolar	2.134	0.773	9
Li-K-NO_3	24.5% Li+	2.083	0.735	10
Li-K-NO_3	37.5% Li+	2.055	0.729	10
Ca-K-NO_3	20% Ca++	2.172	0.735	11
Ca-K-NO_3	30% Ca++	2.192	0.713	11

Correlation of Density and Composition

The molar volume of binary mixtures of molten salts can be obtained by a “volumetric additivity rule” based on the molar volumes of the individual constituents.[10,11] This method is analogous to the Neumann-Kopp rule, which has been applied to calculate heat capacity of molten salts.[14] The volumetric additivity rule computes the molar volume of a mixture, $V_{m,x}$, according to the molar volume of each component, $V_{m,j}$, and its mol fraction, X_j, to as shown in Equation 3.

$$V_{m,x} = \Sigma X_j * V_{m,j} \qquad \text{all components, j} \tag{3}$$

Such a molar volume additivity rule can be applied to calculate the density of multi-component nitrate salts as a function of both composition and temperature. Correlating density with the composition of a molten nitrate salt mixture has a practical application and, possibly, fundamental significance. From a practical aspect, a correlation enables density to be calculated for mixtures other than those subject to measurements and establishes this property for design purposes. With regard to fundamental characteristics of molten salt behavior, the correlation may also indicate if mixtures behave ideally.

Density, ρ, is related to molar volume and molecular weight, MW, according to Equation 4. The molecular weight of a mixture, MW_x, is the weighted sum of its constituents, according to Equation 5. Given the molar volumes of the constituents, the density of a mixture can be calculated by substituting Equations 3 and 5 in Equation 4.

$$\rho = MW / V_m \tag{4}$$

$$MW_x = \Sigma X_j * MW_j \qquad \text{for all nitrate species} \tag{5}$$

The molar volumes for the nitrates of sodium, potassium and lithium were obtained from published density data for the individual salts by rearranging Equation 4, where MW is the molecular weight of the particular nitrate salt. The temperature dependence of the molar volume of individual nitrate salts is effectively linear and can be represented by Equation 6, comparable to the relation for density given earlier. The values of the coefficients A_v and B_v for each pure nitrate salt in the molten state are collected in Table 3.

$$V_m = A_v + B_v * T \qquad \text{where } A_v, B_v \text{ are constants for each nitrate salt} \tag{6}$$

In order to apply the volumetric additivity rule to quaternary mixtures, the molar volume of calcium nitrate must be known as a function of temperature. However, the density of calcium nitrate has not been determined experimentally, presumably because the pure molten salt has limited thermal stability, so its molar volume cannot be calculated directly. Mixtures of $Ca(NO_3)_2$ with alkali nitrate salts in which the latter are the majority constituent are stable at elevated temperature and, in particular, the density of mixtures of K-Ca-nitrate melts have been reported.[10] These density data provide the basis to calculate the molar volume of calcium nitrate in the molten state. The calculation of the molar volume of calcium nitrate is a special case of Equations 3-5 in which only two constituents are present. Solving for the molar volume of calcium nitrate ($V_{m,ca}$) involves a straightforward rearrangement, which produces Equation 7.

Equation 7 may be rewritten using the relation for the temperature-dependent molar volume of KNO_3 ($V_{m,k}$) to yield Equation 8.

Table 3. Temperature dependence of the molar volumes of individual nitrate salts. The coefficients 'A_v' and 'B_v' are defined in Equation 6.

Salt	A_v cm^3/mol	B_v $cm^3/mol\ °C$	Derived from
$NaNO_3$	38.675	0.02032	Ref. 13
KNO_3	45.776	0.02413	Ref. 12
$LiNO_3$	35.456	0.01347	Ref. 10
$Ca(NO_3)_2$	70.378	0.02565	Calculated

$$V_{m,ca} = (1/ X_{ca}) \{ (MW_x / \rho_x) - X_k * V_{m,k} \} \quad (7)$$

$$V_{m,ca} = (1/ X_{ca}) \{ (MW_x / \rho_x) - X_k * (A_{v,k} + B_{v,k} * T) \} \quad (8)$$

The density vs. temperature equations for K-Ca-nitrate mixtures containing mol fractions of 0.2 and 0.3 of $Ca(NO_3)_2$ appearing in Table 2 were applied to Equation 8. The resulting temperature-dependent relation for the molar volume of $Ca(NO_3)_2$ is given in Table 3. The molar volume of these mixtures calculated using the equation in Table 3 agreed with the published density data within 0.3%.

