On-line, In-line, and At-line Monitoring of Molten Salt Systems

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ARGONNE NATIONAL LABORATORY

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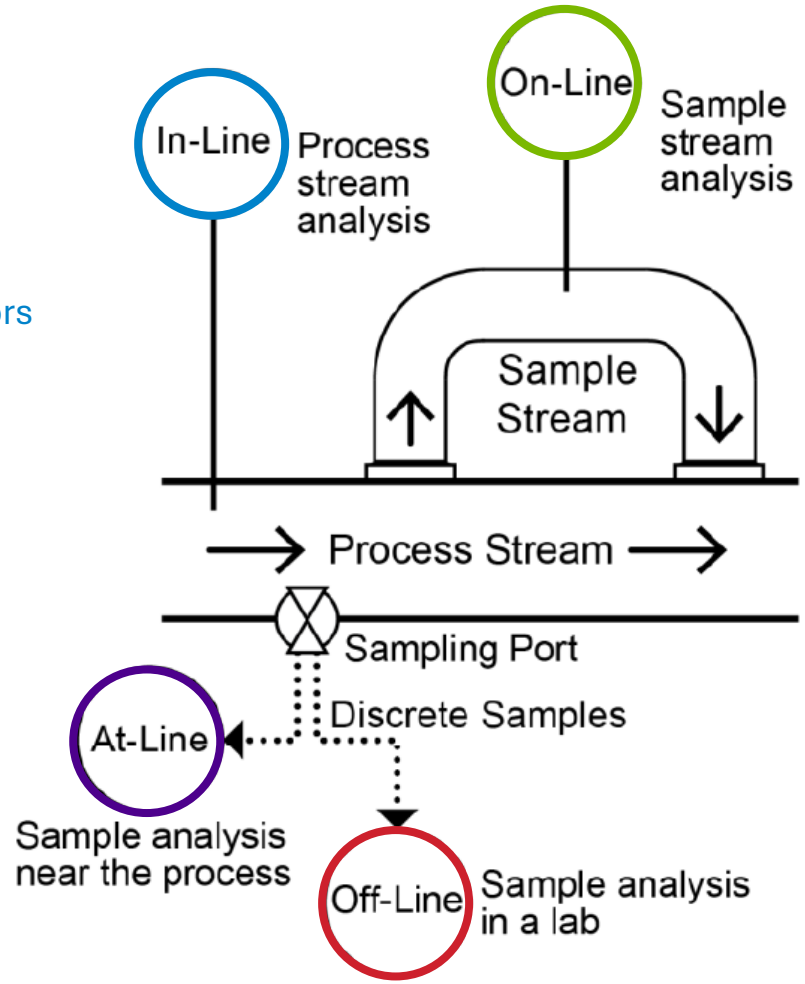
Pros and Cons of Salt Analysis Methods

- **In-line/in-situ**
  - Pros
    - Real time analysis
    - No pathway for diversion
    - Non-destructive analysis
  - Cons
    - Fixed point sample analysis
    - Sensor longevity
    - Cannot use off-the-shelf sensors
- **At-line**
  - Pros
    - Samples remain in “process custody”
    - Allows for lower-temperature, lower-radiation location of analysis
    - Near real time analysis
    - Enables high-throughput samples
    - Can use off-the-shelf sensors
  - Cons
    - Potential diversion path



- **On-line**
  - Pros
    - Enables high-throughput samples
    - Allows for lower-temperature, lower-radiation location of analysis
    - Near real time analysis
    - No pathway for diversion
  - Cons
    - Sensor longevity
    - Cannot use off-the-shelf sensors
- **Off-line**
  - Pros
    - Traditional method of analysis
    - Can use off-the-shelf analysis
  - Cons
    - Often requires large sample of highly radioactive material
    - Delayed analysis
    - Often fixed-point sample analysis
    - Destructive analysis

MATERIAL PROTECTION, ACCOUNTANCY AND CONTROL TECHNOLOGIES PROGRAM

SALT MONITORING DEVELOPMENTS UNDER THE MPACT PROGRAM

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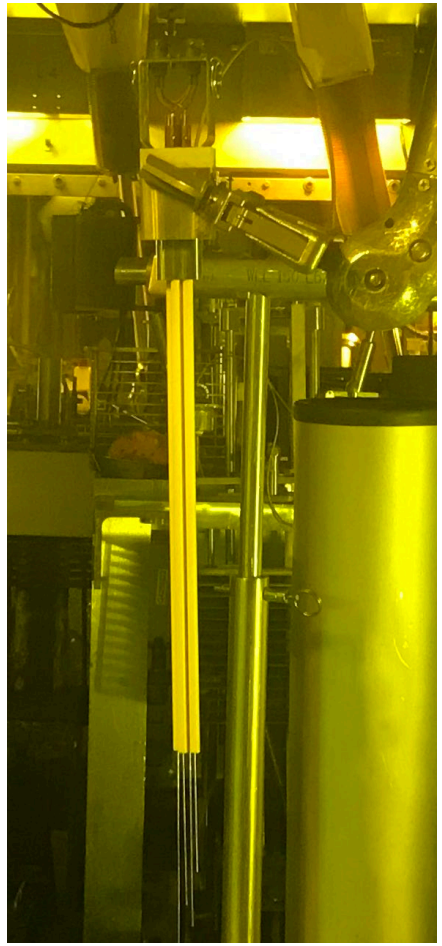
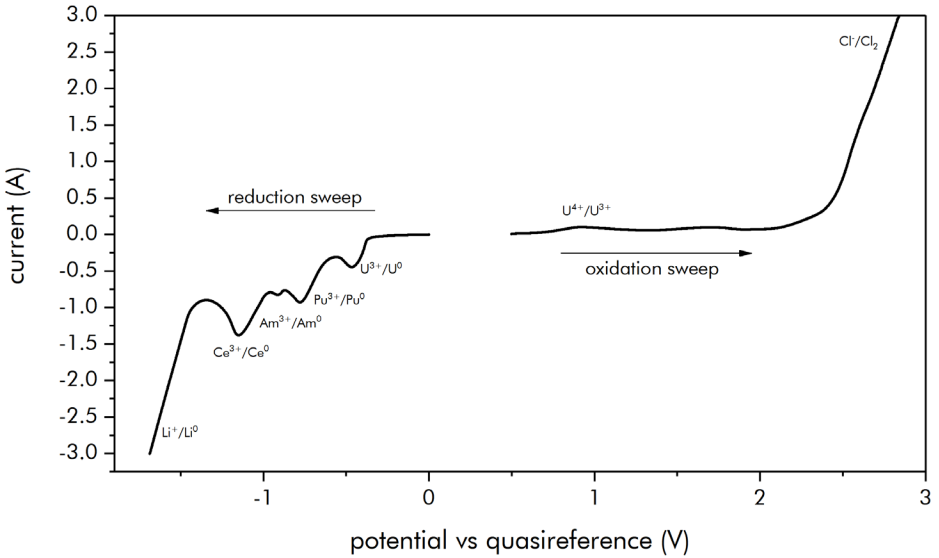
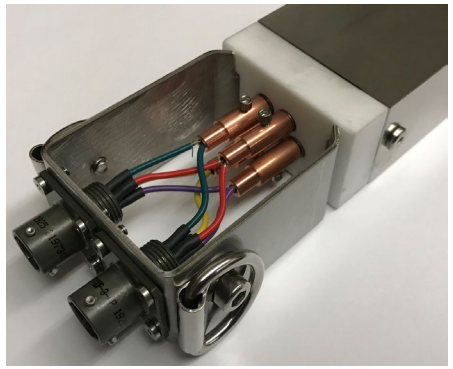
MPACT Voltammetry Sensor Deployment Activities

Project Goals:

- Deploy safeguards-enabling multielectrode array electrochemical sensors to hot cell facilities
- Provide salt composition, salt level, and salt redox potential measurements during a variety of electrorefining operations for spent nuclear fuel
- Assess longevity, uncertainty, stability, and accuracy of the sensors in real-world conditions







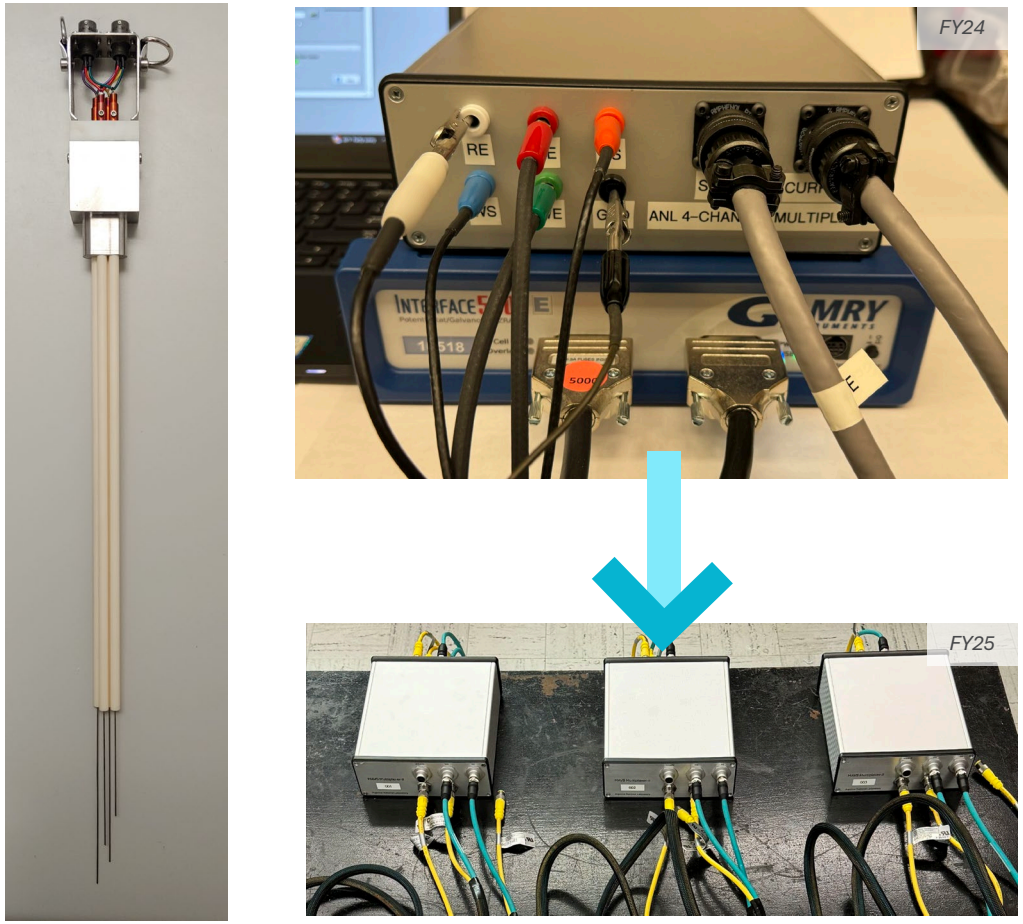
Argonne-developed deployable voltammetry sensor

Updated Hardware for Automated Operations

Argonne has successfully demonstrated voltammetry sensor measurements over years-long operations in spent fuel

A variety of hardware improvements have been pursued to further improve long-term performance

- Replacement probes manufactured as backup in case of damage to deployed system
- Updated wiring and electrical connectors
- New custom multiplexer hardware to control measurements across electrode array
- Improved software and automated analysis tools

