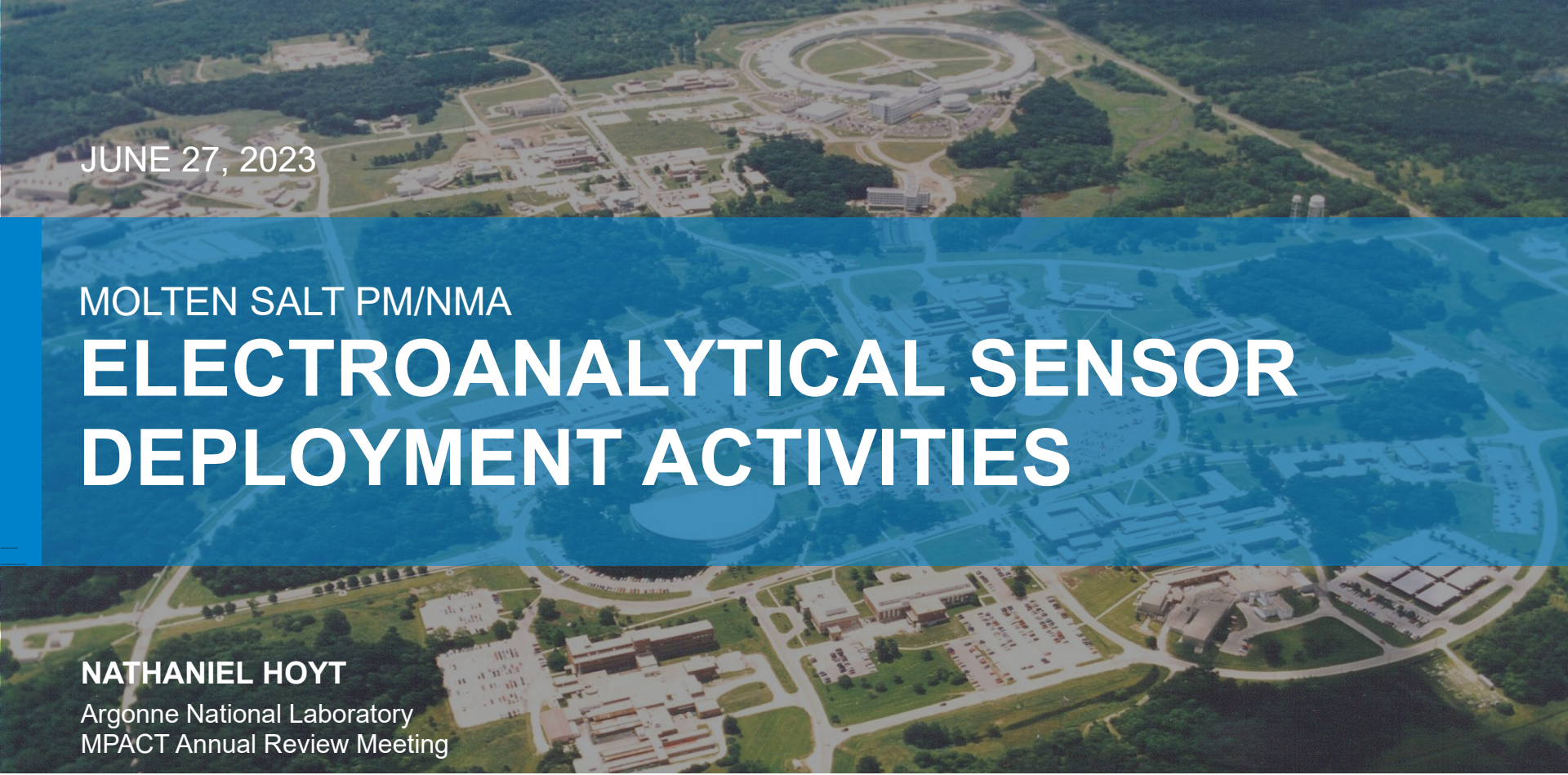


JUNE 27, 2023

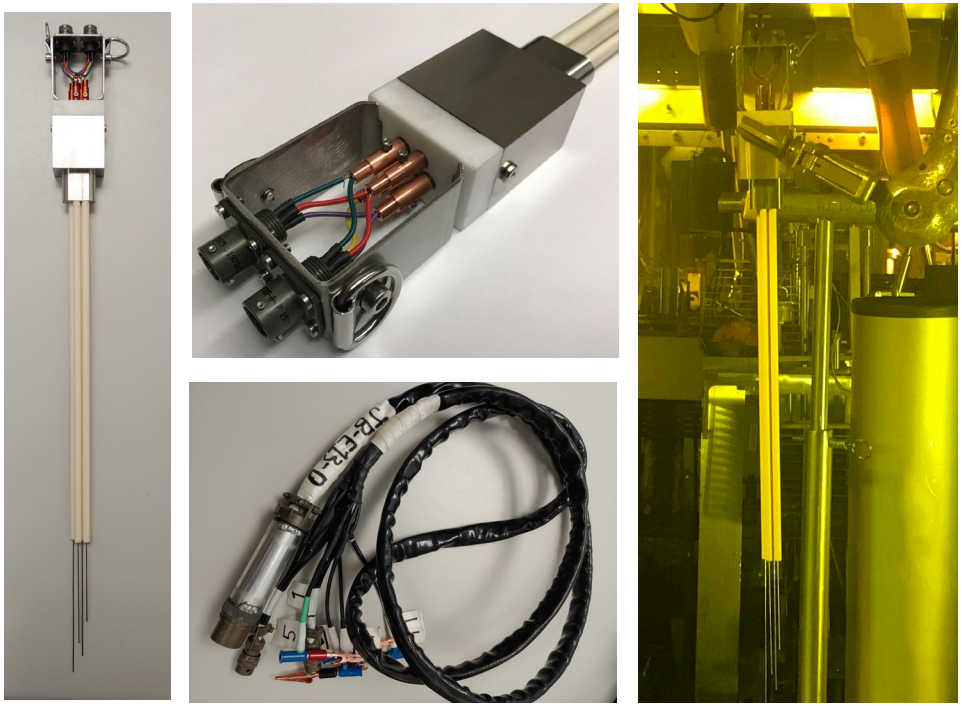
MOLTEN SALT PM/NMA

ELECTROANALYTICAL SENSOR DEPLOYMENT ACTIVITIES

NATHANIEL HOYT
Argonne National Laboratory
MPACT Annual Review Meeting

PROJECT OBJECTIVES

- Deploy safeguards-enabling multielectrode array electrochemical sensors to a hot cell facility
- 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

