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Quality Aspects of Molten Salt Property Measurements

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Advanced Reactor Technologies Program
Molten Salt Reactors Campaign Program Review
June 17, 2021

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Objectives

- Identify quality-affecting aspects of property measurements, sensitivities, and measurement controls to achieve NQA-1 data quality
  - Assess precision of measurements
  - Distinguish effects of uncertainty in salt composition and measurement
- Determine property values of reference salts that can be used to compare methods and determine within-lab repeatability, lab-to-lab reproducibility, and bias in measurements
  - Develop procedures suitable for use as consensus standard practices issued through ASTM-International
  - Generate database for an approved reference material salt (ARM-salt)
- Generate high quality measurements of fuel-bearing salt systems

Quality of Property data

- Affected by
  - Salt composition
  - Environmental factors
  - Instrumental uncertainties
  - Analytical limitations
  - Calculation method
- Data quality assured by
  - Reagent purity and controlled batching
  - Controlled & monitored glovebox atmosphere
  - Reliable measurement procedures and device calibrations
  - Reliable reference and standard materials
  - Fundamental understanding

Argonne approach

- Salt composition
  - Use batched compositions rather than measured compositions when possible
- Environmental factors
  - Require low and constant oxygen and humidity levels in glovebox for data acceptance
- Measurement and instrumental uncertainties
  - Detailed standard operating procedures
  - Routine calibration checks of devices and instruments
  - Routine checks of responses with reference materials
  - Control chart calibrations and checks
- Analytical limitations
  - Replicate samples and replicate analyses
  - Reference materials
- Calculation method
  - Identify quality-affecting parameter values subjected to calibration checks
  - Use reference materials and standards for each parameter used for property determination

Density Measurement by Archimedes method

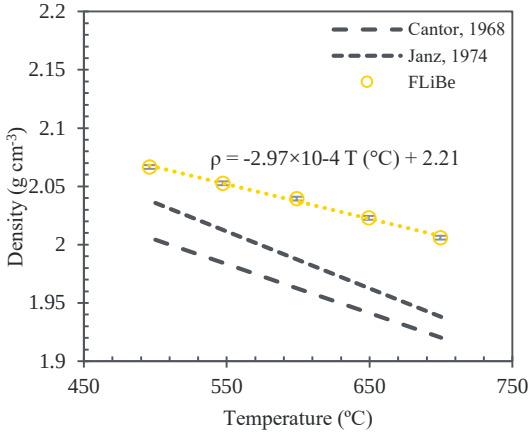
Data quality improvements include:

- Correct for surface tension
  - Depends on T and must be measured
  - Often not considered in the literature
- Checks for preferential volatilization
  - Causes change in salt composition
  - Salt condensing on the suspension wire affects measurement

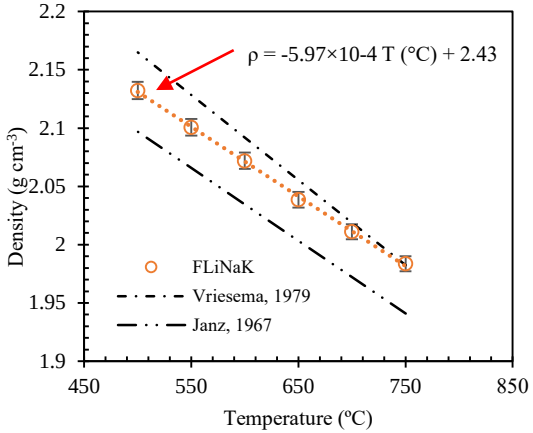
surface tension term

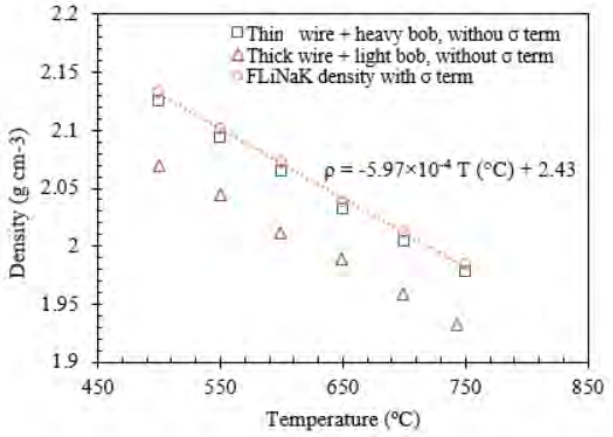
$$\rho = \frac{\Delta m + \frac{\pi D \sigma}{g}}{V_0[1 + \alpha T]^3}$$

FLiBe



FLiNaK





Summary of FY21 Accomplishments

- Identified quality-affecting aspects of differential scanning calorimetry for melting point and heat capacity, density by Archimedes method, and thermal diffusivity measurements
- Demonstrated importance of taking surface tension into account in density calculations
- Assessed repeatability precision of measurements being assessed using replicate samples; reproducibility precision will be assessed using inter-laboratory study results
- Distinguished effects of salt composition and measurement precision using results for multiple batches of FLiNaK

