Mark-18A Cold Runs

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EXECUTIVE SUMMARY

The Savannah River National Laboratory (SRNL) is tasked by the National Nuclear Security Administration (NNSA) to recover highly valued isotopes from irradiated Mark-18A (Mk-18A) targets. The Savannah River Site (SRS) has sixty-five Mk-18A targets available for the recovery of the high valued materials. The sixty-five Mk-18A targets are currently stored in the L-Area Basin and will be removed one at a time and individually transported to SRNL. Upon receipt at SRNL, the Mk-18A target material will be removed from the confinement, dissolved, chemically separated, and calcined to a stable oxide. The flowsheet is designed to recover the plutonium as well as the trivalent actinides. The remaining unrecovered material will be discarded to the high activity drain (HAD) system in SRNL.

A specially designed cask was procured for transport of the targets from L-Area to SRNL. Once received at SRNL, the targets will be loaded into the back of Cell 7 and resized as they enter the cell. The resized targets (1/4 lengths) will then be processed one at a time through the following processes: caustic dissolution and filtration; acidic dissolution and filtration, Reillex anion exchange, diglycolamide (DGA) cation exchange; and DGA calcination. This processing will result in two product streams. The first is an aqueous plutonium solution which will be removed from the shielded cells and taken to a glovebox for further purification and conversion to an oxide. The second is a calcined oxide containing the Am and Cm as well as other lanthanide fission products which will be removed from the shielded cells using a bagless transfer system. Both materials will be packaged for shipment to Oak Ridge National Laboratory (ORNL).

All equipment to carry out this process was designed, procured or fabricated, and installed in a mock-up facility (716-4A) at SRS to allow for simulation testing in a non-radioactive area. This equipment was then dismantled and transferred from 716-4A to 773-A and installed in the SRNL Shielded Cells. After installation in the shielded cells facility testing was performed using water followed by surrogates and cold chemicals. Issues were identified during these evaluations, including equipment issues as well as technical challenges. Many of the issues were rectified during performance of the cold runs, and the remaining have a resolution identified. Table ES-1 provides a summary of all issues identified during the cold run operations, as well as the status and identified resolutions to outstanding issues.

Table ES-1. Summary of Issues Identified During Cold Run Operations

Issue	Status	Resolution
Equipment Checkout/Water Runs		
Quick connect for PW addition to TK-101 not allowing flow.	Able to add water through chemical addition port to complete water runs.	Quick connect to be replaced
Level Elements LE-060 (TK-107), LE-090 (TK-103B), LE-160 (TK-110) failure	Repaired by remaking connection at cable box.	Resolved
TK-108 Level Element LE-150 failure.	Replaced prior to acid simulant processing.	Resolved
P-040 leak	Pump replaced	Resolved
Green deposition on Reillex column	Disappeared at beginning of acid contact.	Resolved
TK-300 supply line leak	Repaired prior to acid simulant processing.	Resolved
Cask Receipt and Target Resizing		
Containment Bags were very difficult to install/remove from the ends of the cask which increases time in high dose area.	Process was demonstrated successfully but raised ALARA and safety concerns.	Resolved: Use of containment bags has been eliminated. Procedure L30-5.10 has been updated to incorporate this change.

Issue	Status	Resolution
An argon leak in the manifold connected to the CITB leg.	Held enough pressure to work for the cold run.	Enter E-079 and perform troubleshooting to determine if the leak is in the manifold or the cell insert seal. Repair or replace as needed.
The Cask sensor, located on the CITB, is too far from the Cask to get the appropriate distance reading.	Able to sense the cask for the cold run	Resolved: Placed a spacer behind the sensor to move it closer to the Cask.
Mk-18A bundle was pushed too far into the cell.	Bundle was pulled back through the cell insert to the OC First Cut position to resume Cold Runs	The bundle length in the software had been changed to accommodate an alignment test bundle that was shorter. The program had not been updated for the Cold Run dummy bundle. The software has been updated with the correct length. This will not happen during actual operations as all the Mk-18A assembly lengths are the same and there will be no changes to the software, without approvals, once all testing and demonstrations are complete and the software is locked down.
During a second demonstration the Sacrificial Plug bound as it entered Cell 7.	Able to use the manipulators in Cell 7 to realign the Sacrificial Plug.	Inspect Sacrificial Plugs and determine if additional deburring/smoothing of edges is needed. To further prevent re-occurrence, the Sacrificial Plug will be pushed further into the cell insert sphincter seal at the end of target resizing. This should center the plug to reduce the chances of binding when the target or dummy bundle pushes it into the cell.
During cutting of the IC bail end, the bail end was not gripped completely and rolled as the saw blade tried to engage.	The operator and SME were able to complete the cut using manual controls on the Cutter System HMI.	Resolved: Information and notes have been added to the procedure to give the operator flexibility to utilize the HMI controls available to recover from events like this.
During last two cuts of the simulated target, the saw paused cutting near the end of the cut just prior to cutting completely through the target.	The operator and SME were able to complete the cut using manual controls on the Cutter System HMI.	Resolved: A thicker spacer on the anvil type device that is used to secure the target for cutting has been fabricated, installed and tested.
Water line to the saw came loose during cutting evolutions.	Hose was temporarily reattached to continue the cold run.	Resolved: After completion of the cold run fittings were tightened down to ensure a good connection.
Low circulation flow was noticed during the fines vacuuming evolution. A kink in the suction line from the catch pan to the hydrocyclone was discovered, causing the accumulation of fines in the line.	The line was disconnected and cleared to allow cold runs to continue.	Resolved: The entire line and pump unit was replaced after cold runs completed to eliminate the kink.