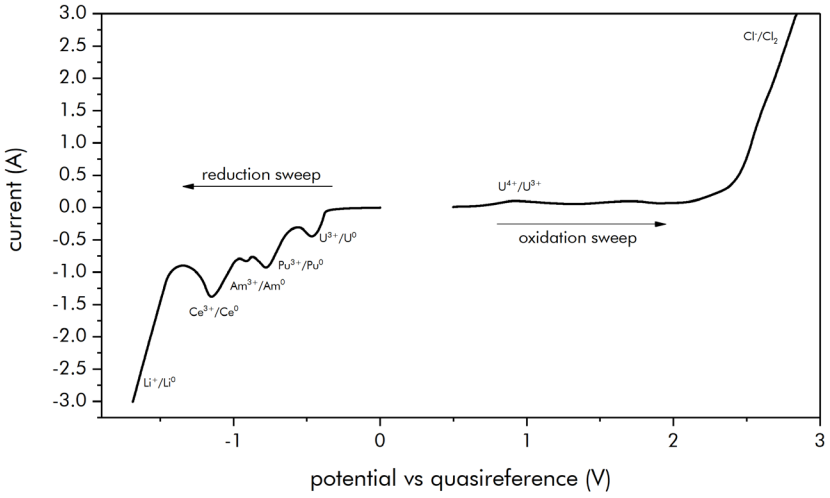


Argonne-developed deployable voltammetry sensor

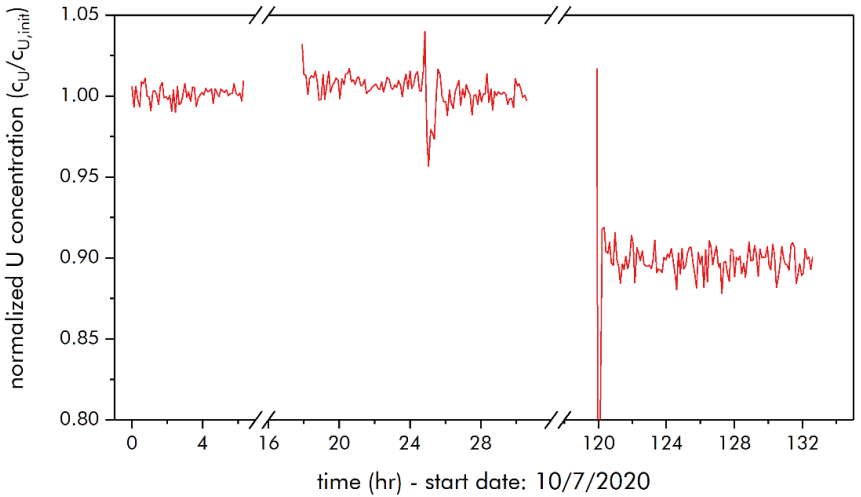
VOLTAMMETRY SENSOR OPERATIONS

MONITORING OF FUEL REPROCESSING

Voltammetry sensors have been deployed to an electrorefiner at INL's Hot Fuel Examination Facility since FY21. These sensors provide measurements of the salt depth, redox potential, and concentrations of species including actinides and lanthanides.



Typical raw electroanalytical signals in electrorefiner salt



U concentration versus time during salt addition test

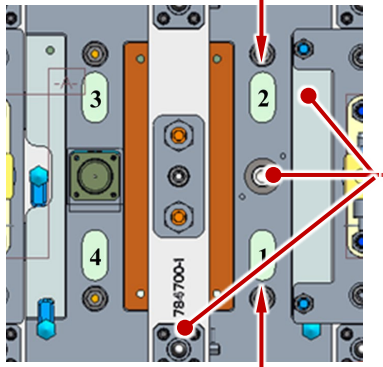
MPACT FY22 ER SAFEGUARDS CAMPAIGN

MPACT funded an electrorefining campaign at HFEF in late FY22 to test and assess the combined performance of a variety of safeguards tools applicable to electrochemical reprocessing

Voltammetry Sensor (N. Hoyt, ANL)
- Provides measurements of salt composition, redox state, and salt level

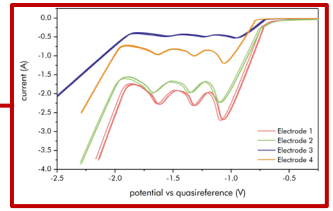
Triple Bubbler (A. Williams, INL)
- Provides measurements of tank level and salt density to determine the total mass of the bulk material in the vessel

Process Monitoring Modelling for ER data (P. Lafrienere, LANL)
- Leverages a variety of ER data streams (voltage, current, temperature, etc.)
- Allows real time assessment of process variables to determine if a given process is operating normally

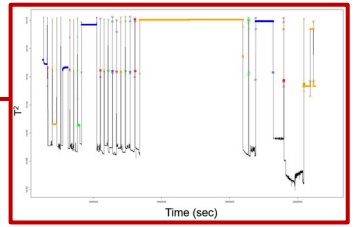


HFEF ER
(INL)

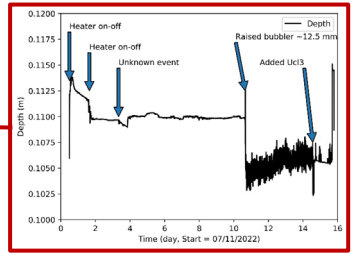
Voltammetry Sensor (ANL)



Process Monitoring Data (LANL)



Tripler Bubbler (INL)



VOLTAMMETRY OPERATIONS

The voltammetry probe was operated throughout a variety of fuel processing activities as part of the MPACT safeguards campaign. The campaign occurred over a period of three months and the sensor's total active operational time was 28 days.

