Molten Salt Reactor PROGRAM

Actinide Salt Loop and Sensor Development at Argonne

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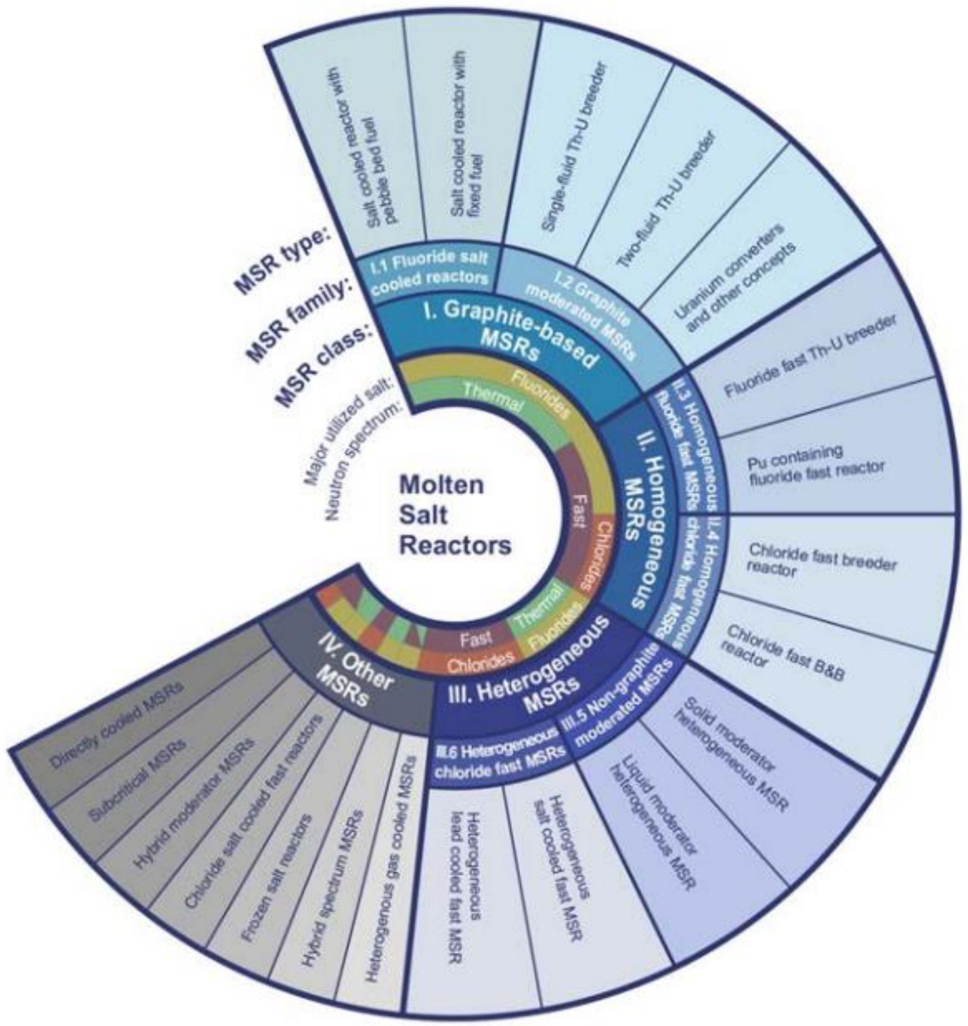
MSR-Relevant Testing Environments

The wide variety of MSR designs creates a need for flexible, modular testing environments to support technology development and fundamental studies.

To address this need, Argonne is currently preparing for installation of a pumped actinide flow loop within one of our large-scale transuranic gloveboxes.

Pumped, non-isothermal loops containing a range of actinides are the ideal analogue for a functional MSR. No such loops are currently operating within the U.S.



Molten salt reactor taxonomy[1]

[1]*Status of Molten Salt Reactor Technology, TR-489 IAEA 2024*

MSR Technology Gaps

Studies of Fundamental Phenomena

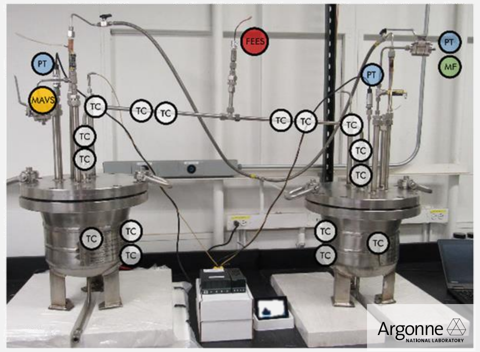
- Non-Isothermal Plate-out Phenomena
- Salt Chemistry
- Corrosion Behavior
- Multiphase Flow Phenomena
- Mass Transport

Technology Development

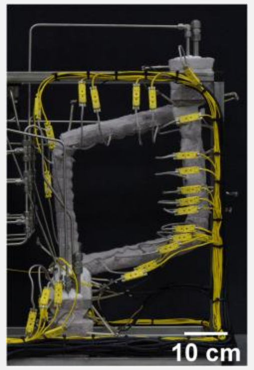
- Sensor Development
- Materials/Seals
- Corrosion Control Systems
- Automated Operations
- Digital Twins

Argonne's pumped actinide loops can help to address all these gaps within a single testing platform

Existing Types of Molten Salt Flow Systems



Twin Tank Pressure-Driven
Pros: Modular, flexible, operates with actinides
Cons: Isothermal, pressure driven with limited durations



[ref 1]

Thermal Convection Loops
Pros: Non-isothermal, can operate with actinides (most don't)
Cons: Very low flow rates; inflexible unless installed in glovebox

Large-scale Pumped Flow Systems
Pros: High flow rates, large geometric scales
Cons: Inflexible, expensive (esp. if using actinides or Be salts)

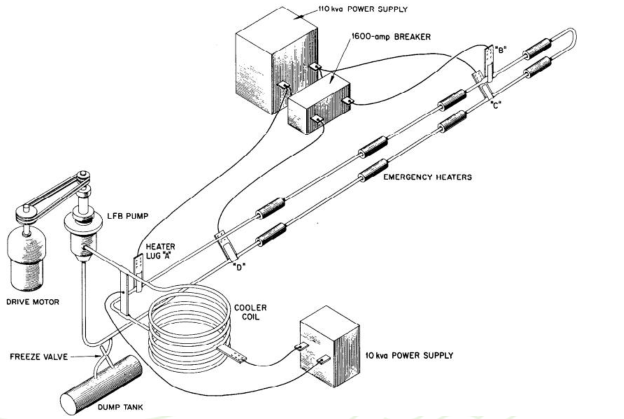
[1]Kelleher, Materials Today Volume 33, December 2022, 104358

Overview of MSR Loops

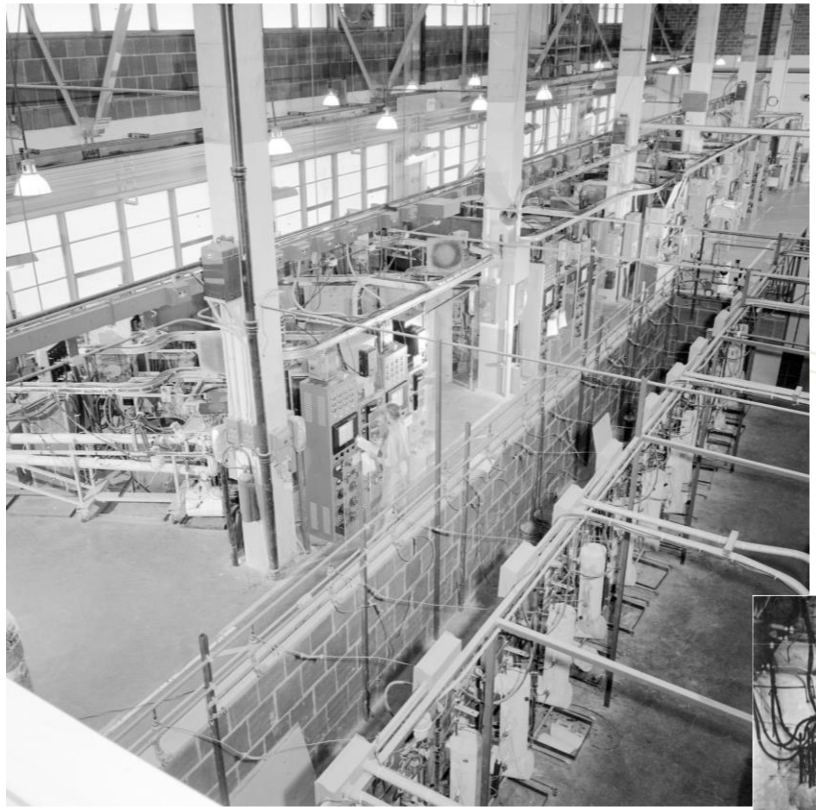
Historically: Thermal and forced convection molten salt loops have been deployed to study material compatibility and corrosion control mechanisms in a variety of salts.