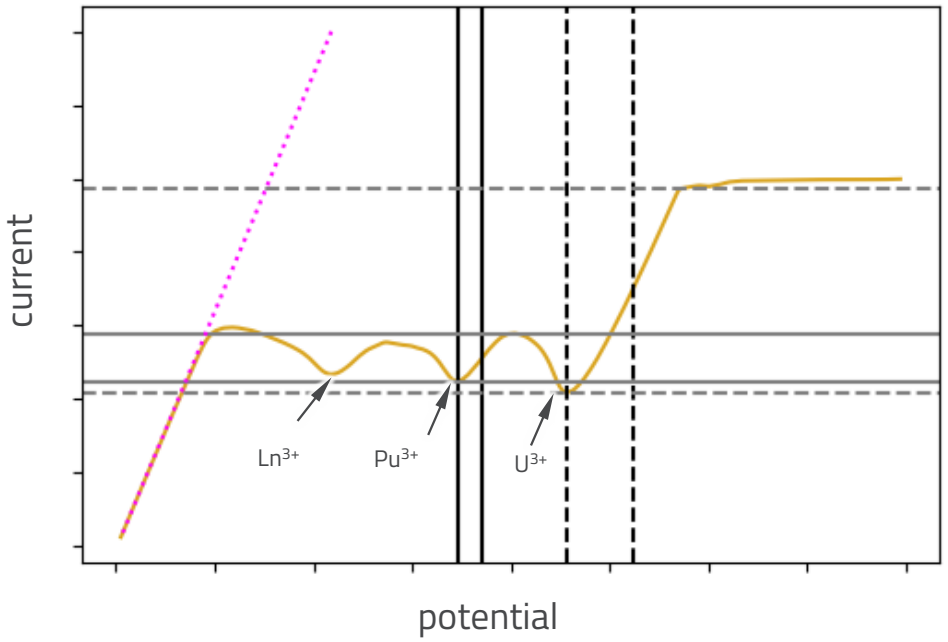


Replacement probe (left), previous custom multiplexer (upper right), and updated custom multiplexers (lower right)

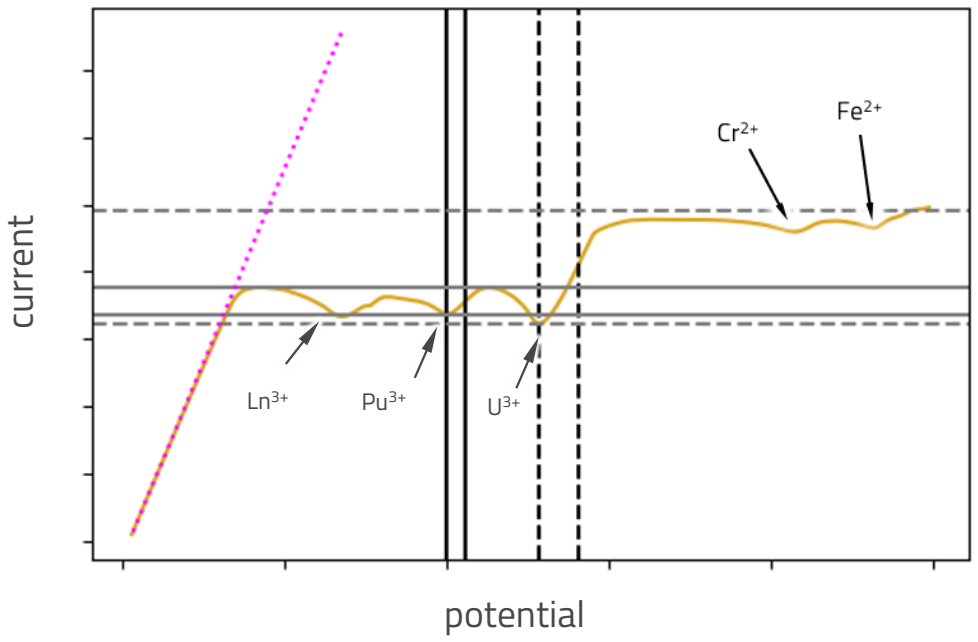
Multianalyte Systems

Spent fuel operations inherently involve multiple analyte species, but real-world operations often leads to the presence of additional complicating impurities

Typical Electroanalytical Sweep in LiCl-KCl Containing Dissolved Spent Fuel

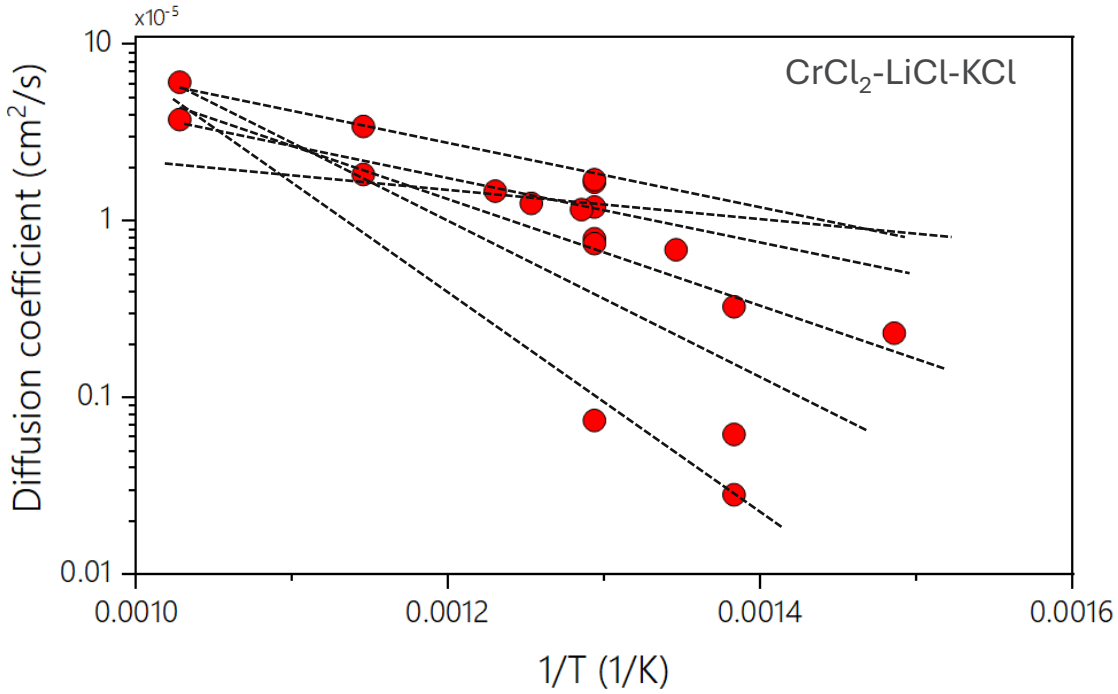


Typical Electroanalytical Sweep in LiCl-KCl Containing Dissolved Spent Fuel and Corrosion Products



Challenges with Multianalyte Impurities

- Alternate techniques can be helpful in quantifying species such as corrosion products
- Accuracy and precision of impurity measurements depend on well-established fundamental values that are not yet realized in the field
  - Major discrepancies in fundamental values such as diffusion coefficients exist in the literature
  - The technique- and apparatus-dependent values suggest unresolved problems associated with electroanalytical practices



$$c_k \sim i_{peak,k} / \sqrt{D_k}$$

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.

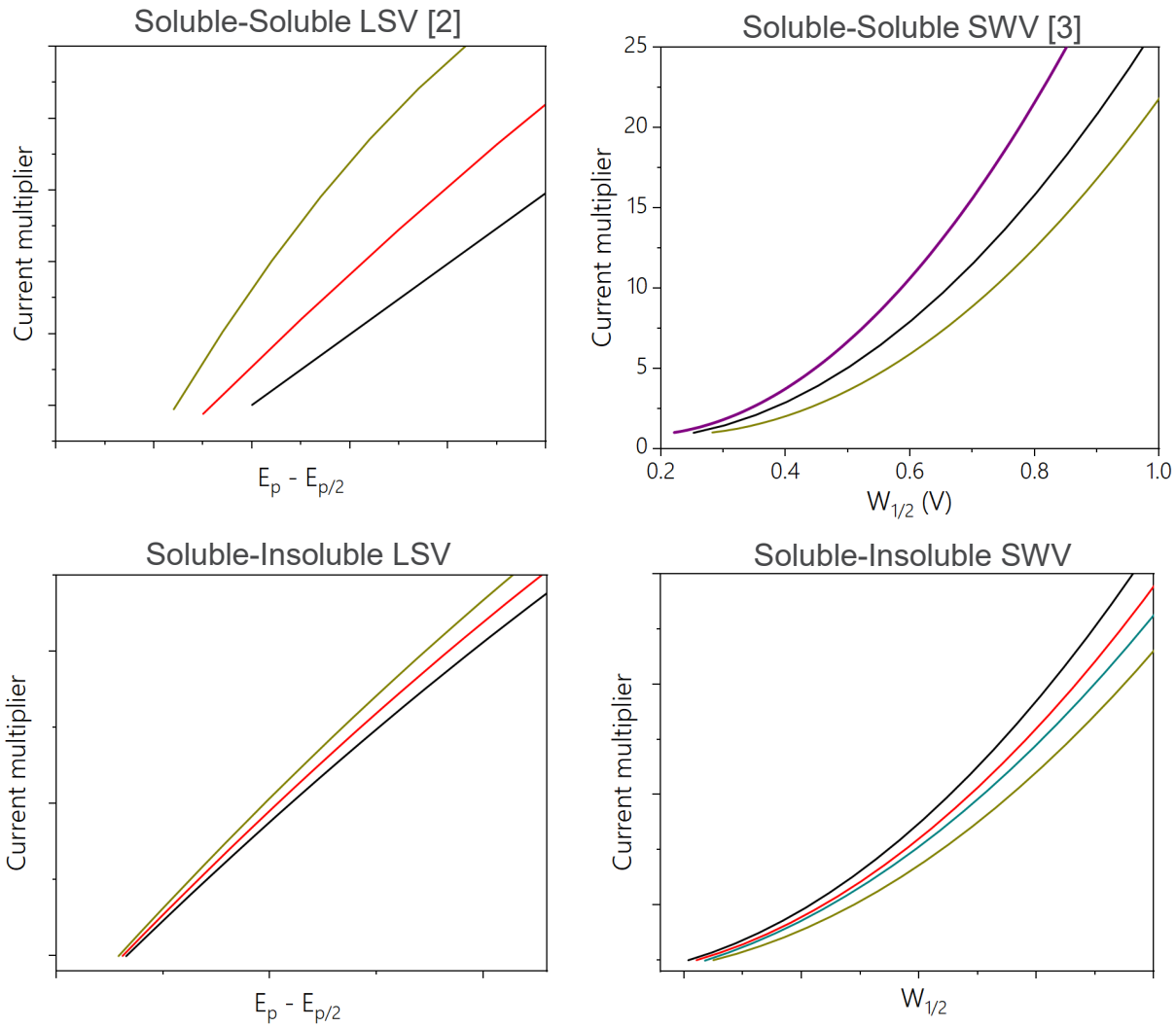
Challenges with Real-world Non-idealities:

Current Multipliers

- Current multipliers (γ) have been developed to resolve major non-idealities
  - LSV current multipliers developed in 2017 [1]
  - Additional multipliers developed in subsequent years
  - Multipliers for soluble-insoluble and soluble-soluble SWV developed this year