Issue	Status	Resolution
Caustic Dissolution		
Transfer paths of chilled water from TK-123 to TK-215 were found to be swapped.	Discharge lines on P-050 and P-060 were changed to the correct configuration prior to proceeding with caustic dissolution.	Resolved
Pump flow totalizers not in agreement with level element indication of volume transferred.	Level elements were used as the basis for volume transferred.	Issue may have been due to communications issues with the software. The communication issues have been resolved, and accuracy of the pump flow totalizer functions will be monitored on future runs.
Release of small amount of liquid/mist from V-110.	Change order of valve closing post-backpulse to avoid residual pressure in TK-121.	Resolved: Changed order of valve closing in R&D directions.
Acid Dissolution		
Unable to achieve target [HNO_3] concentration in the acid dissolver.	Due to the number of backpulses performed, the starting volume in TK-235 was 7.4 L leaving room for only 3.8 L of concentrated acid to be added to the dissolver.	Resolved: In future runs, only a single caustic backpulse will be performed prior to moving to an acidic backpulse if required. Caustic rinse of the filter post-acid backpulse will be transferred to the HAD collection tanks rather than TK-235.
Pump flow totalizer not in agreement with level element indication of volume transferred.	Level element was used as the basis for volume transferred.	Issue may have been due to communications issues with the software. The communication issues have been resolved, and accuracy of the pump flow totalizer functions will be monitored on future runs.
Gelatin solutions began to set up prior to introduction into the acid dissolver due to unusually low temperatures.	Unclear of impact this may have on filtration.	Additional R&D being performed to optimize gelatin addition, including examining the possibility of adding gelatin as a solid.
Effectiveness of gelatin strike unclear.	Sodium metasilicate added as a surrogate for additional Si expected in the irradiated targets. No change in soluble [Si] observed before and after gelatin strike.	Additional R&D being performed to optimize gelatin addition.
LabView labels for solenoid valve V-300 were found to be incorrect.	Solids collected on FLT-315 versus the expected FLT-310.	Resolved. Labels have been corrected in LabView.
Potential solids breakthrough or bypass of FLT-315.	Downstream fouling issues indicated potential solids present after FLT-315.	Alternate filters and/or changes to the filter housing are being evaluated to avoid possibility of solids bypassing filter.
Difficulty in filtering samples in-cell.	Samples filtered in hood.	Evaluating alternate filtering options for in-cell filtrations (filtered centrifuge tubes or screw-driven piston for syringe filters).

Issue	Status	Resolution
Nitric Acid Transfer into Cell		
Initial attempt to transfer acid through the KAPL sleeve into TK-107 was unsuccessful. It was determined the transfer line and vent connections had been inadvertently swapped.	Connections were color coded to prevent reoccurrence.	Resolved
TK-107 level element (LE-060) stopped working due to corrosion from concentrated acid fumes.	The functioning was restored a couple of times during cold runs by cleaning the UT sensor.	Resolved: The sensor has been replaced with a new holder design that allows for rotating the level sensor off the tank (replacing with a cap) when measurements are not actively being taken. This will extend the life of the sensor by minimizing exposure.
Ion Exchange Operations		
Target flowrates unachievable for Reillex INEX column.	Upflow operations temporarily improved flowrates but were observed to decrease with time as additional volume of feed was processed.	Additional characterization of the resin is being performed to determine the source of fouling, but it is likely a result of filtration issues noted above (solids breakthrough or bypass of FLT-315). This issue expected to be resolved with resolution of the filter bypass issue.
Measured acid concentrations lower than expected.	TK-106 target nitric acid concentration was 8 M. Free acid titration result indicated 5.9 M. Ion chromatography analysis indicated 7.9 M nitrate.	A lab prepared standard of known acid concentration will be submitted along with all future sample sets. Additional troubleshooting of the method is needed to determine the cause for discrepancy in some samples.
Low transmitted raw light intensities through UV-vis flowcells.	Sufficient light transmission for cold runs.	Potential to improve light transmission through optimization of instrument set up and arrangement.
Periodic xenon light loss during UV-vis measurements.	Attributed mainly to instrument software real time issues.	Further testing of the instrument hardware and software are being explored to resolve this issue.
DGA Drying, Volume Reduction, and Calcination		
Upon initiation of argon flow to the rotameter, the flow was observed to spike and then drop to zero when the regulator and valve were opened. This suggests a faulty or leaking rotameter.	Rotameter was replaced.	New rotameter needs to be secured in such a way that it remains vertical, and tubing remains clear of potential hot surfaces during operations.
Control valve for argon into the rotameter failed (handle came off).	Resolved with replacement of the rotameter and tubing between the KAPL sleeve and inlet to the DGA column upflow piping.	Resolved
Difficulty aligning catalytic converter within the bucket assembly.	Assembly removed from the top of the furnace and internal alignment of the catalytic converter in the bucket was achieved.	Upon future equipment replacement, the catalytic converter should be attached to the bucket so that it is one integrated piece and does not require alignment of the catalytic converter in the bucket in the cells.

Issue	Status	Resolution
Quad-plug that powers the vacuum pump was found to be mislabeled.	Alternate switch from what is indicated by the color coding was flipped and rotameter on the vacuum pump was used to verify operation.	Quad-plug to be relabeled.
Failure of top heating zone of tube furnace.	Proceeded with middle and lower zones operating.	Based on results of the cold run, modifications have been made to the DGA column to allow it to sit further into the middle zone of the furnace during heating. The heating profile may also be adjusted on subsequent runs to optimize the volume reduction.
Failure of tube furnace over-temperature controller.	Controller was bypassed to allow attended operations only.	Resolved: No unattended operation of the furnace permitted. This restriction is noted in the procedure and R&D Directions.
Incomplete volume reduction of DGA resin.	Slightly overflowed crucible for final calcination resulting in a small amount of loss.	Resolved: Redesign of column to allow it to sit lower in the furnace and adjustment of heating profile (due to failure of top heating zone) expected to resolve this issue. If volume is still too great for calcination tray, final calcination will be performed in batches. Calcination tray will also be placed in secondary container during transfer to allow for recovery of any material that escapes the tray.
Mesh screen completely degraded during calcination.	Likely contributed to higher-than-expected mass of product.	Resolved: Alternate screen designs have been fabricated and will be tested.
Insulating cover for catalytic converter band heater needed.	During cold runs, insulating ceramic batting was used and the Cell 8 right manipulator was tagged out to avoid risk of contacting the exposed leads.	Resolved: An insulating cover has been fabricated and will be installed prior to the next furnace run.
Bagless Transfer and Packaging System		
Difficulty achieving <200 mTorr in the BJIC.	Approval obtained from SME to continue.	Resolved: Braze was evaluated, and it was determined that if the pressure reaches <300 mTorr, the process can continue.
Breaker trip on the brazing unit.	Reset and braze performed.	Resolved: Determine if there is a braze unit fault prior to initiating braze sequence.
Rotary chuck stopped moving towards the can during a cut.	The rotary chuck was manually backed out a few rotations through the HMI and the cut sequence was restarted.	Resolved: The cutting speed has been reduced from 0.5 rps to 0.4 rps to prevent recurrence. In the event of recurrence the cutter will be backed out 10 revolutions and the sequence restarted.