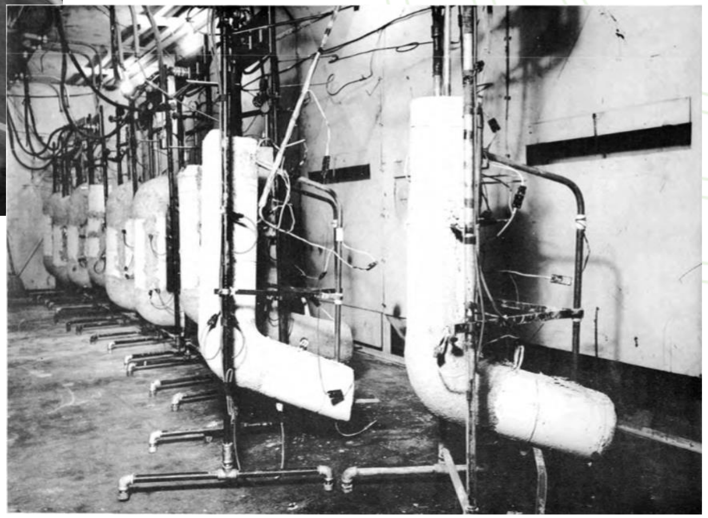
Operation of large numbers of radiological loops was previously possible, but modern safety requirements have made that difficult to achieve for high-hazard salt mixtures (e.g., actinide, Be, and other complex salts mixtures).

An updated approach for flow loops is needed to enable safe, flexible, and inexpensive operations with very high-hazard salts



. ORNL-TM-528, 1963.


ORNL-2684, 1959.

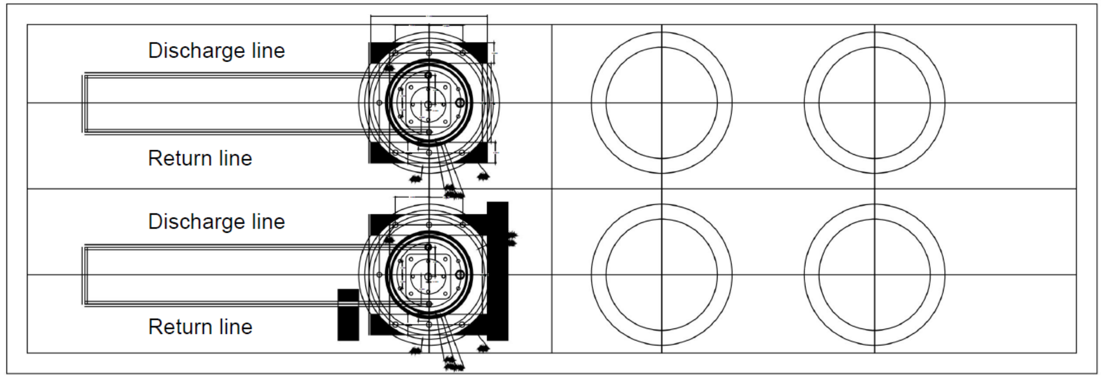

ORNL-2349, 1957.

In-Glovebox Pumped Flow Loops

Argonne's Flow Systems Development Glovebox is an excellent environment to test pumped loops containing MSR-relevant salts

- Located in Pu laboratory
- Glovebox enables easy handling of actinide salts
- Inert atmosphere prevents deleterious moisture and oxygen ingressions
- Enables modular, flexible flow systems that can be readily modified and upgraded

Integration of a loop into the glovebox creates challenges with respect to thermal management, electrical infrastructure, pressure safety, etc.



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Pump and Reservoir Design

A pump suitable for use in the HCA glovebox has many requirements:

- Must fit into existing furnace well
- Must fit into transfer lock for insertion and removal from glovebox
- Installation and mobility via overhead chain hoists
- Sufficient thermal insulation to protect glovebox structures and seals

Argonne has created a combined pump/heat shield assembly that can safely and effectively operate in the glovebox.

Molten salt pump partially inserted into furnace well with salt vessel

Pump and heat shield assembly disassembled into three sections

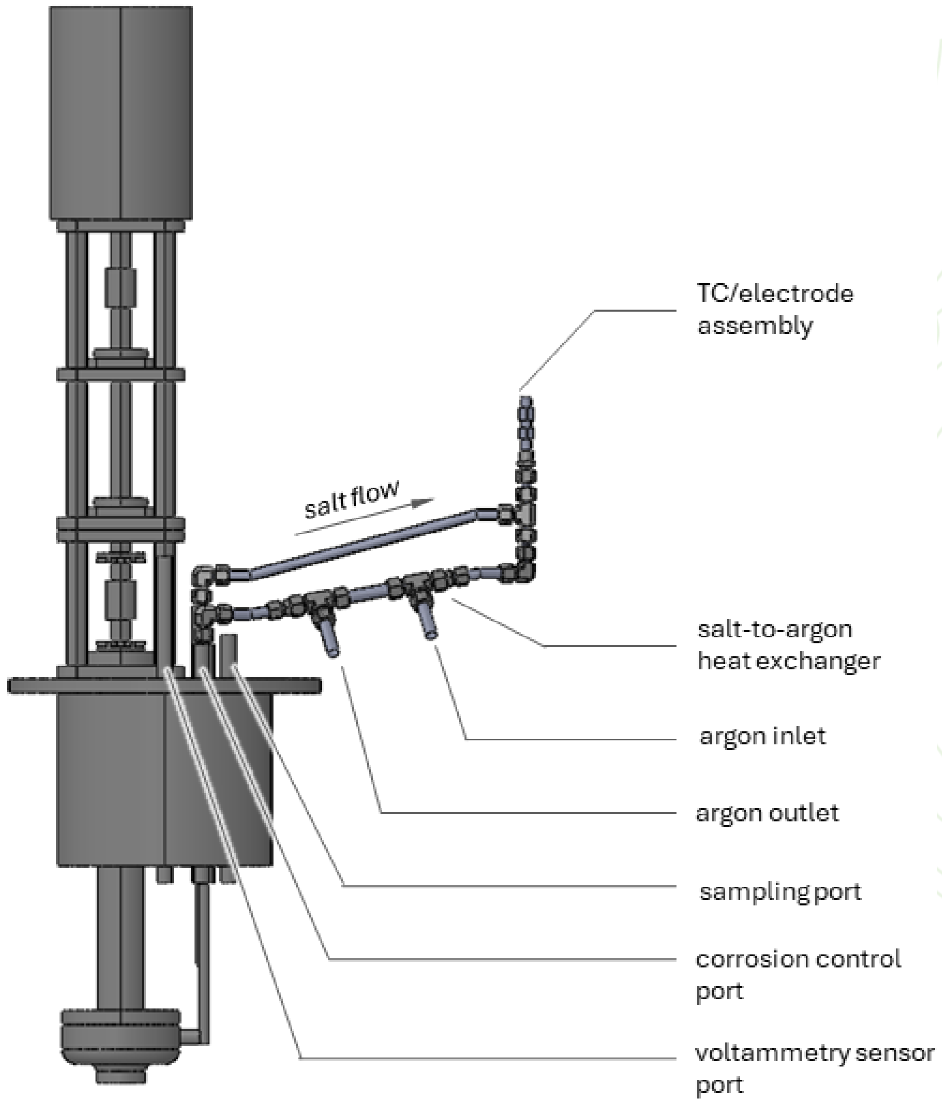
Loop Design

The initial loop design is intended to investigate non-isothermal effects in highly-loaded salts relevant to molten chloride fast reactors