Date	Sequence Identifier	Description of Activities	Total Cycles	Active Electrodes	No. of Measurements	Sequence Length (hr)
5/19/2022	SEQ-01	Initial resumption operations	23	E1,E2	46	4.0
7/7/2022	SEQ-02	Baseline measurements	374	E1,E2	748	94.0
7/11/2022	-	Baseline measurements after basket insertion	N/A	E1,E2	0	0.0
7/12/2022	SEQ-03	Baseline measurements after basket insertion	10	E1,E2	20	8.0
7/13/2022	SEQ-04	Baseline measurements after basket insertion	25	E1,E2	50	4.0
7/14/2022	SEQ-05	Baseline measurements after basket insertion	47	E1,E2	94	7.0
7/18/2022	SEQ-07	Baseline measurements	18	E1,E2	36	4.0
7/21/2022	SEQ-08	More baseline measurements	83	E1,E2	166	18.0
7/25/2022	SEQ-09	Measurements after UCl3 addition	111	E1,E2	222	25.0
7/26/2022	SEQ-10	Measurements during salt addition	28+107	E1,E2;E1	163	12.0
7/27/2022	SEQ-11	Measurements after salt addition	43	E1,E2,E3,E4	172	16.0
7/28/2022	SEQ-12	Measurements after salt samples	277	E1,E2,E3,E4	1108	91.0

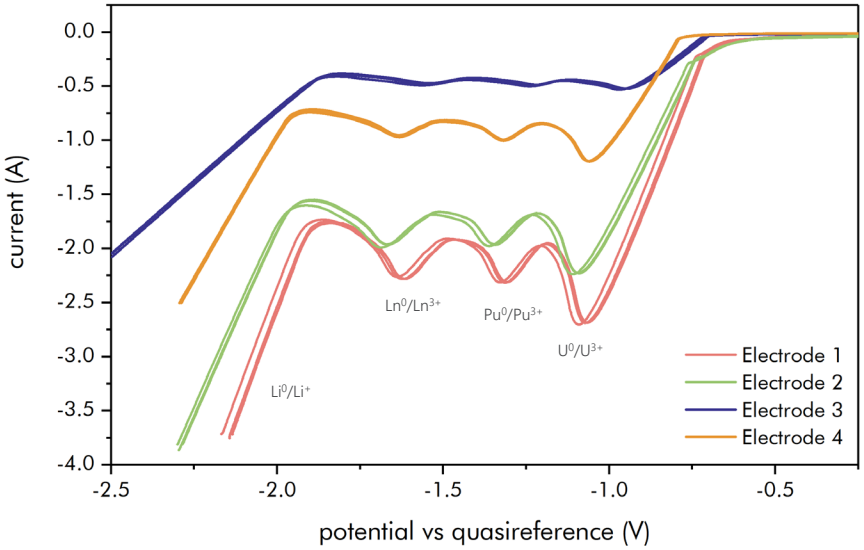
Date	Sequence Identifier	Description of Activities	Total Cycles	Active Electrodes	No. of Measurements	Sequence Length (hr)
8/1/2022	SEQ-13	Continued measurements after salt samples	548	E1,E2,E3,E4	2192	167.0
8/8/2022	SEQ-14	Continued measurements after salt samples	5	E1,E2,E3,E4	20	4.0
8/15/2022	SEQ-15	Continued measurements after salt samples	137	E1,E2,E3,E4	548	42.0
8/17/2022	SEQ-16	Measurements during vessel cleanup	250	E1,E2,E3,E4	1000	78.0
9/21/2022	SEQ-17	Preparation for more ER ops	52	E1,E2,E3,E4	208	16.0
9/22/2022	SEQ-18	Monitoring of ER Operations	324	E1,E2,E3,E4	1296	98.0
9/26/2022	-	More ER Ops monitoring	N/A	E2,E3	-	-
10/5/2022	SEQ-19	More ER Ops monitoring	250	E2,E3	500	-