Issue	Status	Resolution
Robot experienced an "Excessive Disturbance Alarm" and stopped moving while torquing off the SFC cap. The system paused during the 'INSTALL SFC CAP' sequence.	This is normally due to the cap threads not starting to thread quickly enough while the robot moves the cap down.	Resolved; The Lid Shear Drive was manually rotated in the counter-clockwise direction to verify the cap had already been torqued off. The sequence was restarted after updating the SFC state to 'CAP ON'.
Upon attempting to remove the shield plug from the Type A drum, the sequence paused with an indication the robot could not find the item it was looking for.	The 3D vision system was unable to find the shield plug due to lighting discrepancies with the taught reference image. The solution was determined to be re-teaching the vision program for the shield plug.	Resolved
Working from two procedures for disassembly/reassembly of the Type A drum was found to be cumbersome and created confusion.	Recommended to move the HOI steps into the procedure L30-5.12.	Resolved: Revision of the procedure, L30-5.12.

Despite the issues identified above, the cold runs provided the opportunity to perform each of the unit operations necessary for processing of a Mk-18A target and identified areas of improvement that will be implemented prior to receipt of the first target. A dummy target bundle was successfully transferred from L-area and into the SRNL shielded cells, including resizing. A surrogate quarter target (aluminum tube) was successfully dissolved, demonstrating dissolution of the aluminum cladding. Target material surrogates were successfully filtered from the caustic dissolver and backpulsed into the acid dissolver. Neodymium oxide was used as a surrogate of the target material, and this was successfully dissolved in the acid dissolver. Cerium oxide was also included as a surrogate, and as expected, did not dissolve in the acid dissolver, but provide insoluble solids to collect on the filter after acid dissolution. Issues were identified with the filter and housing post-acid dissolution and these are being addressed with modifications to the filter housing. The dissolved Nd^{3+} was partially processed through the Reillex column, but fouling of the column occurred due to inadequate filtration of the feed coming from the acid dissolution process as noted above. The Nd^{3+} was successfully loaded onto the DGA column, and calcination of the DGA provided a surrogate oxide product. This material was loaded in an Mk-vial and successfully transferred out the shielded cells using the bagless transfer process, and then loaded into a special forms capsule and Type A drum to conclude the evolution.

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LIST OF ABBREVIATIONS

AE	Advanced Engineering
AHA	Assisted Hazard Analysis
BP	Boiling Point
BJIC	Bell-Jar Inside Cell
BTC	Bagless Transfer Can
BTPS	Bagless Transfer and Packaging System
CCM	Can Cutting Module
CITB	Cell Insert Transfer Block
CLM	Can Loading Module
CRT	Cask Receipt Table
DGA	Diglycolamide
DSA	Documented Safety Analysis
EDL	Engineering Development Lab
EMITS	Electronic Material Inventory Tracking System
FE	Flow Element
FLT	Filter
FMI	Fluid Metering Inc.
FS	Ferrous Sulfamate
HAD	High Activity Drain
HDPE	High Density Polyethylene
HMI	Human Machine Interface
HMTR	Hazardous Material Transport Representative
HOI	Handling and Operating Instructions
IC	Inner Can
ICPS	In-Cell Process System
INEX	Ion Exchange
KAPL	Knolls Atomic Power Laboratory
LAD	Linear Actuator Drive
LE	Level Element
M&TE	Measurement and Test Equipment
mA	Milliamps
Mk-18A	Mark-18A
NFV	Nuclear Filter Vent
NNSA	National Nuclear Security Administration

OC	Outer Can
ORNL	Oak Ridge National Laboratory
OTC	Over Temperature Control
P	Pump
PW	Process Water
R&D	Research and Development
RPD	Radiological Protection Department
rps	revolutions per second
SB	Sensor Box
scfh	standard cubic feet per hour
SCO	Shielded Cell Operations
SFC	Special Forms Capsule
SMB	Smart Motor Box
SME	Subject Matter Expert
SP	Setpoint
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
TB	Thermocouple Box
TC	Thermocouple
TE	Temperature Element
TID	Tamper Indicating Device
TSR	Technical Safety Requirements
TK	Tank
UET	Use Every Time
UT	Ultrasonic Transducer
UV-vis	Ultraviolet-visible Spectroscopy
V	Valve
VAC	Volts Alternating Current
Vdc	Volts direct current

1.0 Introduction

The Savannah River National Laboratory (SRNL) is tasked by the National Nuclear Security Administration (NNSA) to recover highly valued isotopes from irradiated Mark-18A (Mk-18A) targets. The Savannah River Site (SRS) has sixty-five Mk-18A targets available for the recovery of the high valued materials. The Mk-18A targets were manufactured with ^{242}Pu then irradiated under high neutron flux in K-Reactor at the SRS from 1968 to 1978. The sixty-five Mk-18A targets are currently stored in the L-Area Basin and will be removed one at a time and individually transported to SRNL. Upon receipt at SRNL, the Mk-18A target material will be removed from the confinement, dissolved, chemically separated, and calcined to a stable oxide. The flowsheet is designed to recover the plutonium as well as the trivalent actinides. The remaining unrecovered material will be discarded to the high activity drain (HAD) system in SRNL.

Due to dose concerns, the receipt and processing of the Mk-18A targets will be performed in the SRNL Shielded Cell Facility. The targets are currently stored in a double J-can confinement in the L-Area Basin, and they will be removed one at a time and transported to SRNL for processing. A specially designed cask was procured for transport of the targets from L-Area to SRNL. Once received at SRNL, the targets will be loaded into the back of Cell 7 and resized as they enter the cell. The resized targets (1/4 length) will then be processed one at a time through the following processes: caustic dissolution and filtration, acidic dissolution and filtration, Reillex anion exchange, diglycolamide (DGA) cation exchange, and DGA calcination. This processing will result in two product streams. The first is an aqueous plutonium solution which will be removed from the shielded cells and taken to a glovebox for further purification and conversion to an oxide. The second is a calcined oxide containing the Am and Cm as well as other lanthanide fission products which will be removed from the shielded cells using a bagless transfer system. Both materials will be packaged for shipment to Oak Ridge National Laboratory (ORNL).