- Includes salt-to-argon heat exchanger
- Ports for various sensor technologies
- Port for corrosion control system
- Loop uses inexpensive fittings and materials as risk of leaks and ingressions is mitigated by controlled atmosphere

Additional capabilities and technologies can be readily added

- Modifications possible even during active operations at high temperatures



Molten salt loop including salt-to-Ar heat exchanger, monitoring ports, and chemistry control system

Glovebox Preparation

The flow systems glovebox is being upgraded with improved thermal management capabilities, updated furnace wells and liners, and additional electrical feedthroughs. 90 kg of uranium metal has been procured to support salt synthesis ($NaCl\text{-}KCl\text{-}UCl_3$).

Flow Systems Development Glovebox

Disassembled furnace well with updated cooling system

Uranium metal being prepared for transfer to Bldg 205

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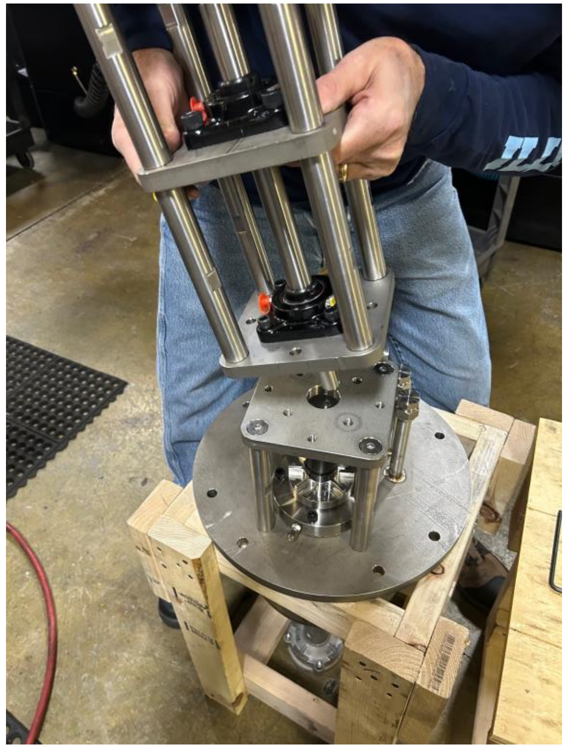
Loop Fabrication and Testing

Fabrication of the pump and heatshield assembly has been completed. Water testing and generation of pump curves is underway. Fabrication of ancillary components is nearing completion.



Pump assembly

Pump and heat shield pack

Assembled pump / heat shields

Electrochemical probe for actinide loop

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Monitoring and Control Systems

Implications of Contaminant Species

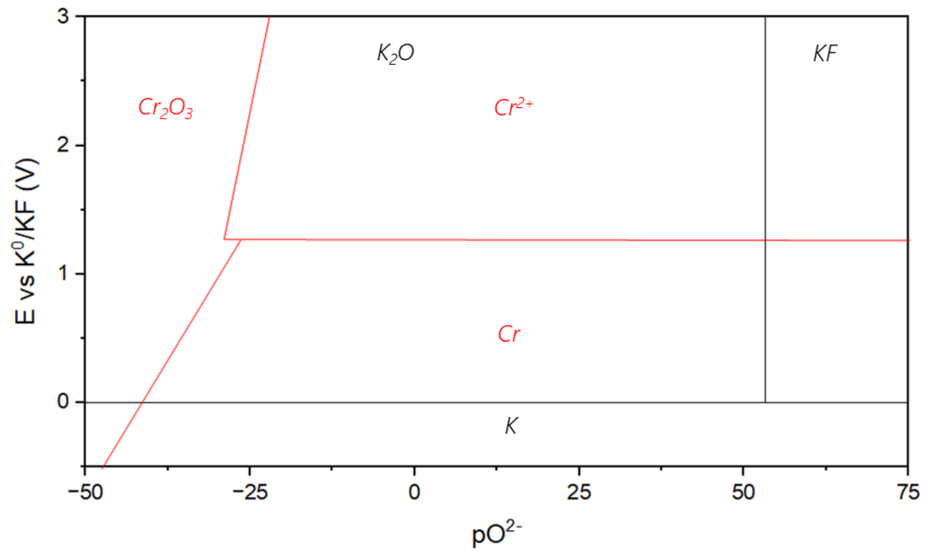
Undesired contaminants may negatively impact operational integrity and safety of MSR system

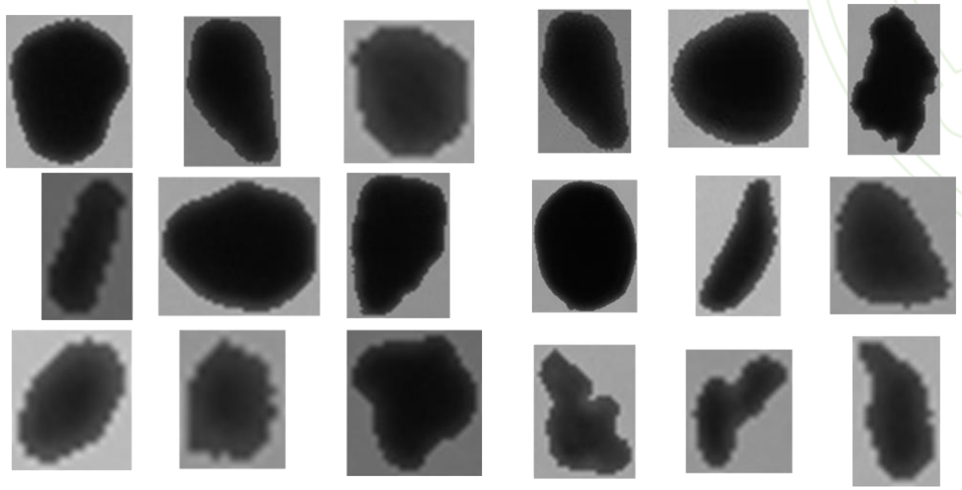
- E.g.: $U^{4+} + 2O^{2-} \rightarrow UO_{2\,(solid)}$

These species are introduced through various pathways, including:

- Structural corrosion
- Atmospheric ingressions
- Inadequate salt purification

Characterization of contaminants informs pathways towards effective corrosion monitoring and control mechanisms

