VISCOSITY MEASUREMENTS IN MOLTEN SALTS

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MOTIVATION

- Accurate viscosity data are needed by molten salt reactor (MSR) developers for range of salt compositions and temperatures to design reactors and present a safety basis for licensing
  - Viscosity affects heat, mass, and momentum transport
  - Viscosity affects thermal hydraulic behavior during normal and transient conditions
  - Viscosity affects flow in an MSR: turbulent or laminar
  - Knowing viscosity is essential to designing pumps for circulating the molten salt
- Viscosities (and other salt property values) have not been measured for many fuel or coolant salts and existing data are inconsistent
- Need to discriminate effects of salt composition, environmental conditions, measurement precision, and artifacts on measured values
- Standardized methods and reference salts are needed to ensure reliable high quality viscosity data are generated

SALT PREPARATION AND CHARACTERIZATION

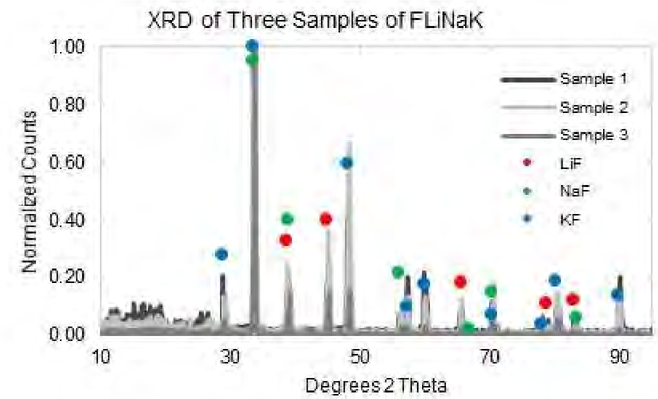
Important to know salt composition and impurity concentrations

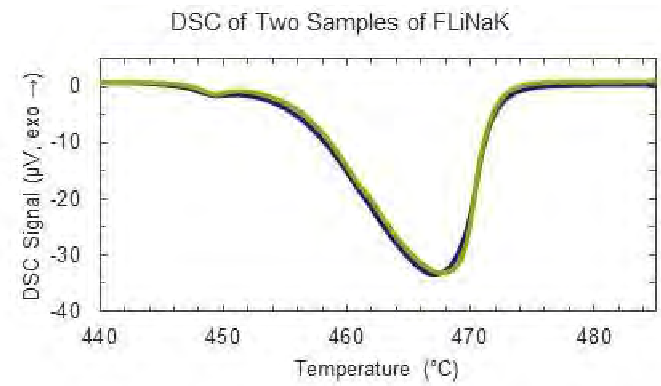
Procure high purity reagent salts and use well-established methods to synthesize

- Inside a glovebox (<10 ppm O_2, <1 ppm H_2O)
- Heat reagents to remove water before batching
- Accurately measure batched compositions
- Fuse and grind salt mixtures at least twice to homogenize

Analyze replicate salt samples before and after measurements

- Inductively coupled plasma – mass spectrometry or optical emission spectroscopy (ICP-MS or OES) for elemental composition and impurities
- LECO analysis for dissolved oxygen and water
- X-ray diffraction for constituent phase identification
- Differential scanning calorimetry (DSC) for melting point determination





MEASURING VISCOSITY

- Relative and absolute measurement methods are used to measure viscosities of molten salts
  - Relative methods:
    - Rely on calibrations with appropriate liquids to determine empirical parameter values
  - Absolute methods:
    - Parameter values have a clear physical meaning and measurements are made under well-controlled conditions to determine dependencies
- For all methods, flow must be laminar and fully developed for accurate measurements
  - Reynolds number (Re) is a dimensionless number quantifying flow conditions
  - Based on ratio of inertial and viscous forces that is geometry-specific

$$Re = \frac{\rho v L}{\eta}$$

where ρ is fluid density, v is fluid velocity, η is dynamic viscosity, L is characteristic length

  - Laminar flow has low Reynolds numbers

RELATIVE METHODS

- Capillary Methods
  - Induce Poiseuille flow in a tube long enough to acquire a parabolic flow profile
  - The pressure drop needed to produce a specific flow rate in a defined geometry can be used to calculate viscosity
  - Tube may deform during measurements due to pressure or temperature
  - Must correct for entrance section geometry by calibration with appropriate liquids in the temperature and viscosity range applicable to molten salts