- The factors holistically correct for a broad range of non-idealities and enable linear responses across typical performance ranges

$$i_{adjusted} = \gamma\left(W_{1/2}, T, n, etc.\right) i_{measured}$$

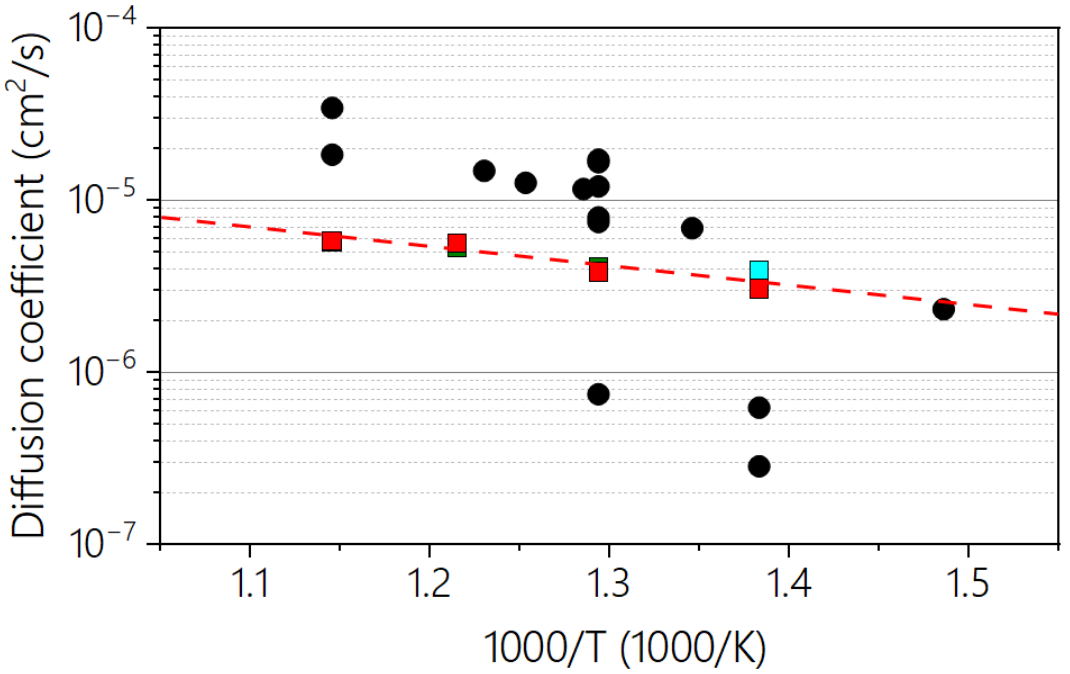


[1] Hoyt, et al. JES (2017)
[2] Doniger, et al. under review.
[3] Shaheen, et al. JES (2024)

Resolved Challenges with Real-world Non-idealities:

Technique-Independent Diffusion Coefficients

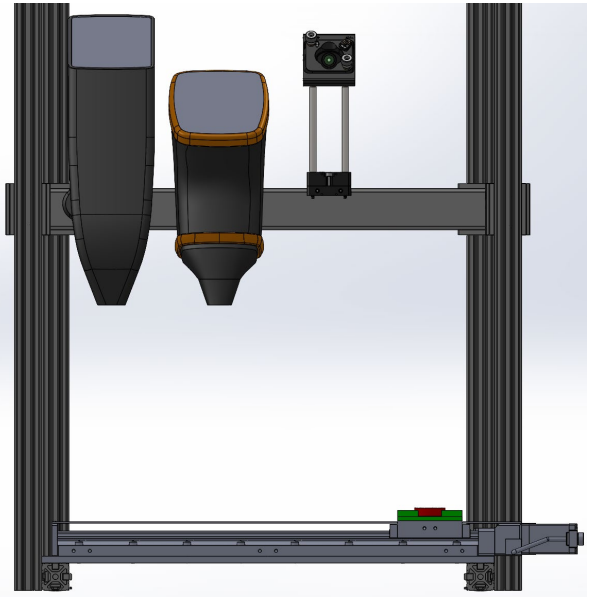
- Using current multipliers, we have been able to achieve accurate, technique-independent values for diffusion coefficients
  - D_{Cr2+} values from soluble-soluble LSV, soluble-insoluble LSV, and soluble-insoluble SWV show excellent agreements
- Ongoing tests are gathering technique-independent diffusion coefficients for additional species



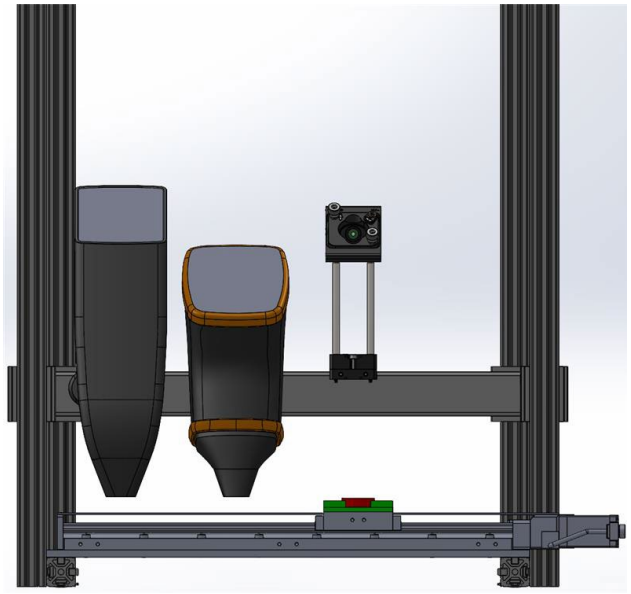
Cr^{2+} diffusion coefficients using Cr^{2+}/Cr^{0} soluble-insoluble LSV, Cr^{2+}/Cr^{3+} soluble-soluble LSV, and Cr^{2+}/Cr^{0} SWV

At-Line Integrated Analysis System (ALIAS)

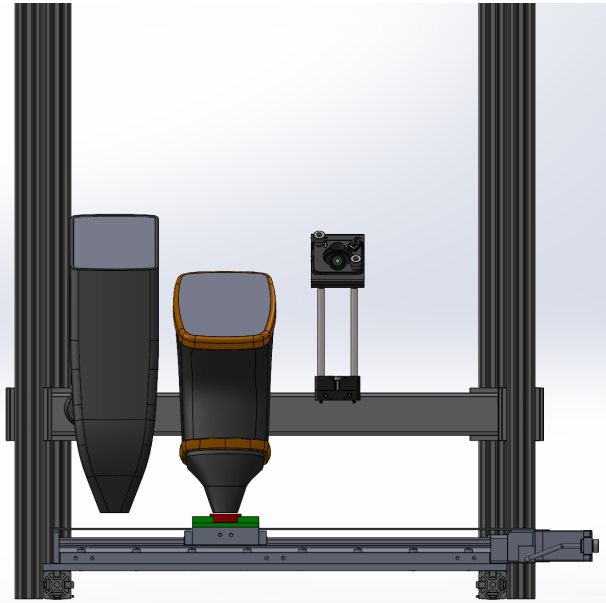
Contact: Amber Polke



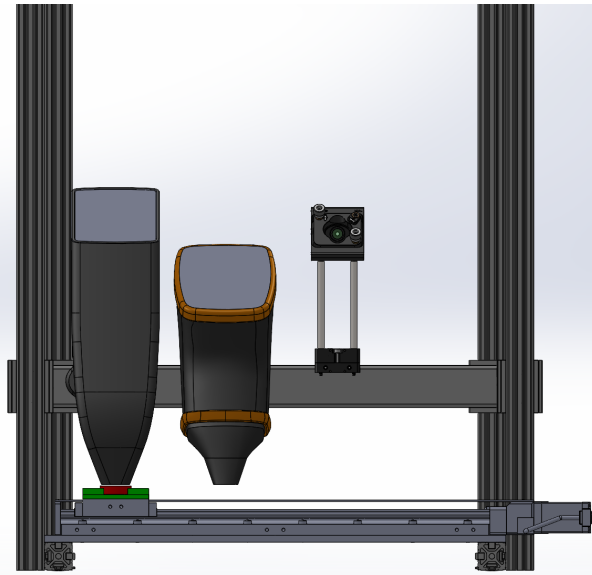
Sample loading



Raman Spectroscopy



X-ray Fluorescence (XRF)



Laser Induced Breakdown Spectroscopy (LIBS)

- Combines multiple optical analysis methods for rapid for near real-time sample analysis
- Automated stage for sample delivery to each instrument.
- Consistent stand off-distance from sample to detector
- Remote automated actuation of each analyzer

Benefits

- High precision analysis
- Improved timeliness
- Non-destructive techniques
- Integration of multiple data streams can average out errors and cross-validate

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SALT MONITORING DEVELOPMENTS FOR MSRs

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MSR licensing is contingent on the development of effective material control and accountancy (MC&A) practices.

Molten salt reactors (MSR) fuel salts safeguards challenges:

- Discrete item counting is likely not possible.
- Large fissile inventory and transient quantities of special nuclear materials.
- Traditional statistical tests are projected to require at or above current destructive analysis levels of precision.

Hogue et al., 2023, ORNL/SPR-2023/3181
Shoman et al., 2023, SAND2023-01845R
Shah et al., 2024, ORNL/SPR-2024/3555

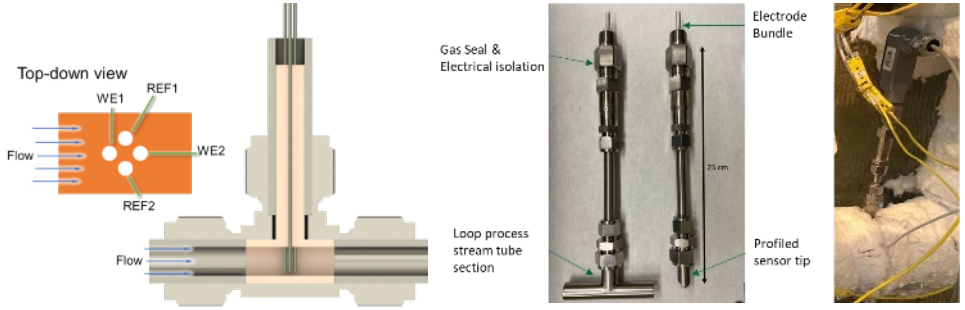
MC&A sensor design requirements:

- High accuracy (10 CFR 74's SEID).
- Deployable in corrosive and radioactive environments.
- Remote operation.
- Minimize the required control elements (surveillance and tamper-safing).

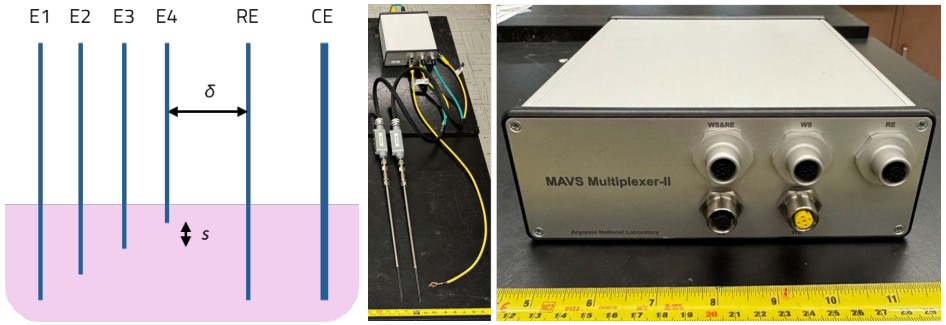
Argonne National Laboratory has developed new monitoring tools to support MC&A licensing for future MSRs

Enabling advanced materials control & accountancy for MSRs through robust, field-deployable electrochemical devices.