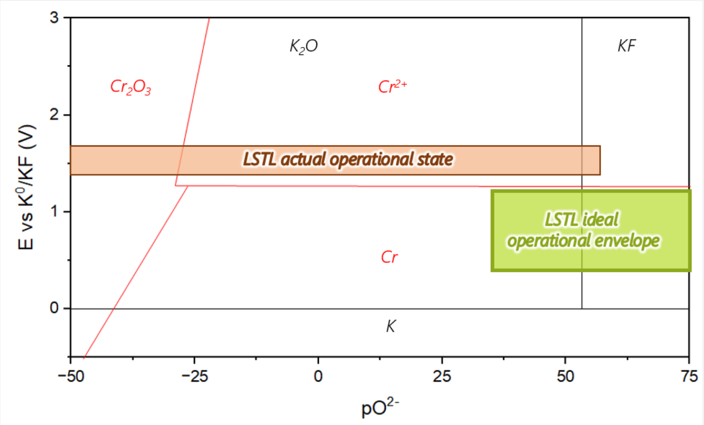




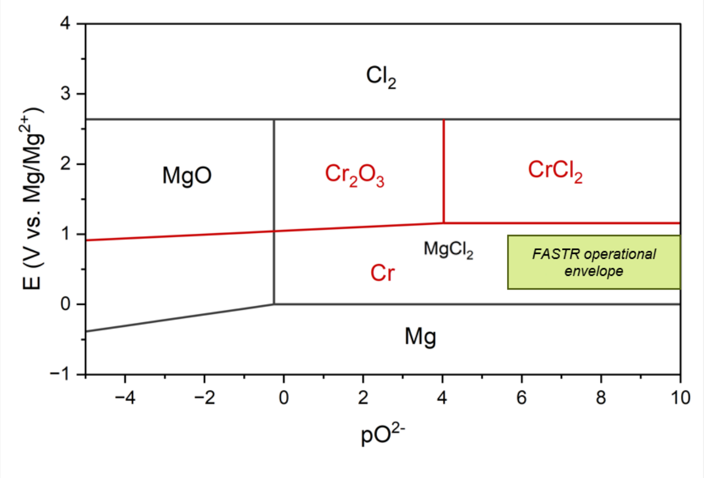
Guo, et al. ANL/CFCT-24/16 2025
(DOE NE MRWFD-funded)

Development and Deployment of Salt Chemistry Monitoring and Control Systems

Operational Envelopes

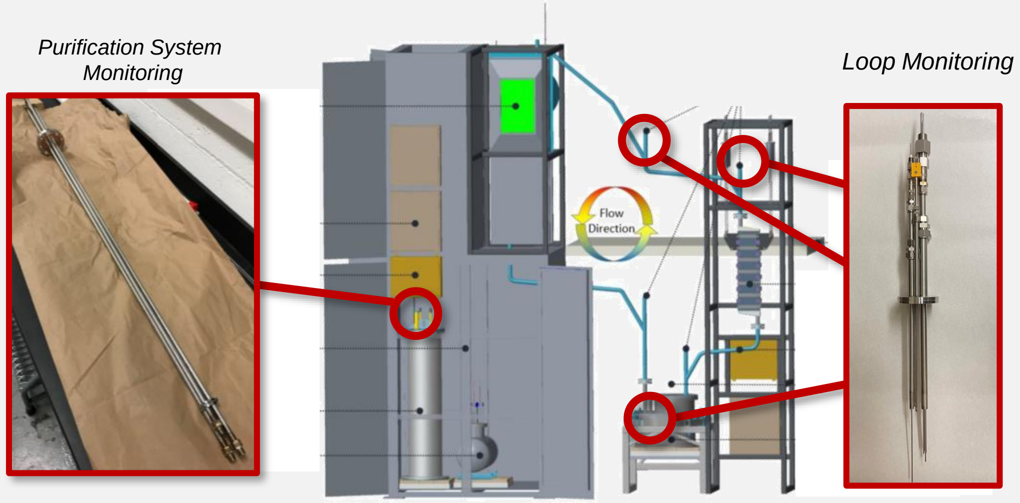


typical fluoride operational envelope

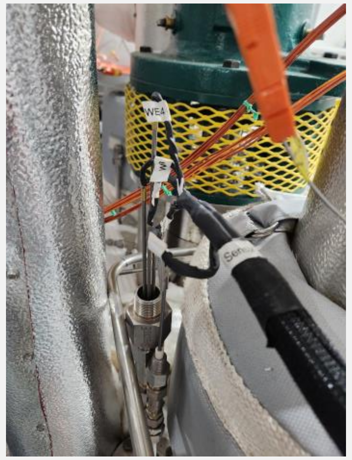


typical chloride operational envelope

Deployed Online Monitoring Tools

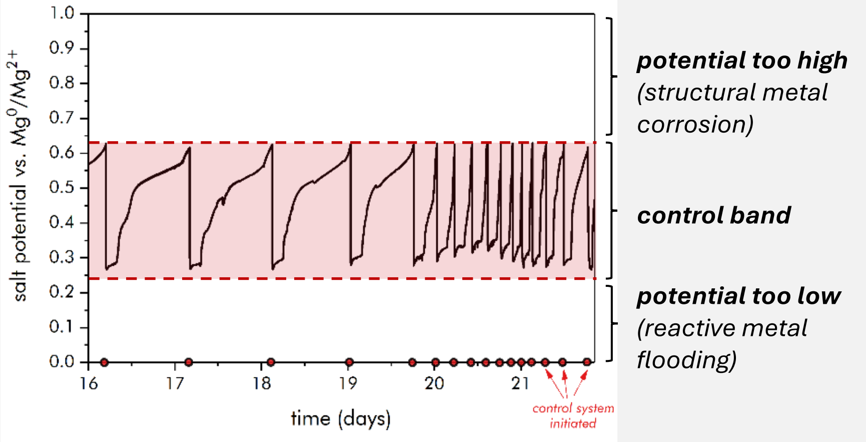


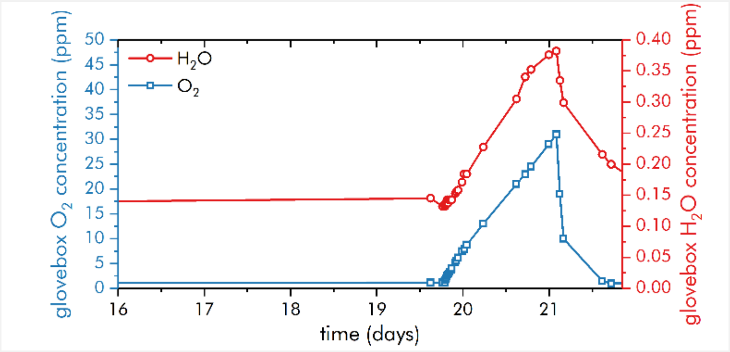
FASTR loop at ORNL



sensor probe assembly after operations

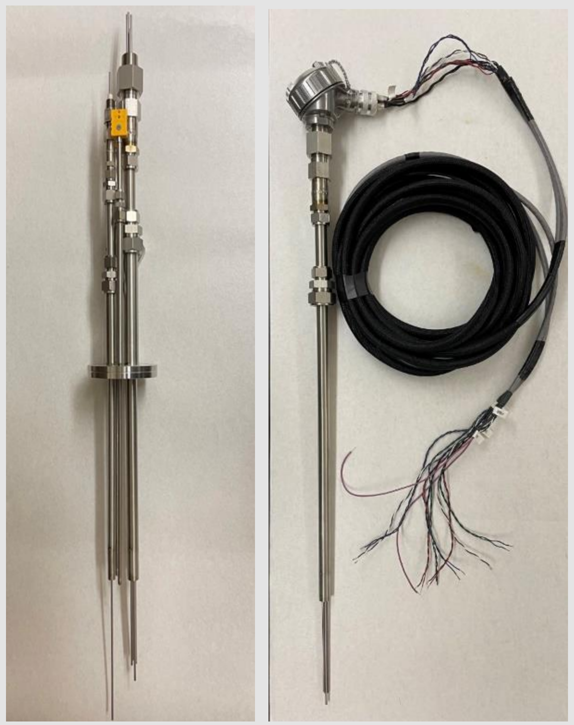
Automated Salt Chemistry Control



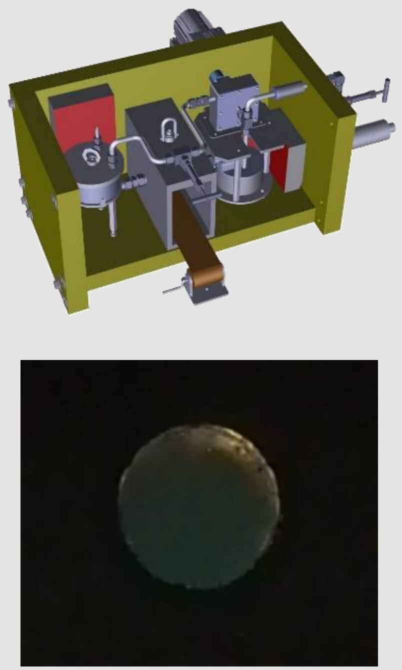


Molten Salt Monitoring Approaches at Argonne

Characterization of solution- and solid-phase species are crucial to maintaining the operational integrity of molten salt systems