Immersion time (months)	Total No. of Measurements	Total Operations Time (hr)
5	8589	688.0

RESUMPTION OF SALT MONITORING

Upon resumption of operations, the sensor was still found to operate as expected

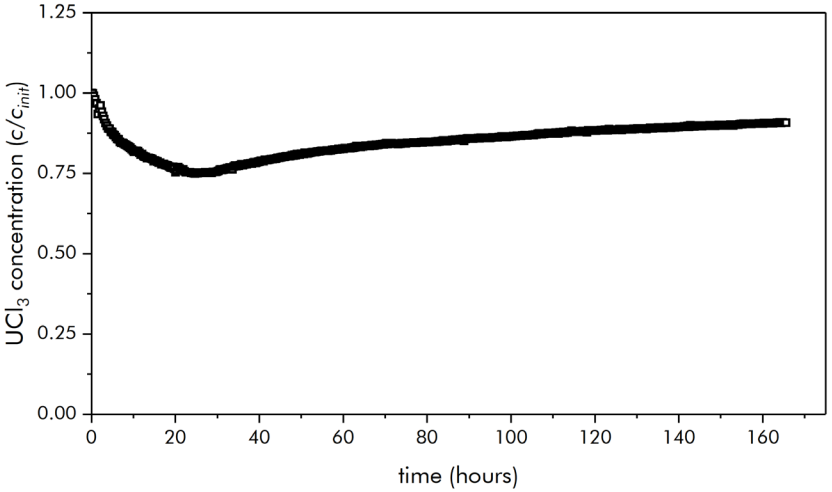
- Similar actinide and lanthanide content detected compared to earlier runs
- Electrode health still suitable for accurate voltammetry (after extensive in situ cleaning procedures)



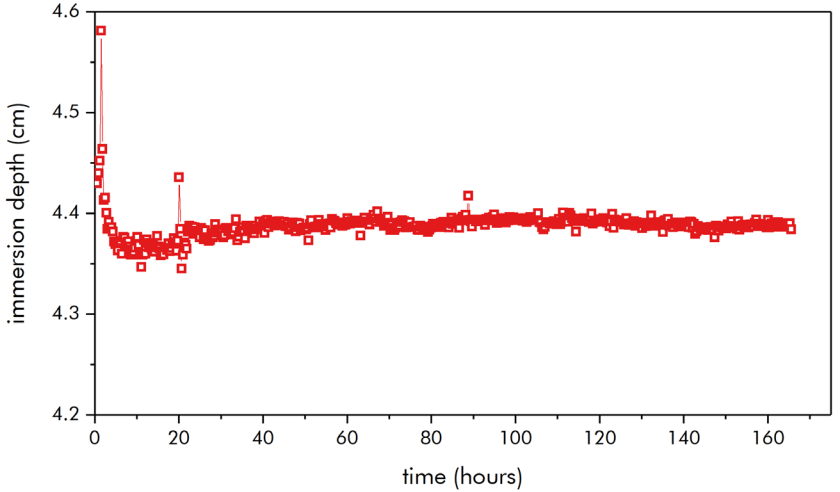
Raw linear sweep voltammograms during electrorefining operations

MONITORING DURING ER OPERATIONS

Monitoring of transient conditions showed good stability during early operations of the electrorefiner. In situ species concentrations and salt level measurements were inline with expectations. Results from salt samples are still pending analysis.