Electrochemical sensors for flowing and stationary environments

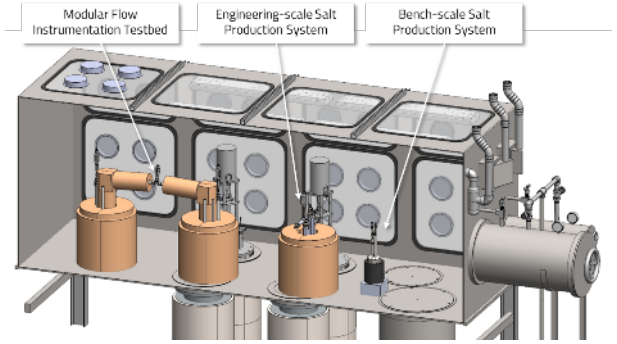


Flow enhanced electrochemical sensors (FEES)



Multielectrode array voltammetry sensors (MAVS)

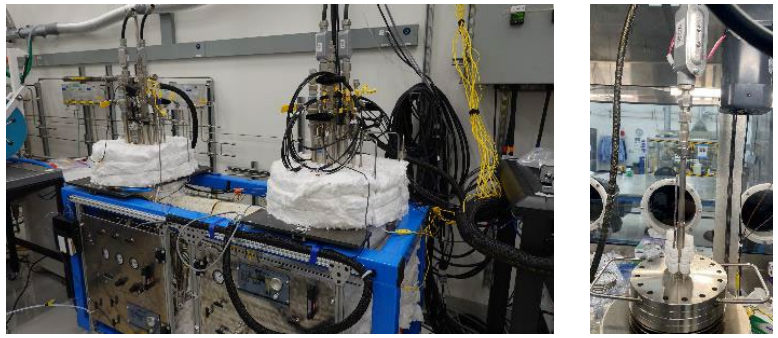
MSR representative environments



Modular Flow Instrumentation Testbed

- High-concentration actinide quantification
- Complex salt systems
- Salt synthesis
- Flowing high-concentration systems

Deployment at industrial partner facilities

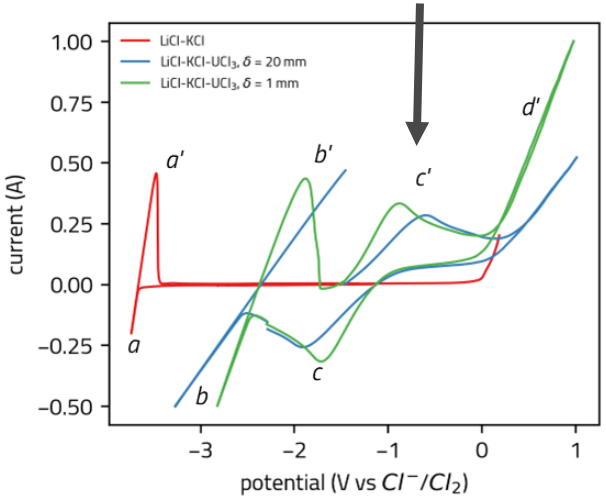


Systems at Kairos Power (left) and TerraPower (right)

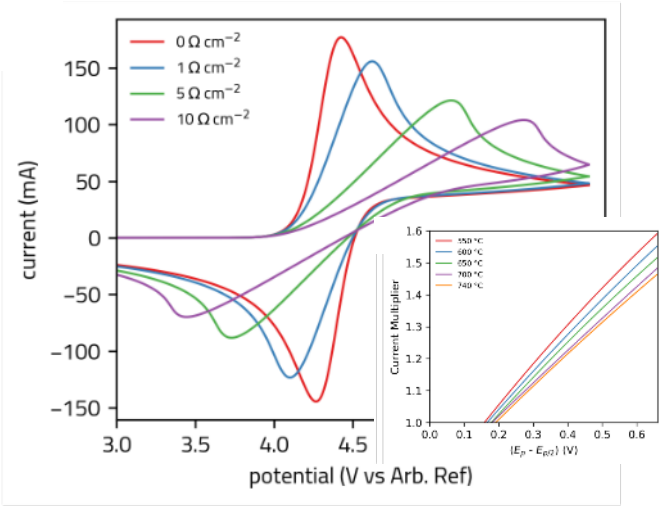
- Real-world conditions
- Remote operation
- Corrosion monitoring

Development of voltammetry techniques for complex high-concentration actinide salts

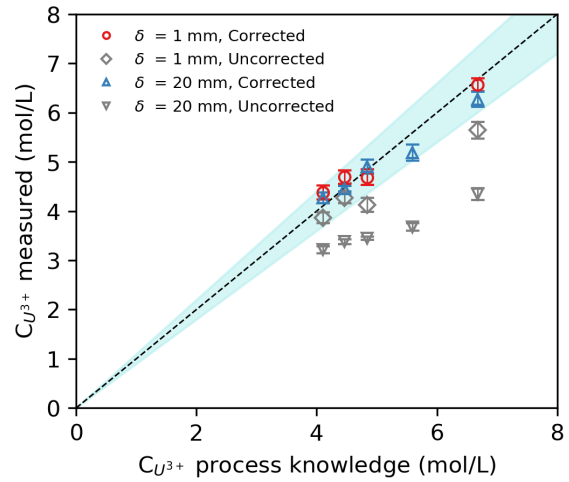
$c': U^{3+} \rightarrow U^{4+} + e^-$



Experimental CVs with various IR_Ω in of LiCl-KCl-UCl_3 (72.3 wt% UCl_3)



Simulated CVs with various IR_Ω & current multipliers



Preliminary measurements using correction procedure showed a mean absolute relative error of 4%.

- The current multiplier approach, as applied to very highly loaded salts, enables strong linear responses suitable for making quantitative measurements of species concentrations.
- Voltammetric measurements of UCl_3 were achieved in salts containing between 55 and 72 wt.% UCl_3.
  - ***5× higher concentration range than any other reported measurements***

Doniger, et al. JES **172** 2025

Engineering-scale UCl_3 salt production and monitoring

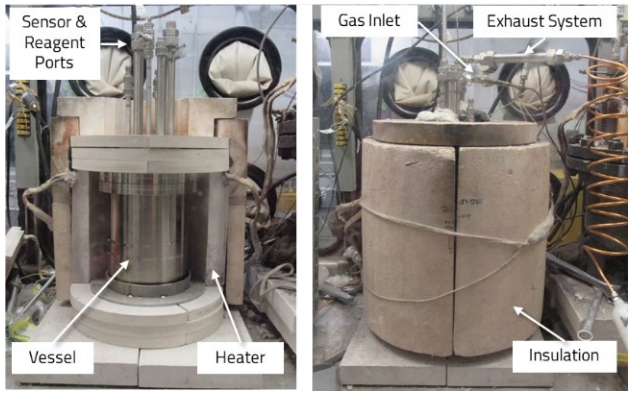
$LiCl$-KCl-UCl_3 salts containing 43, 58, and 72 wt% UCl_3 were produced from depleted uranium metal.

Dissolved $ZnCl_2$ was used as a chlorinating agent.

$$3/2\ ZnCl_2\ + U\ \rightarrow UCl_3 + 3/2\ Zn\ \ (\Delta G = -238.3\ kJ\ at\ 500\ ^\circ C)$$

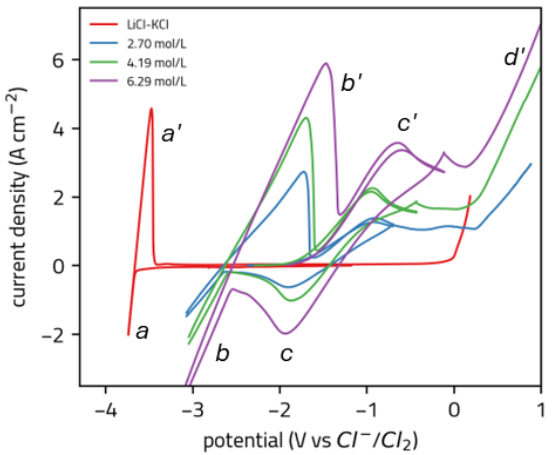
The byproduct Zn was volatilized at high temperature (740 °C) and removed.

- Online monitoring of uranium and zinc concentrations was conducted throughout the purification process.
- Voltammetry used to assess salt purity and establish endpoint of purification process

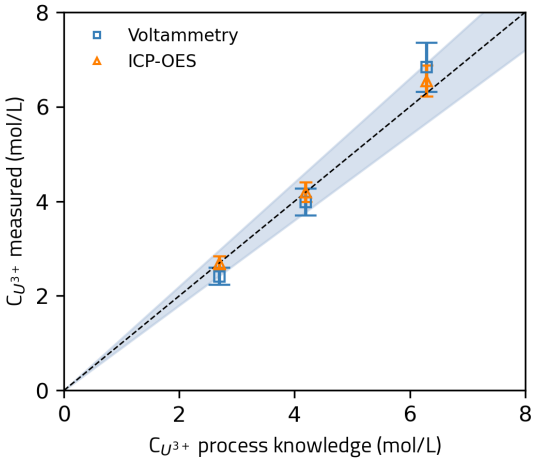


Engineering-scale salt production system (<5 liters)

$LiCl$-KCl-UCl_3 (43.4 wt% UCl_3) salt produced by $ZnCl_2$ chlorination.



CVs of synthesized $LiCl$-KCl-UCl_3 salts at 740 °C.



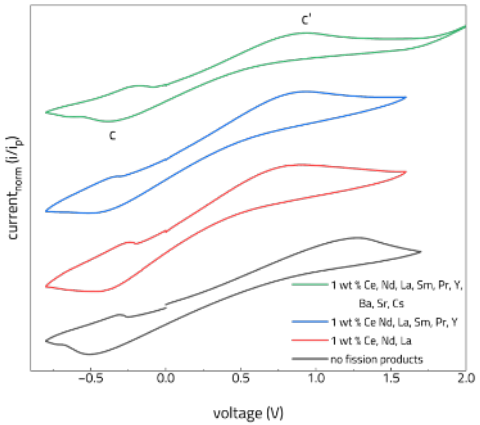
UCl_3 concentration in synthesized $LiCl$-KCl-UCl_3 salts at 740 °C.

Voltammetry measurements of uranium in highly loaded UCl_3-bearing salts in the presence of surrogate fission products

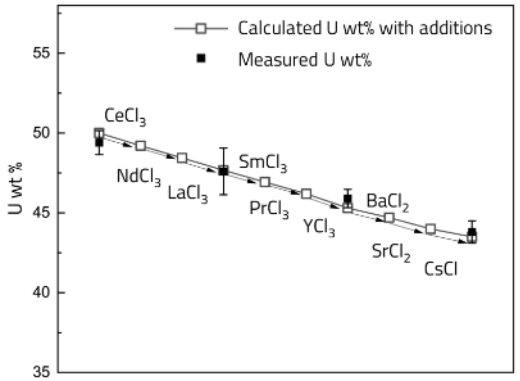
- The fission products Rb, Sr, Y, Zr, Ba, La, and Ce are likely stable and soluble in molten salts.
- To maintain proper accountancy, accurate and selective sensors for species of interest are required.

Soluble fission product half-life and standard reduction potential calculated using HSC.

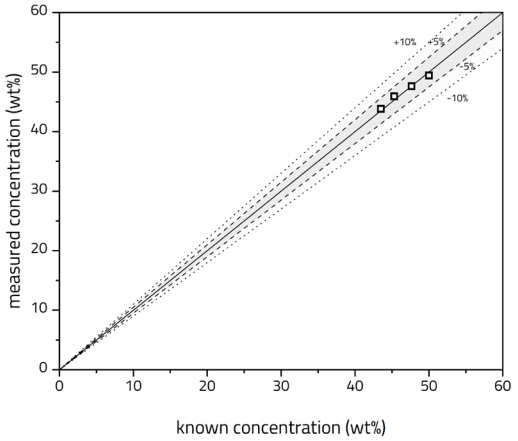
Isotope	Redox Reaction	Half-life	Standard Reduction potential at 550 °C (V)
^{143}Pr	$Pr^{4+} + e^- \rightarrow Pr^{3+}$		5.013
^{148}Nd	$Nd^{4+} + e^- \rightarrow Nd^{3+}$		6.230
^{147}Sm	$Sm^{4+} + e^- \rightarrow Sm^{3+}$		6.505
$^{141}Ce/^{144}Ce$	$Ce^{4+} + e^- \rightarrow Ce^{3+}$		3.133
^{235}U	$U^{4+} + e^- \rightarrow U^{3+}$		0.892
^{235}U	$U^{3+} + 3e^- \rightarrow U^0$		-1.155
^{143}Pr	$Pr^{3+} + 3e^- \rightarrow Pr^0$	13.57 days	-1.781
^{147}Sm	$Sm^{3+} + 3e^- \rightarrow Sm^0$	Stable	-1.781
^{91}Y	$Y^{3+} + 3e^- \rightarrow Y^0$	59 days	-1.756
^{148}Nd	$Nd^{3+} + 3e^- \rightarrow Nd^0$	11.1 days	-1.811
^{140}La	$La^{3+} + 3e^- \rightarrow La^0$	40.3 hours	-1.848
^{141}Ce	$Ce^{3+} + 3e^- \rightarrow Ce^0$	32.3 days	-1.844
^{144}Ce		284 days	
^{140}Ba	$Ba^{2+} + 2e^- \rightarrow Ba^0$	12.8 days	-2.807
^{89}Sr	$Sr^{2+} + 2e^- \rightarrow Sr^0$	52 days	-2.720
^{90}Sr		28.1 years	
^{91}Sr		9.67 hours	
^{133}Cs	$Cs^+ + e^- \rightarrow Cs^0$	Stable	-3.808
^{134}Cs		705 days	
^{137}Cs		29.9 years	



CVs of LiCl-KCl-UCl_3 with various additions of Ce, Nd, La, Sm, Pr, Y, Ba, Sr, & Cs. All species added as 1 wt %.



