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Effects of Cation Concentration on Thermophysical Properties of Solar Salts

Alan Michael Kruizenga, Karla Rosa Reyes, Kirsty Leong-Hau, and April Nissen

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Effects of Cation Concentration on Thermophysical Properties of Solar Salts

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Abstract

An assessment of the effects of cation concentration on the thermophysical properties of salts in the temperature range of 300 to 500°C was investigated. The latent heat and density exhibit a statistically relevant dependence upon mixtures, while heat capacity, viscosity, and thermal conductivity did not exhibit statistical differences among mixtures in the range of temperature studied. Heat capacity tended to be nearly flat while in the liquid state for mixtures at each temperature. Density of the mixtures decreases linearly with temperature. Mixture composition influenced density, with a relative variation up to 2% over the temperature range investigated. Viscosity decreased as a function of temperature in a non-linear fashion and methods used here tended to exhibit a higher value than literature values. Thermal conductivity used laser flash and transient wire methods. Transient wire found no differences between mixtures within repeatability of the measurement, while laser flash was found to not work well for molten nitrate salts due to the large error.

CONTENTS

1. Introduction 9

2. Heat Capacity and Latent Heat of $NaNO_3$-KNO_3 Nitrate Mixtures 11
    2.1 Thermophysical Measurements 11
    2.2 Results and Discussion 12
        2.2.1 Heat Capacity: Accuracy and Reproducibility 12
        2.2.2 DSC-Specific Heat Curve of $NaNO_3$/KNO_3 Mixtures 13
        2.2.3 Latent Heat and Melting Onset Temperature 15
        2.2.4 Heat Capacity at Liquid Phase 18
    2.3 Conclusions 21

3. Density of Molten $NaNO_3$-KNO_3 Nitrate Mixtures 23
    3.1 Introduction 23
    3.2 Measurement Method 23
    3.3 Results and Discussion 26
    3.4 Conclusion 29

4. Viscosity of Molten $NaNO_3$-KNO_3 Nitrate Mixtures 31
    4.1 Experimental Methods 31
        4.1.1 ATS Rheometry Method 31
        4.1.2 SNL Method 31
    4.2 Results and Discussion 33
    4.3 Conclusion 36

5. Thermal Conductivity of Molten NaNO3-KNO3 Nitrate Mixtures 37
    5.1 Method Description 37
        5.1.1 Laser Flash 37
        5.1.2 Transient Wire 37
    5.2 Results and Discussion 37
    5.3 Conclusions 39

6. Conclusions 41

7. References 43

Appendix A: Detailed Salt Compositions 45

Appendix B: Tabulated Data 47

Appendix C: Statistical Analysis of Heat Capacity (memo) 50

Distribution 54

FIGURES

Figure 1: Composition of Mixtures used. Melting Ranges Listed on Plot for Each Composition. Plot Reproduced From Data Available in [3] 10
Figure 2: Accuracy Determination of C_p by SNL Using Sapphire Standard 12

Figure 3: (a) Reproducibility Determination of C_p Taking Independent Measurements of Five 100% $NaNO_3$ Samples. (b) Uncertainty in the RSD of the Measurements 13
Figure 4: DSC-Specific Heat Curve for six $NaNO_3/KNO_3$ Mixtures..14
Figure 5: Example of Latent Heat Using a DSC-Specific Heat Curve......................................15
Figure 6: Latent Heat as Function of $NaNO_3$ Content. Top Plot Compares the Solid-Liquid Transition for Both Methods. The Bottom Plot Compares the Total Latent Heat (Solid-Solid Transitions Plus Solid-Liquid Transition) [6]. Differences of 4.7% are Observed at 64 Mole % (60:40 Mixture) Between Literature and Data in This Study..17
Figure 7: Autosampler Measurements of all Mixtures, Showing Reproducibility Among Measurements (a-e), With the Averages Reported in f. 19
Figure 8: Schematic of Geometric Changes due to Wetting Over the Course of the Test. Top Pictures Were Taken After Removing Sample From Instrument...20
Figure 9: Run-Averages as a Function $NaNO_3$ Composition, Where C_p for Each run was Averaged From 300 to 485°C...20
Figure 10: Comparison Between Data Collected at SNL (current data) and Literature [1,6,9]. Error Bars of 4.5% Were Assigned to SNL. 60% Data Based Upon Repeatability.....................21
Figure 11: Instrument set-up and Apparatus for Density Measurements of Molten Salts. (a) Apparatus of the Mass Balance and Furnace, (b) Crucible Used in the Molten Salt Furnace, (c) Titanium Sinker and Wire, and (d) Sinker Submerged in Molten Salt Crucible..........................25
Figure 12: (Top) Density of DI water and (Bottom) Density of Ethanol (EtOH) Both as a Function of Temperature (°C) 26
Figure 13: Density as a Function of Temperature Compared to Previous Work. Differences are Less than 0.6%..28
Figure 14: Mixtures as a Function of Temperature (°C) of Less than 60% $NaNO_3$ Have Lower Density Values, While Mixtures With Higher $NaNO_3$ Appear to Have Higher Density Values. Maximum Difference of 1.82% is Observed Between 60% and 75.98% Mixtures. Uncertainty Bars are 0.4% Based on RSD From Table 5. 28
Figure 15: ATS Replicates Were Made for all Mixtures With Relative Differences Resulting in Smaller Than 1%. 60:40 was Shown as an Example and the Behavior is Typical for all Mixtures 32
Figure 16: Geometries use for Viscosity Measurement. (a) Flat Plates and (b) Flat Plate/cup....32
Figure 17: SNL Accuracy Data for Plate/cup Method (top). Data was Taken Either High Temperature to low Temperature (High-low) or low Temperature to High Temperature (low-High) 100cP Standard Could Have Differences up to 13-14% Based on Loading. Replicates Were Made for all Mixtures With Relative Differences Resulting in 1%. 60:40 was Shown as an Example (Bottom)..33
Figure 18: Viscosity Results for 60/40 Nitrate Salt. ATS and SNL Data Were Both Performed Using a Flat Plate-Type Method, While SAND2001-2100 Used an Under-Damped Oscillating Method 34
Figure 19: SNL Data Display Similar Trends vs. Temperature and is Relatively Uniform Among all Mixture Compositions..35
Figure 20: Transient Wire Measurements are Within 7-11% of Literature Values.....................38
Figure 21: Transient Wire Data for all Mixtures. Repeat Measurements Were Taken for 100% $NaNO_3$ at 330°C and for the 45% Mixture at 400°C 38
Figure 22: Heat Capacity Data for Five Solutions of Varying $NaNO_3$ Concentrations With a Plot Showing Average Data as a Function of Temperature 51

Figure 23: Run-Averages Over 300°C to 485°C for Varying Levels of....................................52

TABLES

Table 1: Thermal Properties Measured With Corresponding Institution Listed 10
Table 2: Heat Capacity Parameter 12
Table 3: Latent Heat (J/g), Onset Temperature and Peak Temperature (°C) for the Melting of 100% $NaNO_3$ Salt 16
Table 4: Latent Heat [J/g], Onset Temperature and Peak Temperature (°C) for the Melting of 6 $NaNO_3$/KNO_3 Mixtures..16
Table 5: Density of the Nitrate Salt Mixtures Measured at Various Temperatures.....................27
Table 6: SNL Density Fitting Parameters 29
Table 7: Viscosity SNL Fitting Parameters...35
Table 8: Thermal Conductivity Fitting Parameters for Transient Wire Method.........................39
Table 9: SNL Heat Capacity Data 47
Table 10: Heat Capacity Data Extrapolated up to 600°C for NETZSCH...**Error! Bookmark not defined.**
Table 11: Density of the Nitrate Salt Mixtures Measured at Various Temperatures...................47
Table 12: Viscosity ATS Data...47
Table 13: Viscosity SNL Data...48
Table 14: Thermal Conductivity Data From Transient Wire Method. (Duplicate Measurements Listed)..48

NOMENCLATURE

CI	Confidence Interval
C_p	Heat Capacity
CSP	Concentrated Solar Power
DI	Deionized
DSC	Differential Scanning Calorimetry
Ethanol	EtOH
InSb	Indium Antimonide
KNO3	KNO_3
SCR	Solar Central Receiver
SNL	Sandia National Laboratories
NaNO3	$NaNO_3$
SQM	Sociedad Quimica y Minera S.A.
RSD	Relative Standard Deviation
TES	Thermal Energy Source

1. INTRODUCTION

Molten nitrate salts are currently used as a heat transfer fluid and thermal energy storage (TES) material in central solar receiver plants. These systems are designed and built using a mixture of salts composed of sodium ($NaNO_3$) and potassium (KNO_3) nitrates at a weight ratio of 60:40 (known as solar salt). The thermal properties of solar salt 60:40 (i.e. density, heat capacity, latent heat, viscosity, thermal conductivity) are well known and have been extensively studied at Sandia National Laboratories (SNL) since the 1980s [1]. The Solar Power Tower design basis document uses both historical measurements and experiences from the operation of Solar Two and is considered one of the best references by many in the industry.

In production of solar salt, there is a trade-off that must be considered. Should purified streams of $NaNO_3$ and KNO_3 be purchased and mixed on-site, as was done at Solar Two, or would it be better to purchase a premixed stream with known tolerances on cation concentration. The impact of differing concentrations of $NaNO_3$ and KNO_3 may alter the thermal properties from the previously compiled data in SAND2001-2100.

To investigate the effects of a pre-mixed salt versus an on-site mixed salt on its thermophysical properties, five mixtures of $NaNO_3/KNO_3$ at varying $NaNO_3$ mole % plus pure $NaNO_3$ were chosen as indicated by dotted lines in Figure 1. Solar salt is an off-eutectic mixture, so to determine if there were any trends in the thermal properties, different compositions that were both eutectic (44.7:55.3) and widely off-eutectic (75.98:24.02) were selected.

The density, heat capacity, latent heat, viscosity, and thermal conductivity of these mixtures were measured in this study, with an emphasis on liquid phase values. Molten salts, within central solar plants, are in liquid phase during normal operation, thus plant design hinge upon liquid values. Differences in liquid properties have been noted in literature for $NaNO_3$ and KNO_3 [2]. To assess the differences between institutions, attempts were made to duplicate measurements by multiple institutions. Table 1 lists the thermal property measurements made and the corresponding institution.

$NaNO_3$ and KNO_3, both refined grade – thermosolar product, were received from SQM in a powdered form (see Appendix A: Detailed Salt Compositions for further salt information). Nitrates were mixed in an alumina crucible, in proportions as given in Figure 1, heated to 500˚C, and held at temperature for several hours. Upon melting, the mixtures were turbid, presumably due to the decomposition of trace amounts of magnesium nitrate present in the salts. After approximately two hours, the solution became transparent, resembling water. Once transparent, the mixtures were removed, quenched in an actively cooled stainless vessel, and stored in a dry environment.

This report is a compilation of measurements made, where each section contains the given thermal property data. Values of data tabulated as a function of temperature were provided in Appendix B: Tabulated Data.

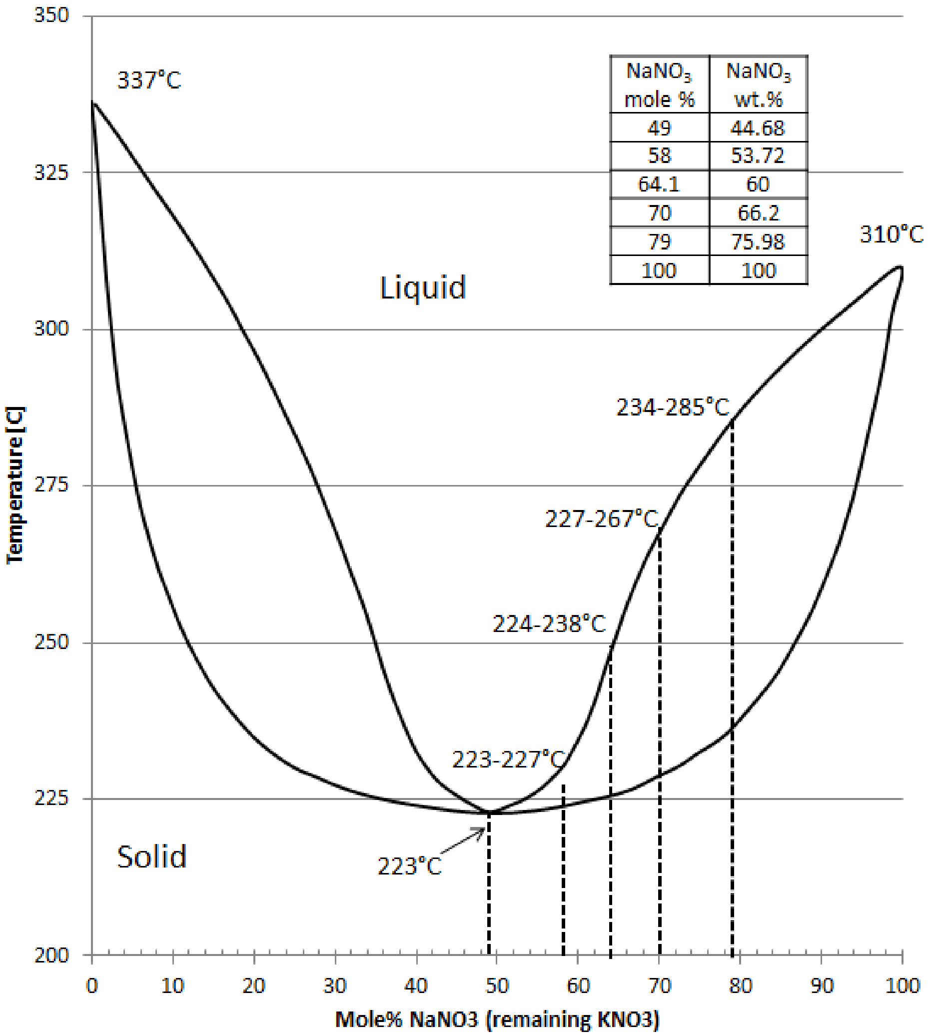


Figure 1: Composition of Mixtures used. Melting Ranges Listed on Plot for Each Composition. Plot Reproduced From Data Available in [3]

Table 1: Thermal Properties Measured With Corresponding Institution Listed

Thermal Property	**Institution Conducting the Measurements**	
Heat Capacity	SNL	-
Viscosity	SNL	ATS Rheometry
Thermal Conductivity	Dynalene	-
Density	SNL	-

2. HEAT CAPACITY AND LATENT HEAT OF $NaNO_3$-KNO_3 NITRATE MIXTURES

2.1 Thermophysical Measurements

The heat capacity measurements were carried out in Sandia SNL (Livermore, CA). In SNL, a simultaneous thermal analyzer (NETZSCH STA 449 F3) with a Pt furnace was used to collect the measurements. Data analysis at SNL was carried out by a comprehensive PC software package that allows for the computation of peak picking and onset temperatures, inflection points, partial area integration, specific heat, transformation energetics.

The Differential Scanning Calorimetry (DSC) measures heat capacity by heating a sample and measuring the difference between the heat flows from the sample and a reference (always an empty crucible) sides of a sensor as function of temperature. Heat flow values from three different measurements are required for the calculation of the heat capacity: baseline (two empty crucibles), standard (sample side: crucible with sapphire disc, 0.75 mm thick and reference side: empty crucible) and sample (sample side: crucible with sample and reference side: empty crucible). The baseline measurement is the response with both crucibles empty, yielding a signal bias inherent in the system. For the reference measurement, a sample with a well-known specific heat is tested. NETZSCH-supplied sapphire was chosen as the heat capacity reference and used by both institutions. The heat capacity of the experimental samples was calculated using the C_p ratio method define by the following equation:

$$C_{p\,sample} = C_{p\,ref} \times \frac{HF_{sample}}{HF_{ref}} \times \frac{m_{ref}}{m_{sample}} \qquad \textbf{Eqn1}$$

Where the heat capacity of the sample $C_{p\,sample}$ is calculated by using the known values for the heat capacity of the sapphire reference material ($C_{p\,ref}$), the heat flow of the sapphire (HF_{ref}) and the sample (HF_{sample}), and the mass of the sapphire (m_{ref}) and sample (m_{sample}).

All three of these measurements were performed in immediate succession. Accurate results are obtainable when the test parameters are identical for all three measurements. The parameters listed in Table 2 were easily controlled and remained constant over all measurements. Other parameters, such as the crucible's position on the sensor and the sample's contact on the bottom of the crucible, especially during melting and wetting out of the salts, were difficult to keep constant. These parameters led to irreproducible data and/or large standard deviations. Measurements could be made manually or with a robotic autosampler, ultimately the autosampler was used for salt mixtures, while positioning manually was used for accuracy of standards.

The advantage with the robotic autosampler was nearly continuous operation of the DSC, resulting in larger datasets. The robot could move crucibles, but could not add or remove anything within the crucible. Thus different aluminum crucibles were used for each step in the measurement process. The disadvantage is that any differences between aluminum crucibles used during the baseline, sapphire standard, and sample will contribute to error in determined value of heat capacity.

Table 2: Heat Capacity Parameter

Parameter	Condition
atmosphere (purge gas)	N_2
flow rate	40 mL/min
temperature range	35 – 500 °C
heating rate	20 K/min
mass of crucible and lid	Same mass
crucible material	Aluminum

2.2 Results and Discussion

2.2.1 Heat Capacity: Accuracy and Reproducibility

As mentioned in Section 2.1 Thermophysical Measurements, slight differences in the experimental parameters among the reference, baseline, and sample measurements can have a drastic effect on calculating the heat capacity. To determine the accuracy of the measurements, the heat capacity of a sapphire disc (thickness = 0.75 mm, obtained from NETZSCH company) was measured and the values were compared with standard known sapphire values. As shown in Figure 2, the heat capacity values measured at SNL and those collected from the standard have less than 0.7% difference. Therefore, all measurements were collected using this standard method prior to measuring the salts. The test method used is reflective of the manual method and indicates the best attainable accuracy, meaning that any uncertainty related to crucible location on the sensor or variability between crucibles as realized in the autosampler method would act to increase the error.