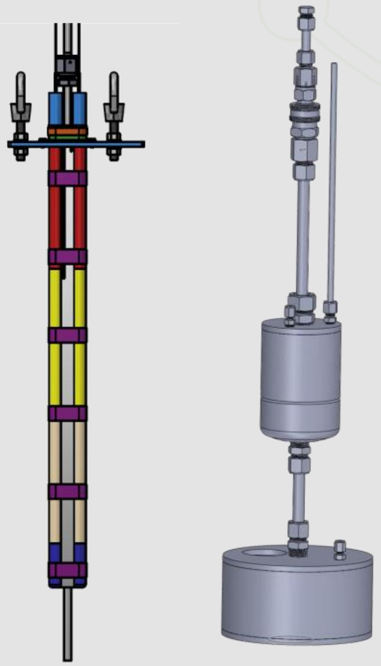


Electrochemical Monitoring of Salt Composition



Windowless Optical Monitoring of Composition

NA-22 Safeguards Portfolio



Automated Salt Sampling

DOE NE MPACT

Particulate Monitoring

DOE NE MRWFD

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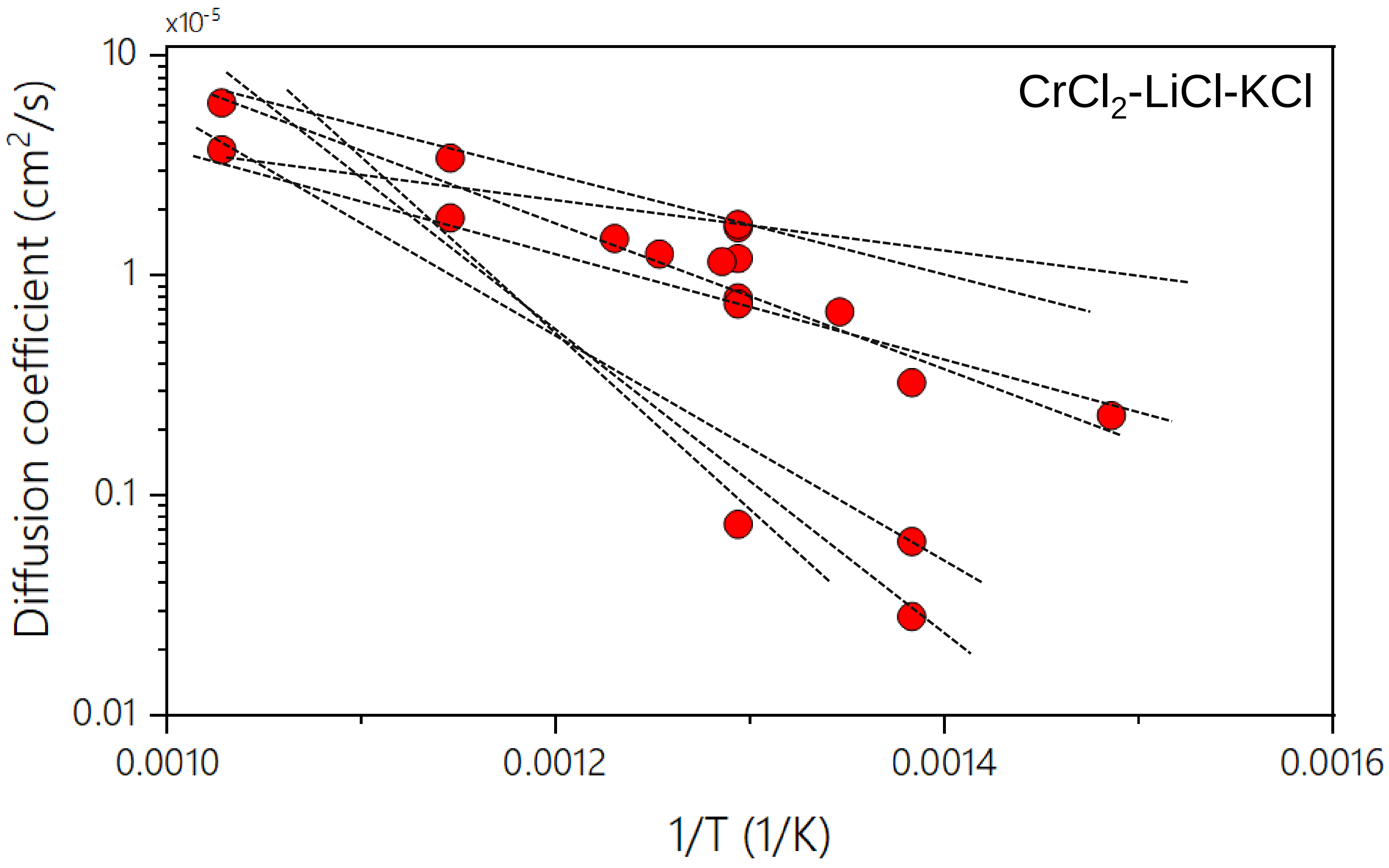
Electrochemical Sensor Development

Electrochemical sensors can be deployed inline and at temperature for extended periods of times

- E.g.: Argonne's multi-electrode array sensor at INL

Accuracy and precision of sensor readout depends on well-established fundamental values that are not yet realized in the field

Diverging and technique-dependent values reported in literature indicate improper care towards accounting for non-idealities



Argonne is developing deployable sensors that can reliably meet practical process design and regulatory requirements

F. Lantelme et al. J. Electroanal. Chem., 1992,337(1), 325.
S. Yoon et al., JES, 2021, 168, 013504.
N. A. Shaheen et al., JES, 2024, 171(12), 126502.
J. Park et al., JES, 2017, 164, D744.
A. Cotarta et al.,R. Roumaine de Chimie, 2002, 47(7), 597.

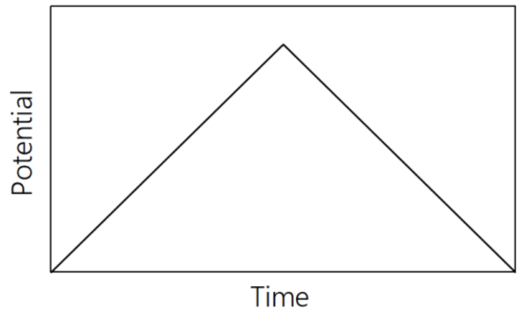
Development of Electroanalytical Techniques

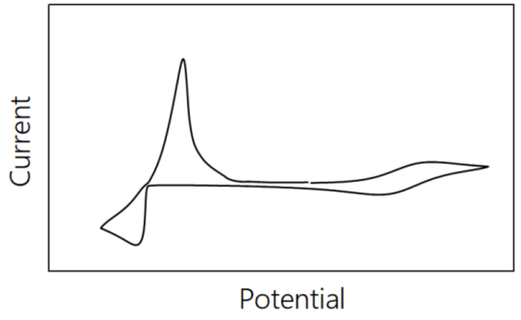
Argonne is developing complementary electroanalytical approaches rooted in fundamental theory to advance electrochemical sensors capable of deployment in MSRs