All equipment to carry out this process was designed, procured or fabricated, and installed in a mock-up facility (716-4A) at SRS to allow for simulated testing in a non-radioactive area. The mock-up facility included a mock-up of SRNL Shielded Cells 7, 8, and 9, as well as two manipulators that could be moved between the mock-up cells. This allowed for verifying all operations and equipment manipulations could be performed in the space provided using manipulators. This equipment was then dismantled and transferred from 716-4A to 773-A and installed in the SRNL Shielded Cells. After installation was complete, check outs were performed with water to ensure all equipment had been reconnected such that there were no leaks and electrical equipment was responding as expected. The water runs were then followed by cold-chemical runs to demonstrate the process using actual chemicals and non-radioactive surrogates for the actinide elements present in the Mk-18A targets.[1] B-block of the SRNL shielded cells was de-inventoried of nuclear material prior to the window replacements that occurred prior to Mk-18A equipment installation. Under the current Documented Safety Analysis (DSA) B-block is considered de-inventoried and therefore, Mk-18A cold runs required the use of non-radioactive surrogates as no new radioactive materials could be introduced. This document describes the results of the initial equipment checkouts and the cold chemical runs in the shielded cells facility. Process diagrams of the equipment installed in Cells 7-9 are provided in references 2 – 4.[2,3,4]

All chemical processing activities were covered under a Hazard Analysis Package (SRNL-ACT-01985). Assisted Hazard Analyses (AHAs) were performed for the cask receipt, target cutting, and bagless transfer and packaging operations (AHA-SRT-34532, AHA-SRT-34752, and AHA-SRT-34753). Research and Development (R&D) personnel led the in-cell chemical processing operations with support from Shielded Cell Operations (SCO) personnel for performing the hands-on operations and technical support from Advanced Engineering (AE) personnel. Operations personnel led the cask receipt, target resizing, and bagless transfer and packaging operations with support from AE and R&D personnel.

Actual processing of Mk-18A targets will require nuclear material accountability and tracking using the Electronic Material Inventory Tracking System (EMITS). As no nuclear material was used in the cold run demonstration, EMITS transactions were simulated using EMITS Version 3.0 set up on a separate server dedicated to the Mk-18A cold runs. This allowed for simulating the transactions without impacting the actual SRNL nuclear material inventory system. The composition of the expected first target (FT-80-03) was used for the mock EMITS transactions.

2.0 Electrical Checkout and Water Runs

2.1 Electrical Checkout

The In-Cell Process System (ICPS) pertains primarily to the In-Cell chemical processing components in Cell 8 and Cell 9. These systems include the caustic dissolver, metal filter, back pulse tank, acid dissolver, chiller skid, bag filter housing, Reillex ion exchange (INEX), DGA INEX, tube furnace, muffle furnace, and the associated tanks, tubing, manual valves, automated valves, sensors, pumps, and mixers. These systems were fully tested in the mockup facility (716-4A), but needed to be re-checked after disassembly, transport, and re-installation in the shielded cells.

This equipment is controlled by the Mk-18A In-Cell HMI (Human Machine Interface) software running on the Mk-18A In-Cell System control computer. This computer is connected via an ethernet connection to the Mk-18A Main Electrical Box located in room E-023. Numerous cables from the Main Electrical Box are routed through the KAPL sleeves and connected to seven junction boxes mounted on the cell liners' back wall. These junction boxes include two motor control boxes, two sensor control boxes, two thermocouple junction boxes, and one solenoid control box. Additional connections are also made to one junction panel at the chiller skid, the caustic heater assembly, the acid dissolver heater assembly, and the catalytic converter heater. Also, cables from the tube furnace and muffle furnace controllers, which are external to the cells, pass through the KAPL sleeves to the respective furnace. The cables provide power and control signals from the main box to the junction boxes and return sensor data to the main electrical box.

The method to check out the equipment prior to chemical runs required a two-step process. The first step was to verify connection to the devices (power and signal). The second step was performed during water runs and cold chemical runs where function over a range of conditions was possible. The first step of this process was to perform a function check of each component. This verified the cable connections between the Main Electrical Box, cables to the KAPL sleeves, the KAPL sleeve cables, the In-Cell junction box, the In-Cell cables and the component.

The two motor control boxes are connected to a network of motors connected to FMI pump heads or mixer heads. Both Smart Motor Boxes (SMB-1 and SMB-2) have a power cable and an RS-485 communication (Comms) cable coming from the Main Electrical Box. SMB-1 has 6 mixers and 2 pumps connected. SMB-2 has 6 mixers and 3 pumps connected. Each motor has a power and Comms cable from the SMB. At the HMI software, it was determined by the feedback that each motor had power and was communicating. There were some inadequate connections that were rectified at this stage. Mixer operation can only be determined by removing the mixer from the tank and visually observing the mixer blade turning, which was not practical in the cells. FMI pump operation can be verified visually only in cases where the motor rotation is observable, otherwise verification was made during the water runs.

For the motor networks, feedback from the individual motors from the RS-485 network port indicated that the motor had power and communications. It was verified that most of the motors were communicating. Motors that were not communicating required checking the power and communication cable at the junction box. Eventually all motors for the FMI pumps and mixer heads were verified operational. However, this check did not verify that the motor was on the correct channel. That was validated later during water runs.

Each of the two sensor boxes (SB-1 and SB-2) have three cables from the Main Electrical Box, one providing 24 Vdc power and two signal cables. SB-1 has seven level sensors, five flow meters, and two pressure sensors attached. SB-2 has eleven level sensors and three flow meters attached. Each sensor has a single cable to the respective SB.

The sensor output was checked to ensure the signal was in the range of outputs for the sensor type. For example, for a 4-20 mA signal, sensors should be reading near 4 mA. A zero-mA reading indicates that the sensor was not functional or not properly connected. Several connections were found to be flawed on several channels. Most issues were corrected by reattachment of the Amphenol connectors at the SB. One level element (LE-150) was found to be faulty and was replaced. A logical method to ensure the sensor is connected to the correct channel would be to disconnect it from the SB, however, it is not a good practice to disconnect the sensor with power on due to the possibility of damaging the sensor. Therefore, validation of the correct channel connection was performed during the water runs.

The two Thermocouple Boxes (TB-1 and TB-2) are connected to Type J and Type K thermocouples (TCs). TB-1 has eleven Type J TCs and TB-2 has seven Type J and four Type K TCs attached. For the thermocouple checkout, it was verified that the TC was reading correctly near room temperature, and the channel was verified by unplugging it from the junction box to ensure correct connections. Unlike the sensors discussed above, TCs can be unplugged during operation without causing damage. In this way, all the TC channels were verified. However, the final verification was performed during water runs to verify that the temperatures were increasing during heating evolutions.

The valve box is connected to five solenoid valves. Operation of the valves through the valve box could not be tested. The valves are normally closed and a 24 Vdc digital output is supplied to the coil of the solenoid to open the valves. The state of the valves cannot be determined visually. The only way to validate solenoid valve operation was during water runs.