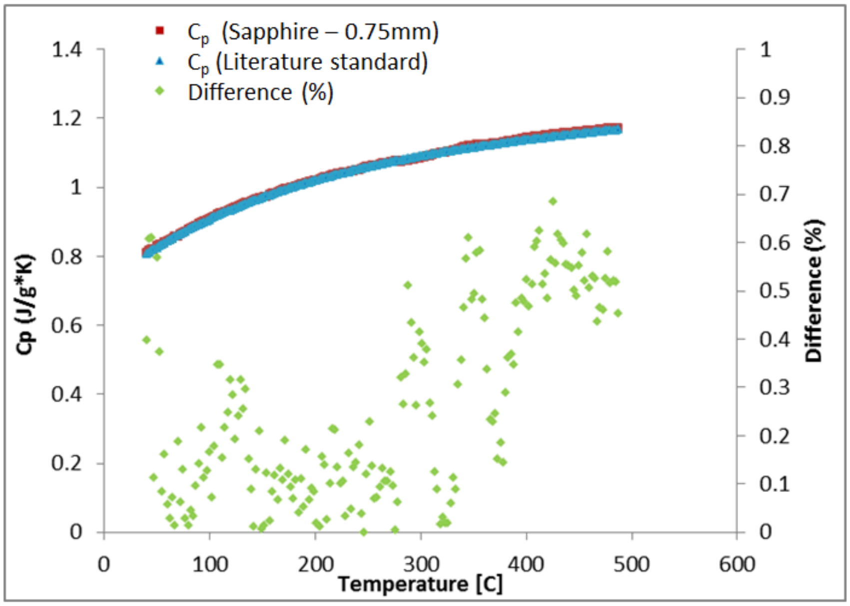


Figure 2: Accuracy Determination of C_p by SNL Using Sapphire Standard

To determine the reproducibility of the heat capacity measurements, five samples of $NaNO_3$ were measured. As shown in Figure 3 (a), the five samples have similar C_p curves. The relative deviation among the measurements was around 4% in solid phase and slightly above 5% close to near melting transients (in liquid phase). The uncertainty was determined by normalizing the standard deviation of measurements by the average, as shown in Figure 3 (b) and determined to be 5%.

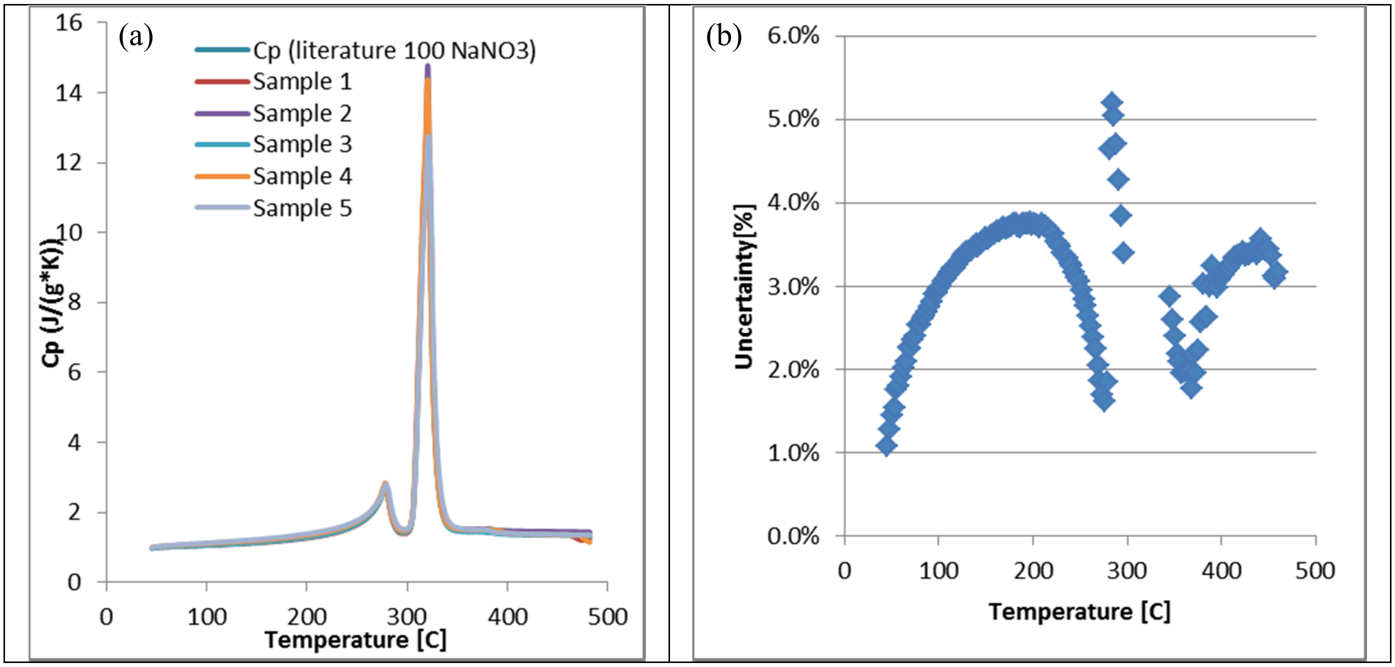


Figure 3: (a) Reproducibility Determination of C_p Taking Independent Measurements of Five 100% $NaNO_3$ Samples. (b) Uncertainty in the RSD of the Measurements

2.2.2 DSC-Specific Heat Curve of $NaNO_3$/KNO_3 Mixtures

The DSC-specific heat curves for six $NaNO_3$/KNO_3 mixtures, independently collected by SNL, are shown in Figure 4. For all six mixtures, a small peak in the temperature range of 100-150°C is due to the presence of KNO_3 nitrate transforming from an orthorhombic to a rhombohedral structure [2, 4]. For 100% $NaNO_3$, another peak at a higher temperature (250-300°C) is observed marking a structural order-disorder transition [5]. Although the underlying transition is distinctly different, these lower heat flow peaks correspond to a solid-solid phase transition in both cases. Upon melting the salts repeatedly, the 100-150°C peak disappeared.

In all six mixtures, the predominant peak denotes the solid-liquid phase transition. The 76% mixture, which is the mixture furthest from the eutectic, had a nonsymmetrical melting peak as evidenced by a double peak due to the existence of multiple phases.

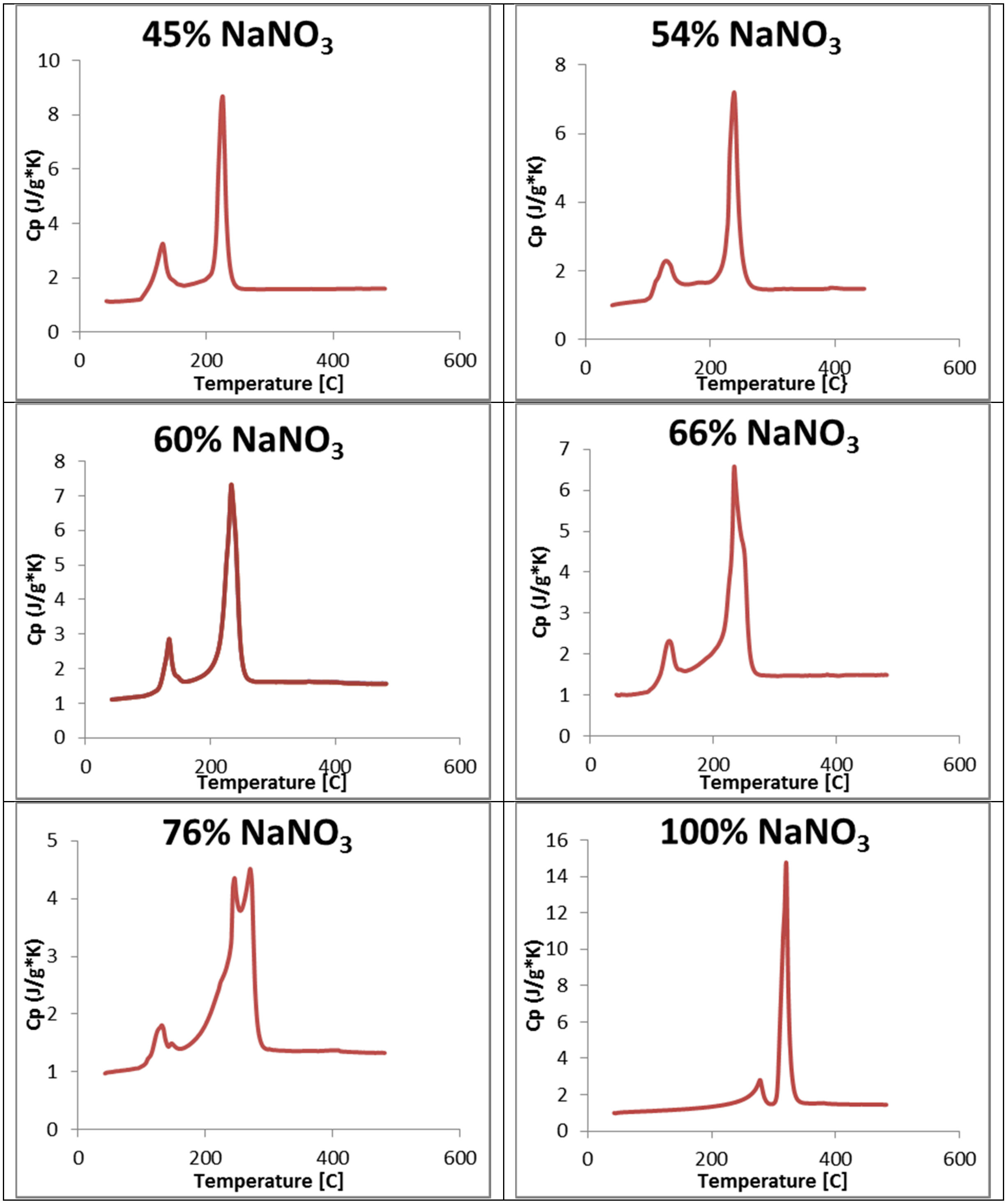
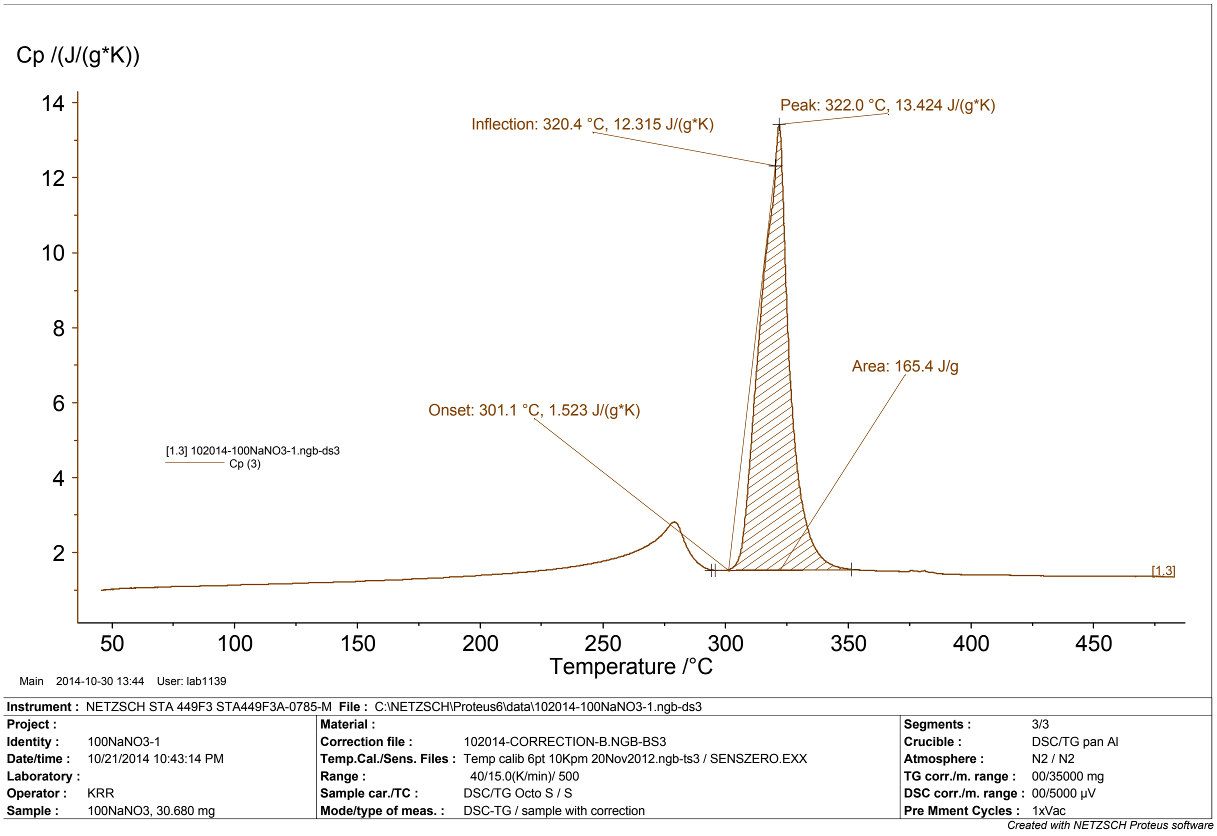


Figure 4: DSC-Specific Heat Curve for six $NaNO_3/KNO_3$ Mixtures

2.2.3 Latent Heat and Melting Onset Temperature

Using the Proteus software, the latent heat and the melting temperature of the salt mixtures were determined (Table 3). The latent heat was determined by integrating the area under the heat capacity curve in which the melting temperature was defined as the onset temperature. It should be noted that only the solid-liquid phase transition was investigated, which is the larger of the two peaks shown in Figure 4, and this is best illustrated in Figure 5. This procedure was utilized for replicate data in Figure 3a with the results summarized in Table 3.

The latent heat for $NaNO_3$ was found to be 162.5 J/g, which is within 6.5% of the existing data [2] as shown in Table 4. Figure 6 shows that for both the autosampler and manual methods that the data increases linearly with changing concentrations of cation. This behavior can be understood by simple rules of mixing between the $NaNO_3$ and KNO_3. One interesting observation is that all mixtures, with the exception of the 45 wt% mixture, are off-eutectic. Based on the phase diagram (Figure 1) it would be expected that a double peak would be observed in the solid-liquid endotherm. The 76% mixtures was the only example where this occurred and may simply indicate that within the resolution of the instrument any small differences in melting were not resolved.



Instrument : NETZSCH STA 449F3 STA449F3A-0785-M **File :** C:\NETZSCH\Proteus6\data\102014-100NaNO3-1.ngb-ds3

Project :		**Material :**		**Segments :**	3/3
Identity :	100NaNO3-1	**Correction file :**	102014-CORRECTION-B.NGB-BS3	**Crucible :**	DSC/TG pan Al
Date/time :	10/21/2014 10:43:14 PM	**Temp.Cal./Sens. Files :**	Temp calib 6pt 10Kpm 20Nov2012.ngb-ts3 / SENSZERO.EXX	**Atmosphere :**	N2 / N2
Laboratory :		**Range :**	40/15.0(K/min)/ 500	**TG corr./m. range :**	00/35000 mg
Operator :	KRR	**Sample car./TC :**	DSC/TG Octo S / S	**DSC corr./m. range :**	00/5000 µV
Sample :	100NaNO3, 30.680 mg	**Mode/type of meas. :**	DSC-TG / sample with correction	**Pre Mment Cycles :**	1xVac

Created with NETZSCH Proteus software

Figure 5: Example of Latent Heat Using a DSC-Specific Heat Curve

The Relative Standard Deviation (RSD) found here is 1.5%, indicating a difference when compared to literature. Observed differences between literature and data here may be due to baseline offsets. The example in Figure 5 has a flat baseline, making the integration more reliable. Unfortunately, not all of the mixtures were well-behaved in this way. Figure 4 show that the 76% mixture had the largest varying baseline.

Table 3: Latent Heat (J/g), Onset Temperature and Peak Temperature (°C) for the Melting of 100% $NaNO_3$ Salt

100% $NaNO_3$	*T onset (°C)*	*T peak (°C)*	*Latent Heat (J/g) [Solid-Liquid Transition]*
1	301.1	322	165.4
2	*	320.6	164.8
3	301.3	321.2	157.6
4	301.8	320.9	164.9
5	304.5	325.1	161.3
6	*	322	164.2
7	*	325.3	162.8
8	305.8	323.6	160.9
9	309.4	325.6	159.5
10	309.6	325.8	163.1
Average	304.8	323.2	162.5
std dev	3.4	2	2.4
Error (as RSD) %	1.1%	0.6%	1.5%

*Onset temperature could not be identified due to peak overlap.

