REPROCESSING SAMPLER

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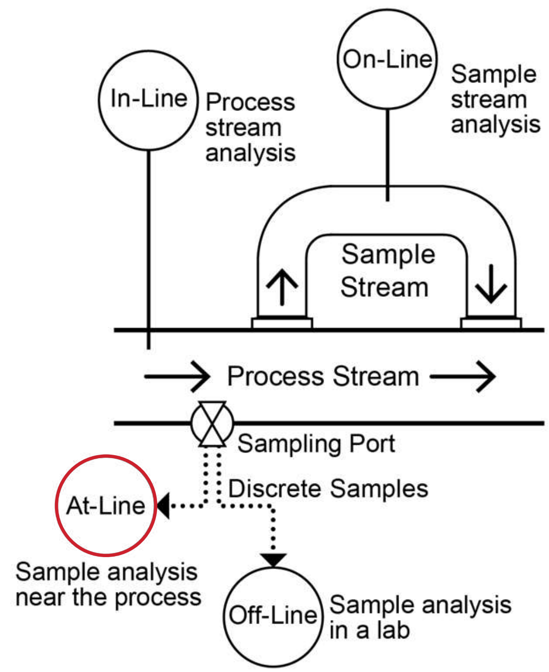
Project Objectives and Approach

- Develop and deploy automated molten salt sampling approaches for interfacing relevant unit operations with salt analysis
- Improve timeliness of sampling-based analysis
- Reduce statistical uncertainties of measurements
- Assessment of sampler in engineering-scale electrorefiner in FY23 with transition to hot cell deployment in FY24

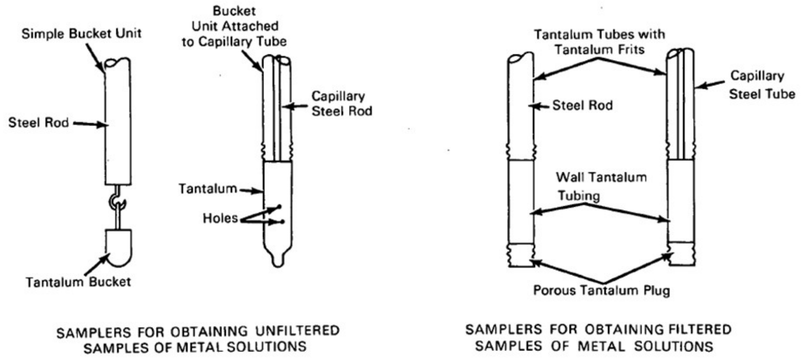
Approach:

- Replace manual off-line sample processing and analysis with automated at-line analysis
- Use novel sample tube mechanisms to support rapid sample delivery to lower temperature/lower radiation areas for high-precision non-destructive analysis

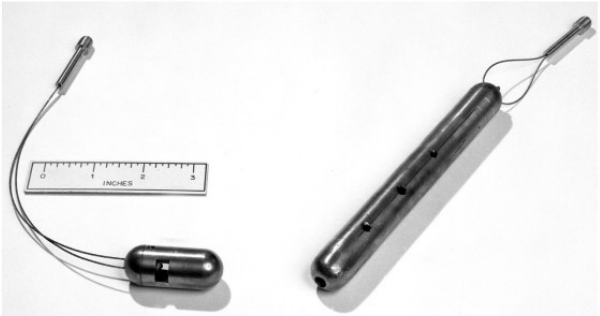


Historical Salt Sampling Procedures

- Reprocessing requires salt sampling to enable measurements for material accountability and process monitoring
  - Redox potentials
  - Corrosion rates
  - Impurities concentration
  - Particulate formation
- Traditional nuclear safeguards process monitoring requires a significant amount of manual sampling (e.g., fritted Ta tubes), sample processing and destructive analysis
  - High temperatures, air sensitivity, potential contamination and radioactivity of the salt
  - the sample is a poor representative of the bulk process material
- Assessment of historical practices and measurement uncertainties is underway
  - FT-23AN04010310: Molten Salt Sampling & Analysis – ANL



Winsch, I.O., ANL-7088 (1965)