The chiller skid has three cables from the Main Electrical Box. Two cables are for the control of two 24 Vdc pump motors. The third cable provides 220 VAC power to the fan motor. Operation was verified by observing the electrical current in the HMI software using current transducers in the Main Electrical Box. However, this does not confirm fluid flow or airflow from the fan across the radiator, this was verified during the water runs.

There are three pneumatic pumps that are operated by compressed air (plant air) controlled by solenoid valves that are not connected to the valve junction box in Cell 9. These solenoids are controlled by sending a command from the ICPS HMI to the Cell 7 Cutter System HMI. The operation of these pumps was performed during water runs as there are no other indications that the pumps are running.

The catalytic converter heater, caustic dissolver heater, and acid dissolver heater modules are connected to the Main Electrical Box via cables through the KAPL sleeves. Furthermore, cables from external furnace controllers pass through the KAPL sleeves to the Tube Furnace and the Muffle Furnace. The Main Electrical Box provides an interlock to the tube furnace controller and RS-485 communication to both the tube furnace and muffle furnace. Both furnaces can be controlled by the ICPS HMI software or the Eurotherm controllers on the external controls.

The tube furnace, muffle furnace, caustic dissolver heaters, acid dissolver heaters, catalytic converter heaters and related thermocouples were tested. This was done by setting the power output to 5–10% or by setting a low temperature setpoint on the controllers (tube and muffle furnace). On the caustic dissolver heaters, acid dissolver heaters, and catalytic converter heaters, the output current is measured on current transducers and current reading is a confirmation of operation. In addition, the associated TCs also indicated higher temperature with heating. In the case of the tube furnace and muffle furnace, indication of % power output and TC temperature rising indicated proper connections. The connections and low power operations of the caustic dissolver, acid dissolver, catalytic converter heaters, and muffle furnace were verified, but high temperature testing was performed during the water or cold chemical runs.

Unfortunately, during check out of the tube furnace, it was discovered that the over temperature control (OTC) thermocouple and top zone heating element were damaged during the move or installation in the shielded cells. It was deemed impractical to repair these faults in the shielded cells. The OTC thermocouple was bypassed in the external furnace control; however, this disables OTC protection and will necessitate 100% surveillance while heating. The furnace can be operated without the top zone, and actions will be taken to minimize impact to the thermal cycle for the DGA drying operation. This will be discussed further in Section 3.6 when discussing the cold runs.

2.2 Water Runs

Water runs were performed prior to the start of the cold chemical runs to verify all flow paths and connections and ensure there were no leaks. Process water (PW) additions were made to all tanks capable of receiving process water additions. Prior to execution of this portion of the water run, procedure TO-13-001 was executed to start up the E-wing process water system. The process water is introduced through a KAPL sleeve on the left side of Cell 8, to the Mk-18A process water manifold, MANIFOLD-002. The plant water supply has a flow meter and a solenoid valve that were verified functional as the tanks were filled. Adding PW to the tanks also allowed verification of the level sensors over the range of operation, whereas earlier checkout only verified the empty signal. This process also verified that tubing connections from the water manifold to each tank were correct. Table 2-1 summarizes the tanks PW was added to, and the level elements used to monitor additions.

Attempts to add PW to TK-101 were initially unsuccessful. Troubleshooting identified the issue to be the quick connect fitting on the top of TK-101 not opening when tubing was connected. As a temporary work around, the chemical addition port on the top of TK-101 was opened and the PW tubing to the TK-101 quick connect was disconnected and fed into the opening on the top of TK-101. A replacement quick connect fitting has been obtained, installed, and is awaiting testing. During PW additions, it was also confirmed that the LE-150 on TK-108 was not functioning and needed to be replaced. This was done prior to the start of cold chemical runs. For the water runs, a small amount of PW was transferred to TK-108 to confirm no leaks in the flow path.

Table 2-1. Initial Process Water Additions to Various Tanks

Tank ID	Purpose	Level Element ID	Volume PW added (L)
TK-101	$NaNO_3$ reagent	LE-020	90.2
TK-102	NaOH reagent	LE-030	32.6
TK-123	Chiller	LE-050	47.9
TK-108	Wash acid	LE-150	N/A
TK-109	Elution acid	LE-140	5.1
TK-110	DGA Raffinate	LE-160	3.8
TK-215	Caustic Dissolver	LE-040	8.6
TK-235	Acid Dissolver	LE-080	2.1
TK-121	Backpulse tank	LE-070	0.8
N/A	Chip pan	N/A	N/A

Water was transferred into Tank TK-107 located in Cell 9 using the nitric acid transfer apparatus. It includes a 57-L (15-gallon) stainless steel drum with secondary containment located outside of the cells and a peristaltic pump located inside the cell. This was performed using L33-0255 as a reference (technical reference) procedure.[a] The 57-L drum outside of the cells was filled with process water for this evolution, and the water was transferred from the drum through a KAPL sleeve penetration and into TK-107. Initially the level element in this tank (LE-060) was not working but was fixed by disconnecting and reconnecting a cable. During the next few days, water from TK-107 was transferred into all the tanks expected to receive nitric acid utilizing pump P-070 and the nitric acid manifold, MANIFOLD-003. Table 2-2 summarizes these transfers. As noted above, the level element in TK-108 (LE-150) was not working, and it was later replaced. The volume transfer to TK-108 was determined using the level element on the sending tank, TK-107.

Table 2-2. Water Transfers from TK-107 to Various Tanks (Simulating Nitric Acid transfers)

Tank ID	Purpose	Level Element ID	Volume added (L)
TK-103B	HAD Residue	LE-090	5.1*
TK-103C	HAD Residue	LE-110	8.0
TK-109	Elution acid	LE-140	6.0
TK-110	DGA Raffinate	LE-160	6.1*
TK-106	Reillex Feed	LE-130	7.4
TK-108	Wash Acid	LE-150	5.4*
TK-235	Acid Dissolver	LE-080	5.0
TK-121	Backpulse tank	LE-070	0.4

*LE of receiving tank not working at time of transfer, but later fixed by reconnecting cable at junction box. Volume based on LE-060 (sending tank).

After adding water to the various tanks, either through the process water system or as simulated nitric acid transfers from TK-107, transfers between tanks were made. This allowed for verification of pump operation, flow meter operation, and level sensor operation in source and target tanks. The general procedure to verify flow was to look for agreement between flow measured on flow meters, compared to flow calculated from feedback from the motors, and compared to changes in level sensor readings (in volume) from the source and target tanks.