Table 4: Latent Heat [J/g], Onset Temperature and Peak Temperature (°C) for the Melting of six $NaNO_3/KNO_3$ Mixtures

			Solid-Liquid Phase Transition (J/g)		*Total Value* (J/g)*
wt% $NaNO_3$	*T_{onset} (°C) [Manual Method]*	*T_{onset} (°C) [Autosampler]*	*Latent Heat [Manual Method]*	*Latent Heat [Autosampler]*	*Latent Heat [Autosampler]*
45	214.6	212.1	88.13	104.5	134.9
54	228.1	220.6	**	124.8	148.8
60	225.9	224.2	130.8	126.2	146.6
66	229.3	223.4	131.3	132.0	151.5
76	(double peaks)		223.4	150.0	163.9
100	305	-	162.5	-	
	* Sum of all phase transitions, solid-solid and solid-liquid, **Instrument error				

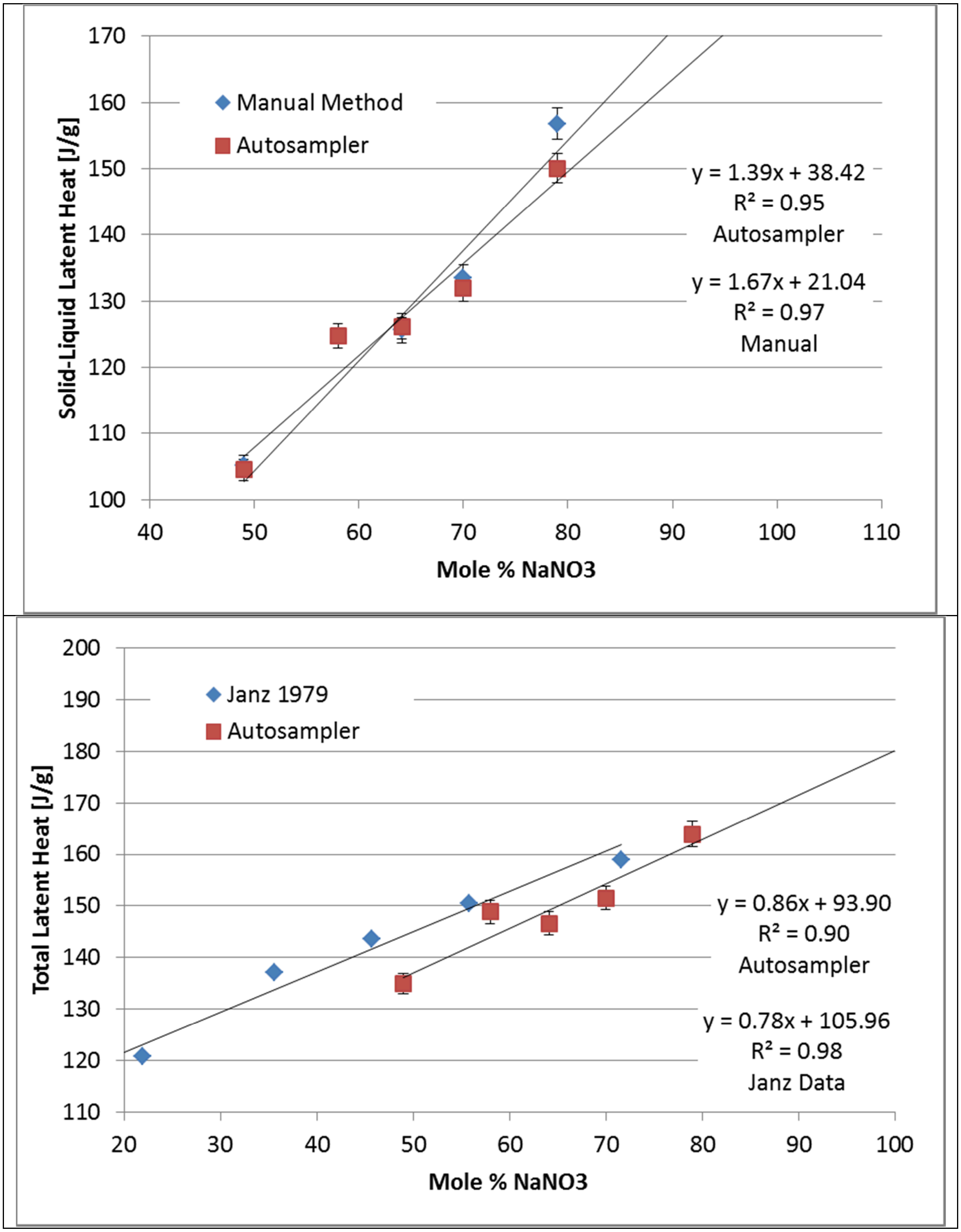


Figure 6: Latent Heat as Function of $NaNO_3$ Content. Top Plot Compares the Solid-Liquid Transition for Both Methods. The Bottom Plot Compares the Total Latent Heat (Solid-Solid Transitions Plus Solid-Liquid Transition) [6]. Differences of 4.7% are Observed at 64 Mole % (60:40 Mixture) Between Literature and Data in This Study

2.2.4 Heat Capacity at Liquid Phase

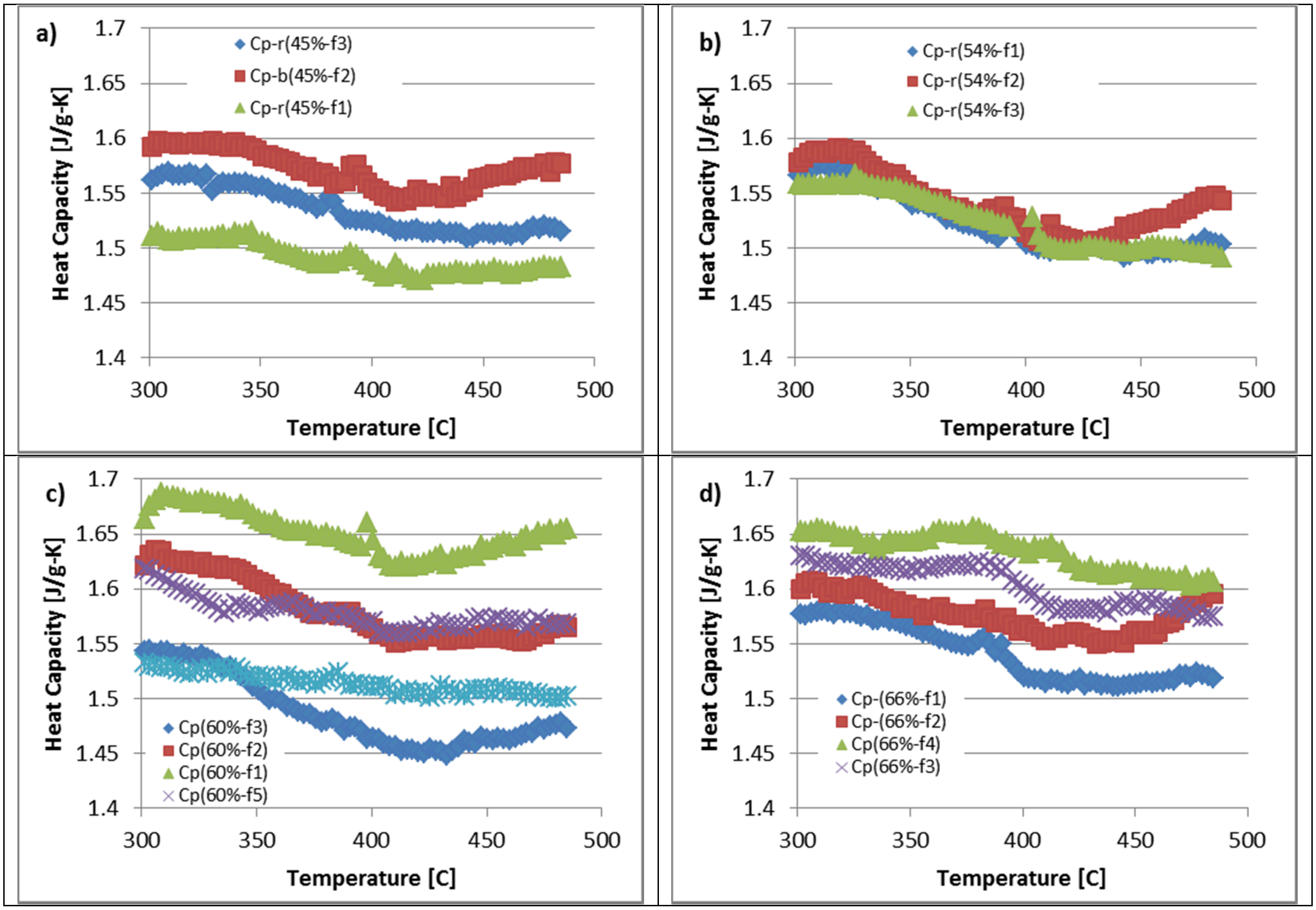
The heat capacity measurements of molten salts at liquid phase is challenging for several reasons. The primary challenge was with the tendency of molten nitrates to wet out and creep out of the measurement crucible, as was noted elsewhere [7]. In the NETZSCH's user manual, *Onset*, high temperature measurements (>400°C) should be avoided due to the salt creeping out of the crucible container and to slight changes in nitrite formation [8]. However, the timescale for nitrite formation is relatively slow (hours) in comparison to duration of the measurement (minutes). Due to these considerations measurements above 500˚C were not acquired.

Figure 7 shows the results of the autosampler test where at least three replicates were performed with each mixture. Five independent measurements were made of the 60:40 mixture due to its relative importance in this study. In Section 2.2.1 Heat Capacity: Accuracy and Reproducibility proved that there is nothing inherently wrong with the instrument or method that was used during the course of the study, so any issues with reproducibility must be due to a factor related to the sample. It was observed that wetting during the course of the experiment led to nearly no salt remaining on the bottom of the sample, as shown in Figure 8. Different crucibles were utilized in an attempt to mitigate this problem (aluminum and platinum), but the behavior did not change. The inherent lack of reproducibility sample-to-sample at a fixed composition is thought to be due to an evolving geometry over the course of the measurement. Due to this complexity it is difficult to understand with certainty what the difference in heat capacity is between salt mixtures.

In view of the scatter of the data an analysis was performed to clarify if any dependence on heat capacity could be observed as a function of mixture composition. The approach taken generated a single C_p average per run (or "run-average") by averaging the heat capacity from each temperature point over the range of 300°C to 485°C. Sequential heat capacity measurements on a given experiment, i.e., for a determined mixture, should be highly correlated and cannot be treated as an individual measurement. Results of this analysis are shown in Figure 9, where each data is the output of analysis using run-averages values for the mixtures in the whole range of composition. Statistical tests for a difference in mean heat capacity (assuming constant variance) include analysis of variance, which also assumes that the data are normal in distribution, and the Kruskal-Wallis test. The P-value for analysis of variance is 0.066 and the P-value for the Kruskal-Wallis test is 0.077. Within the 5% significance level there is no difference in the mean heat capacity for different $NaNO_3$ concentrations. For information on the statistical treatment please see

Appendix C: Statistical Analysis of Heat Capacity (memo) for further details.

Figure 10 is a graphical comparison between the SNL data generated in this study and mixtures found in literature, for 45% and 60% mixtures [1, 6, 9]. Based upon repeatability of data, as shown in Figure 7, an error of 4.5% was assigned to the 60% mixture. Maximum differences between the 60% mixture taken here and literature range from 6-10% at 300°C and tend to decrease with increasing temperature. The 45% mixtures have a maximum difference of 10%, which also decreases with temperature. It should be noted that trends in the heat capacity are different in literature. The 60% mixtures both increase with temperature, while the 45% mixture decreases as a function of temperature. It also should be noted that data found here has some slight decline with temperature, but the relative difference from values at 300°C and 475°C are all approximately 3% more over flat, which is different relative to these literature data. By contrast Janz data has a 13% (45% mixture) and Dudda has a relative change of 7% (60% mixture), while the SAND2001-2100 data is relatively flat, only a 2% change.



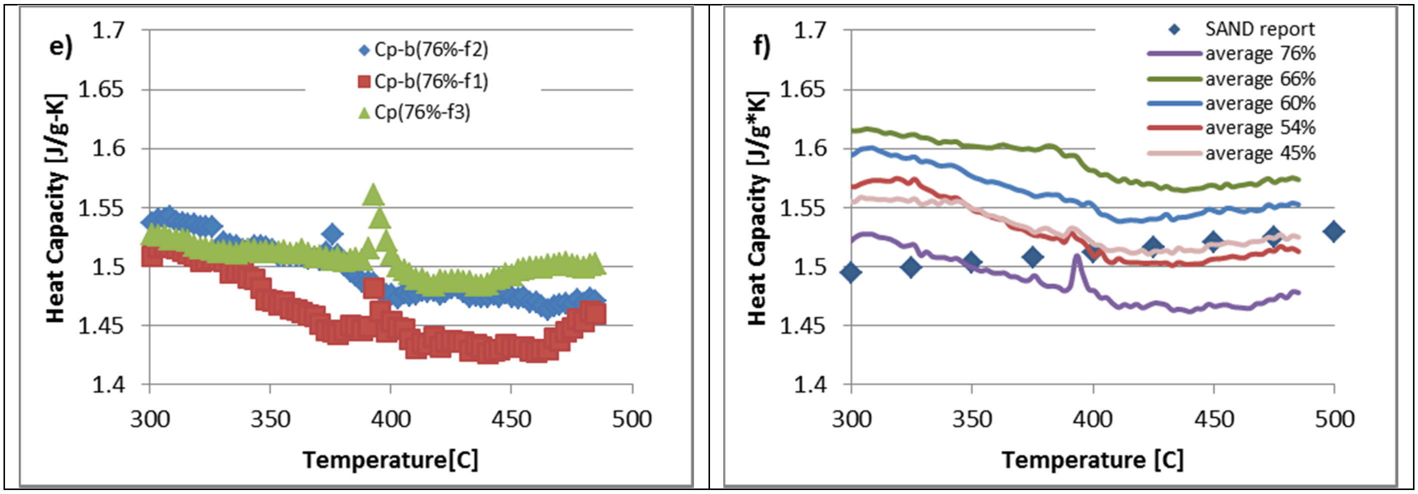


Figure 7: Autosampler Measurements of all Mixtures, Showing Reproducibility Among Measurements (a-e), With the Averages Reported in f.

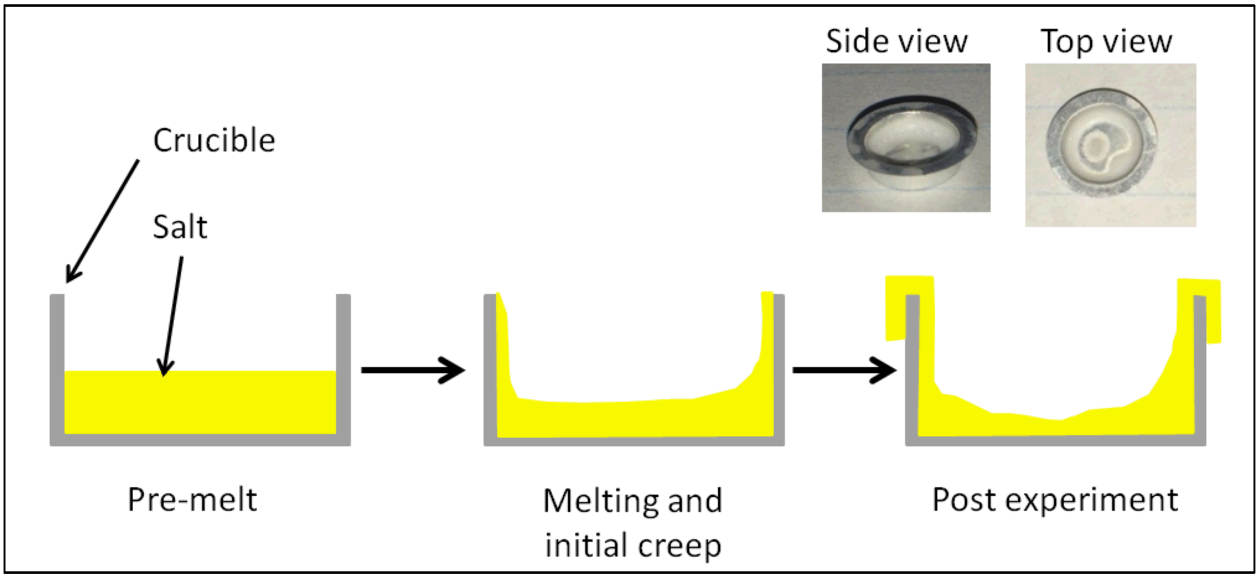


Figure 8: Schematic of Geometric Changes due to Wetting Over the Course of the Test. Top Pictures Were Taken After Removing Sample From Instrument