- Falling and Rolling Body Methods
  - The time required for an object (ball or cylinder) of defined geometry to fall a measured distance at terminal velocity used to determine viscosity
  - Geometry parameters established at relevant temperature and viscosity ranges by calibrations
  - Falling ball can experience random slip and spin
  - Reynolds number must be less than 10 to ensure laminar flow

ABSOLUTE METHODS

- Oscillation Methods
  - Use an oscillating body (sphere, cup, cylinder or disk) with salt outside or inside the body
  - The body is set into oscillation by a small perturbation, then the frequency and damping decrement are used to determine viscosity of the fluid
- Vibration Methods
  - Use piezoelectric resonators with oscillator circuit, quartz crystal, vibrating wires and cantilevers
  - A vibration is induced to produce a viscous wave in the surrounding fluid, which changes the resonance frequency of the vibrating medium in a measureable way
- Rotating Cylinder
  - A cylinder is rotated in a molten salt at a specific speed to produce shear at the cylinder wall and induce Couette flow
  - Method used at Argonne because it does not require complex electronics, is easily adapted to a glovebox environment, and does not require complex modeling to process the data

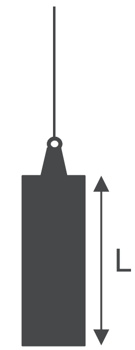
ROTATIONAL VISCOMETER

- Viscosity (μ) is the ratio of the shear rate (τ) to the shear stress (γ) produced by the applied torque (M)

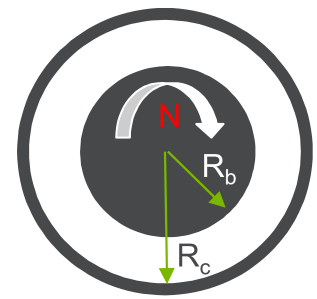
$$\mu = \frac{\tau}{\gamma} \qquad \tau = \frac{M}{2\pi R_b^2 L} \qquad \gamma = \frac{2\left(\frac{2\pi}{60}N\right)R_c^2}{\left(R_c^2 - R_b^2\right)}$$

$$\mu = \frac{M\left(R_c^2 - R_b^2\right)}{8\pi^2 R_c^2 R_b^2 L\left(\frac{N}{60}\right)}$$

- The measured torque (M) required to rotate a spindle at a specific rotational velocity (N) used to calculate viscosity



Side view of spindle



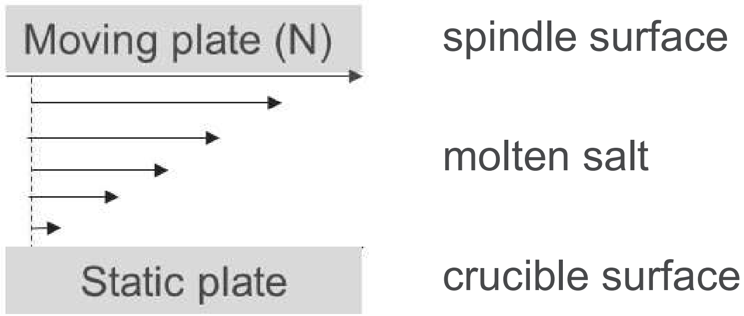
Top view of spindle in crucible

FLUID DYNAMICS OF ROTATIONAL METHOD

- Rotational method requirements:
  - Fluid must be incompressible
  - System must be isothermal to avoid axial convective flow
  - Flow must be laminar and fully developed (Re<370 for Couette flow)

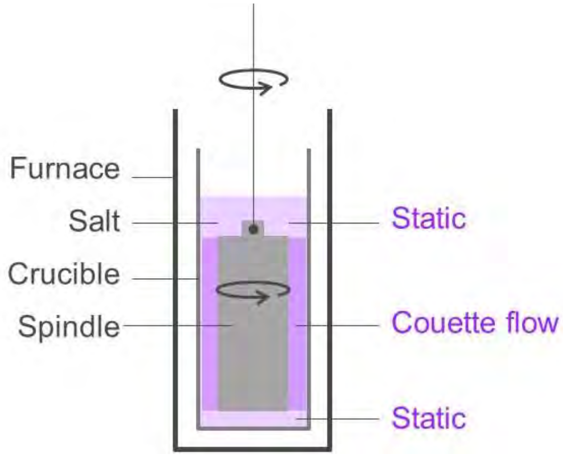
$$Re = \frac{\rho(T) N d_{spindle}(d_{crucible} - d_{spindle})}{\eta(T)}$$