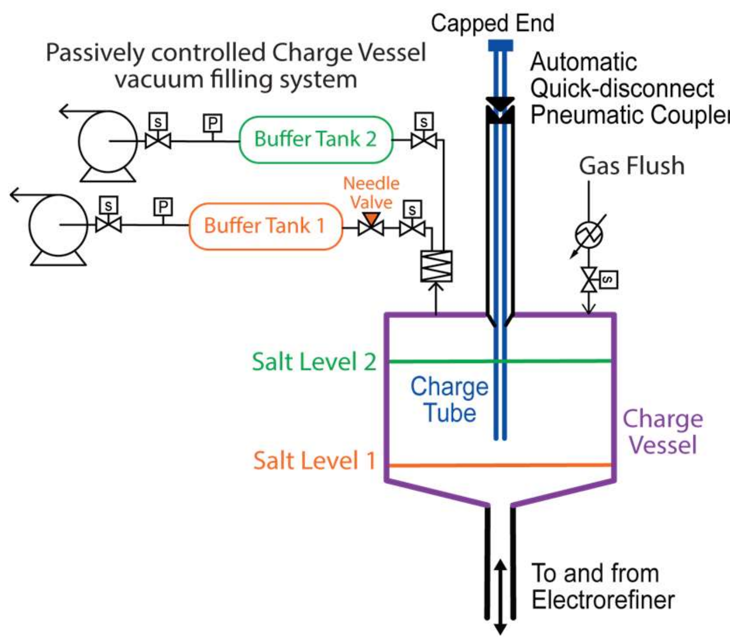
Gallaher, R. B. *Operation of the sampler-enricher in the Molten Salt Reactor experiment*. No. ORNL-TM--3524. Oak Ridge National Lab., 1971.

Rapid Sample Tube Charging Operation

- Dynamic filling process combines vacuum sampling and tube sampling
- Quick-connect tube assemblies to enable rapid/high-frequency sampling
- More representative samples versus traditional dip-tube point sampling by providing access to a larger cross-section of salt

Filling operation for sample tube assembly:

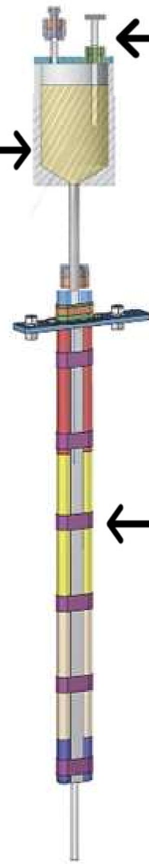
- Push-connect fresh charge tube
- Slowly vent charge vessel to first buffer tank until vacuum pressure equilibrates at first salt level
- Vent charge vessel to second buffer tank for immediate gas pressure drop in charge vessel and charge tube
- Equilibration fills charge vessel and charge tube to second salt level
- Pull-disconnect charge tube



Testing of Rapid Tube Charging Sampler

Rapid tube sampler insulated in glovebox

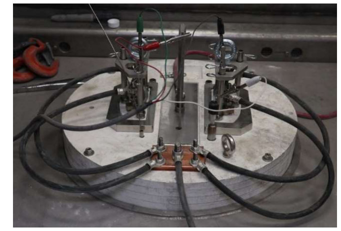
Upper sample vessel



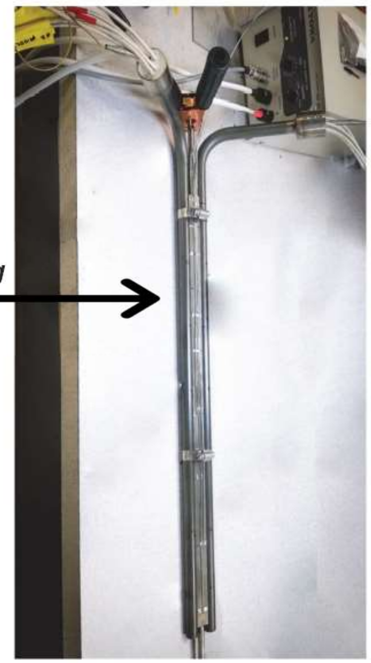
Sample extraction tube

Sampling line

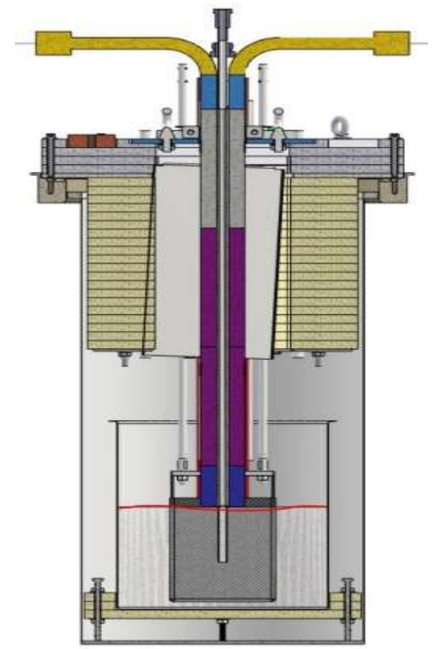
Rapid tube sampler module with heated sampling line