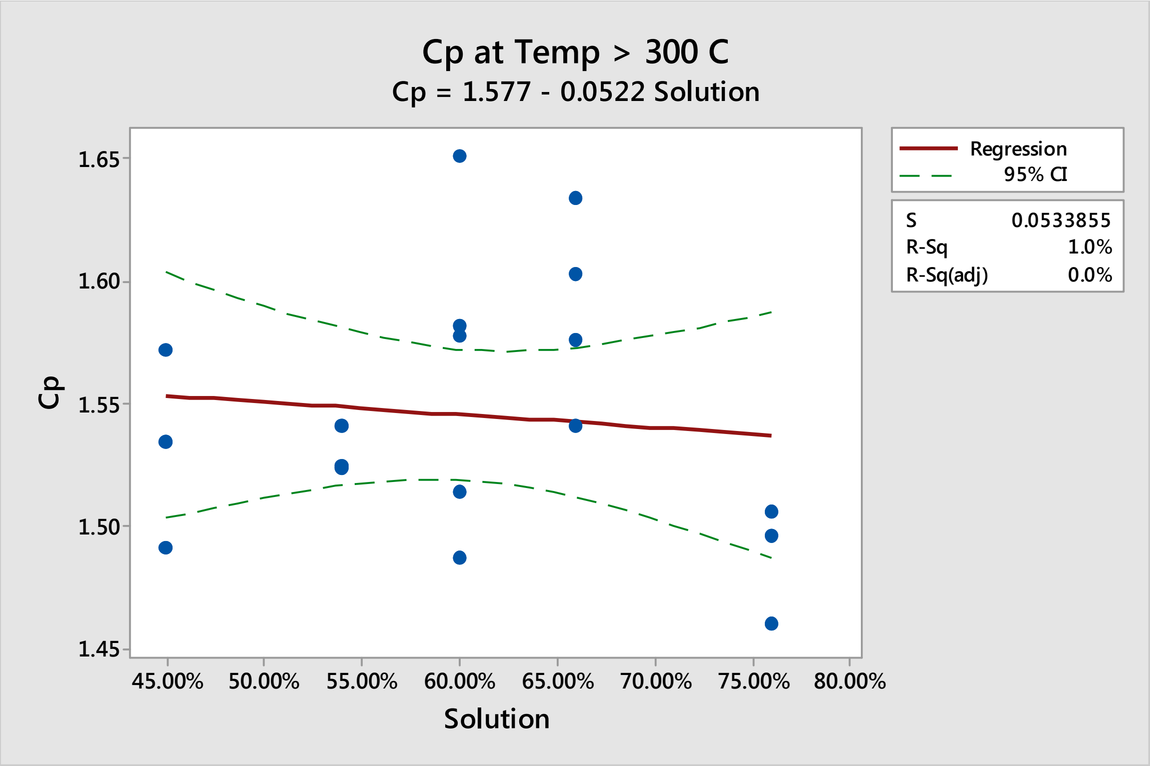
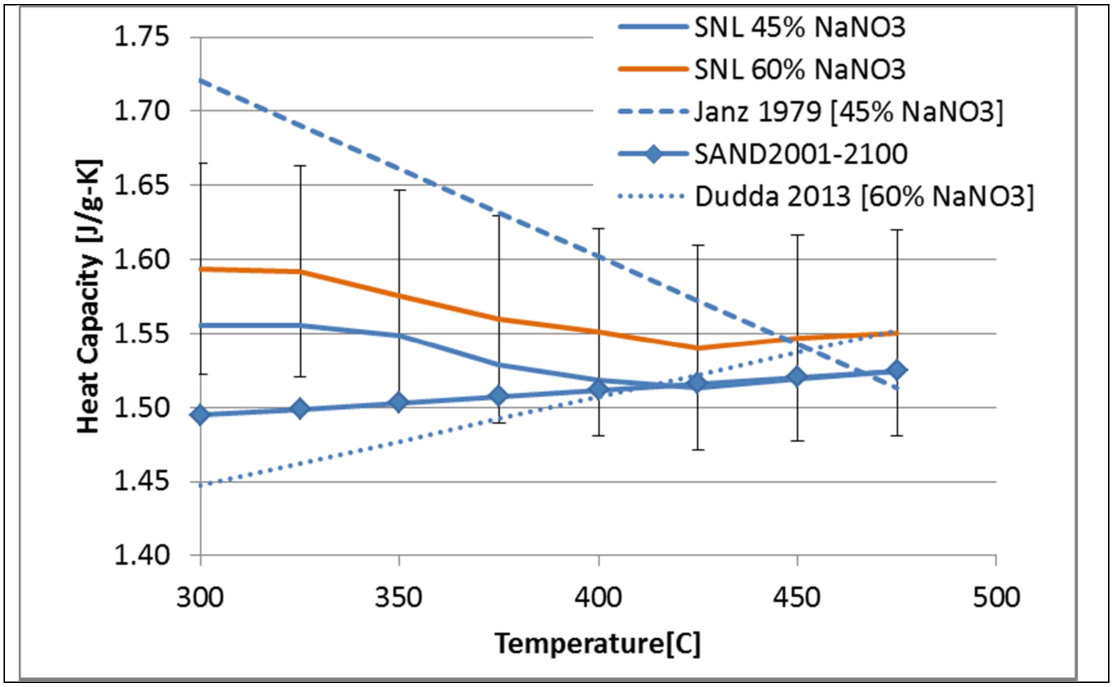


Figure 9: Run-Averages as a Function NaNO$_3$ Composition, Where C_p for Each run was Averaged From 300 to 485°C



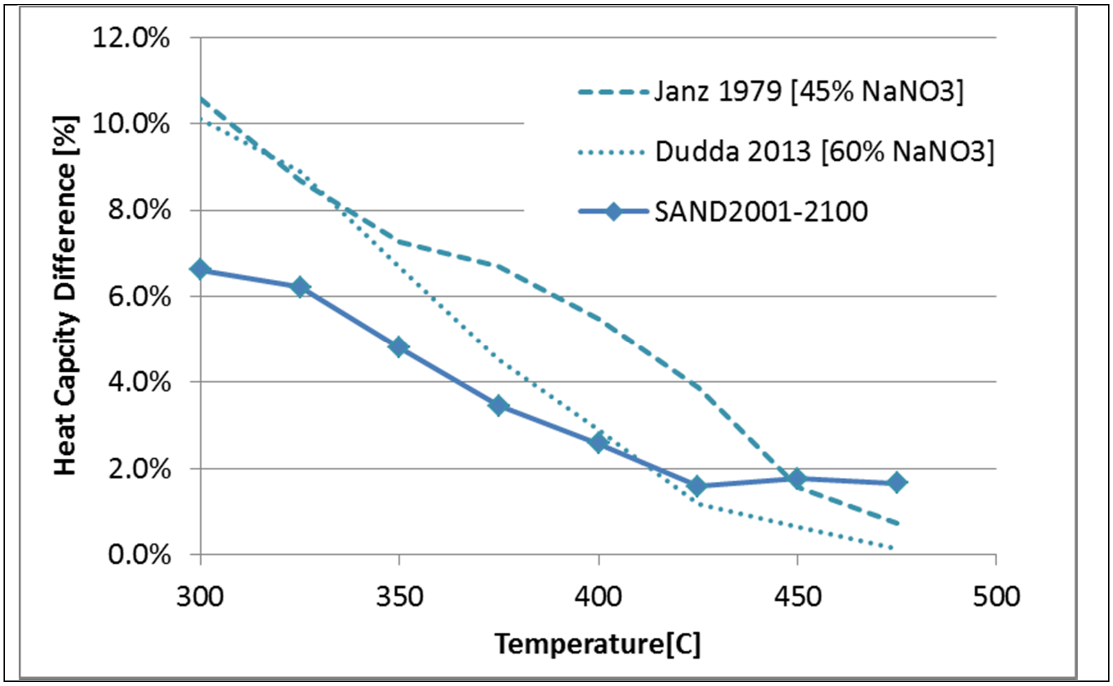


Figure 10: Comparison Between Data Collected at SNL (current data) and Literature [1, 6, 9]. Error Bars of 4.5% Were Assigned to SNL. 60% Data Based Upon Repeatability of Measurements

2.3 Conclusions

The heat capacity and latent heat of five mixtures were investigated. It was found that the method used when compared to a sapphire standard was accurate to within 1% or less and was repeatable. The latent heat increased linearly as function of $NaNO_3$ content. In comparison to literature, the maximum difference in latent heat was 4.7% difference at 60:40.

The method used was checked for reproducibility with 100% $NaNO_3$ from room temperature to 500°C and in the liquid state had a 5% RSD. However, experience here indicates that mixtures tended to be less repeatable, due to the salt wetting out of the crucible. Data was taken in at least triplicate for each mixture over the temperature range of interest. No trend with $NaNO_3$ concentration in the 45% - 66% range between 300 – 485 °C could be discerned within the uncertainty of the measurement at the 5% significance level.

3. DENSITY OF MOLTEN $NaNO_3$-KNO_3 NITRATE MIXTURES

3.1 Introduction

Fluid density is important for the purpose of gauging the heat transport capability of coolant. Multi-component molten salts are utilized as heat transfer fluid in parabolic trough solar fields to improve system performance and to reduce the cost of electricity [10, 11]. These salts typically are composed from nitrates of $NaNO_3$, KNO_3, lithium, and calcium, and display liquidus temperature below 100°C [10, 11]. Physical property data on these multi-component nitrate containing molten salts are needed to properly design such systems and their components. Density data as a function of temperature is useful for assessing the stability of thermoclines in such types of systems.

$NaNO_3$ and KNO_3 nitrates are inorganic phase change materials. These salts are commonly used in large-scale solar power plants producing electricity. The molten salt consisting of a mixture of $NaNO_3$ and KNO_3, or so called solar salt, with composition 60:40 wt.%, has a melting point of 223-227˚C (according to Figure 1) and has been used successfully as a thermal energy collection and storage fluid in a large-scale solar central receiver (SCR) demonstration system at temperatures up to 565°C [12].

This section describes the results of experimental measurements on the density of variable composition solar salt, around the 60:40 composition. The effects of cation concentration on the density of molten nitrate salts were determined over the temperature range of 325°C to 625°C.

3.2 Measurement Method

The density of molten salts can be calculated using the experimental apparatus shown in Figure 11 and the Archimedean's principle, which governs the buoyant forces acting on immersed bodies [13]. The principle states that the upward or buoyant force acting on an immersed body is equal to the weight of fluid displaced by the body. 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 2 below:

$$\boldsymbol{\rho = -(M_i - M_0) / V_s [1 + 3C(T - T_0)]} \qquad \textbf{Eq. 2}$$

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 2 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 is configured to enable density measurements from 150°C to at least 650°C and consists of a balance, crucible furnace and sinkers machined from titanium. A Mettler Toledo

Model PB1502 balance is 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 is a Thermo Scientific Lindberg Blue M Model 1200 5″ diameter crucible type with detached controller. The molten salts are contained in a stainless steel crucible fabricated from a 2.75″ Conflat® nipple with one end welded closed. The crucible is fastened to a supporting plate with stainless steel all-thread, which allows its position to be adjusted in relation to the level of molten salt in the crucible and the distance below the balance (Figure 11[b]). The fixture is supported by the circular flange at the top, which rested on the insulation at the top of the furnace. Figure 11(d) shows the sinker immersed in the molten salt crucible. Thermal insulation is placed below the balance platform to avoid heating the balance during the measurement periods and an air draft shield is placed to prevent air convection from impinging upon the top-loading balance pan and destabilizing readings.

Titanium is 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 [13]. Thus, a significant buoyancy force will result when the sinker is immersed. The sinker weighs about 10 grams (Figure 11[c]). 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 effect the density determinations. The coefficient of thermal expansion of titanium is 8.6 x 10^{-6} m/m/K at 25°C [14]. The sinker is suspended by 0.008″ 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 also may 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.

The correct functioning of the apparatus and measurement technique was confirmed by measuring the density of deionized (DI) water and ethanol at ambient temperature (20°C) and at a temperature ranging from ambient to the solvent's boiling point. For DI water, a value of 0.9981 g/cm^3 is obtained compared to the published value of 0.9982 [15]. The calculated percent error is 0.01%. For ethanol, a value of 0.7859 g/cm^3 is obtained compared to the published value of 0.7874 g/cm^3. The calculated percent error is 0.19% [15]. The calibration standard graphs of density versus temperature are shown in Figure 12. Density measurements were repeated twice for reproducibility.

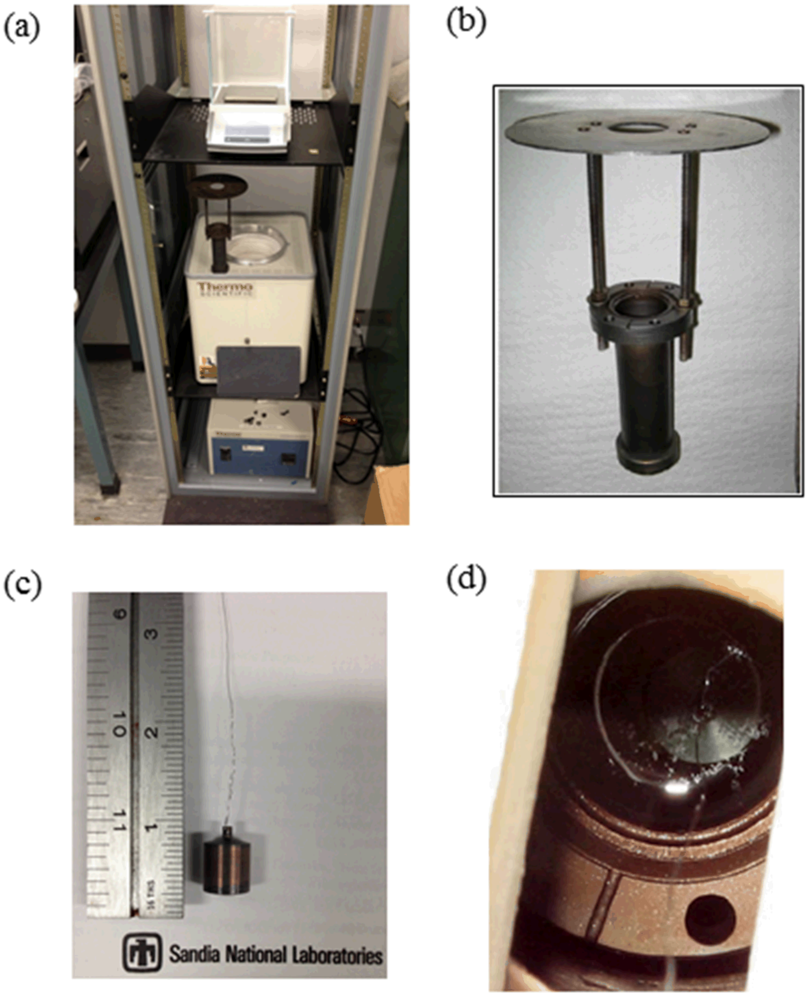
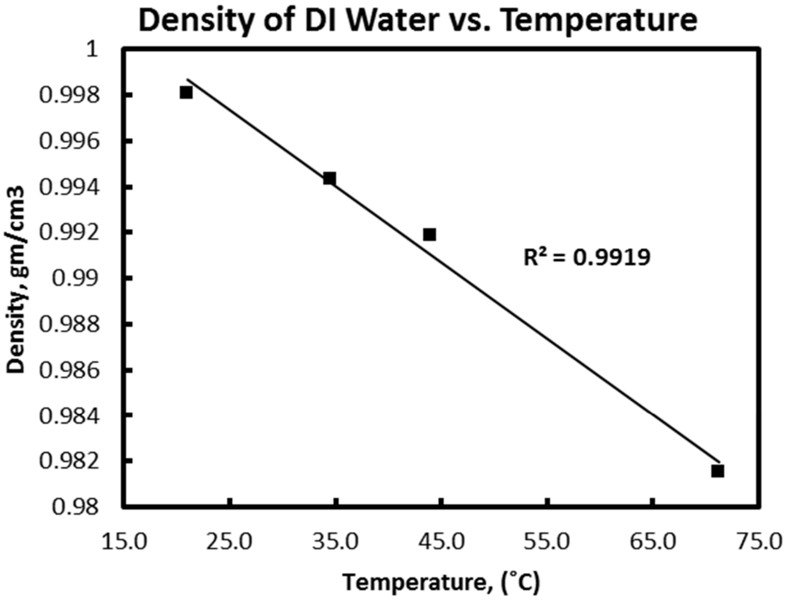


Figure 11: Instrument set-up and Apparatus for Density Measurements of Molten Salts. (a) Apparatus of the Mass Balance and Furnace, (b) Crucible Used in the Molten Salt Furnace, (c) Titanium Sinker and Wire, and (d) Sinker Submerged in Molten Salt Crucible



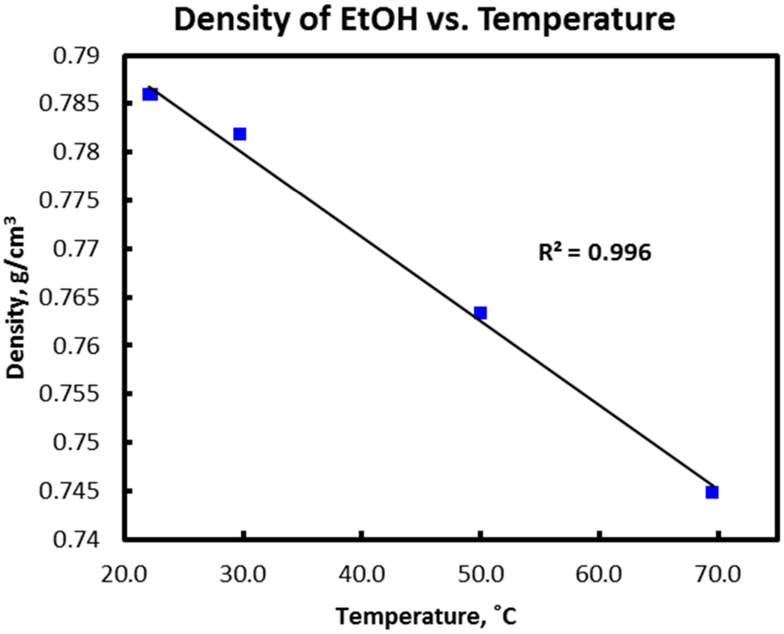


Figure 12: (Top) Density of DI water and (Bottom) Density of Ethanol (EtOH) Both as a Function of Temperature (°C)

3.3 Results and Discussion

The densities of the nitrate salt mixtures were measured from 325˚C to 625˚C (calculated by using Equation 2)). The compositions of these nitrate mixtures vary in mol % or wt.% of $NaNO_3$ and are specified in Figure 1. The densities of the various salt mixtures range from approx. 1.694 gm/cm^3 to 1.908 gm/cm^3 (Table 5), clustering around 1.90 gm/cm^3 at 325˚C and 1.694 gm/cm^3 at 625˚C. The density increases as the wt.% of $NaNO_3$ increased in the mixture from 49 to 100 mol

%. These density values are similar to those published in literatures for solar salts with molar ratios of $NaNO_3$ to KNO_3 is 60:40[15]. For all the nitrate salt mixtures, the salt density decreases linearly with increasing temperature

The R-squared values (R^2) listed in Table 5 is near unity. As the wt % of $NaNO_3$ increases in each mixture, the density closely resembles that of pure $NaNO_3$. Interestingly, the slopes of the density data for the mixtures are relatively similar.