Measured and calculated concentrations of U in LiCl-KCl-UCl_3 with sequential additions of surrogate fission products.



Measured and known uranium concentrations during additions of fission product surrogates.

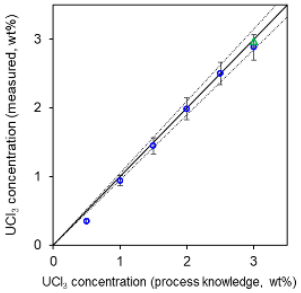
1 wt% additions of each species resulted in a corresponding decrease in UCl_3 concentration.

Voltammetric UCl_3 concentration measurements demonstrated a mean absolute relative error of only 0.8% even in the presence of up to nine fission product surrogates

Electrochemical sensor technologies for MSR-relevant salts have displayed steadily improving capabilities over the past several years

Low U Concentrations (< 5 wt%) – FY22-FY24

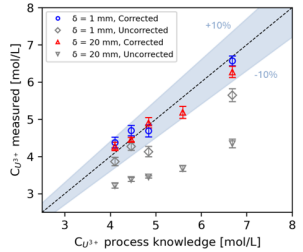
Measurement	Uncertainty Component (%rel.)		Total uncertainty (%rel.)
	u(r)	u(s)	
Concentration	<1%	~2.5%*	~2.6%*



FEES sensor UCl_3 concentration measurements show mean relative standard error of 2.5%

Initial Measurements of Very High U Concentrations (> 55 wt%) – FY24

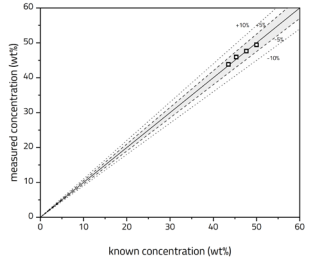
Measurement	Uncertainty Component (%rel.)		Total uncertainty (%rel.)
	u(r)	u(s)	
Concentration	<1%	~4.0% *	~4.0%*



Predicted U^{3+} concentration vs. process knowledge in LiCl-KCl-UCl_3 at 550 °C.

Optimized Measurements of Very High U Concentrations (> 55 wt% with and without surrogate fission products) – FY25

Measurement	Uncertainty Component (%rel.)		Total uncertainty (%rel.)
	Random Error	Systematic Error	
Concentration	1.3%	0.8%	~1.3%*



Predicted U^{3+} concentration vs. process knowledge in LiCl-KCl-UCl_3 during additions of fission product surrogates at 550 °C.

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SALT MONITORING DEVELOPMENTS UNDER ARPA-E

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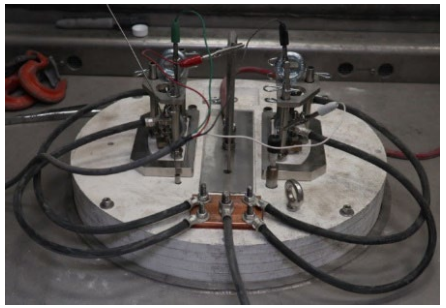
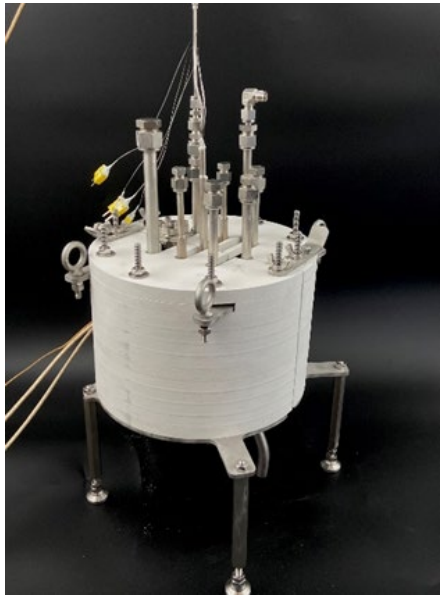
Multimodal Safeguards Monitoring Unit (MSMU)

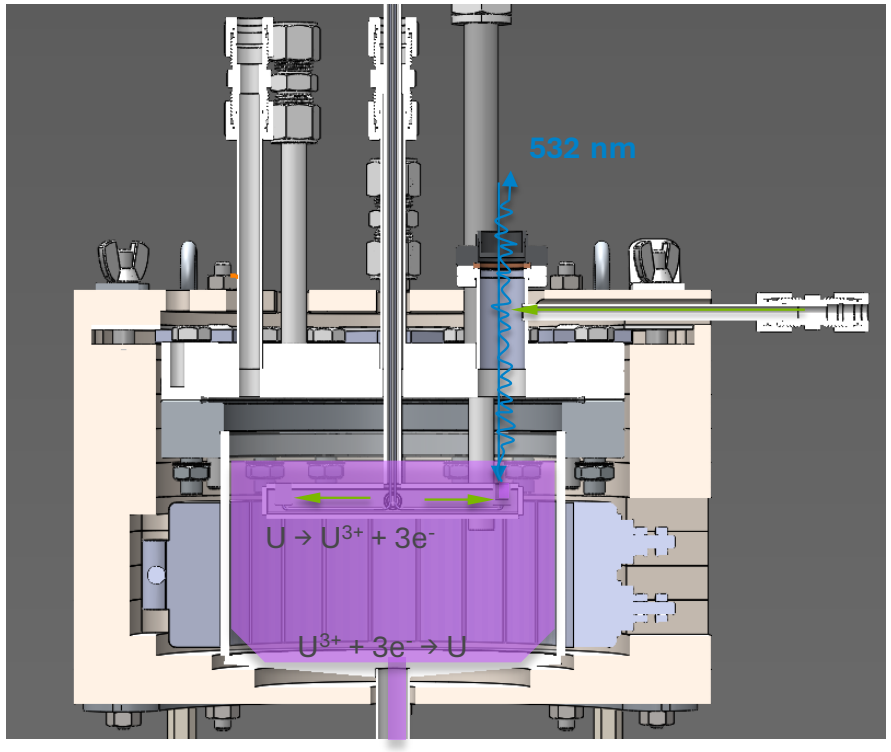
Online sampling system that combines multiple analytical techniques to make high-fidelity material accountancy measurements

- **Online Monitoring:**
  - Electroanalytical/EIS suite
  - Raman spectroscopy with consistent sample standoff distance
  - DTA probe with active cooling
- **At-line and Offline Monitoring:**
  - Discrete sampling port
- Unit successfully operated on an engineering scale electrorefiner for four months

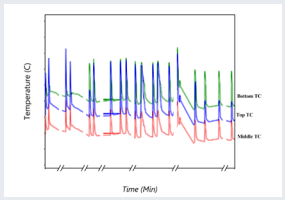
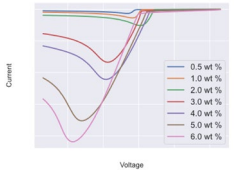
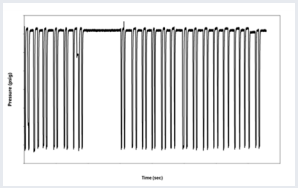
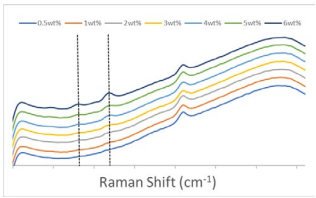
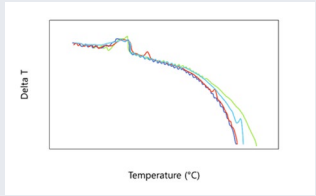




MSMU OPERATIONS



Multimodal sensors facilitate on-line measurements with multiple analytical techniques in a compact and robust system allowing for real-time analysis.

Step	Data	Measurements
Salt is charged		pressure, temperature, **level/volume**, **density**
Electroanalytical/ EIS performed		salt level, **salt composition**, **salt redox state**
Discharge salt		pressure, temperature, **level/volume**, **density**
Raman spectroscopy performed		**salt composition**, **salt chemical state**
Differential Thermal Analysis (DTA) performed		temperature, **salt composition**, enthalpy

Conclusions

- Salt monitoring tools are crucial for the operations and licensing of MSRs
  - Corrosion monitoring and control
  - Salt composition information to inform control of reactor neutronics
  - MC&A

- Argonne has developed and developed a variety of salt monitoring tools
  - Tools ushered from low TRL up to TRL 6-7
  - Sensors deployed at multiple universities, national labs, and industry partners

- Multimodal monitoring, sensor fusion, and automation can help to reduce O&M costs that could otherwise make MSRs uneconomical

Acknowledgements

- Thanks to:
  - Robert Hoover
  - Ammon Williams
  - Stephen Warmann
  - Phil Lafrienere

- Government License Notice
  - *The submitted manuscript has been created by UChicago Argonne, LLC, Operator of Argonne National Laboratory ("Argonne"). Argonne, a U.S. Department of Energy Office of Science laboratory, is operated under Contract No. DE-AC02-06CH11357. The U.S. Government retains for itself, and others acting on its behalf, a paid-up nonexclusive, irrevocable worldwide license in said article to reproduce, prepare derivative works, distribute copies to the public, and perform publicly and display publicly, by or on behalf of the Government.*

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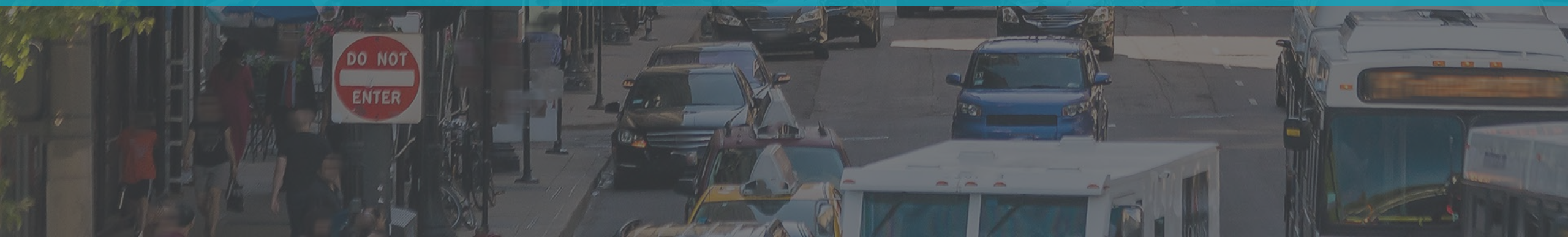