The chiller pumps P-050 and P-060 were both operated to recirculate water through the heat exchangers. Transfers were then made from TK-101 and TK-102 to TK-215 to simulate $NaNO_3$ and NaOH additions to the caustic dissolver. These transfers utilized pumps P-020 and P-030. P-020 was operated at a flow rate of 1.0 L/min while P-030 was operated at 250 mL/min. The heater elements in TK-215 were also tested once the water had been added.

Due to an incorrect interpretation of the P&ID, an attempt was made to recirculate the contents of the caustic dissolver using P-040 and the flow path from the bottom of the dissolver, through V-100, and return to the tank through V-130 as shown in Figure 2-1 below. Upon initiation of this valve alignment and pump operation, a leak was observed from P-040. The pump was stopped, and it was then determined that this valve alignment was not meant for recirculation of the caustic dissolver, and that the tank return from V-130 was meant as a return line from FLT-305, as necessary. Attempts were made to tighten the bolts on P-040 to stop the leak but were unsuccessful. P-040 was replaced with a new pump.

After replacing P-040, transfers were made from TK-215 through FLT-305 to the HAD residue collection tanks, TK-103B and TK-103C. It was noted during this evolution that once P-040 stopped, the volume continued to transfer from TK-215 to the HAD residue collection tank until the valves were closed. Under normal operations, it is expected that the full volume of TK-215 will be transferred to either TK-103B or TK-103C in a single evolution. However for the water runs, the flow path to both residue collection tanks was being tested, transferring approximately half of the volume in TK-215 to each tank. Using argon,

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[a] L33-0255 has since become a Use Every Time (UET) procedure.

backpulsing of FLT-305 was also tested by pressurizing TK-121, which contained 1.2 L of water. The backpulse of FLT-305 was routed to TK-235.

The heater elements in TK-235 were tested and shown to be functional. The water in TK-235 was then pumped through filters FLT-310 and FLT-315 and into TK-106 using P-100. After about half of the volume of TK-235 had been transferred through FLT-310, the pump was stopped, and valve alignments were made to pump the remaining volume through FLT-315. Bag filters were installed in the filter housings for this operation.

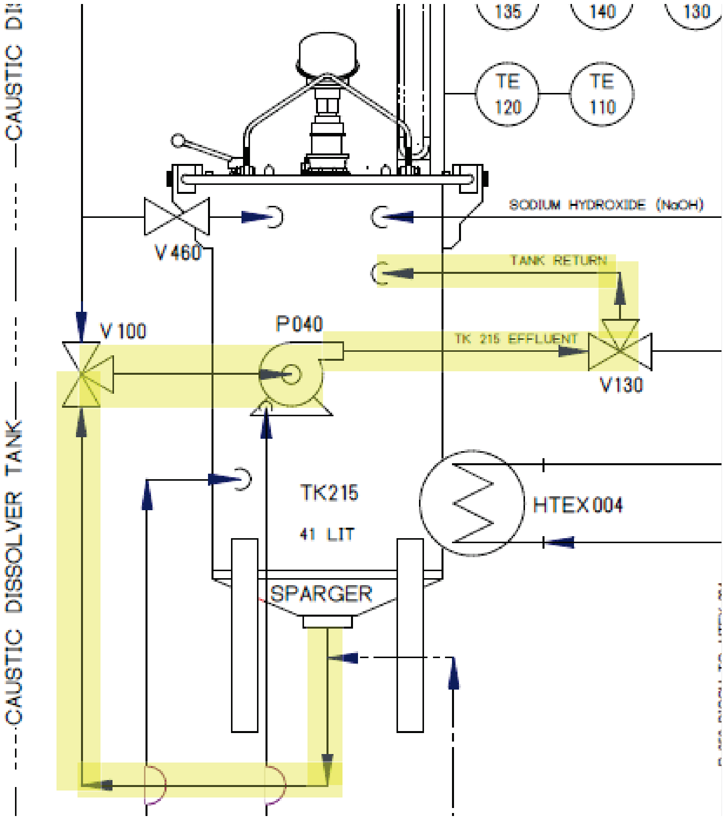


Figure 2-1. Flowpath of attempted recirculation of TK-215

Water was subsequently processed through Reillex/water-filled glass column INEX-003 from tanks TK-106, TK-108, and TK-109 into TK-124. Water was processed through the column by upflow and downflow through the Reillex standpipe (TK-305). The standpipe heights required to produce downflow water flowrates ranging from 5-60 mL/min were determined and the data is plotted in Figure 2-2 where a linear response is observed in the standpipe height and volume versus flowrate. In general, the Reillex system performed as expected except for the TK-108 level element, which was not functioning and was replaced. A green algae color was observed in the first couple of inches of the top of the Reillex column after water runs which is presumably bacterial growth present in the water that adhered to the column. The green color was monitored during subsequent nitric acid processing (see discussion below). This is not expected to be a concern in the future as the column will be stored in dilute acid. Tubing connecting valves V-700 and V-670 to downstream tanks was incorrectly installed, but this issue was corrected upon discovery. Elution hearts and tails steps were conducted by collecting column effluent water in TK-111 (2 L poly bottle) and TK-112, respectively. Pd stripping steps were conducted by processing water from TK-113, through the Reillex column, and into TK-114 (both of these tanks are 2-L bottles).

Water was also processed from TK-124 through column INEX-001 (which did not contain DGA resin) into TK-110. The DGA column also did not contain water. Initial water flow was in an upward direction to remove air from below the resin support screen and completely fill the column with water. Since flow resistance from the DGA resin bed was minimal, faster than normal flow rates were required during downflow operations to see significant response in the standpipe liquid level element (LE-200 on tank TK-300). Water was processed through the DGA column and standpipe by upflow and downflow. Standpipe heights were not meaningful to any subsequent testing since the column was not loaded with resin. A leak was observed in the supply tubing leading to TK-300 which was addressed upon observation by tightening the associated Swagelok connections. In general, the DGA system performed as expected to the extent that evaluation was possible without a packed resin bed.