Values obtained for 60:40 mixtures were compared to previous measurements compiled in a design basis document [1]. Figure 13 shows the difference in density data produced in this study compared to literature values, which also was measured using similar buoyancy techniques. The maximum observed difference was less than 0.6%.

Figure 14 is a plot of data obtained over all mixtures for this study. Trends for all mixtures are similar in that the density decreases linearly as a function of temperature. Mixtures at or below 60% $NaNO_3$ have lower densities in comparison to mixtures of higher $NaNO_3$ concentrations. The maximum difference in density between 60:40 is 1.82% as observed in the difference calculation on the left axis of
Figure 14.

Finally, Table 6 is a compilation of these data with an estimated uncertainty of 0.4% based upon RSD over two measurements. Behavior of all mixtures is linear and fits are generated using least squares methods (Table 6).

Table 5: Density of the Nitrate Salt Mixtures Measured at Various Temperatures

	Density (g/cm^3)					
Temp (°C)	**44.68wt.%**	**53.72wt.%**	**60wt.%**	**66.23wt.%**	**75.98wt.%**	**100 wt%**
325	1.863 (0.007)	1.883 (0.001)	1.885 (0.001)	1.908 (0.000)	1.912 (0.003)	1.897
375	1.839 (0.008)	1.851 (0.001)	1.851 (0.001)	1.878 (0.004)	1.878 (0.004)	1.867
425	1.809 (0.002)	1.818 (0.001)	1.817 (0.002)	1.849 (0.002)	1.849 (0.005)	1.838
475	1.784 (0.002)	1.786 (0.001)	1.785 (0.001)	1.818 (0.003)	1.818 (0.002)	1.809
525	1.759 (0.001)	1.758 (0.002)	1.756 (0.002)	1.788 (0.001)	1.788 (0.003)	1.783
575	1.732 (0.004)	1.731 (0.001)	1.734 (0.001)	1.761 (0.003)	1.759 (0.001)	1.765
625	1.694 (0.007)	1.703 (0.000)	1.701 (0.006)	1.735 (0.004)	1.727 (0.000)	1.743
*Average values over two sample measurements. The standard deviation is listed in parentheses following the density values.						

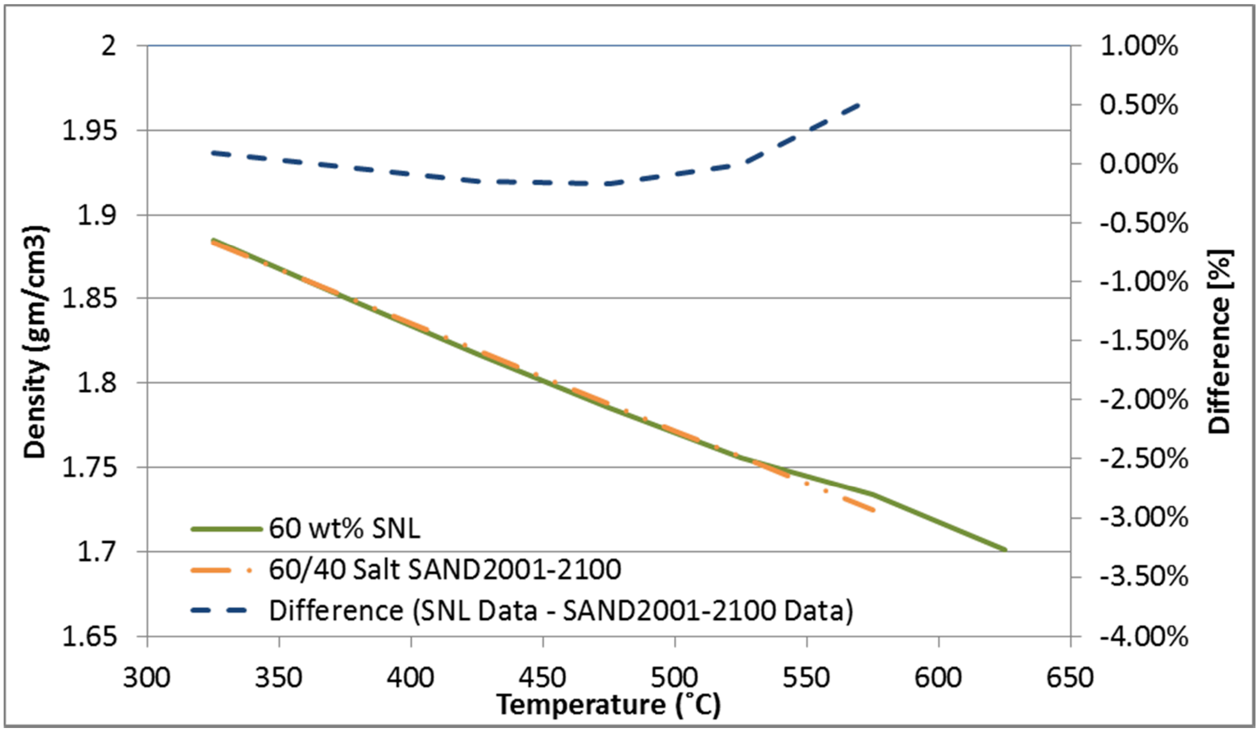


Figure 13: Density as a Function of Temperature Compared to Previous Work. Differences are Less than 0.6%

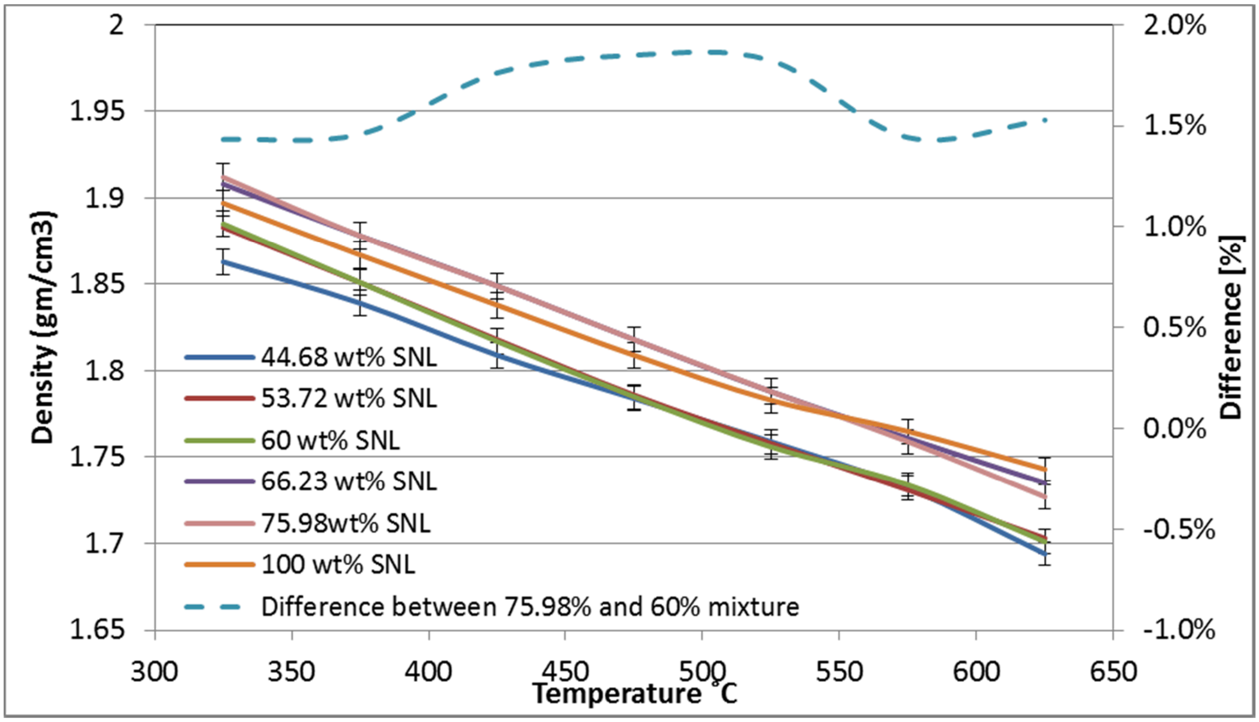


Figure 14: Mixtures as a Function of Temperature (°C) of Less than 60% $NaNO_3$ Have Lower Density Values, While Mixtures With Higher $NaNO_3$ Appear to Have Higher Density Values. Maximum Difference of 1.82% is Observed Between 60% and 75.98% Mixtures. Uncertainty Bars are 0.4% Based on RSD from Table 5

Table 6: SNL Density Fitting Parameters

$NaNO_3$ wt%	Coefficient values Y=A+B*T [g/cm^3]	Estimated % uncertainty of measurement	R^2 value
100%	A= 2.0585 B= -0.0006	0.4%	0.992
75.98%	A= 2.1094 B= -0.0006	0.4%	0.999
66.73%	A= 2.0959 B= -0.0006	0.4%	0.999
60%	A= 2.0772 B= -0.0006	0.4%	0.996
53.72%	A= 2.0744 B= -0.0006	0.4%	0.998
44.68%	A= 2.0448 B= -0.0006	0.4%	0.996

3.4 Conclusion

The density of nitrate containing molten salts was experimentally measured to determine the effects of $NaNO_3$ nitrate concentration on the thermal properties. 60:40 measured in this report was compared to data from SAND2001-2100. Maximum relative difference of 0.6% is observed from the measurements. RSD found a maximum error of 0.4%, as determined through duplicate measurements.

These results demonstrate that changing the cation's concentration does result in density changes. Based on the data, increasing $NaNO_3$ content above 60% may cause a change in density by up to 1.82%. The temperature dependence of density is described by a linear equation regardless of composition.

4. VISCOSITY OF MOLTEN $NaNO_3$-KNO_3 NITRATE MIXTURES

4.1 Experimental Methods

4.1.1 ATS Rheometry Method

Rheological measurements were conducted using a NOVA Rheometer with a 40 mm parallel plate with a gap of approximately 0.25 mm. Test temperatures were controlled using an ETC-3 temperature cell with the novel Joule-Thomson Vortex cooling system. After zeroing the gap at the test temperature, a small amount of the sample was loaded on the lower plate. When the sample melted, bubbles appeared in the liquid (attributed to absorbed moisture in the sample), and could be removed using a metal sample spoon. The upper plate was then lowered to a gap height of 0.25 mm. With the oven closed, the sample was allowed to reach thermal equilibrium for 15 minutes prior to starting the test. The sample was then trimmed to remove all excess material from around the plates.

Shear rate sweep measurements were conducted in stress controlled mode on all samples in the stress range of 2 – 5 Pa. The stress ranges for the measurements were chosen so that the lower limit generated enough torque to give a good signal to noise ratio, and the upper limits were chosen so as to avoid sample loss at high shear rates, as well as to keep the flow from getting turbulent. A delay time of 20 seconds and an integration time of 100 seconds were given for each data point. Repeat measurements were conducted for a few samples in order to verify the reproducibility and accuracy of the data. Viscosity measurements were conducted at 320 ˚C, 353˚C, 386 ˚C and 420˚C for all six samples. The temperature calibration of the instrument was verified prior to beginning the measurements. Replicate measurements were found to have less than a 1% variation (Figure 15).

4.1.2 SNL Method

The nitrate salt mixtures were measured on a TA Instruments DHR-2 rheometer with the ETC furnace, using a 40 mm steel upper plate (P/N 527400.937) and 45 mm steel cup (P/N 533281.901) on the disposable parallel plate geometry. The cup and plate were reused for multiple measurements, and between measurements the parallel surface of each was abraded using 600 grit wet/dry aluminum oxide paper to remove any discoloration caused by exposure to the salts at elevated temperatures (Figure 16).

For each measurement, the plates were installed on the geometry, the gap was zeroed and then increased to 3000 μm, and the geometry was allowed to equilibrate at 320°C for one hour. The gap was re-zeroed at 320°C and then increased to 20000 μm, and 3g of salt mixture was placed in the cup and equilibrated at 320°C for one hour. A small spatula was used to agitate the mixture in order to remove any obvious bubbles in the molten mixture. The gap was then set to the measurement height of 800 μm and allowed to equilibrate at 320°C for one hour.

Measurements were made in three consecutive temperature sweeps, 320°C to 550°C at 2°C/min. The shear speed was set at 1000 [1/s] for the first sweep, 500 [1/s] for the second sweep, and 200 [1/s] for the third sweep. In between sweeps, the temperature was allowed to cool uncontrolled back to 320°C and held for five minutes before beginning the next sweep. Loading had some effect on viscosity as shown in on the top of Figure 17. The final loading used in molten salt measurements was 3 g as determined through full wetting and complete coverage of the plate. The data was repeatable within 1% as shown on the bottom of Figure 17.

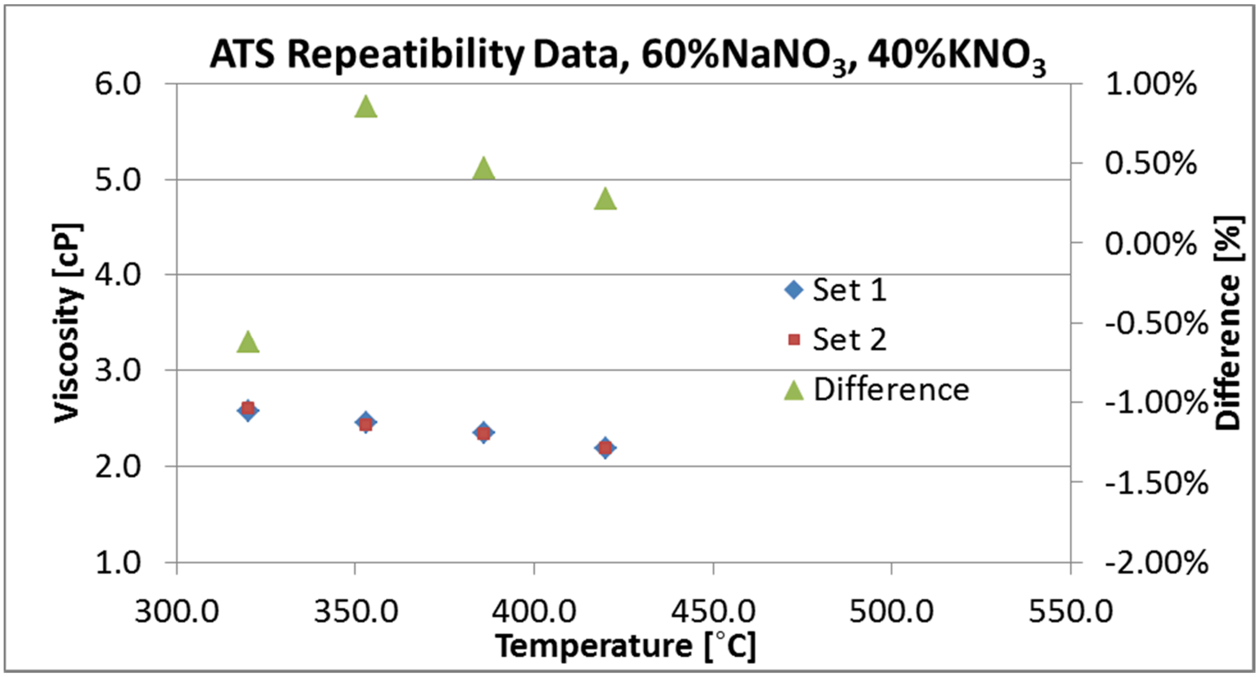


Figure 15: ATS Replicates Were Made for all Mixtures With Relative Differences Resulting in Smaller Than 1%. 60:40 was Shown as an Example and the Behavior is Typical for all Mixtures

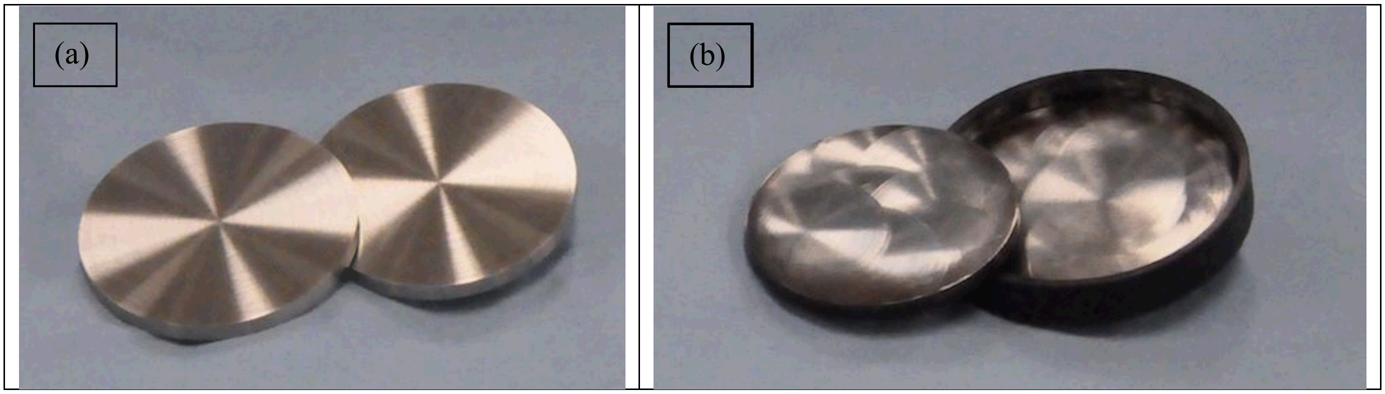


Figure 16: Geometries use for Viscosity Measurement. (a) Flat Plates and (b) Flat Plate/cup