The molar volumes of the four constituent nitrate salts were used to calculate the density of quaternary mixtures, according to Equation 9. Although somewhat awkward to write in closed form, it is a simple calculation to implement by a spreadsheet, so that density may be computed for any composition and temperature within the range studied here. The results are shown in Table 4 and demonstrate that the calculated values agree well with the experimental data. The average deviation, over a range from 150°C to 500°C, was less than 0.4% for the four quaternary mixtures studied and only a single datum deviated by more than 0.5%.

The molar volume of the ternary eutectic mixture, Li-Na-K-NO_3, was also calculated using the volumetric additivity rule and was compared with the density data obtained by experimental measurements. The agreement between measured and calculated values was very close, as shown in Table 4. The average deviation, over a range from 150°C to 500°C, was less than 0.15% for the ternary mixture and the largest deviation was only 0.26%.

$$\rho = \Sigma X_j * MW_j \ / \Sigma X_j * (A_{v,j} + B_{v,j} * T) \qquad \text{for all nitrate salts, j} \quad (9)$$

The excellent agreement of density data and calculations implies that the multi-component molten salt mixtures behave ideally with regard to the space-filling properties of the individual ions. The ions appear to behave as "hard bodies" that fill space independently of their neighbors. The space-filling characteristics prevail despite the fact that the nitrate ion is planar but the cations are spherical.[15] The introduction of divalent cations in $Ca(NO_3)_2$ means that the cation-anion ratio of mixtures composed solely of alkali metal nitrates the structure of the liquid is not disrupted by additional anions. One must also recognize that the volumetric additivity rule applies over the relatively low temperature range at which multi-component molten nitrates are liquid despite the fact that the molar volume equations for the individual components were extrapolated well below their individual liquidus temperatures. Regardless, the volumetric additivity approach was demonstrated to work well and can be used to calculate density of multi-component molten salt mixtures.

Table 4. Goodness-of-fit of density calculated according to the volumetric additivity rule vs. experimental data for several multi-component nitrate salt mixtures.

$NaNO_3$ mol%	KNO_3 mol%	$LiNO_3$ mol%	$Ca(NO_3)_2$ mol%	Mean deviation %	Maximum deviation %
Quaternary Mixtures					
15	45	25	15	0.17	0.43
20	38	30	12	0.35	0.93
18	45	29	8	0.29	0.42
23	46	26	5	0.22	0.32
Ternary Mixture					
18	45	37	---	0.15	0.26

Summary

Experimental measurements of the density of multi-component molten nitrate salts were conducted to determine the effects of the constituents which included various proportions of the nitrates of potassium, sodium, lithium and calcium. Measurements of the density of multi-component mixtures were compared to data reported in the literature for the individual constituent salts or simple combinations, such as the binary Solar Salt mixture of $NaNO_3$ and KNO_3. The results demonstrate that calcium nitrate increases density, relative to potassium nitrate or sodium nitrate, while lithium nitrate decreases density, in agreement with observations for binary mixtures. The net result with regard to quaternary mixtures containing these constituents, as well as potassium nitrate and sodium nitrate, is to offset the contributions of the former salts. The density of quaternary salt mixtures that have relatively low liquidus temperatures is quite similar to that of binary Solar Salt. The temperature dependence of density is described by a linear equation regardless of composition.

The molar volume of multi-component mixtures can be calculated using a linear volumetric additivity rule based on the properties of the individual constituents. The calculation required deriving the molar volume of calcium nitrate, as the density of the pure salt has not been measured directly. Given the molar volume, the density of multi-component mixtures can be calculated as a function of composition and temperature with a high degree of accuracy. The molten salt mixtures shown ideal behavior in this regard.

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