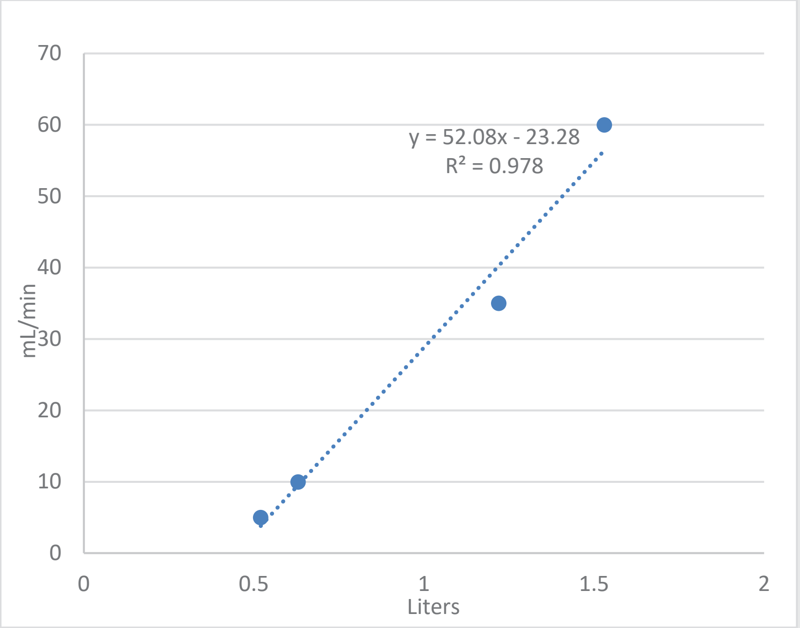


Figure 2-2. Reillex Standpipe Liquid Volume Versus Flow Rate for Water Runs January 2024.

Following testing, the water in the various processing tanks was transferred to HAD residue collection tanks TK-103B and TK-103C. Water was then transferred from these tanks to TK-103A in Cell 8 where it was subsequently transferred to the SRNL HAD. The water heel volumes remaining in the tanks after complete pump down based on the LabView Level Element readings are provided in Table 2-3 below. These heel volumes should be considered approximate.

Table 2-3. Cell 9 Tank Heel Volumes

Tank	Heel Volume (L)
TK-103A	6.5
TK-103B	0.3
TK-103C	not recorded
TK-106	2.5
TK-107	3.3
TK-108	unknown*
TK-110	0.4
TK-112	0.2
TK-124	2.0

* Level element not working.

Equipment issues observed during the water runs are summarized in Table 2-4.

Table 2-4. Mark-18A Equipment Issues during Water Evaluations and Status/Resolution

Issue	Status/Resolution
Quick connect for PW addition to TK-101	To be replaced
Level Elements LE-060 (TK-107), LE-090 (TK-103B), LE-160 (TK-110) failure	Repaired by remaking connection at cable box
TK-108 Level Element LE-150 failure	Replaced prior to acid simulant processing
P-040 leak	pump replaced
Green deposition on Reillex column	disappeared at beginning of acid contact
TK-300 supply line leak	repaired prior to acid simulant processing

3.0 Cold Runs

3.1 Cask Receipt and Target Resizing

3.1.1 Receipt of the Mk-18A Cask

Mark-18A targets are currently stored in a double-can confinement, referred to as a “J-can”. The Mk-18A fuel is 4.22” in diameter and between 90.3” and 92” in length. The “inner J-can” is 4.875” in diameter and 13’ in length. The “outer J-can” is 5.563” in diameter and 14’ in length. See Figure 3-1 for a diagram of the target assembly. The dummy bundle was fabricated from aluminum tubing with the same dimensions. The dummy target bundle was loaded into the Mk-18A shipping cask in L-Basin. The dummy bundle consists of an outer can, an inner can, and a dummy target, all of which have the same dimensions as the Mk-18A target and J-cans. The dummy bundle was then delivered to SRNL in the Mk-18A Shipping Cask which consists of a shielded sleeve and a structural frame. A simulated EMITS transaction was initiated upon receipt of the Mk-18A Shipping Cask. The Shipping Cask was backed into Room E-055 and disconnected. The Radiological Protection Department (RPD) ensured that all surveys were completed, the truck was removed, and the Room E-055 shield doors were closed. The simulated EMITS transaction was completed.

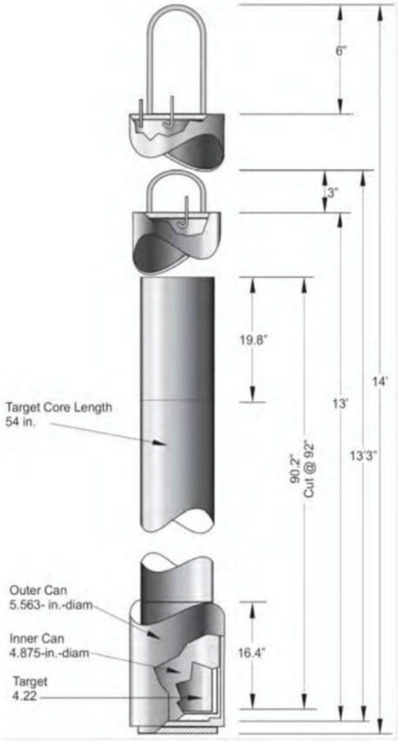


Figure 3-1. Target Assembly in Double J-Can Confinement

The following day, another simulated EMITS transaction was initiated for an internal move. The radiological control bag was unzipped and rolled down far enough to access the cask ball lock pins. See Figure 3-2 for a photo of the cask in the radiological control bag before unzipping. RPD verified that surveys were completed. The radiological control bag support structure was removed from the Mk-18A Shipping Cask and stored in Room E-055. The drip pans and absorbent pads were verified and surveyed. Visual verification was performed to ensure all pieces of the Mk-18A Cask were present, with no signs of damage. The Mk-18A Cask ID was verified that it was traceable to the authorization ID listed on the shipping operations homepage. The 4 ball lock pins were removed from the 4 pivot arms and the pivot arms were lowered out of the way. The approved rigging was installed on the Mk-18A Cask lifting bail and connected to Room E-055 10-ton crane hook. Containment bags were installed on each end of the cask to capture any potential water that dripped out of the cask during movement. The top cable-operated door and the E-079 double doors were opened.

Figure 3-2. Photo of Cask Inside Radiological Control Bag

Prior to moving the Cask into E-079 the [Prepare To Load Cask] automatic sequence on the HMI was selected and executed as shown in Figure 3-3. The following configuration was verified on the Cask Receipt Table (CRT) after completion of the automatic sequence:

- Plug Platform (Shelf) is in the STOWED position.
- Cradle Lift (Trough) is in the FULLY DOWN position.
- Cask Table is in the LOAD position.
- Chain Drive (Push Plug) is in the RETRACTED position.
- Shield Door Plug is in the FULLY DN position.
- South End Shield Plug Lift Table is in the DN position.