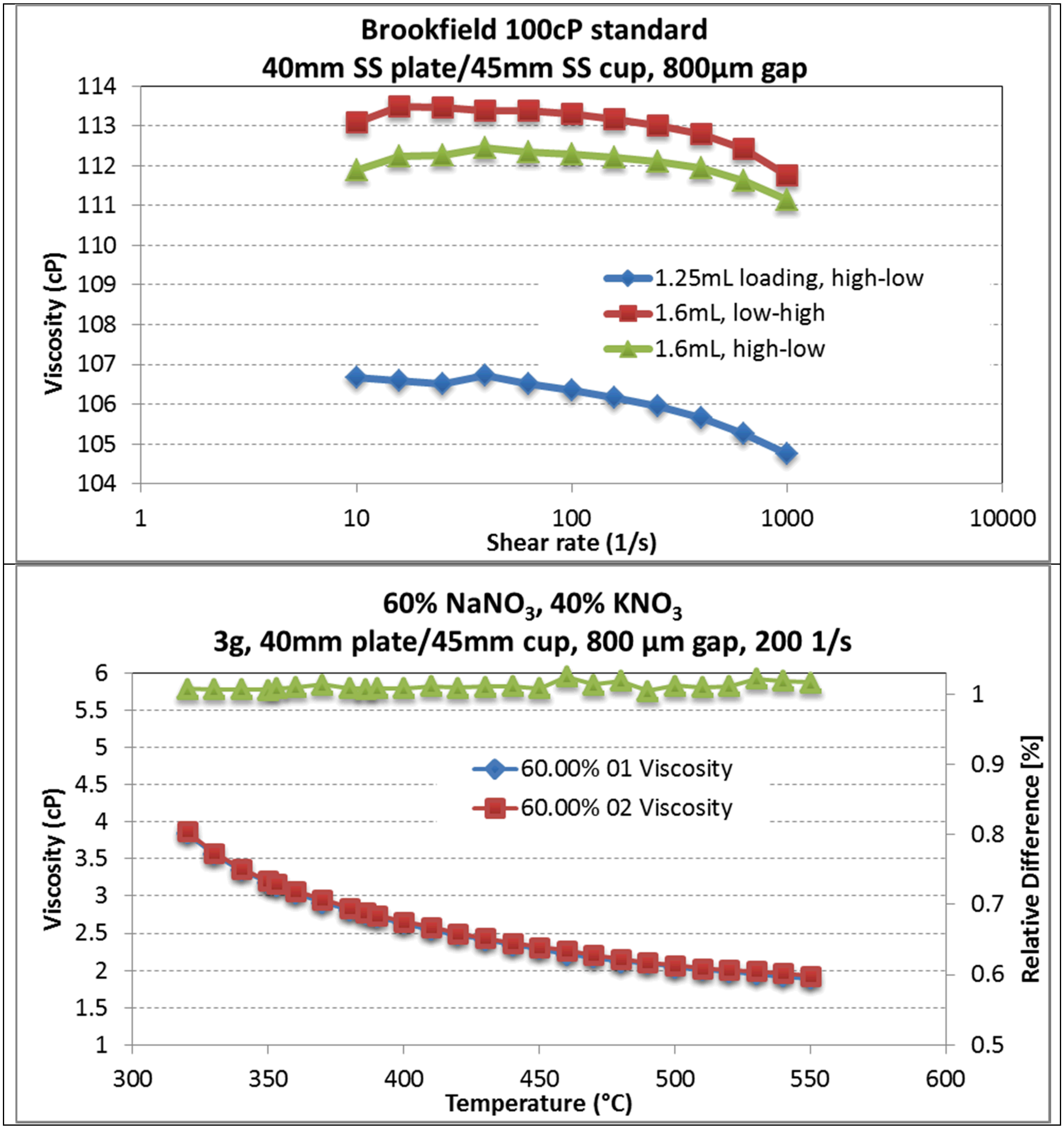


Figure 17: SNL Accuracy Data for Plate/cup Method (top). Data was Taken Either High Temperature to low Temperature (High-low) or low Temperature to High Temperature (low-High) 100cP Standard Could Have Differences up to 13-14% Based on Loading. Replicates Were Made for all Mixtures With Relative Differences Resulting in 1%. 60:40 was Shown as an Example (Bottom)

4.2 Results and Discussion

ATS and SNL data for 60% $NaNO_3$ were compared to viscosity data from [1] as shown in Figure 18. The difference between literature and the data collected at SNL is 60% at the maximum. The reasons for these differences are unclear, even though the measurements in this work used parallel plate methods SAND2001-2100 which was obtained from a custom harmonic oscillator method [16].

Based on the different methodologies between these measurements, a quantitative comparison seemed inappropriate, and qualitative trends were determined instead. Based on measurements made at SNL,

Figure 19, the appearance of cation content does not seem to appreciably affect the viscosity. A 95% Confidence Interval (CI) was determined, based on statistics from 60:40 replicates. 66% $NaNO_3$ was the lowest of all the measurements and at temperatures below 425°C did not fall within this confidence band. Tabulated fitting parameters are provided for ease of use in Table 7.

Data acquired from ATS exhibited larger differences up to 1cP at 320°C, it is unclear what caused this behavior. One possibility is the use of parallel plates versus a cup / parallel plate geometry. At the beginning of this work, SNL utilized parallel 40 mm plates, but later discovered the data was difficult to reproduce, while ensuring the salt did not over flow and damage the rheometer. Given the limited amount of data from ATS, further evidence to support the hypothesis was inconclusive.

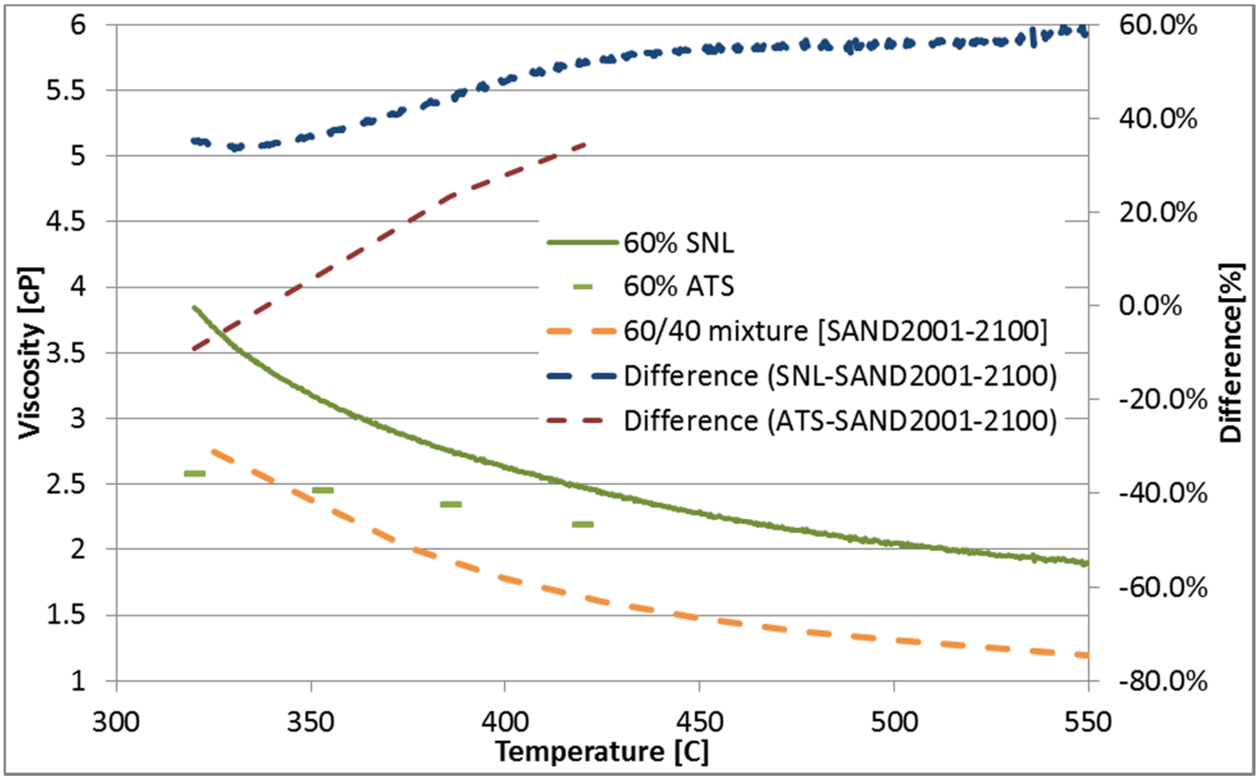


Figure 18: Viscosity Results for 60/40 Nitrate Salt. ATS and SNL Data Were Both Performed Using a Flat Plate-Type Method, While SAND2001-2100 Used an Under-Damped Oscillating Method

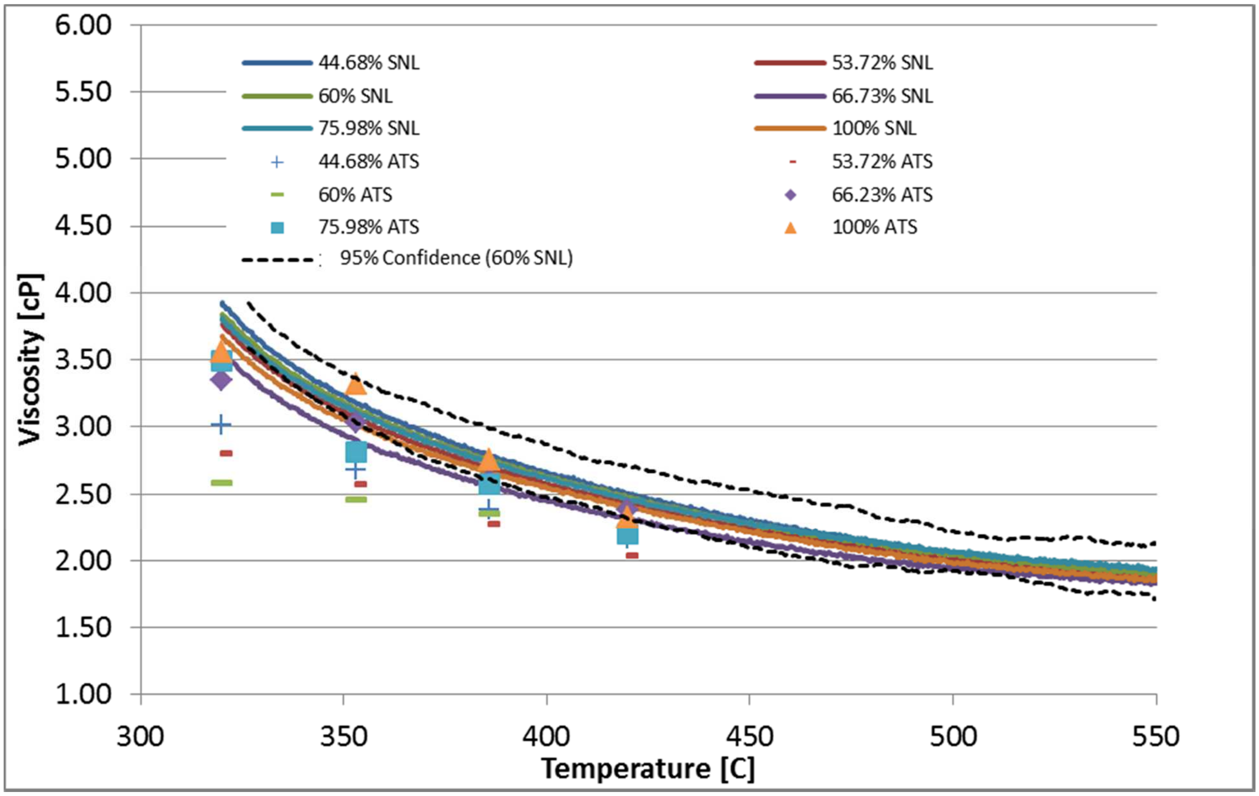


Figure 19: SNL Data Display Similar Trends vs. Temperature and is Relatively Uniform Among all Mixture Compositions

Table 7: Viscosity SNL Fitting Parameters

$NaNO_3$ wt%	Coefficient values $Y= A*T^3+B*T^2+C*T+D$ [cP]	Estimated % uncertainty of measurement	R^2 value
100%	A= -1e-7 B= 0.0002 C= -0.1096 D= 21.825	5%	0.998
75.98%	A= -2e-7 B= 0.0002 C= -0.1235 D= 24.1	5%	0.9984
66.73%	A= -1e-7 B= 0.0002 C= -0.1148 D= 22.418	5%	0.998
60%	A= -2e-7 B= 0.0002 C= -0.1254 D= 24.443	5%	0.9983
53.72%	A= -2e-7 B= 0.0002 C= -0.1253 D= 24.257	5%	0.9981

44.68%	A= -2e-7 B= 0.0002 C= -0.129 D= 25.151	5%	0.9981

4.3 Conclusion

The parallel plate methods resulted in statistically different results compared to literature values, which utilized a damped oscillator method. However, relative comparisons were made between mixtures on viscosity data. It was determined that within a 95% CI, noticeable differences between mixtures were minimal. The range of viscosity measurements was about 4% over all mixtures taken by SNL. Data from ATS showed no observable trends with relation to $NaNO_3$ concentration.

5. THERMAL CONDUCTIVITY OF MOLTEN NANO3-KNO3 NITRATE MIXTURES

5.1 Method Description

5.1.1 Laser Flash

Laser flash was performed by NETZSCH using the NETZSCH LFA 427 instrument. The sample disk is aligned between a neodymium YAG laser (1.06 mm wavelength, 0.3-1.2 ms pulse width) and an indium antimonide (InSb) IR detector in a high temperature furnace.

It was found during this measurement series that laser flash results in largely variable data with compounding error. The opinion was that this method should not be used without further developmental efforts, thus data was not reported here.

5.1.2 Transient Wire

Transient wire method was performed by Dynalene, Inc. (Whitehall, PA). This technique employs a fine platinum wire which is delicately placed in the melted salt samples. A current is applied to the wire and the voltage change over time is measured. The resistance change is obtained from the voltage data. The temperature change versus logarithmic time is then calculated through calibration of the platinum wire in water. The slope from the temperature versus logarithmic time graph, in conjunction with a reduced differential equation, provides the thermal conductivity value. Similar methods have been used elsewhere, but are specifically focused upon measuring the conductivity of solid medium [17].

It was found during the course of the work that temperature limitations in the measurement were primarily driven by cracking and subsequent failure of the ceramic coating over the platinum wire, thus measurements were limited to 400°C.

This set up is custom and does not have associated standards. Water was used as a standard and was found to be 0.60±0.01 [W/m-K], where accepted standards list this value as 0.58 [W/m-K].

5.2 Results and Discussion

Transient wire results for the 60% mixture were compared to literature in Figure 20. Estimated error plotted for data was determined through reproducibility experiments on transient wire data. Duplicate measurements of 100% $NaNO_3$ at 330°C and 45% $NaNO_3$ at 400°C were taken, as shown in Figure 21. The 45% mixture had 0.55±0.05 [W/m-K], while 100% $NaNO_3$ was 0.55±0.02 [W/m-K]. The maximum error was used for the error plot in Figure 20.

Considering uncertainty, which ranged from ±5% to ±9%, there was no discernable difference between mixtures for transient wire measurements (Figure 20) over all three temperatures. Ease of use linear fitted parameters is available in Table 8.