- Fully developed Couette flow is approximated by linear flow between moving and static plates



EXPERIMENTAL APPARATUS

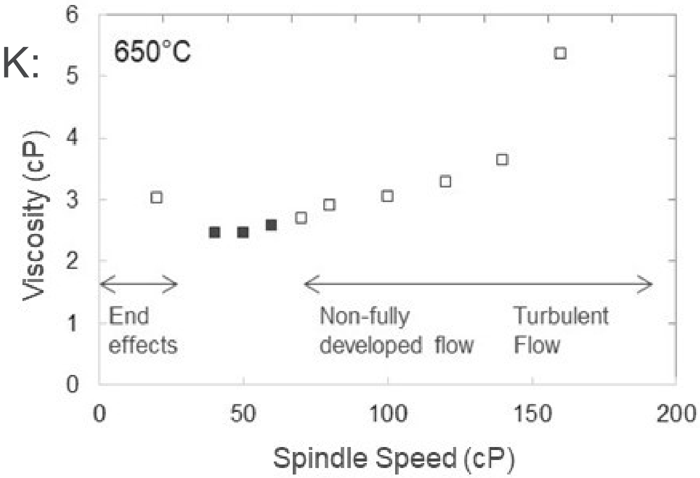
- Commercial viscometer head paired with custom metallic spindle and crucible
  - spindle and crucible manually polished to remove oxide layers prior to use
- Salts contained in an inert crucible
- Thermal insulation to maintain uniform temperature

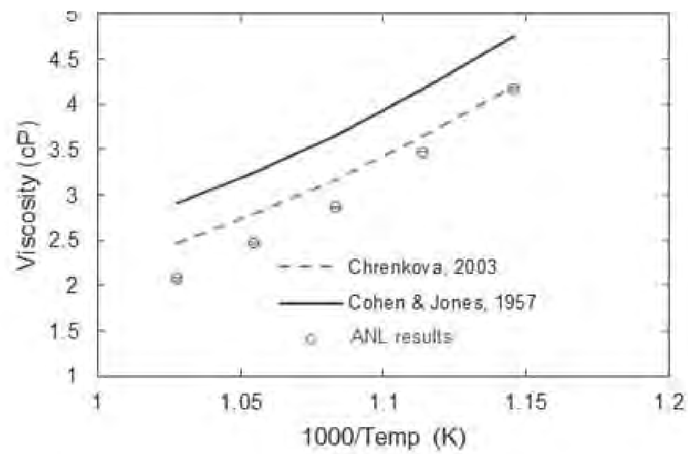


VISCOSITY DATA

Eutectic FLiNaK:

- Must avoid turbulent flow regimes in the annulus for a reliable measurement
- Viscosity depends on temperature and measurement is sensitive to rotational speed
  - Speeds that are too low have significant end effects
  - Speeds that are too high result in turbulence
  - Both end effects and turbulence artifacts increase measured torque and calculated viscosity, so minimum values are most reliable
- Sufficient startup time required to develop coquette flow
  - Lower temperatures require longer startup times due to more viscous flow conditions
- Take measurements at several spindle speeds and start up times to determine appropriate values for each salt mixture at every temperature





TIPS

- Maintain stable environment with low oxygen and moisture contents to prevent contamination of salt during preparation and measurements
- Properly insulating device to ensure isothermal operation
- Characterize salt composition before and after testing to detect preferential volatilization and contamination (e.g., DSC)
- Make measurements at several spindle rotational speeds to identify range of conditions providing stable laminar flow
- Use long (and different) start up times before taking measurements to ensure fully developed flow
- Make replicate measurements to verify results are not affected by volatilization, corrosion of spindle, or contamination of salt during measurements

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QUESTIONS