<u>Linear Sweep Voltammetry (LSV)/ Cyclic Voltammetry (CV)</u>

- Analytically simple
- LOD: 1 ppm

- Non-idealities are less apparent than pulse techniques

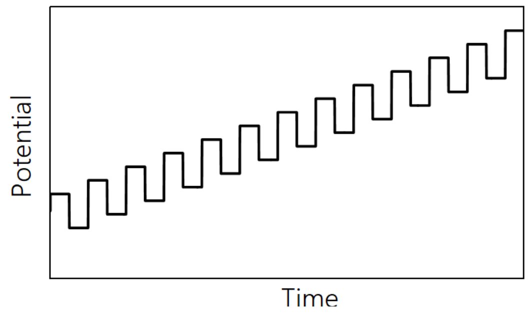


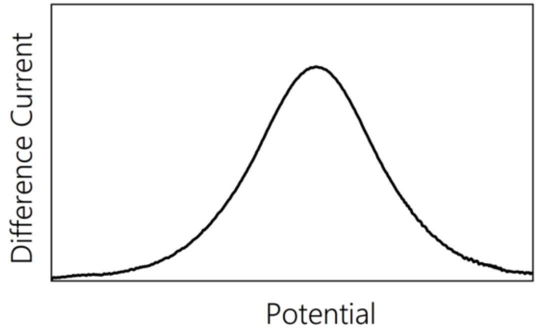


<u>Square Wave Voltammetry (SWV)</u>

- Rapid measurement
- LOD: 1 ppb

- Analytically intensive
- <u>Very sensitive to non-idealities compared to LSVs/CVs</u>

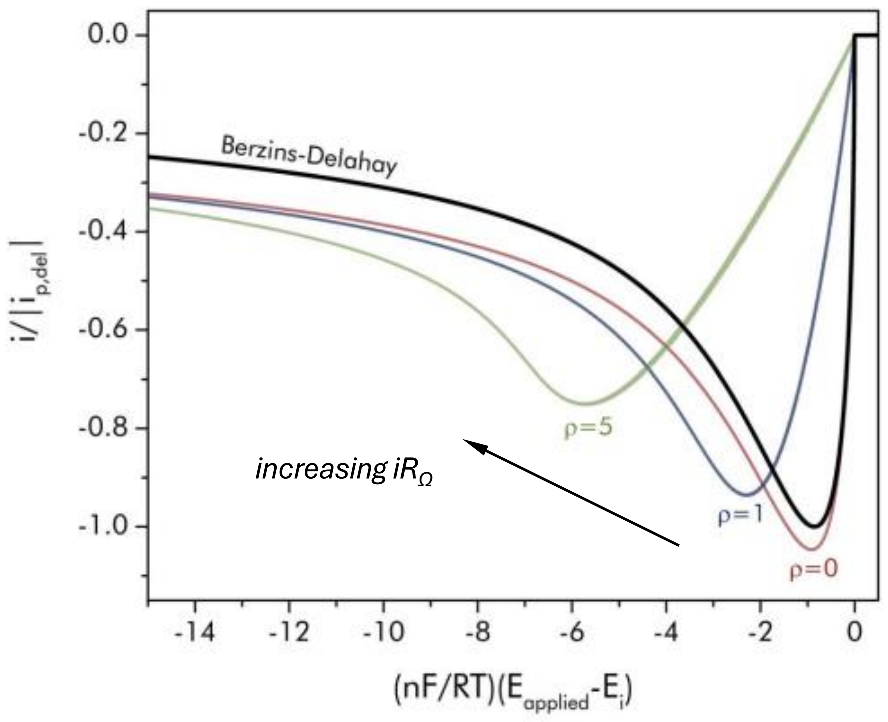




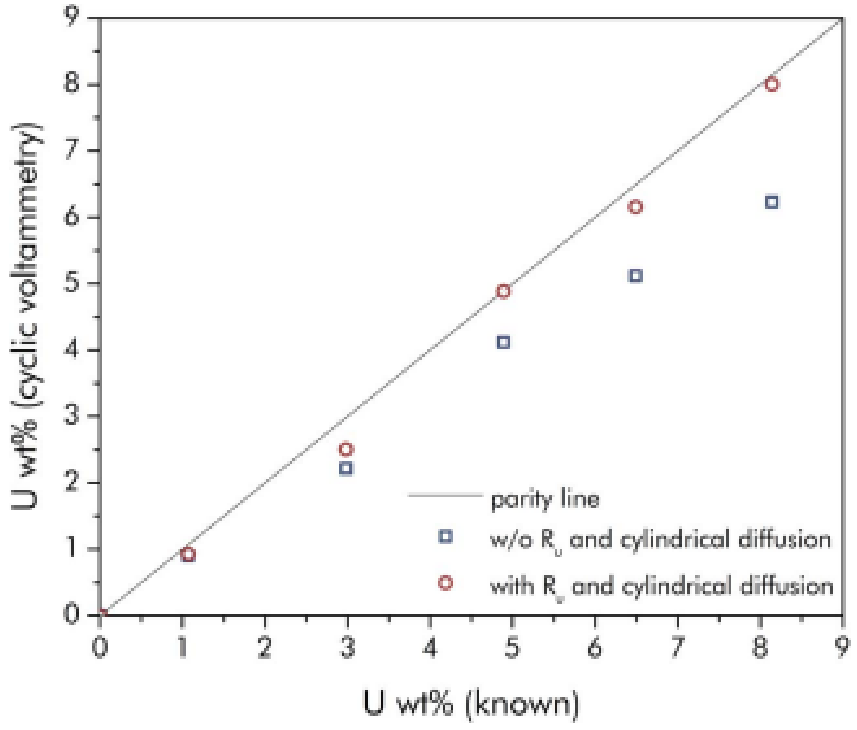
Accounting for Non-Idealities in LSVs

Previous work conducted by our group focused on simulating non-idealities in LSVs to address poor performance of LSVs at MSR-relevant concentrations

- These analyses successfully addressed Ohmic resistance effects that otherwise cause LSV measurements to be attenuated by a factors of 2x or more



Simulated LSVs with various amounts of Ohmic resistance



Parity plot of U with and without correction

N. C. Hoyt, et al., *JES*, 164(2) H134 (2017).

work funded by DOE NE MPACT

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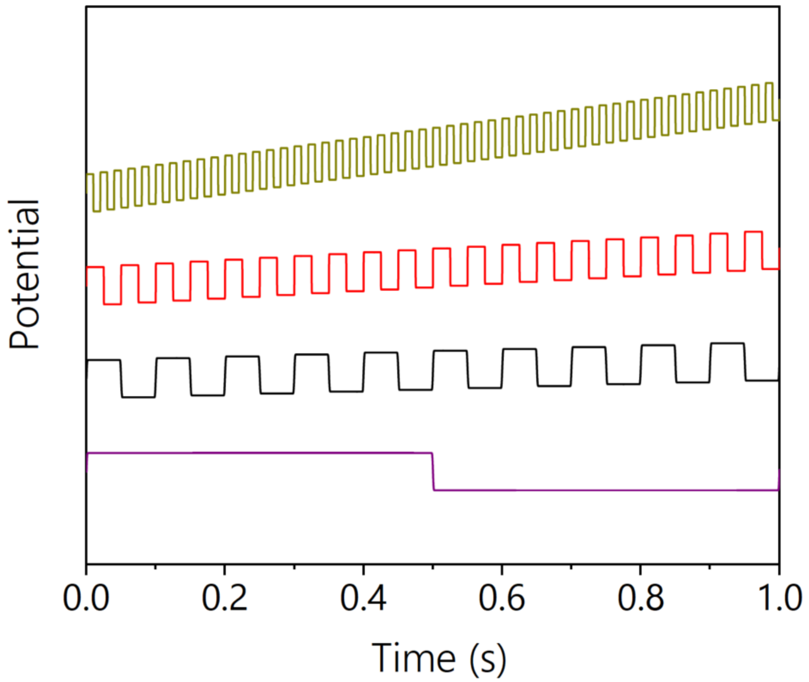
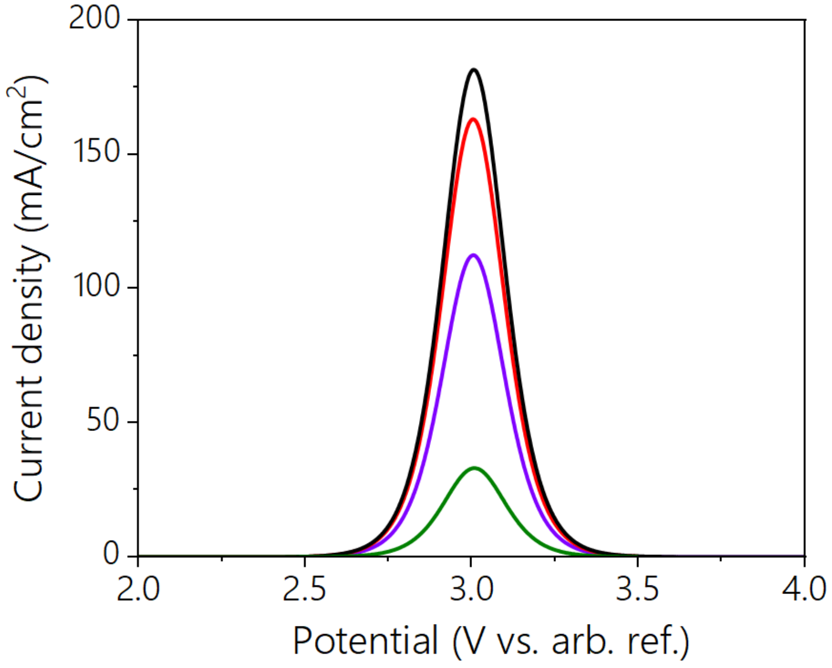
Accounting for Non-Idealities in SWVs