Top of Electrorefiner



Heated sampling line



Sampling line in electrorefiner

Samples obtained in extraction tubes

Sample extraction tubes and charge vessel interfaces with diameters from ¼" to ½"

Sampler Results from Argonne Engineering-Scale Electrorefiner

- A measurement campaign was performed with 65 samples (to date) pulled with the reprocessing sampler
- Various parameters were tested
  - Tube diameter
  - Buffer tank pressure
  - Operator recovery
  - Tube discharge time
- A standard operating procedure for the reprocessing sample was developed

1/4" 3/8" 1/2"

Results from molten salt sampling campaign

Tube Diameter (in)	Average Mass (g)	Standard Deviation
¼ A	1.34	1.07
¼ B	1.69	1.12
¼ C	1.24	1.20
¼ All	**1.36**	**1.10**
⅜ B	4.59	1.51
⅜ C	5.94	1.94
⅜ All	**5.13**	**1.73**

Sampler Test Observations

- Some samples were lost during removal from the sample due contact of the tube with the wall of the charge vessel interface
  - Determined a mechanical mechanism for stabilizing extraction tube upon removal is necessary
- ½″ tubes did not pull consistent sample possibly due to:
  - Lower relative surface tension forces
  - Large diameter of tube causing contact with the walls of the charge vessel interface
- Samples were observed to freeze towards the wall of the extraction tubes leaving a hole in the sample which released the vacuum in the tube
  - Will be leveraged for sample recovery

Upper sample vessel with heaters and sample extraction tubes

Samples obtained in extraction tubes

Sample Recovery Unit (Generation 1)

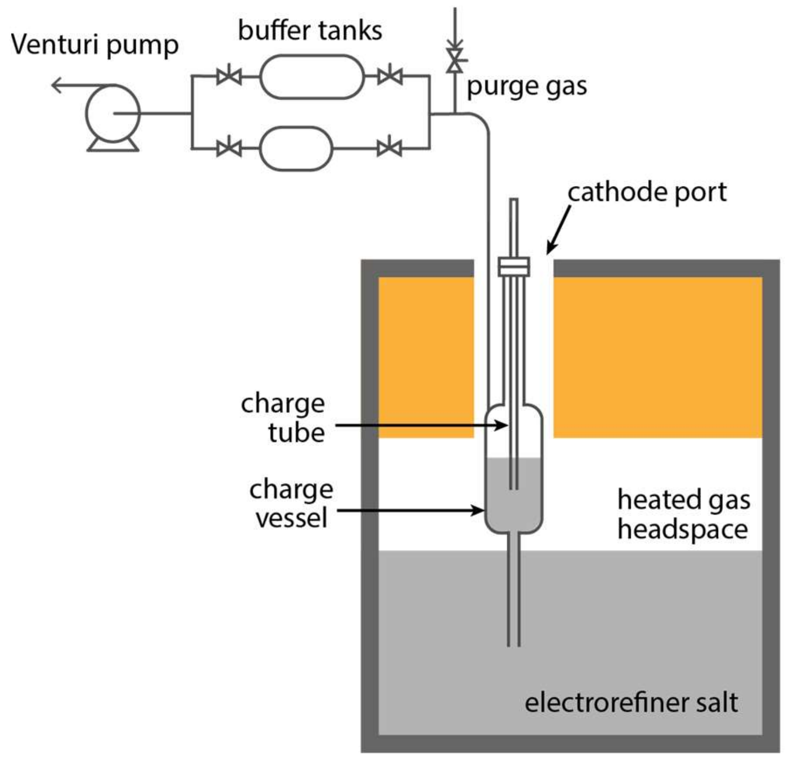
- A prototype for the sample recovery unit generation 1 for off-line analysis of the samples is currently under development
- This prototype will be used to recover samples from the reprocessing sampler on Argonne's ER
- Will be leveraging how the salt freezes to sides of extraction tubes
- Additionally, valves have been added to sample extraction tubes is vacuum is not fully released
- Recovered samples will be sent for analysis to determine salt inhomogeneity, particle composition and distribution