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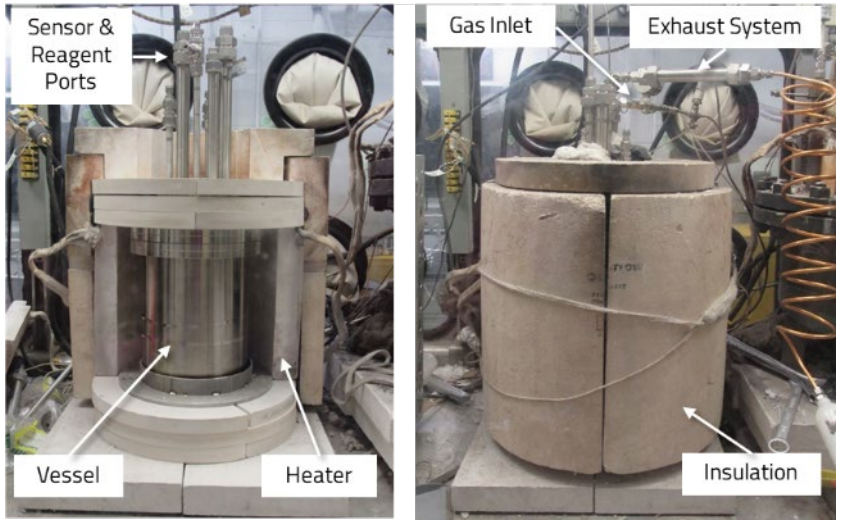
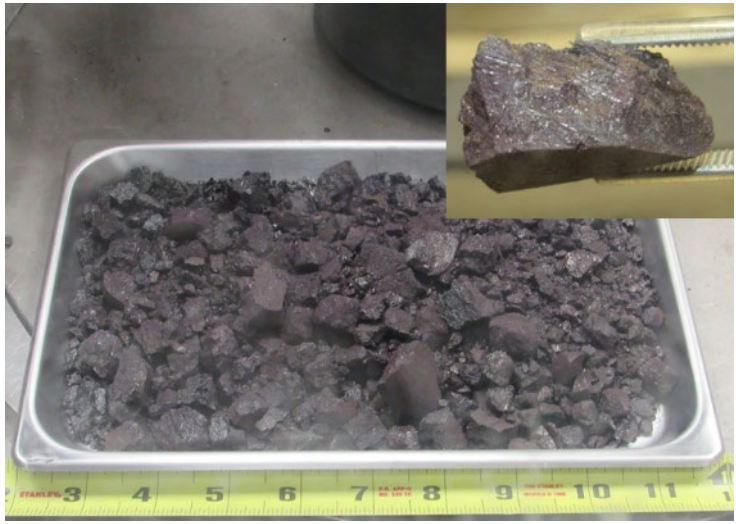
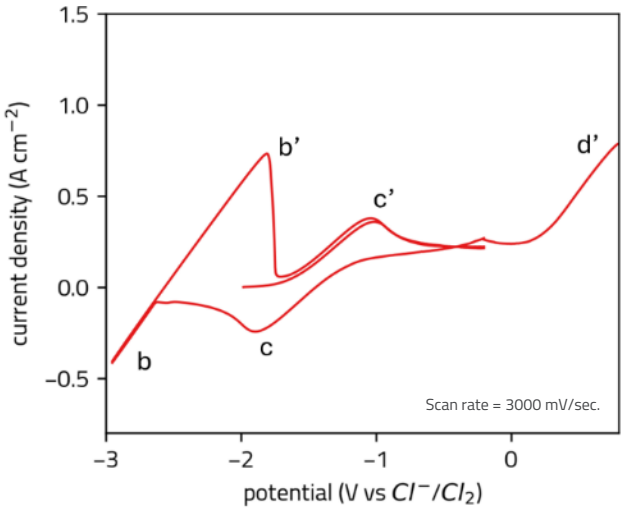
ENGINEERING-SCALE UCl_3 SALT PRODUCTION AND MONITORING



Engineering-scale salt production system (<5 liters)



LiCl-KCl-UCl_3 (43.4 wt% UCl_3) salt produced by $ZnCl_2$ chlorination.



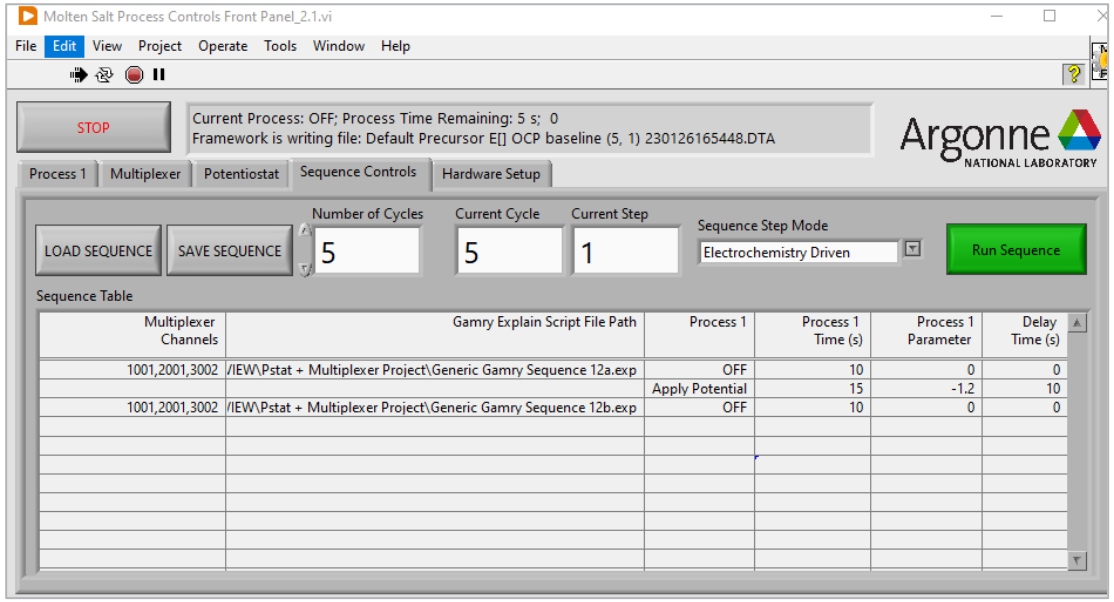
CVs of LiCl-KCl-UCl_3 (43.4 wt% UCl_3) at 610 °C.

- In FY25, a ~1 kg batch of LiCl-KCl-UCl_3 (43.4 wt% UCl_3) was produced to support electrochemical sensor research.
- Voltammetry was used in the engineering-scale vessel to monitor salt composition during chlorination and confirm the salt purity.

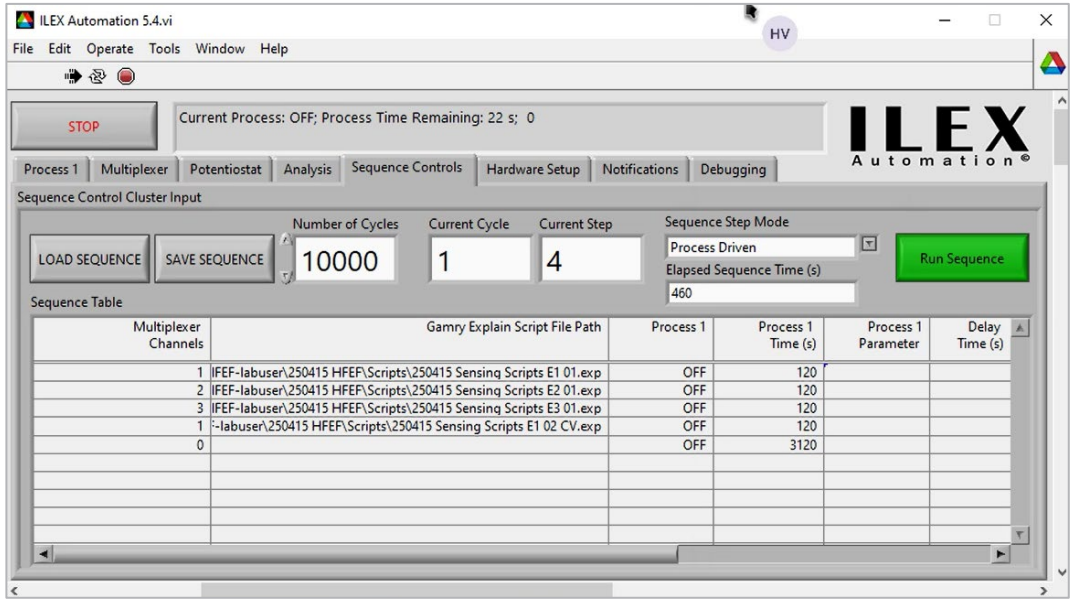
Updated Software for FY25 Operations

A variety of software improvements have been implemented to improve long-term operations of the sensor when installed in fuel processing equipment

- Sensor feedback to enable automated electrorefiner operations
- Updates to automated analysis scripts and output



ILEX Version 2.1



ILEX Version 5.4

Note: ILEX was originally developed for the ARSS program; additions and updates have been added under MPACT to enable pyroprocessing-specific operations

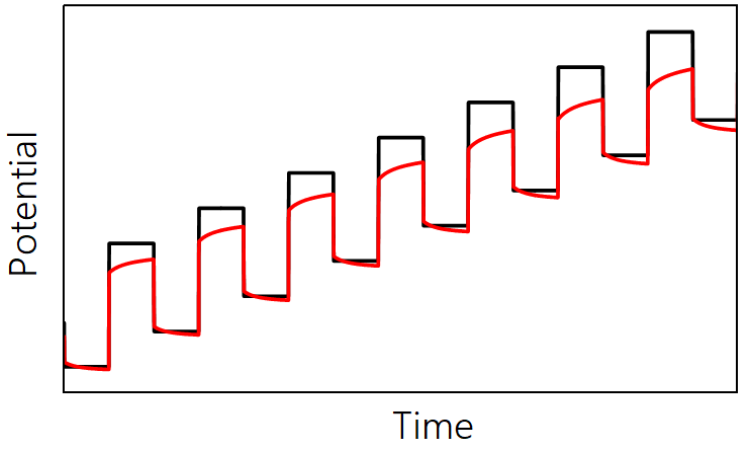
Challenges with Real-world Non-idealities:

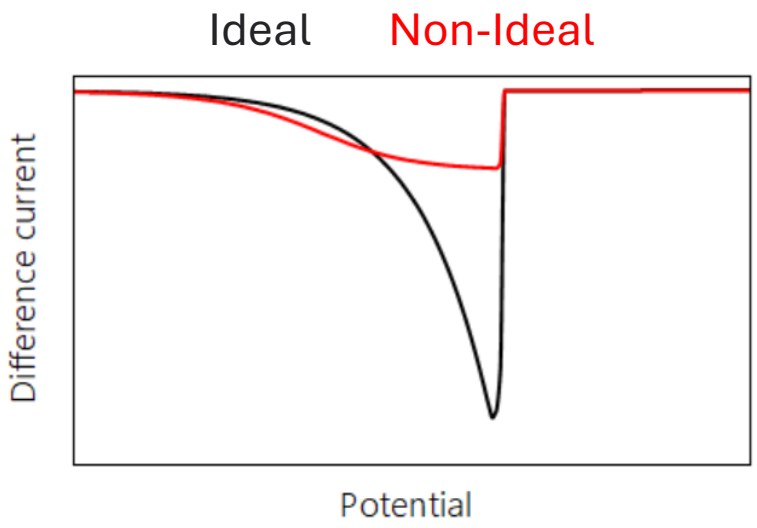
Solution Resistance

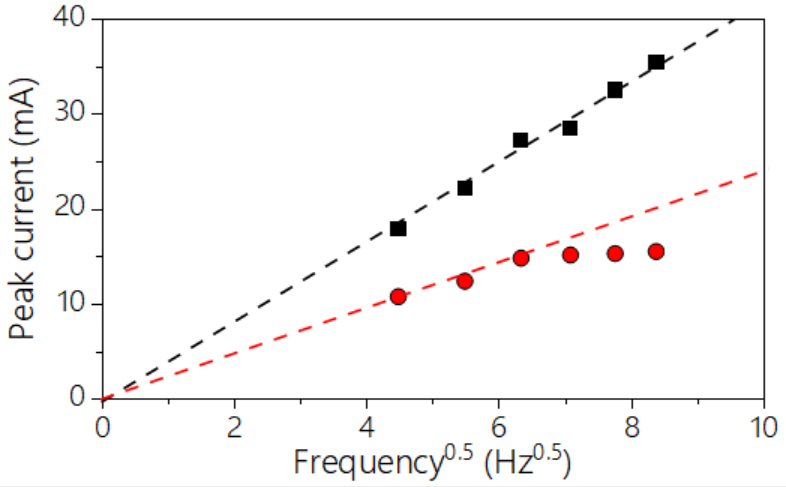
Simulations offer avenues towards quantitatively correcting for non-idealities introduced in square wave voltammetry (SWV)