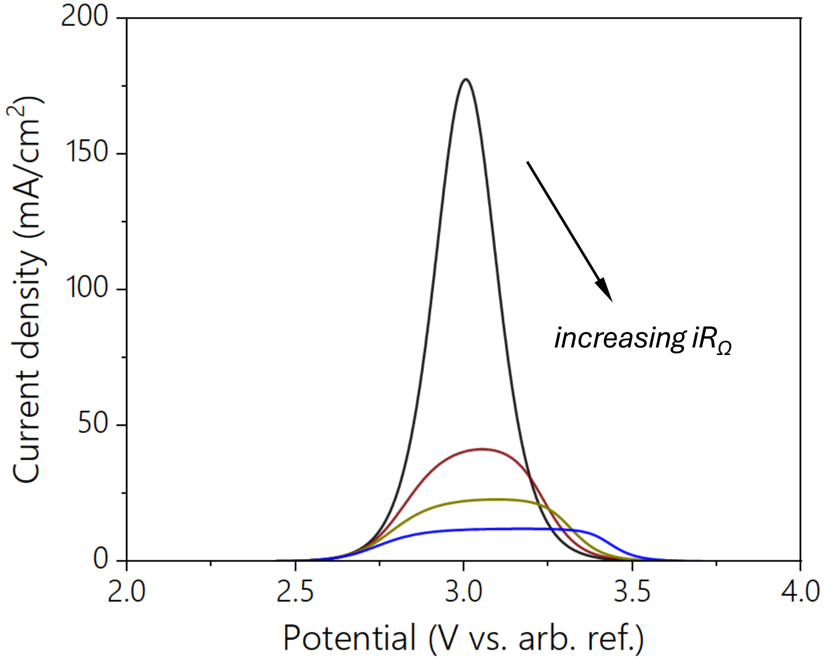
Square wave voltammograms were simulated with varying degrees of Ohmic resistance contributions to generate relationships between observed responses and those from predicted from theory



Example potential-time profile of SWVs at various frequencies



Example simulated SWVs at various frequencies



Example simulated SWVs with increasing amount of IR

N. A. Shaheen, et al. *JES*, 2024, 171, 126502.
N. A. Shaheen, et al., ANL/CFCT-23/39.

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Implications of Non-Idealities on Sensing

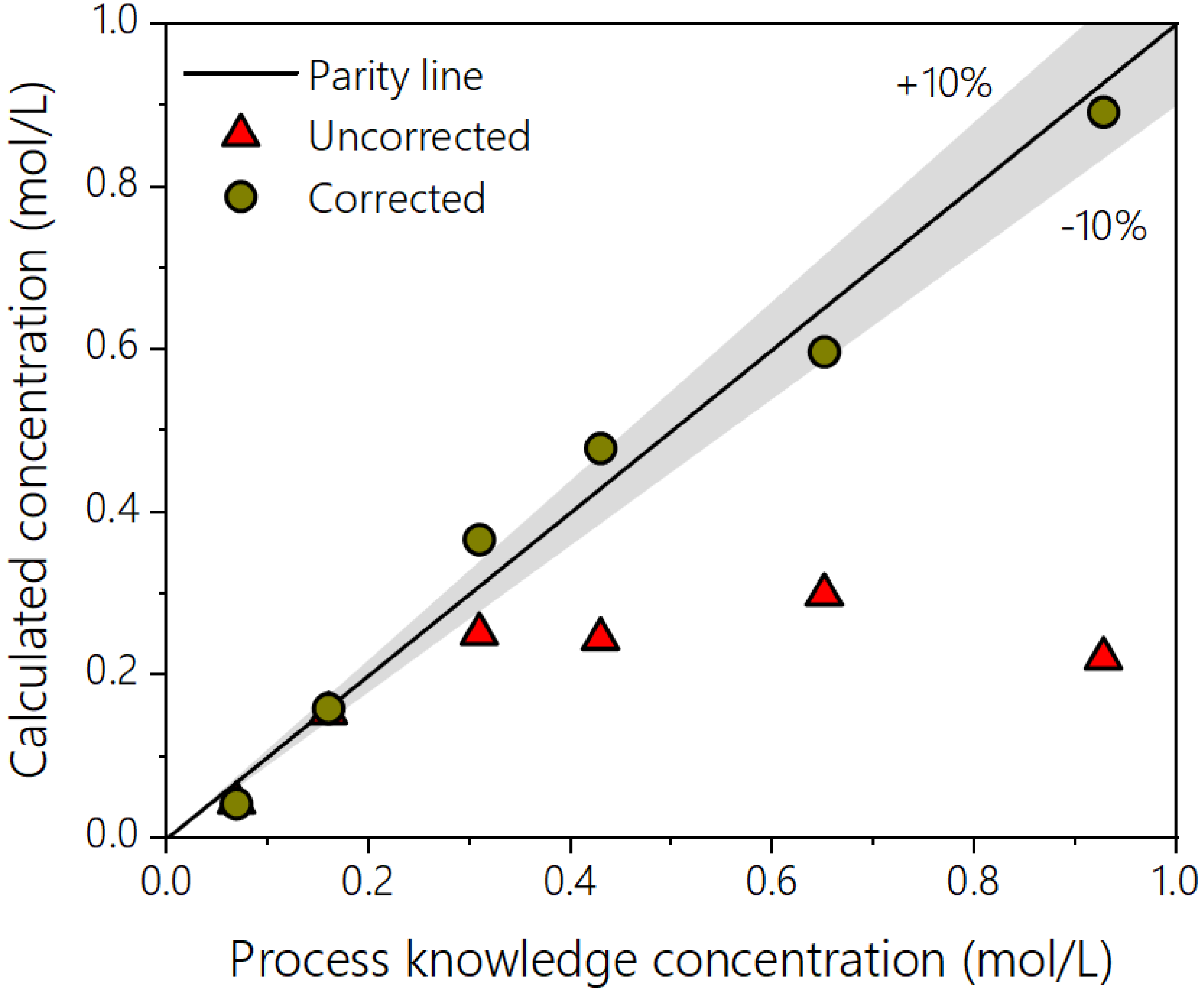
CVs were collected to independently determine diffusion coefficients

- Used to calculate concentrations from SWV measurements

Parity plot shows that failure to use correction factors can lead to:

- Erroneous, technique-dependent diffusion coefficients
- Substantial underpredictions of solubility limits