UCl_3 concentration during active electrorefining operations



Salt level during active electrorefining operations

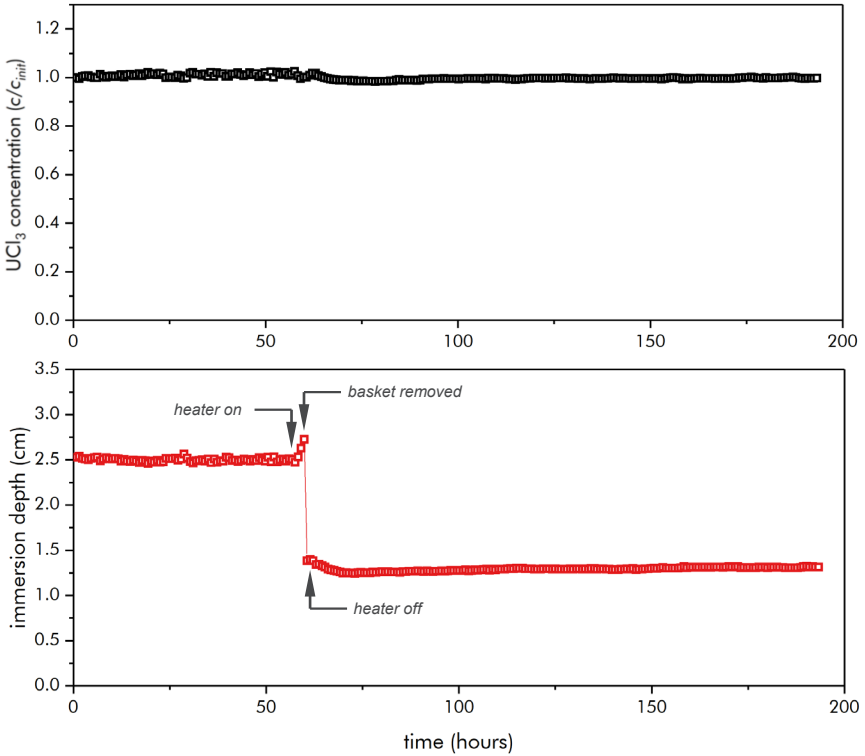
MONITORING DURING ANCILLARY OPERATIONS

Monitoring during ancillary operations such as basket insertions, basket removals, salt additions, etc. were also conducted.

All relevant operations were coordinated via an ER operations log maintained by INL.

Example ER Operations Log:

1110AM : Turned on upper zone for pulling cathode
1210AM : Pulled up cathode to drain position
1233PM : Turned off upper heater
1325PM : Cathode out / plug back in

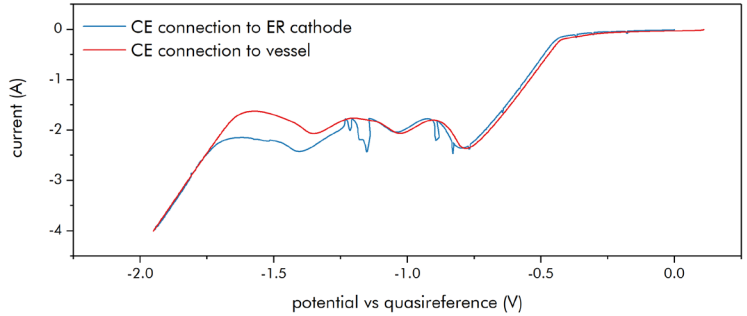


Concentration (top) and immersion depth (bottom) versus time during basket removal

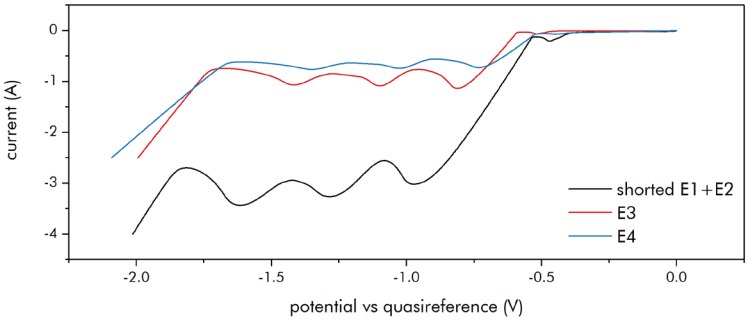
SENSOR PERFORMANCE

Periods of low data quality were encountered throughout the safeguards measurement campaign for a variety of reasons

- Counter electrode disconnections
- Noise from anode basket
  - Resolved by connecting counter electrode to vessel
- Disruptions from deterioration of hot cell atmosphere
- Shorting of electrodes to anode baskets and cathodes
  - Resolved by readjusting probe insertion
- Inter-electrode shorting from material buildup