Sample Recovery Unit for Argonne ER operations

Sampler Module for HFEF Deployment

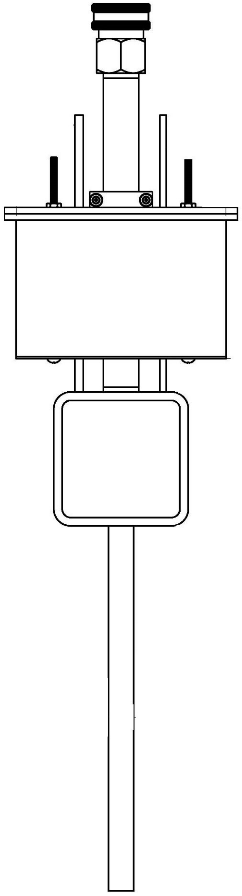
Maximizes use of existing infrastructure and tooling

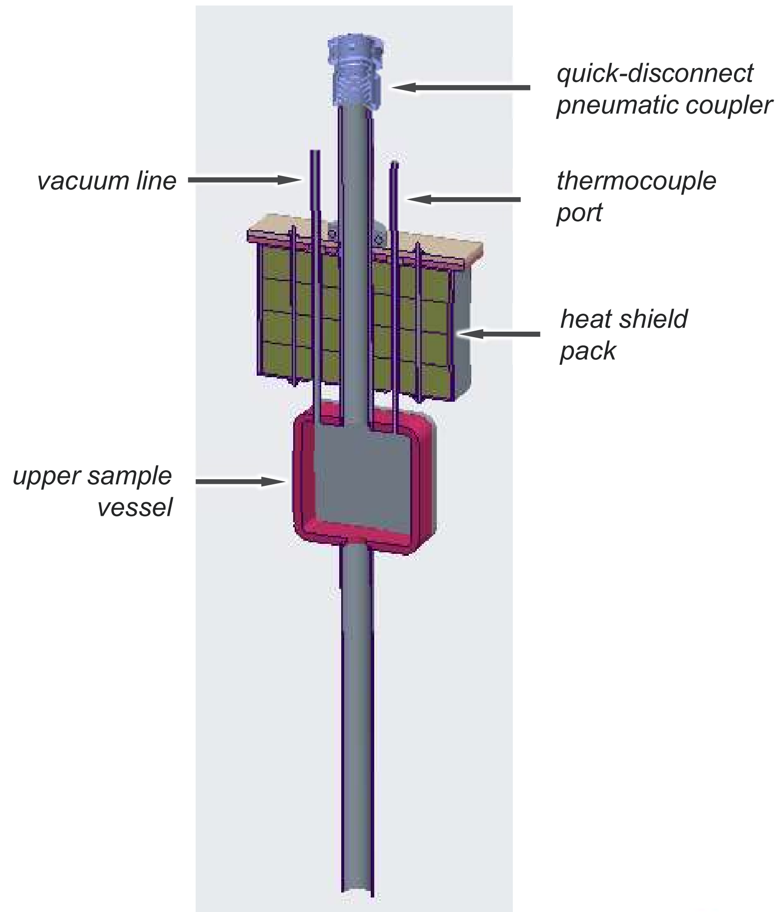
- Leverage the ER’s heated gas headspace
- Existing compressed gas supply to operate Venturi vacuum pump and gas purge
- Passive pressure control: buffer tanks sized to reach negative gas pressure set-points at steady-state Venturi pumping
- Existing visual pressure monitoring approach
- Manipulator valve-actuation and sample handling
- Utilize existing equipment for sample stabilization