Presentations

Sharing our capabilities, data and data quality with developers

“Molten Salt Modeling, Thermophysical Measurements, and Database Development” at the 2019 Chemistry and Corrosion Workshop at ORNL

“Viscosity Measurements in a Molten Salt” at the 2020 Molten Salt Thermal Properties Working Group Workshop (held virtually July 2020)

“Argonne’s Molten Salt Property Measurements” at the 2020 MSR Developer Workshop at ORNL (held virtually in October 2020)

“Measuring Thermophysical Properties of Molten Salts” at 2019 GLOBAL Conference in Seattle, WA

“Quality aspects of molten salt property measurements” at MSR PIRT Meeting on March 31st, 2021

Reports

Documenting measured data and technique improvements

“Salt Production and Analysis in Support of the MSR Campaign” –submitted in PICs March 2020

“Thermochemical Property Measurements of FLiNaK and FLiBe in FY 2020”- submitted in PICs Nov. 2020

“Thermophysical Property Measurements: Improved Density, Viscosity and Thermal Diffusivity Methods” – Submitted in PICs Nov. 2020

“Production and Chemical Analysis of NaCl-KCl-UCl_3 Salts” – submitted in PICs Feb 2021

Milestones for FY21

High Quality Molten Salt Property Measurements

Milestone Number	Title	Date
M3RD-21AN0703021*	Evaluate salt property measurement data quality and controls	8/23/21

* Under RD-21AN070302 Salt Data Quality Assurance Needs Assessment – ANL

All Milestones On Schedule

Future Direction

- Identify quality controls for property measurements made using other methods
- Develop consensus standards for property measurement methods
- Develop and produce salt mixture for use as a reference test material

Funding Timeline

- FY2021 $250K RD for Technology-based Licensing Assessment (Uncertainty)
- FY2021 $100K RD for Data Quality Assurance Needs Assessment

3 Year Plan and Budget Request

Significance of research: This work assesses the quality of experimental property data for relevant salt mixtures used in process models supporting the design and licensing of MSRs.

- FY 2022 $200K
  - In collaboration with modelers, identify controls for salt property measurement methods necessary to meet precision requirements of quality data for process and safety modeling ($125K)
  - Draft property measurement methods for standardization through ASTM International that integrate results of intra-laboratory and inter-laboratory (round robin) studies ($75K)
- FY 2023 $250K
  - Develop standard salt for use as reference test material and produce source batch for use as reference test material in inter-laboratory studies
  - Submit methods for measuring density, thermal properties, and heat capacity of molten salts for standardization by ASTM International
  - Draft ASTM methods for measuring viscosity and thermal diffusivity
- FY 2024 $200K
  - Develop ASTM International methods for measuring viscosity and thermal diffusivity of molten salt
  - Conduct inter-laboratory study (round-robin) using reference salt test material

Salt data quality - ANL

OVERVIEW

Purpose: Establish quality standards for property measurements with MSR-relevant salts to support reactor design and safety analyses.

Objectives:
Identify and implement controls to generate NQA-1 quality data and property values
Establish methods to assess quality of property data, precision and bias

IMPACT

Logical Path: Description (or flowchart) of logical path to accomplish work

- Identify controls to ensure quality measurements, including calibrations and environment controls.
- Develop technically sound approaches to analyze measured data and determine property values and uncertainties

Outcomes: Brief summary of expected project outcomes

- Document methods, controls, results, and derived property values in project reports.
- Draft methods for standardization through ASTM-international
- Promulgate methods through programmatic presentations and reports, and consensus standards.

DETAILS

Principal Investigator: Melissa Rose
Institution: Argonne National Lab

Collaborators:
FY 2022 Total Funding Requested: $200k
3 scenarios

- $200k Continue to document controls, and pursue ASTM standardization of methods
- $125k Continue to document controls
- $75k Pursue ASTM standardization of methods

DELIVERABLES

Milestones Level 2, 3, 4
M3 Proposed ASTM International standard methods

Journal papers on improved measurement methods for molten salts
Present at MSR developer workshop, MSTPWG Workshops, and any additional relevant conferences to promulgate methods.

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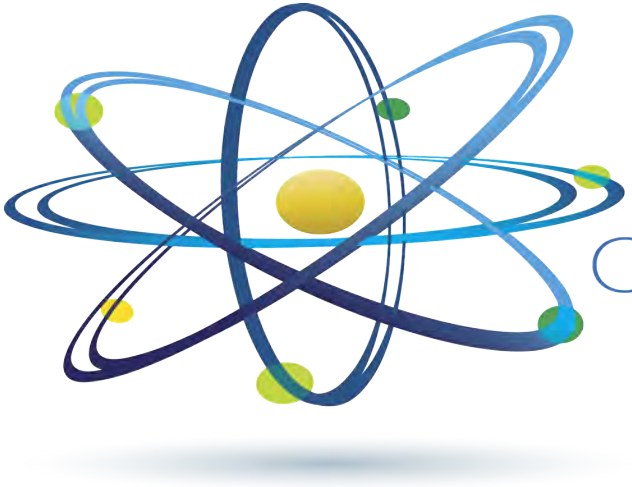

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