LSVs before and after noise mitigation



Electrode array response with shorted E1 & E2

SENSOR PERFORMANCE

The voltammetry sensor has been in the hot cell for a period of three years and is beginning to show signs of wear

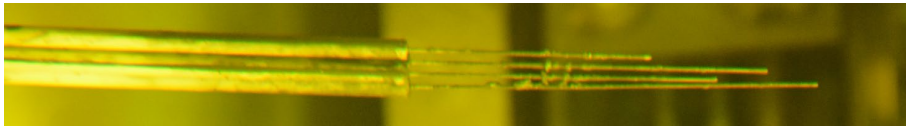
- At least eight months of total salt immersion time
- Multiple installation/removal cycles

Bent electrodes and material buildup has been observed in photos

- No attempts have yet been made to physically remove the built up material

Prior to Installation in Electrorefiner (August 2020)

After Temporary Removal from Electrorefiner (December 2020)

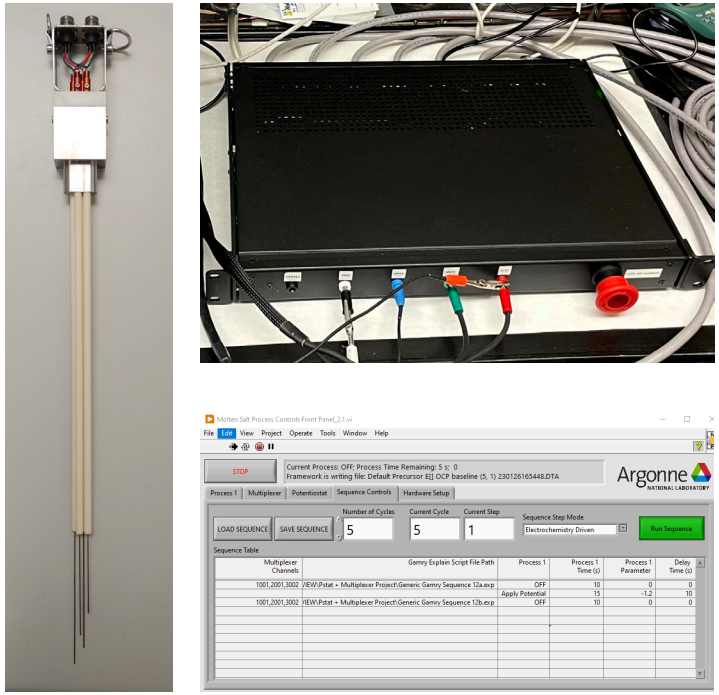


After FY22/23 Safeguards Campaign (February 2023)

UPDATED HARDWARE FOR FUTURE OPERATIONS

A variety of hardware improvements are being pursued to improve long-term operations of the sensor

- Replacement probe manufactured to address degradation of current probe
- Increased automation through the use of a new control interface
- New custom multiplexer hardware to control measurements across electrode array



Replacement probe (left), custom multiplexer (upper right), and updated control interface software (lower right)

ANTICIPATED ISSUES AND IDENTIFIED CARRY-OVER

- Currently on track to complete FY23 milestones.
  - M3FT-23AN040103052 - Report on Monitoring Campaign and Sensor Development Activities
  - M3FT-23AN040103053 - Publication Detailing INL ER Monitoring

- Anticipated carryover level for FT-23AN04010305 is ~20%

SUMMARY

- MPACT conducted a measurement campaign to develop and exercise sensor technologies for the safeguarding of pyroprocessing equipment
- Argonne's voltammetry sensor provided monitoring throughout this measurement campaign
  - Performance was generally good but various issues compromised measurement quality during certain periods of operations
- New hardware and software has been prepared to improve sensor performance and to make operations more automated