Sampler Module for HFEF Deployment Progress

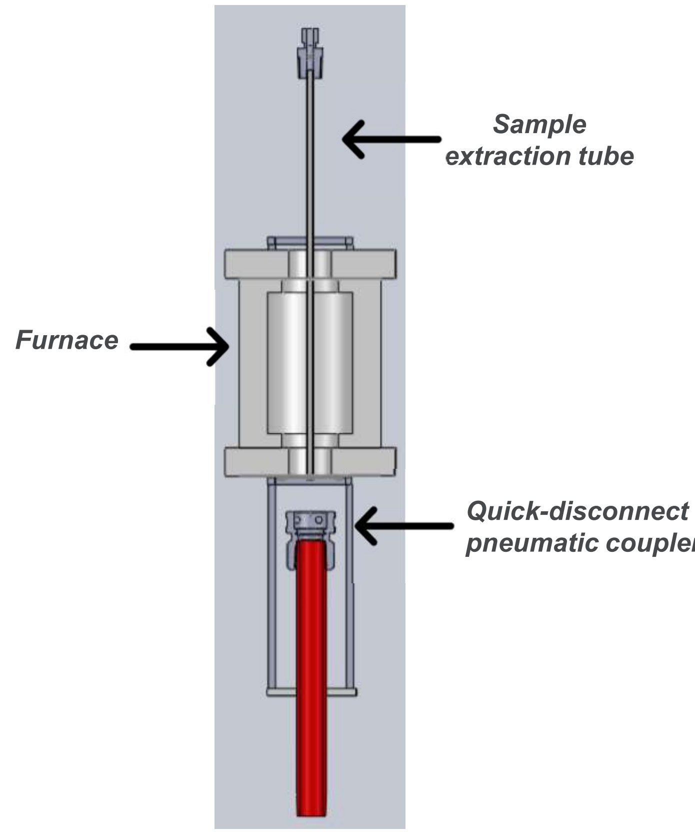
- Drawings of sampler module for HFEF deployment have been completed and approved by INL
- Materials have been purchased
- Argonne central shops has been sent drawings to begin manufacturing the sampler module
- Generation 1 sample recovery module for off-line analysis is under development





Generation 1 Sample Recovery Module for HFEF

- Generation 1 Sample Recovery Module will allow for rapid tube sampling discharging for offline analysis
- Furnace will be mounted directly above automatic quick-disconnect pneumatic coupler
- Extraction tube will be pulled into furnace where sample will initially freeze
- A collection chamber will be installed below furnace before heating
- Sample will drop into collection chamber upon melting
- Historically analytical labs found melting samples out of Ta tubes and then dissolving allowed for greater surface area available to react with water or acid resulting in more dissolution of the sample

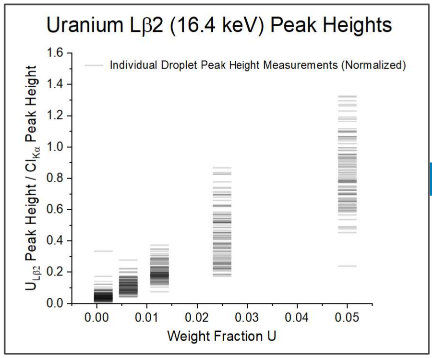


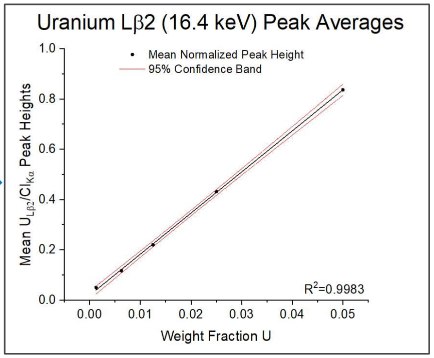
Rapid Sample Tube Discharging for At-line Analysis

- Rapid molten salt discharge to replace manual off-line aqueous dissolution of tube samples
- Molten salt sample tube discharge operation:
  - Drop tube segments into pneumatic sample generator chamber
  - Apply gas pulses to chamber to eject precision samples from tubes

Sample tube segments in sample generator

Spotted salt samples for XRF or µCAL analysis





High-throughput high-precision at-line salt sample analysis

Anticipated Issues and Identified Carry-Over

- Currently on track to complete M2 milestone design and delivery of sampler to HFEF facility for testing in an engineering scale electrorefiner.

- Anticipated carryover level for FT-23AN04010308 is ~20%
  - Some uncertainty as sampler fabrication costs are unclear

Summary

- Salt sampling tools under development to support rapid, high-precision salt characterization
- Sampler designed to improve accuracy of analysis by improving sample representativeness
- Novel dynamic vacuum sampling operation with rapid sample tube coupling and charging demonstrated with molten salt in an ER
- Working to balance the Argonne sampler operational needs with preparation for deployment of the HFEF system
  - HFEF ER sampler optimized to enable proof-of-concept testing with minimal facility disruption

Future work

- Sampler delivery to INL during FY23 with operations planned for FY24
- Utilize Sample Recovery Unit for sampling campaign with off-line analysis of ER salt
  - inhomogeneity, particle composition and distribution
- Development of Sample Recovery Unit Generation 2 for at-line analysis of ER salt
  - Integration with precision pneumatic sample generation
    - Argonne and HFEF
  - Integration with analytical techniques (particularly microcalorimetry) and vetting for inclusion in an integrated safeguards monitoring system

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