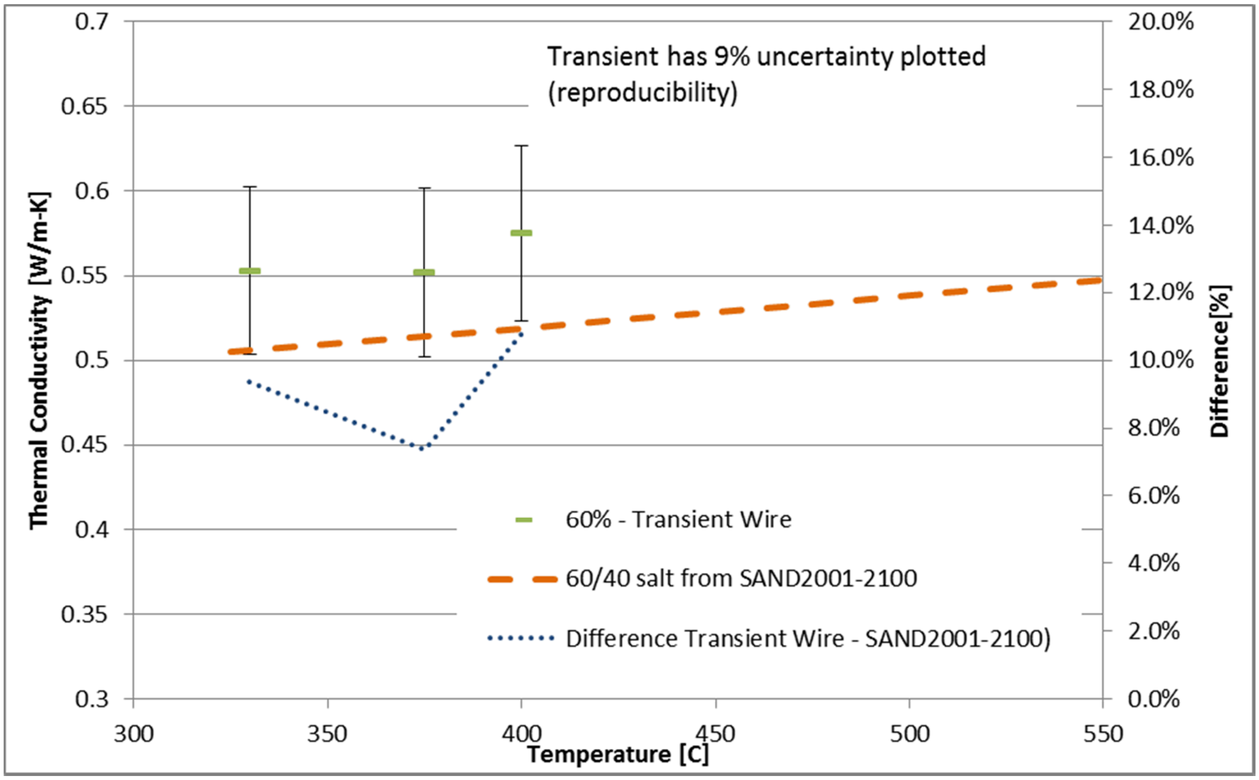


Figure 20: Transient Wire Measurements are Within 7-11% of Literature Values

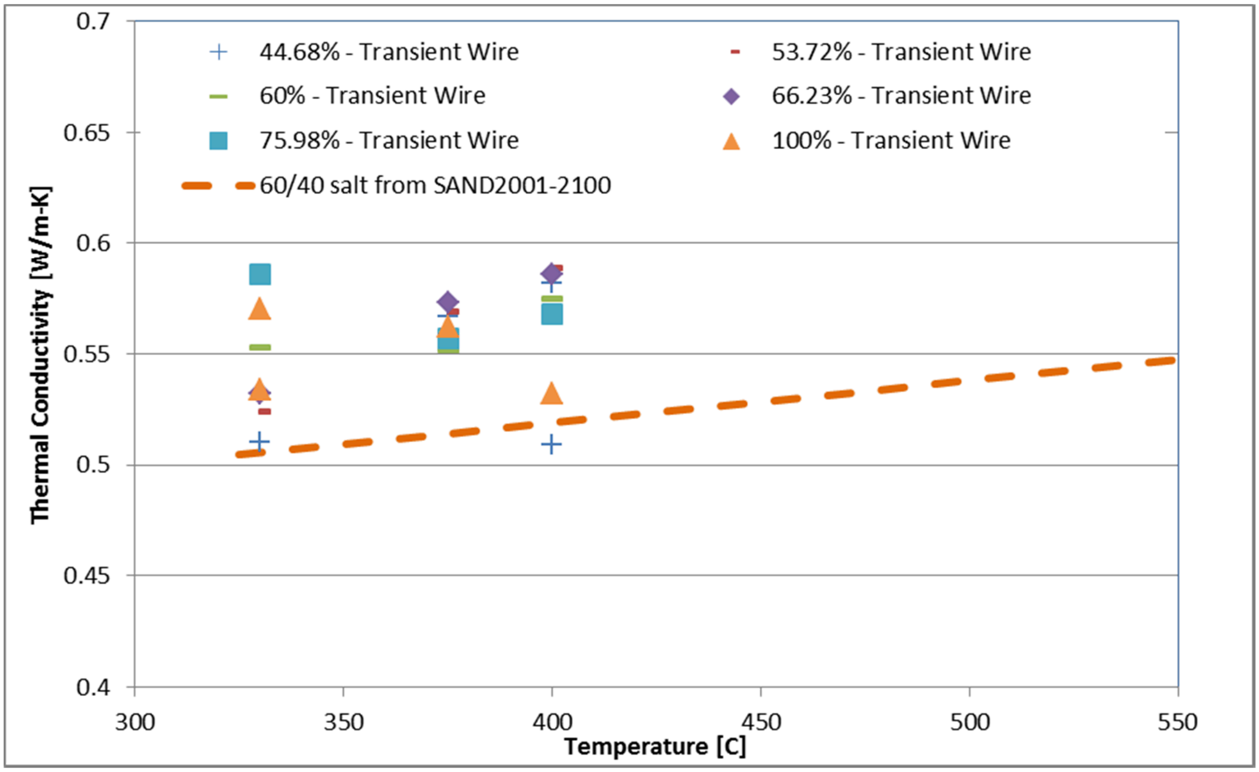


Figure 21: Transient Wire Data for all Mixtures. Repeat Measurements Were Taken for 100% $NaNO_3$ at 330°C and for the 45% Mixture at 400°C

Table 8: Thermal Conductivity Fitting Parameters for Transient Wire Method

$NaNO_3$ wt%	Coefficient values Y=A+B*T [W/m-K]	Estimated % uncertainty of measurement	R^2 value
100%	A= 0.5246 B= 5e-5	10%	0.0107
75.98%	A= 0.6821 B= -0.0003	10%	0.5401
66.73%	A= 0.2734 B= 0.0008	10%	0.9839
60%	A= 0.459 B= 0.0003	10%	0.5597
53.72%	A= 0.2176 B= 0.0009	10%	1
44.68%	A= 0.5147 B= -1e-5	10%	1

5.3 Conclusions

Transient wire method was used to measure thermal conductivity using a platinum wire submerged in the molten salt. The upper limit of the measurement was limited to 400°C based upon wire breakdown. This method was compared to literature values of 60:40 as a function of temperature. Transient wire data was within 7-11% of literature values. Repeatability measurements were variable, ranging from ±5% to ±9%. Based on this data, no trend in $NaNO_3$ concentration vs. thermal conductivity was observed within the uncertainty of the measurement.

6. CONCLUSIONS

Thermal properties of heat capacity, latent heat, density, viscosity, and thermal conductivity were measured for five mixtures of $NaNO_3$ nitrate and KNO_3 nitrate to determine if the dependency of properties on $NaNO_3$ nitrate concentration.

Heat capacities were measured for all mixtures from ambient conditions to 500°C. Accuracy and repeatability when compared to sapphire standards was less than 1%. Mixtures were not as repeatable due to salt wetting and creep during the measurement. This resulted in geometrical changes influencing the data repeatability. No trend could be determined as a function of $NaNO_3$ concentration within a 5% significance level. Latent heat increased linearly as a function of $NaNO_3$ content.

Density of the 60:40 mixture measured in this report was compared to data from SAND2001-2100. A maximum relative difference of 0.6% observed from measurements. RSD found a maximum error of 0.4%, determined through duplicate measurements. Results show that changing the cation's concentration does result in density changes. Increasing $NaNO_3$ content above 60% may cause a change in density up to 1.82% above 60:40 density. Density linearly decreases as a function of temperature regardless of the composition.

Viscosity was measured using parallel plate methods. It was found these methods did not compare well with harmonic oscillator methods used in literature. As such, relative comparisons were made among the mixtures. Within a 95% CI there were no appreciable differences between mixtures, with the range of viscosities among mixtures being ~4% of the mixture. Viscosity decreased non-linearly as a function of temperature.

Thermal conductivity was made with two different methods, laser flash and transient wire. Transient wire method was within 10% of literature values and indicated no trends among mixtures within error. Laser flash was found have large error due to a propagation of error, as thermal diffusivity was directly measured in this method. At this time laser flash should not be used for such molten salt measurements until refinements to the method can ensure better data.

7. REFERENCES

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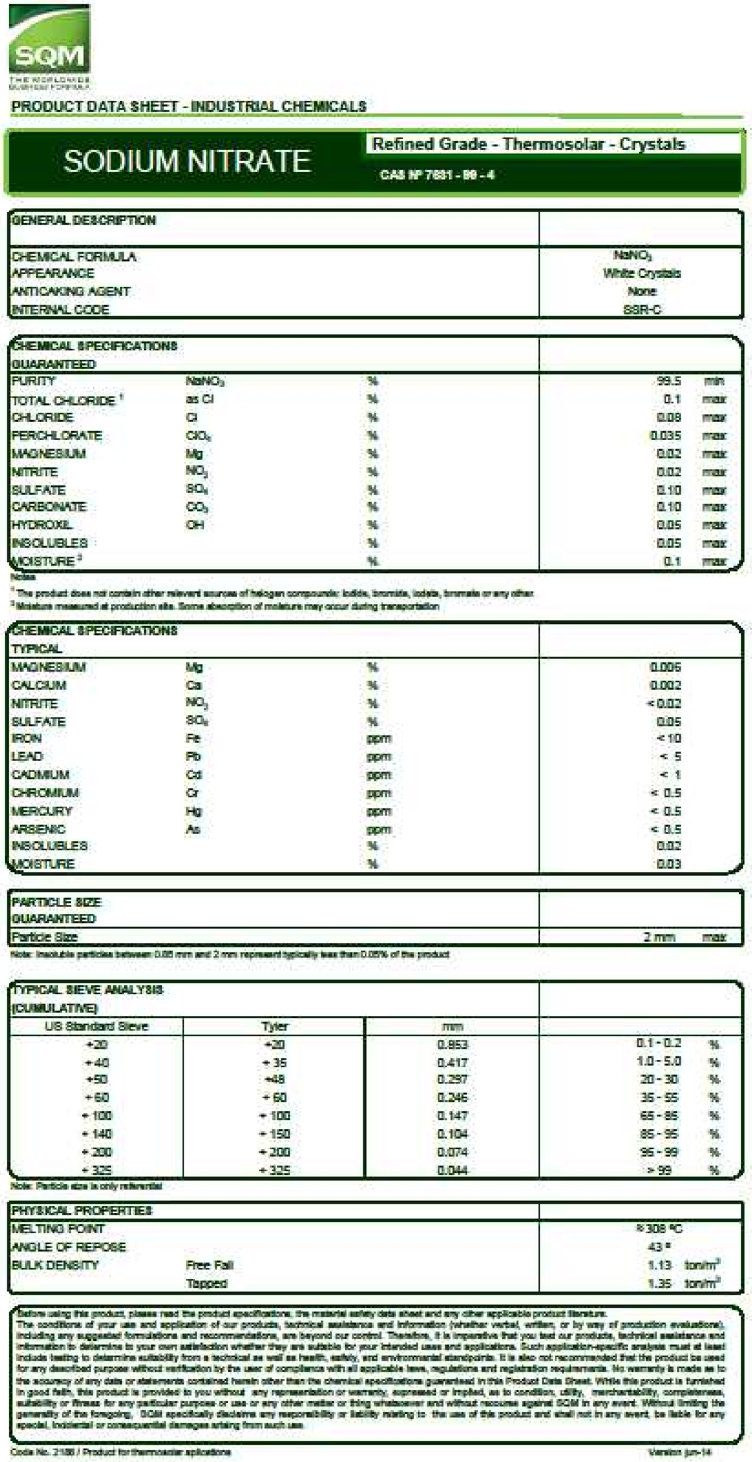
APPENDIX A: DETAILED SALT COMPOSITIONS



PRODUCT DATA SHEET - INDUSTRIAL CHEMICALS

SODIUM NITRATE

Refined Grade - Thermosolar - Crystals

CAS Nº 7631 - 99 - 4

GENERAL DESCRIPTION	
CHEMICAL FORMULA	$NaNO_3$
APPEARANCE	White Crystals
ANTICAKING AGENT	None
INTERNAL CODE	SSR-C

CHEMICAL SPECIFICATIONS GUARANTEED				
PURITY	$NaNO_3$	%	99.5	min
TOTAL CHLORIDE [1]	as Cl	%	0.1	max
CHLORIDE	Cl	%	0.08	max
PERCHLORATE	ClO_4	%	0.035	max
MAGNESIUM	Mg	%	0.02	max
NITRITE	NO_2	%	0.02	max
SULFATE	SO_4	%	0.10	max
CARBONATE	CO_3	%	0.10	max
HYDROXIL	OH	%	0.05	max
INSOLUBLES		%	0.05	max
MOISTURE [2]		%	0.1	max

Notes

[1] The product does not contain other relevant sources of halogen compounds: iodide, bromide, iodate, bromate or any other.

[2] Moisture measured at production site. Some absorption of moisture may occur during transportation

CHEMICAL SPECIFICATIONS TYPICAL			
MAGNESIUM	Mg	%	0.005
CALCIUM	Ca	%	0.002
NITRITE	NO_2	%	< 0.02
SULFATE	SO_4	%	0.05
IRON	Fe	ppm	< 10
LEAD	Pb	ppm	< 5
CADMIUM	Cd	ppm	< 1
CHROMIUM	Cr	ppm	< 0.5
MERCURY	Hg	ppm	< 0.5
ARSENIC	As	ppm	< 0.5
INSOLUBLES		%	0.02
MOISTURE		%	0.03

PARTICLE SIZE GUARANTEED		
Particle Size	2 mm	max

Note: Insoluble particles between 0.05 mm and 2 mm represent typically less than 0.05% of the product

TYPICAL SIEVE ANALYSIS (CUMULATIVE)				
US Standard Sieve	Tyler	mm		
+20	+20	0.853	0.1 - 0.2	%
+ 40	+ 35	0.417	1.0 - 5.0	%
+50	+48	0.297	20 - 30	%
+ 60	+ 60	0.246	35 - 55	%
+ 100	+ 100	0.147	65 - 85	%
+ 140	+ 150	0.104	85 - 95	%
+ 200	+ 200	0.074	95 - 99	%
+ 325	+ 325	0.044	> 99	%

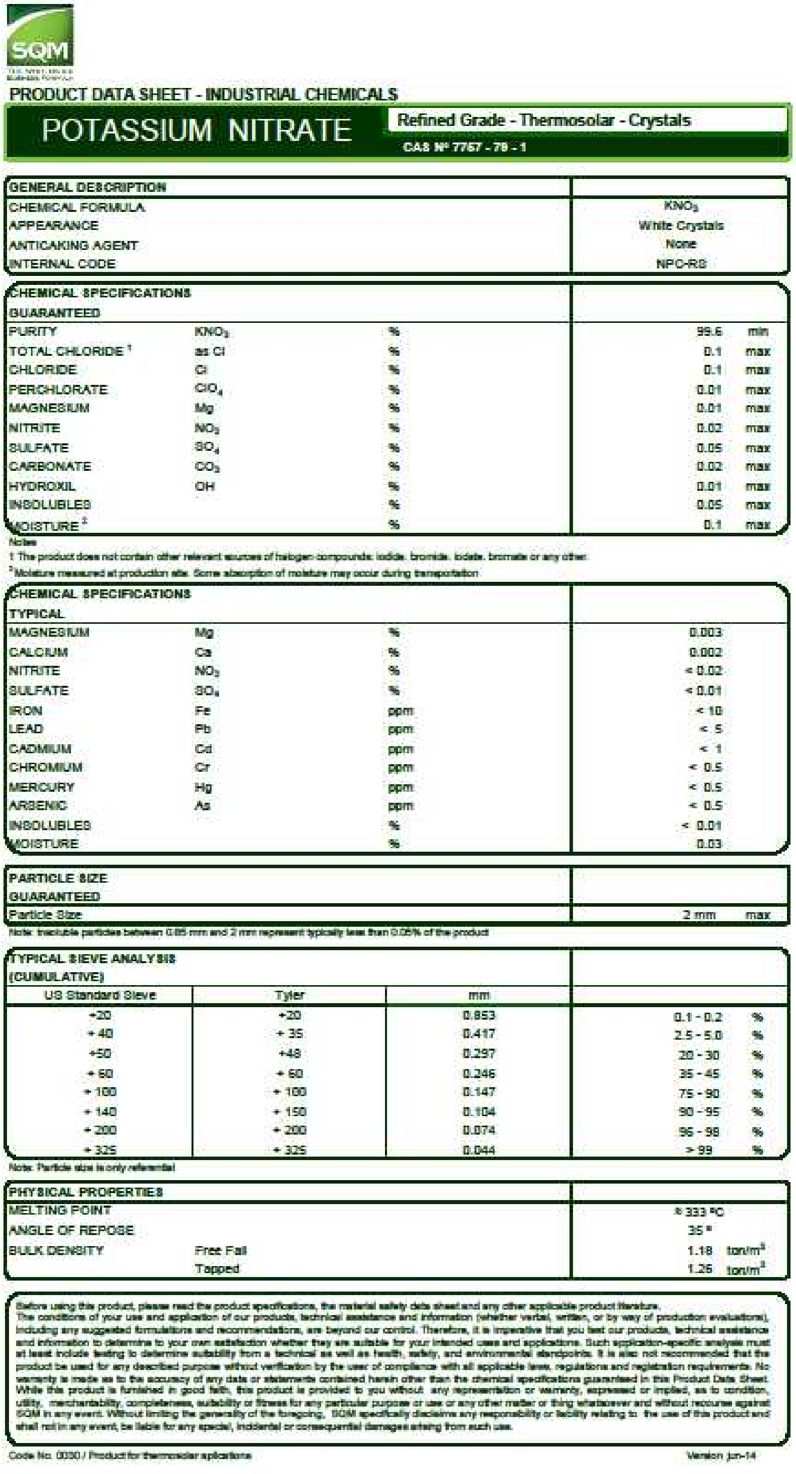
Note: Particle size is only referential

PHYSICAL PROPERTIES			
MELTING POINT		≈ 308 ºC	
ANGLE OF REPOSE		43 º	
BULK DENSITY	Free Fall	1.13	ton/m^3
	Tapped	1.35	ton/m^3

Before using this product, please read the product specifications, the material safety data sheet and any other applicable product literature.
The conditions of your use and application of our products, technical assistance and information (whether verbal, written, or by way of production evaluations), including any suggested formulations and recommendations, are beyond our control. Therefore, it is imperative that you test our products, technical assistance and information to determine to your own satisfaction whether they are suitable for your intended uses and applications. Such application-specific analysis must at least include testing to determine suitability from a technical as well as health, safety, and environmental standpoints. It is also not recommended that the product be used for any described purpose without verification by the user of compliance with all applicable laws, regulations and registration requirements. No warranty is made as to the accuracy of any data or statements contained herein other than the chemical specifications guaranteed in this Product Data Sheet. While this product is furnished in good faith, this product is provided to you without any representation or warranty, expressed or implied, as to condition, utility, merchantability, completeness, suitability or fitness for any particular purpose or use or any other matter or thing whatsoever and without recourse against SQM in any event. Without limiting the generality of the foregoing, SQM specifically disclaims any responsibility or liability relating to the use of this product and shall not in any event, be liable for any special, incidental or consequential damages arising from such use.