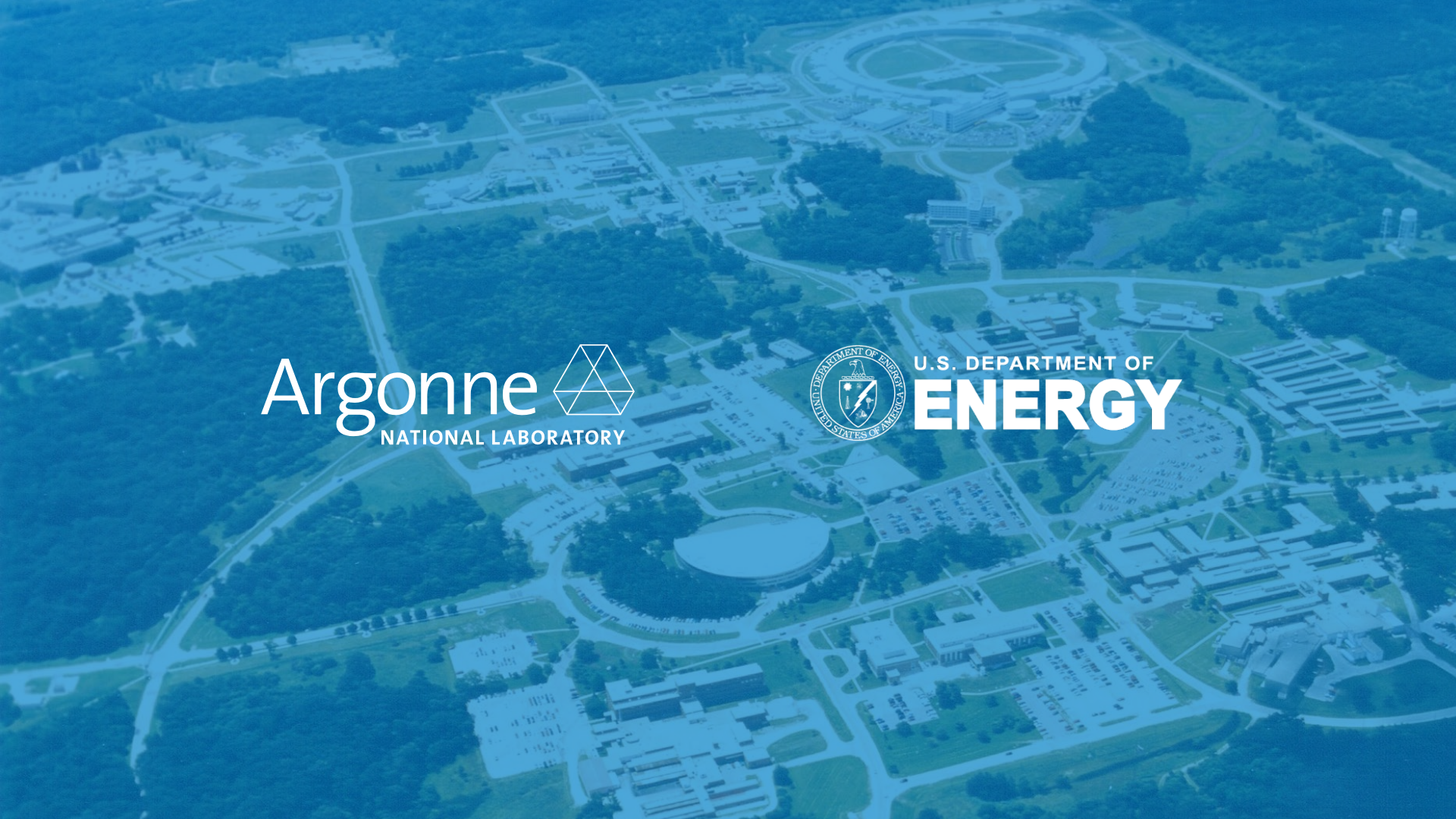
FUTURE WORK

- Resumption of sensor operations in HFEF electrorefiner FY24
  - Testing with new multiplexing hardware and sensor automation software
- Coordination with INL and LANL regarding analysis of data from ER safeguards campaign
- Continued development and deployment of safeguards-enabling tools

ACKNOWLEDGEMENTS

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

GOVERNMENT LICENSE NOTICE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