Implications on a number of process design and safeguards considerations



Parity plot of corrected and uncorrected concentrations

N. A. Shaheen, et al. *JES*, 2024, 171, 126502.

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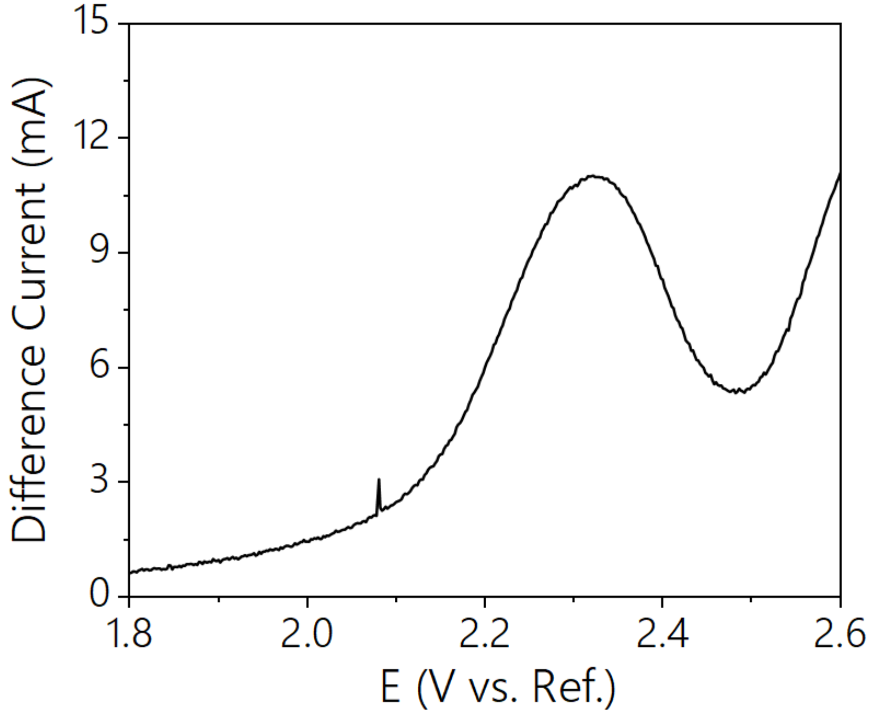
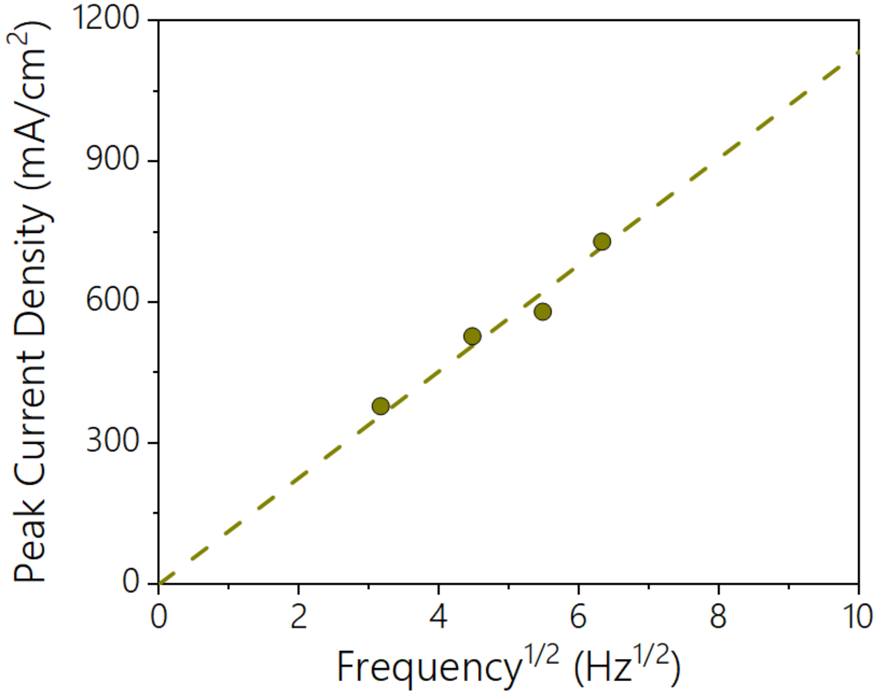
Extending Approach to Oxides and Other Contaminants

Dissolved oxides are particularly challenging to detect and characterize on account of their reactivity and potential for phase changes

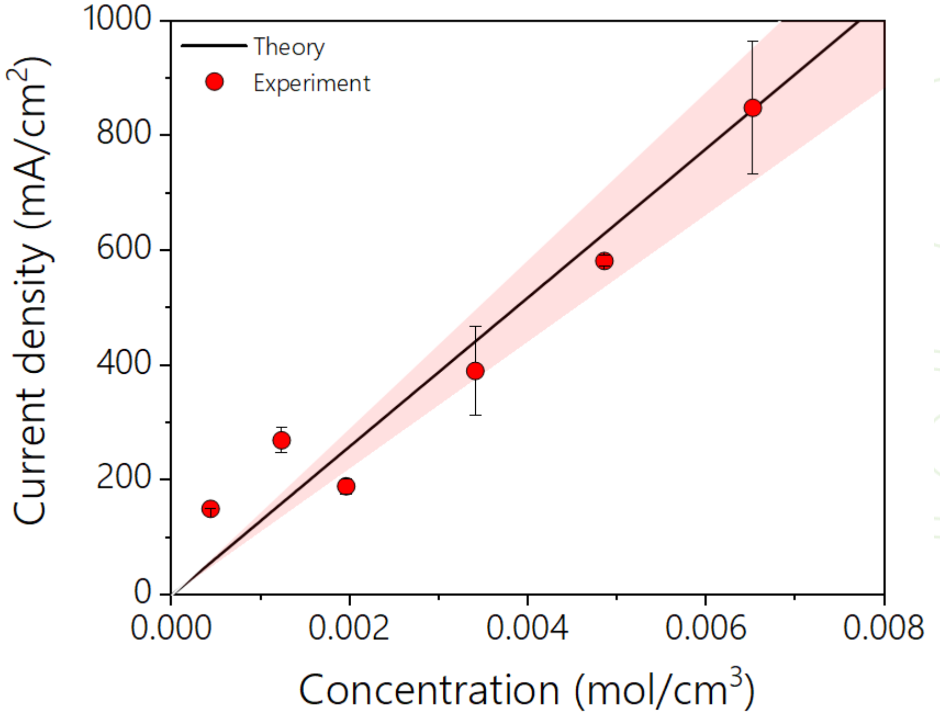
- Our group is characterizing fundamental properties associated with reactive oxygen species in MSR-relevant salts
- **We have been able to achieve accurate, linear oxide measurements over a very wide range of concentrations**



Representative SWV of dissolved oxides



Representative relationship between peak current density and frequency



Representative relationship of current density versus concentration

N. A. Shaheen and N. C. Hoyt, *JES*, under review.

Milestones and Achievements

Milestone Number	Title	Due Date	Status
M3AT-25AN0702031	Installation of Molten Salt Flow Loop with Chemistry Monitoring and Control System	09/30/2025	On time
M4AT-25AN0702032	Molten Salt Sensor Operations	09/30/2025	On time

Presentations and Publications:

Conference Presentations:

N. A. Shaheen, J. Guo, and N. C. Hoyt (2024) "Quantification and correction of impedance effects on square wave voltammetry for high concentration soluble-soluble reactions." ECS PRiME.

N. A. Shaheen and N. C. Hoyt (2025) "On the electro-oxidation of oxides in molten FLiNaK." 248th ECS Meeting.

Publications:

N. A. Shaheen, J. Guo, and N. C. Hoyt (2024) "Quantitative Correction of Ohmic Effects on Square Wave Voltammetry for High-Concentration Soluble-Soluble Redox Reactions in Molten Salts." *J. Electrochem. Soc.*, 171,126502.

N. A. Shaheen and N. C. Hoyt (2025) "Electrochemical characterization of dissolved oxides in molten FLiNaK." *Submitted.*

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Thank you

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