- Uncompensated Ohmic resistance can significantly attenuate SWV responses and reduce sensor reliability for MC&A applications

- Correction factors obtained from simulations can account for attenuation such that experimental data agrees with theory



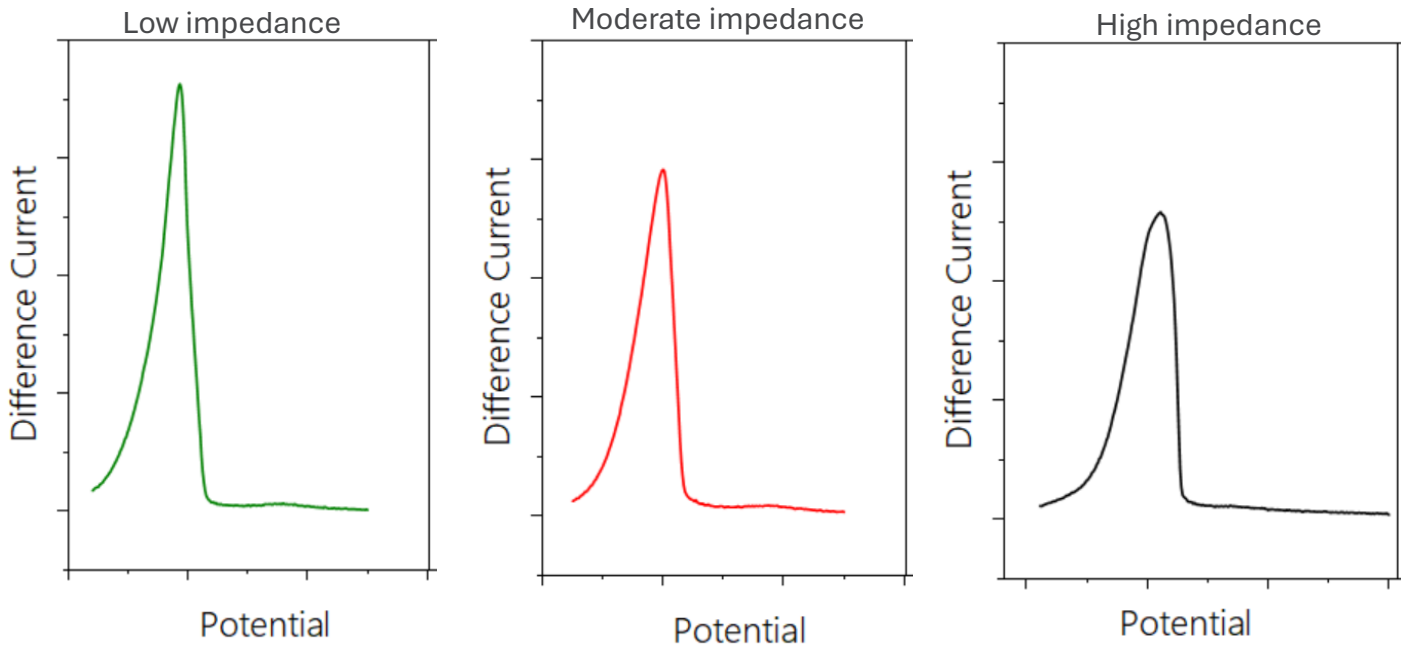




Challenges with Real-world Non-idealities:

Wiring Impedance

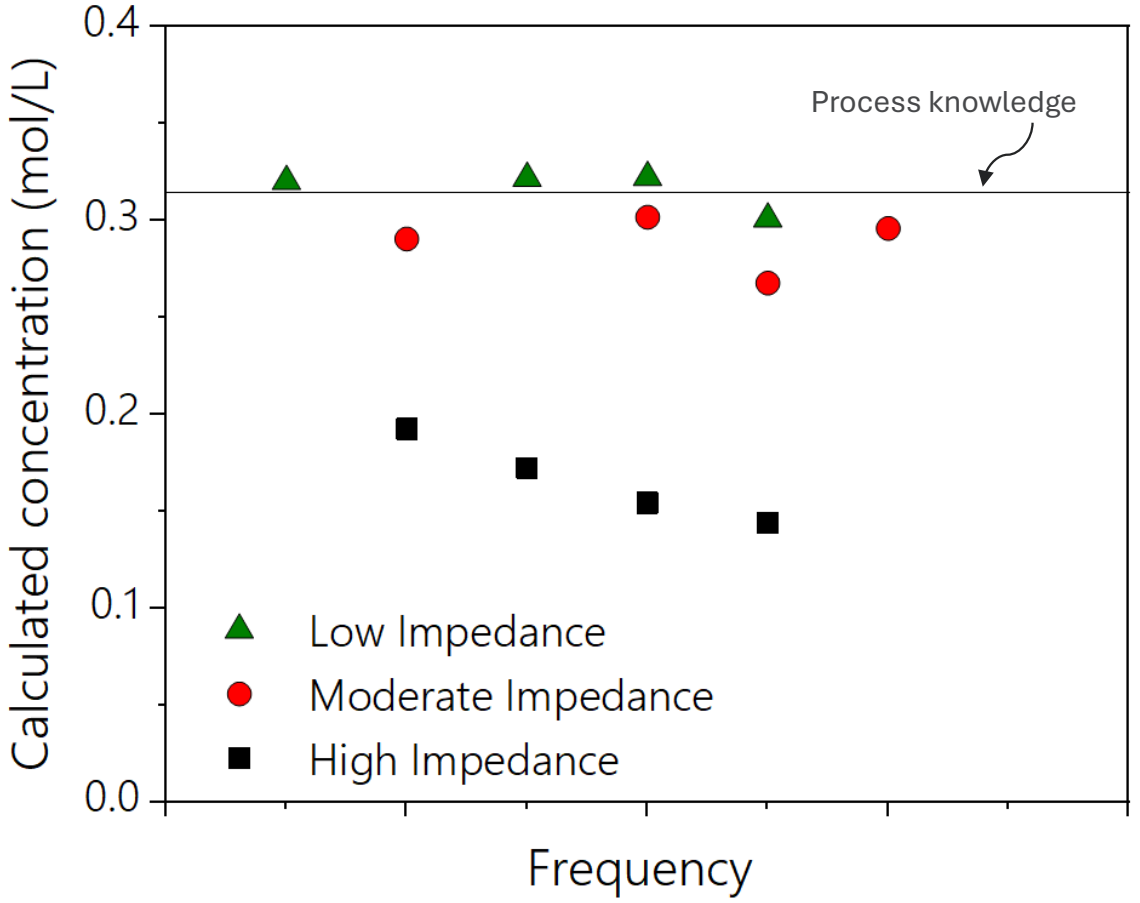
- Unaccounted for wiring impedance is appreciable in practical industrial settings, particularly when utilizing long cable lengths through high noise environments
- Left unaddressed, MC&A capabilities become severely compromised as sensor response is attenuated



Challenges with Real-world Non-idealities:

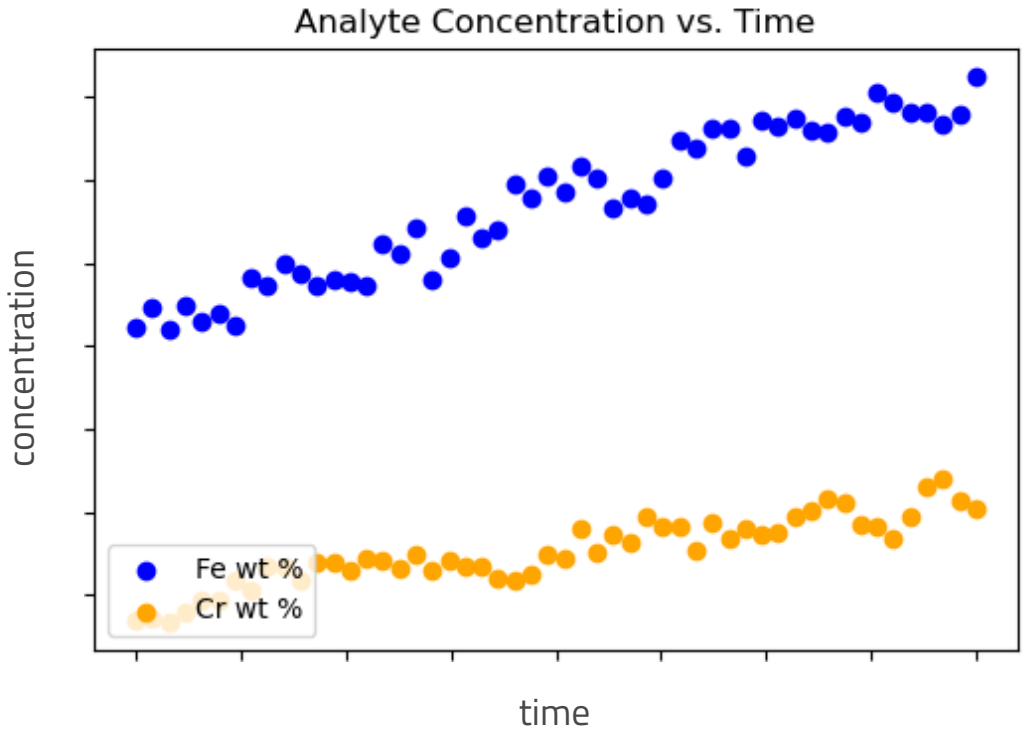
Wiring Impedance

- At sufficiently high frequency scans, sensor-calculated concentrations are no longer constant and deviate from known process knowledge concentrations
- Correction factors previously developed to account for uncompensated ohmic resistance are insufficient to correct for industrial-scale impedance effects
- Ongoing tests are aimed at holistically characterizing and correcting for these non-idealities



Multianalyte Monitoring During ER Operations

- Updated approaches and fundamental data have enabled improved measurements of impurity species during ER operations
  - Cr^{2+} and Fe^{2+} corrosion products can be readily quantified
- Operations over the past several months have enabled further understanding of long-term ER performance



Corrosion product concentrations over time during spent fuel processing operations

Anticipated Issues and Identified Carry-Over

- Currently on track to complete FY25 milestones.
  - **M3FT-25AN040103072** *Sensor Deployments, Operations, and Improvements*
    - Due 9/30/2025