Code No. 2186 / Product for thermosolar aplications

Version jun-14



PRODUCT DATA SHEET - INDUSTRIAL CHEMICALS

POTASSIUM NITRATE

Refined Grade - Thermosolar - Crystals

CAS Nº 7757 - 79 - 1

GENERAL DESCRIPTION	
CHEMICAL FORMULA	KNO_3
APPEARANCE	White Crystals
ANTICAKING AGENT	None
INTERNAL CODE	NPC-RS

CHEMICAL SPECIFICATIONS GUARANTEED				
PURITY	KNO_3	%	99.6	min
TOTAL CHLORIDE [1]	as Cl	%	0.1	max
CHLORIDE	Cl	%	0.1	max
PERCHLORATE	ClO_4	%	0.01	max
MAGNESIUM	Mg	%	0.01	max
NITRITE	NO_3	%	0.02	max
SULFATE	SO_4	%	0.05	max
CARBONATE	CO_3	%	0.02	max
HYDROXIL	OH	%	0.01	max
INSOLUBLES		%	0.05	max
MOISTURE [2]		%	0.1	max

Notes

[1] The product does not contain other relevant sources of halogen compounds: iodide, bromide, iodate, bromate or any other.

[2] Moisture measured at production site. Some absorption of moisture may occur during transportation

CHEMICAL SPECIFICATIONS TYPICAL			
MAGNESIUM	Mg	%	0.003
CALCIUM	Ca	%	0.002
NITRITE	NO_3	%	< 0.02
SULFATE	SO_4	%	< 0.01
IRON	Fe	ppm	< 10
LEAD	Pb	ppm	< 5
CADMIUM	Cd	ppm	< 1
CHROMIUM	Cr	ppm	< 0.5
MERCURY	Hg	ppm	< 0.5
ARSENIC	As	ppm	< 0.5
INSOLUBLES		%	< 0.01
MOISTURE		%	0.03

PARTICLE SIZE GUARANTEED		
Particle Size	2 mm	max

Note: Insoluble particles between 0.85 mm and 2 mm represent typically less than 0.05% of the product

TYPICAL SIEVE ANALYSIS (CUMULATIVE)

US Standard Sieve	Tyler	mm		
+20	+20	0.853	0.1 - 0.2	%
+ 40	+ 35	0.417	2.5 - 5.0	%
+50	+48	0.297	20 - 30	%
+ 60	+ 60	0.246	35 - 45	%
+ 100	+ 100	0.147	75 - 90	%
+ 140	+ 150	0.104	90 - 95	%
+ 200	+ 200	0.074	95 - 98	%
+ 325	+ 325	0.044	> 99	%

Note: Particle size is only referential

PHYSICAL PROPERTIES			
MELTING POINT		≈ 333 °C	
ANGLE OF REPOSE		35 °	
BULK DENSITY	Free Fall	1.18	ton/m³
	Tapped	1.26	ton/m³

Before using this product, please read the product specifications, the material safety data sheet and any other applicable product literature.
The conditions of your use and application of our products, technical assistance and information (whether verbal, written, or by way of production evaluations), including any suggested formulations and recommendations, are beyond our control. Therefore, it is imperative that you test our products, technical assistance and information to determine to your own satisfaction whether they are suitable for your intended uses and applications. Such application-specific analysis must at least include testing to determine suitability from a technical as well as health, safety, and environmental standpoints. It is also not recommended that the product be used for any described purpose without verification by the user of compliance with all applicable laws, regulations and registration requirements. No warranty is made as to the accuracy of any data or statements contained herein other than the chemical specifications guaranteed in this Product Data Sheet. While this product is furnished in good faith, this product is provided to you without any representation or warranty, expressed or implied, as to condition, utility, merchantability, completeness, suitability or fitness for any particular purpose or use or any other matter or thing whatsoever and without recourse against SQM in any event. Without limiting the generality of the foregoing, SQM specifically disclaims any responsibility or liability relating to the use of this product and shall not in any event, be liable for any special, incidental or consequential damages arising from such use.

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APPENDIX B: TABULATED DATA

Table 9: SNL Heat Capacity Data

Temperature [C]	*45% $NaNO_3$ [J/g-K]*	*54% $NaNO_3$ [J/g-K]*	*60% $NaNO_3$ [J/g-K]*	*66% $NaNO_3$ [J/g-K]*	*76% $NaNO_3$ [J/g-K]*
300	*1.556*	*1.568*	*1.594*	*1.615*	*1.521*
325	*1.556*	*1.571*	*1.592*	*1.611*	*1.518*
350	*1.549*	*1.547*	*1.575*	*1.602*	*1.499*
375	*1.529*	*1.526*	*1.560*	*1.599*	*1.493*
400	*1.519*	*1.509*	*1.551*	*1.581*	*1.480*
425	*1.513*	*1.503*	*1.540*	*1.569*	*1.469*
450	*1.519*	*1.507*	*1.547*	*1.569*	*1.466*
475	*1.524*	*1.513*	*1.550*	*1.571*	*1.472*
*500**	*1.505*	*1.485*	*1.529*	*1.556*	*1.452*
*525**	*1.499*	*1.475*	*1.521*	*1.548*	*1.444*
*550**	*1.492*	*1.464*	*1.514*	*1.540*	*1.435*
*575**	*1.486*	*1.454*	*1.506*	*1.533*	*1.427*
*600**	*1.480*	*1.444*	*1.498*	*1.525*	*1.418*
**linear extrapolation*					

Table 10: Density of the Nitrate Salt Mixtures Measured at Various Temperatures.

	Density (g/cm^3)					
Temp (°C)	**45 wt%**	**54 wt%**	**60 wt%**	**66 wt%**	**76 wt%**	**100 wt%**
325	1.863	1.883	1.885	1.908	1.912	1.897
375	1.839	1.851	1.851	1.878	1.878	1.867
425	1.809	1.818	1.817	1.849	1.849	1.838
475	1.784	1.786	1.785	1.818	1.818	1.809
525	1.759	1.758	1.756	1.788	1.788	1.783
575	1.732	1.731	1.734	1.761	1.759	1.765
625	1.694	1.703	1.701	1.735	1.727	1.743

Table 11: Viscosity ATS Data

	Viscosity (cP)					
Temp. (°C)	45 wt%	54 wt%	60 wt%	66 wt%	76 wt%	100 wt%
320	3.02	2.80	2.58	3.35	3.49	3.56
353	2.68	2.57	2.45	3.03	2.81	3.32
386	2.38	2.27	2.35	2.60	2.58	2.75
420	2.16	2.04	2.19	2.38	2.20	2.32

Table 12: Viscosity SNL Data

Temp. (°C)	45 wt%	54 wt%	60 wt%	66 wt%	76 wt%	100 wt%
325	3.76	3.62	3.70	3.41	3.66	3.53
350	3.23	3.12	3.18	2.94	3.16	3.05
375	2.91	2.80	2.86	2.66	2.84	2.77
400	2.66	2.58	2.63	2.45	2.61	2.55
425	2.47	2.39	2.43	2.28	2.42	2.37
450	2.31	2.24	2.29	2.16	2.27	2.21
475	2.17	2.12	2.15	2.03	2.16	2.08
500	2.06	2.00	2.04	1.95	2.07	1.98
525	1.99	1.92	1.96	1.88	2.00	1.91
550	1.93	1.86	1.89	1.84	1.94	1.88

Table 13: Thermal Conductivity Data From Transient Wire Method. (Duplicate Measurements Listed)

Temp. [˚C]	45 wt%	54 wt%	60 wt%	66 wt%	76 wt%	100 wt%
330	0.51	0.524	0.553	0.532	0.586	0.534 0.570
375	0.567	0.569	0.552	0.573	0.557	0.562
400	0.509 0.582	0.589	0.575	0.586	0.568	0.532

APPENDIX C: STATISTICAL ANALYSIS OF HEAT CAPACITY (MEMO)

Date: April 14, 2015
To: Alan Kruizenga

Original Signed by
From: Alix Robertson (8225)

Subject: Statistical Analysis of Experimental Data for Heat Capacity of Solutions

I took a look at the experimental heat capacity data provided in your Excel workbook Changed_Cp-mixtures-autosampler-V6.xlsx. Based on the data plots that you provided, I looked at the range of temperatures from 300 C to the maximum temperature tested of 485 C. These data are plotted on Figure 22.

Based on our discussion I understand that there is no theoretical reason for heat capacity to change with temperature during a run, and we do not believe that the observed trends with temperature have any physical significance for heat capacity. However, it is possible that measurement error could grow smoothly during a run, due to physical processes such as the sample solution progressively wetting the side walls of the container. Therefore, sequential measurements within a run will be highly correlated, and consequently the individual measurements cannot be treated as statistically independent samples.

It is theoretically possible that heat capacity could vary with NaNO3 concentration. In order to test for this difference one can look at "run-averages" calculated as the average heat capacity measured over each run from 300 C to 485 C. The individual run-averages are plotted on Figure 23 which also shows a regression of heat capacity against NaNO3 concentration. The data do not show any monotonic trend of heat capacity with NaNO3 concentration, and (as can be seen from the confidence intervals) the regression line slope is not significantly different from zero. The P-value for the regression is 0.693.

Finally, one can then test whether the observed run-averages are consistent with the hypothesis that there is no difference in true heat capacity of the solutions. Statistical tests for a difference in mean heat capacity (assuming constant variance) include analysis of variance, which also assumes that the data are normal in distribution, and the Kruskal-Wallis test. The P-value for analysis of variance is 0.066 and the P-value for the Kruskal-Wallis test is 0.077. These results indicate that the null hypothesis of no difference in mean heat capacity for different NaNO3 concentrations cannot be ruled out at the five percent significance level.

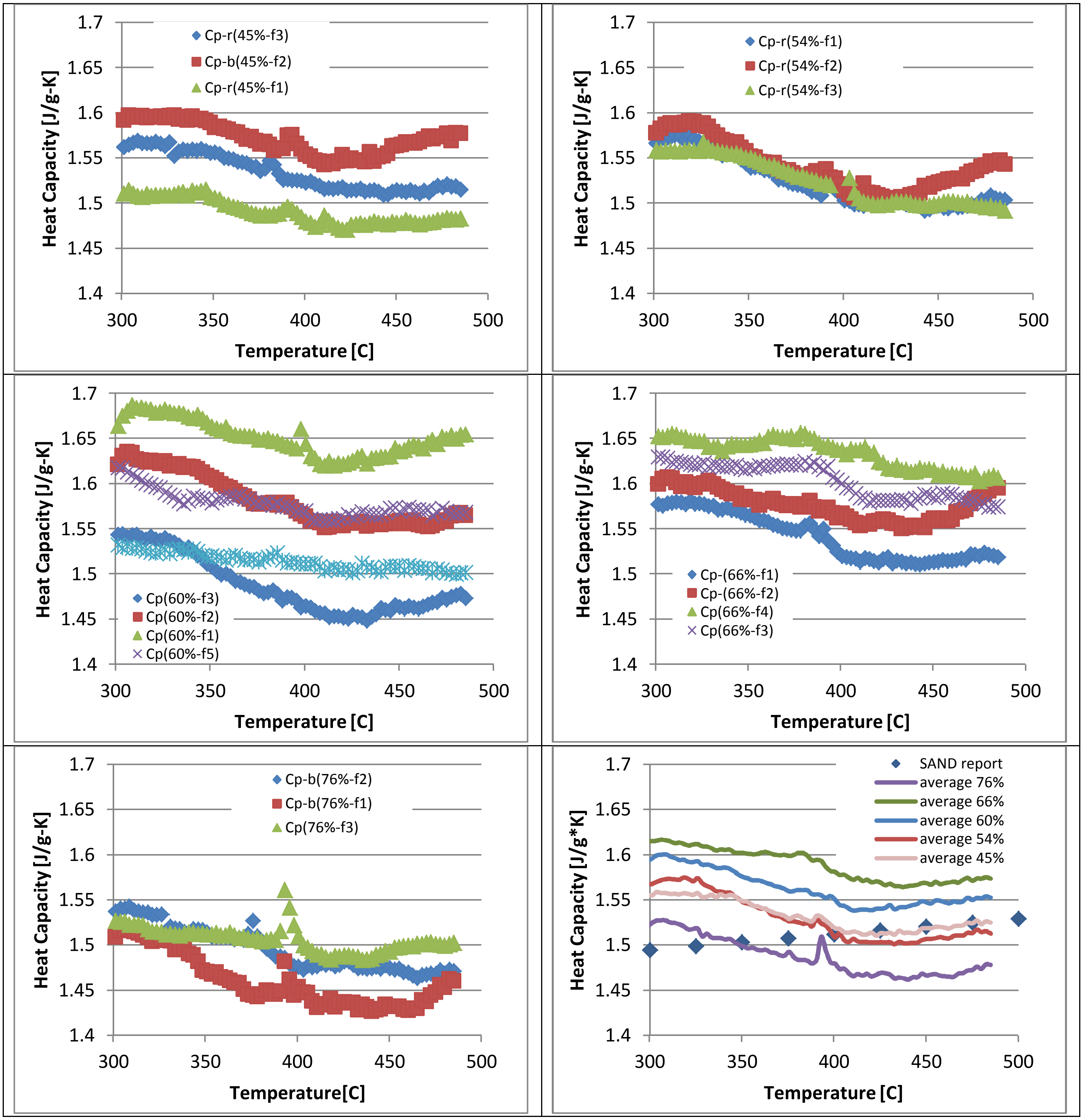

Figure 22: Heat Capacity Data for Five Solutions of Varying $NaNO_3$ Concentrations With a Plot Showing Average Data as a Function of Temperature

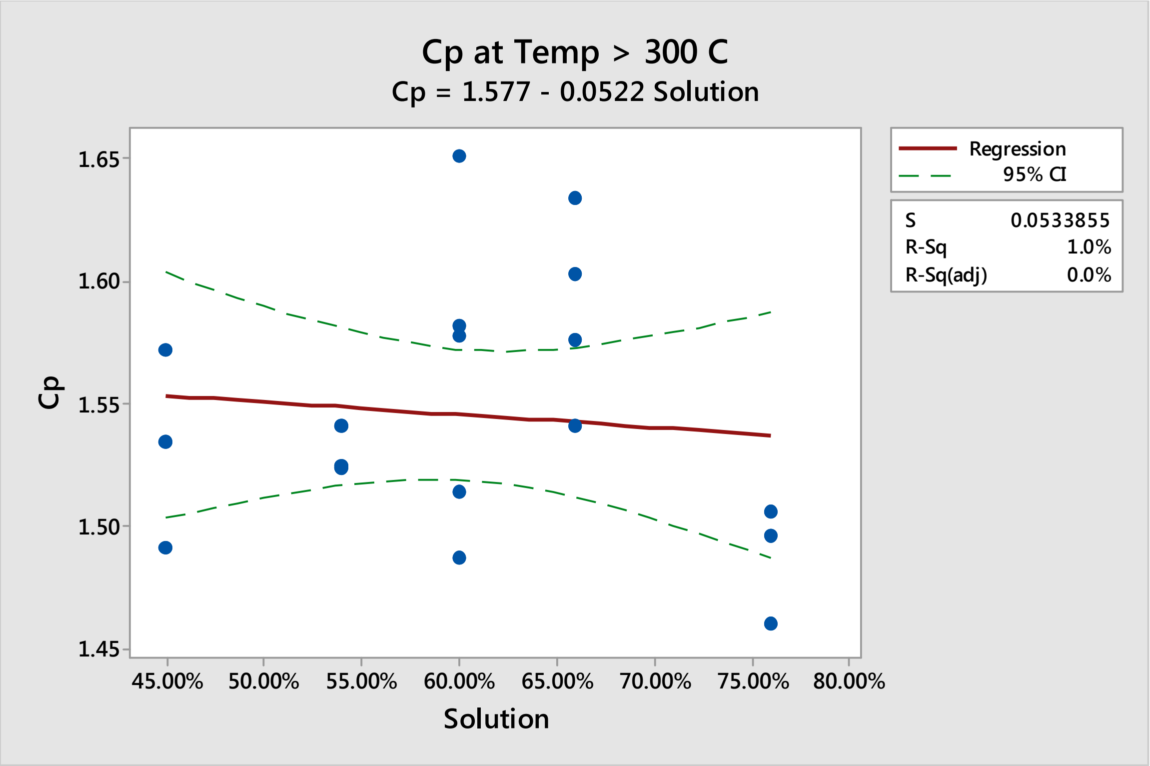

Figure 23: Run-Averages Over 300°C to 485°C for Varying Levels of $NaNO_3$ Concentrations

References

1. Excel workbook Changed_Cp-mixtures-autosampler-V6.xlsx.

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