- Anticipated carryover level for FT-25AN04010307 is ~15%

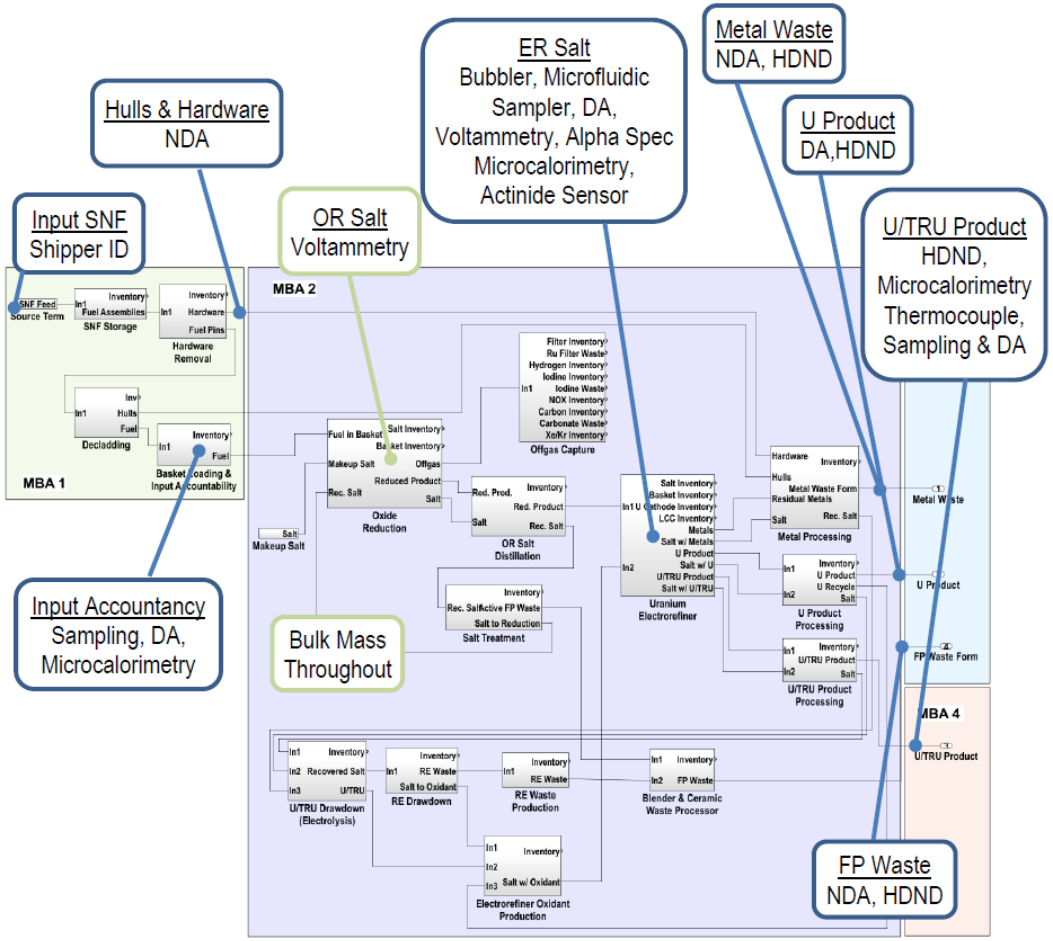
Safeguards Technologies for Pyroprocessing Flowsheets

Pyrochemical processing can enable recycling of spent nuclear fuel derived from many fuel cycles

- Light water reactor fuel
- Sodium fast reactor fuel
- Other advanced reactors

Safeguarding of special nuclear materials is essential for licensing and commercialization of pyroprocessing

New technologies and approaches must be developed to ensure compliance with domestic MC&A requirements

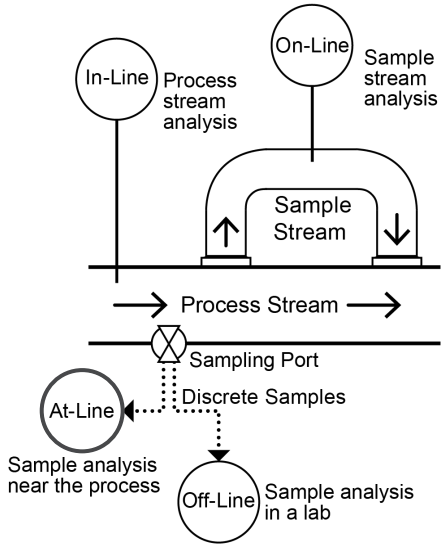


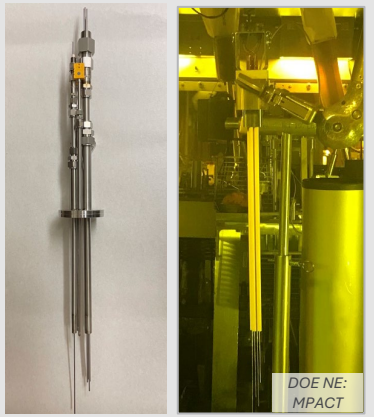
Cipiti, B.; Reim, M., Browne, M.; The MPACT 2020 Milestone: Lessons Learned. SAND2020-13367PE (downloaded from: https://arpa-e.energy.gov/sites/default/files/BenCipiti.pdf)

Argonne Technologies for Molten Salt Monitoring, Control, and Safeguards

Key monitoring capabilities needed for molten salt systems

- Salt composition
- Isotopics
- Salt level
- Redox state
- Particulates
- Density
- Etc.





Electrochemical Monitoring of Salt Composition

Windowless Optical Monitoring of Composition

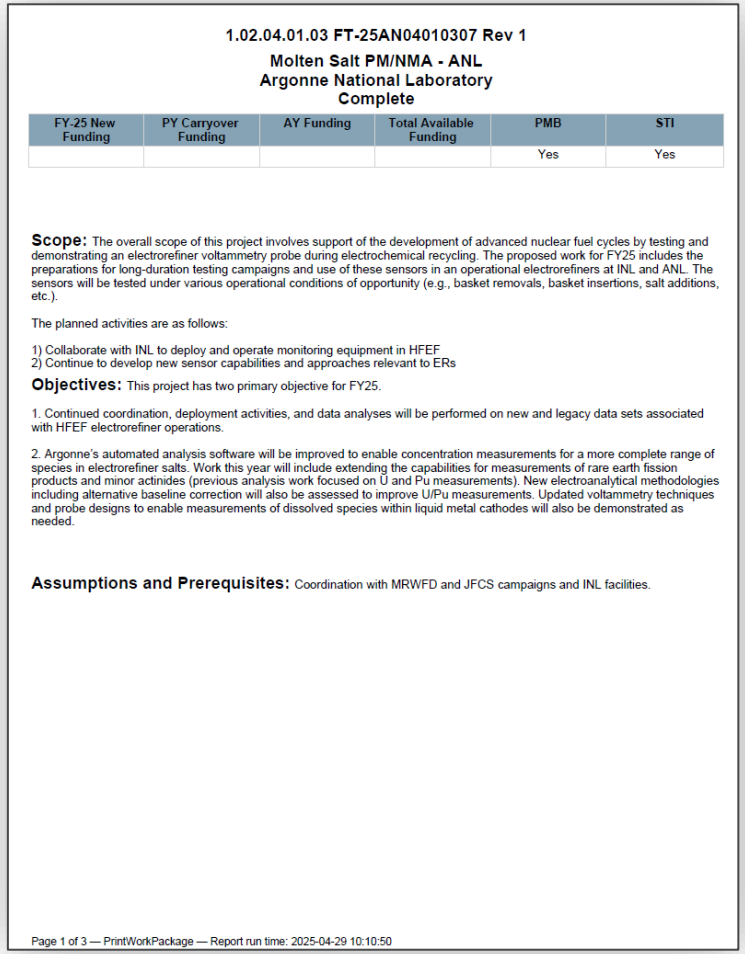
Particulate Monitoring

Automated Salt Sampling

Argonne has demonstrated a wide variety of monitoring technologies to enable safe operations and material accountancy for nuclear-relevant systems.

FY25 Project Objectives

- Deployment/Operations
  - HFEF spent fuel processing
  - Argonne co-deposition electrorefiner
- Sensor improvements
  - Updated instrumentation/automation
  - Expanded multianalyte systems
  - Expanded non-ideal effects
- Development/testing of new sensor concept



1.02.04.01.03 FT-25AN04010307 Rev 1

Molten Salt PM/NMA - ANL
Argonne National Laboratory
Complete

FY-25 New Funding	PY Carryover Funding	AY Funding	Total Available Funding	PMB	STI
				Yes	Yes

Scope: The overall scope of this project involves support of the development of advanced nuclear fuel cycles by testing and demonstrating an electrorefiner voltammetry probe during electrochemical recycling. The proposed work for FY25 includes the preparations for long-duration testing campaigns and use of these sensors in an operational electrorefiners at INL and ANL. The sensors will be tested under various operational conditions of opportunity (e.g., basket removals, basket insertions, salt additions, etc.).

The planned activities are as follows:

1) Collaborate with INL to deploy and operate monitoring equipment in HFEF
2) Continue to develop new sensor capabilities and approaches relevant to ERs

Objectives: This project has two primary objective for FY25.

1. Continued coordination, deployment activities, and data analyses will be performed on new and legacy data sets associated with HFEF electrorefiner operations.

2. Argonne's automated analysis software will be improved to enable concentration measurements for a more complete range of species in electrorefiner salts. Work this year will include extending the capabilities for measurements of rare earth fission products and minor actinides (previous analysis work focused on U and Pu measurements). New electroanalytical methodologies including alternative baseline correction will also be assessed to improve U/Pu measurements. Updated voltammetry techniques and probe designs to enable measurements of dissolved species within liquid metal cathodes will also be demonstrated as needed.

Assumptions and Prerequisites: Coordination with MRWFD and JFCS campaigns and INL facilities.

Page 1 of 3 — PrintWorkPackage — Report run time: 2025-04-29 10:10:50

FY25 Project Objectives

- Deployment/Operations
  - HFEF spent fuel processing
  - Argonne co-deposition electrorefiner

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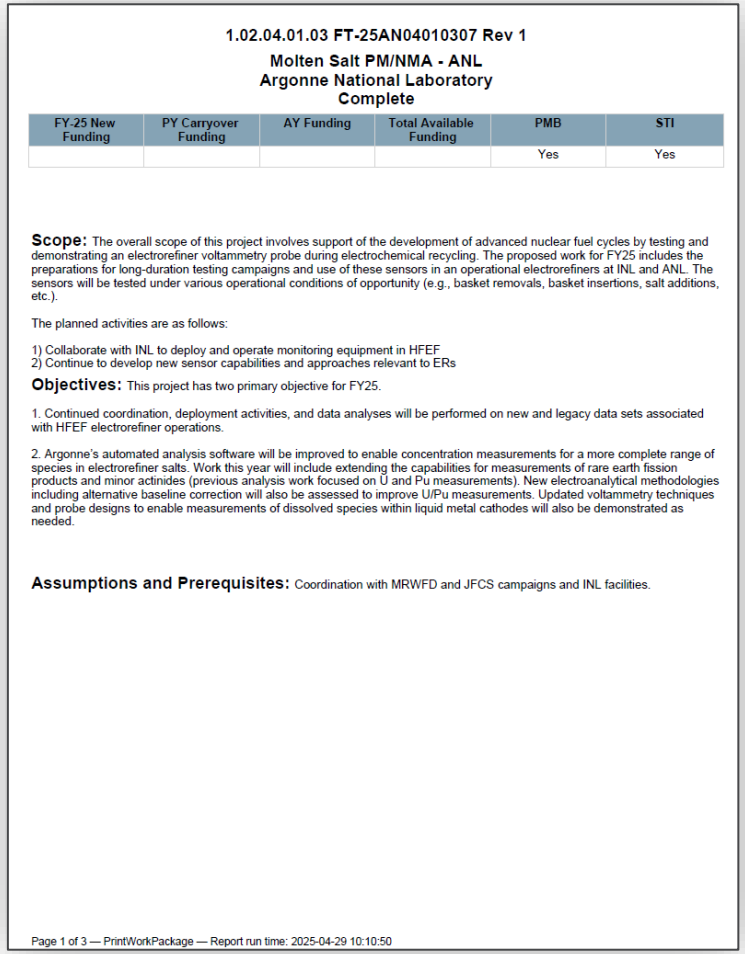

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Page 1 of 3 — PrintWorkPackage — Report run time: 2025-04-29 10:10:50

Real-World Deployment Challenges for Voltammetry Sensors

- **Problem:** Slow data turnaround; excessive personnel burden
  - **Solution:** Improvements to automated operations and data analysis

- **Problem:** Presence of corrosion products and other impurities
  - **Solution:** Improvements for analysis of multianalyte systems

- **Problem:** Distortions from non-ideal effects
  - **Solution:** Updated simulations to account and adjust for distortions