HMI MODE: TROUBLESHOOTING
HMI LOCATION: BACK OF CELL 7
SEQ PREPARE TO LOAD CASK
11:18:42 AM
06/29/2021

AUTOMATIC OPERATIONS STATE: INSERT SHIELD DOOR PLUG DN COMMAND EXECUTING: Elapsed Time 4 of 40 Seconds

	POS DESCRIPTION	POS DEG.	VEL DEG/SEC
PLUG PLATFORM (SHELF)	STOWED	0.00	0.00
CRADLE LIFT	FULLY DOWN	0.00	0.00

	POS DESCRIPTION	POS IN.	VEL IN/SEC
CASK TABLE	LOAD	0.03	0.00
CHAIN DRIVE (PUSH PLUG)	RETRACTED	0.63	0.00

MOVING CASK TABLE TO 0.000 Inches : LOAD: Elapsed Time 4 of 75 Seconds : Current Position = 0.0 Inches ...COMPLETE

CASK DISTANCE (Inches): -1.000
SHIELD DOOR PLUG
PLUG POSITION: PARTIALLY UP/DN
CASK SEAL STATE IND.: DEFLATED
PRESSURE(PSIG): 0.0

INSERT SHIELD DOOR PLUG DN COMMAND EXECUTING: Elapsed Time 4 of 40 Seconds

Figure 3-3. [PREPARE TO LOAD CASK] Automatic Sequence execution display on CRT HMI

The sacrificial plug was then placed into position on the Cradle Lift (Trough). The Mk-18A Cask was then lifted out of the cask frame and placed onto the correct position of the CRT in Room E-079. See Figure 3-4 for a photo of the cask on the CRT. The north end drip pan was then moved to the proper location to capture any water that may drain from the cask during shield plug removal. The rigging was removed from the crane hook and left to rest on top of the cask. The crane was moved back into E-055 and the craneway door, and the E-079 shield doors were closed. The simulated EMITS transaction for internal move was then completed.

Figure 3-4. Photo of Cask on CRT Behind Cell 7

3.1.2 Preparation of Mk-18A Cask for Unloading

RPD continued to perform Radiation, Contamination, and Airborne Radioactivity Monitoring throughout the receipt and staging of the Mk-18A cask to verify the expected conditions. A simulated EMITS transaction for an internal move was initiated. The containment bags installed on each end of the cask while on the trailer were removed. The drain rig hose was routed to a poly bottle and the drain rig valve was opened to allow water to drain to the poly bottle. The Plug Platform (Shelf) on the north end of the Cask, was lowered under the north end Shield Plug by using the Control Pendant. The Shield Plug Holder was un-clamped and moved south on the Plug Platform to line up with the Shield Plug. The 2 bolts securing the North Shield Plug were removed. Then, the Plug Platform was raised/lowered while sliding the Plug Holder to engage the Shield Plug. Once the Plug Holder engaged the Shield Plug, the Plug Holder was pulled back to the center of the Plug Platform with the Shield Plug and clamped down. The Plug Platform was then moved to the STOWED position. The Cradle Lift was then raised to the CHAIN EXTEND position. The Chain Drive was extended until the Sacrificial Plug began to enter the cask. The South End Plug Lift Table was raised to align the Plug Holder under the Shield Plug. As required, the CRT was extended/retracted to get it into position. The South Shield Plug bolts were removed, and the Plug Holder engaged the South Shield Plug. The Plug Holder and Shield Plug were moved to the south end of the Lift Table. The CRT was then retracted to the LOAD POSITION, and the South End Plug Lift Table was lowered to the fully DOWN position and verified using the CRT HMI. The drain rig valves on both ends were verified to be closed and the water will be disposed of per RPD and GCO instructions.

3.1.3 Prepare for Resizing the Mk-18A Target Bundle

In preparation for Target Resizing, The Mk-18A Cutter System was powered on and made ready for operations. Using the Cutter System HMI as shown in Figure 3-5, the Cutter System was confirmed to be in the following configuration:

- Saw Tilt is in the CLEAR position
- OC Clamp is OPEN
- Saw Clamp is OPEN

Once the Cutter System was in the required configuration, the [PREPARE TO CUT ASSEMBLY] button was selected and executed on the CRT HMI. This automated sequence prepares the Mk-18A Assembly for segmentation by the Mk-18A Cutter System. The sequence performed the following actions:

- Moved the Shield Door Plug to the FULLY UP position.
- Ensured the Plug Platform (Shelf) was in the STOWED position.
- Moved the Cradle Lift (Trough) to the CHAIN EXTEND position.
- Moved the Cask Receipt Table to the CELL SEAL position at the front of the CITB.
- Inflated the Cask Seal to a minimum of 2 PSIG.
- Using Chain Drive (Push Plug), pushed the Mk-18A Assembly into the cell but went too far as identified in Table 3-1. The assembly was supposed to pause at the OC CLAMP FRONT position to allow removal of the previous Sacrificial Plug. The bundle was pulled back to the OC FIRST CUT position and operations resumed. Table 3-1 also lists an off normal event that happened during a second demonstration where the sacrificial plug bound up as it began to enter cell 7. After successful realignment using the manipulator, operations proceeded without incident.

During the Prepare To Cut Assembly phase, there were 5 issues that occurred, all of which could be temporarily fixed for the cold run. Table 3-1 shows the issues and planned resolutions.

HMI MODE: OPERATIONS
HMI LOCATION: LOCAL
WAITING ON COMMAND
05:06:52 PM
07/12/2021
MAN OPS 1

HORIZONTAL
POS DESCRIPTION: CUT OUTER CAN
POS INCHES: 0.01
VEL (IN/SEC): 0.00
AXIS 3 (HOR): STOP: STOP CMD... COMPLETE

SAW TILT
POS DESCRIPTION: CLEAR
POS INCHES: 1.25
VEL (IN/SEC): -0.00
AXIS 4 (SAW TILT): STOP: STOP CMD... COMPLETE

SAW FEED
POSITION: FULLY UP
POS INCHES: 0.000
VEL (IN./SEC): 0.0000
AXIS 2 (FEED): STOP: STOP CMD... COMPLETE

SAW CUT
POSITION: READY
VEL (RPS): 0.00
AXIS 1 (CUT): STOP: STOP CMD... COMPLETE

* PRESS THE GOLD SECTION BARS TO GOTO DETAILED SCREEN FOR ITEM

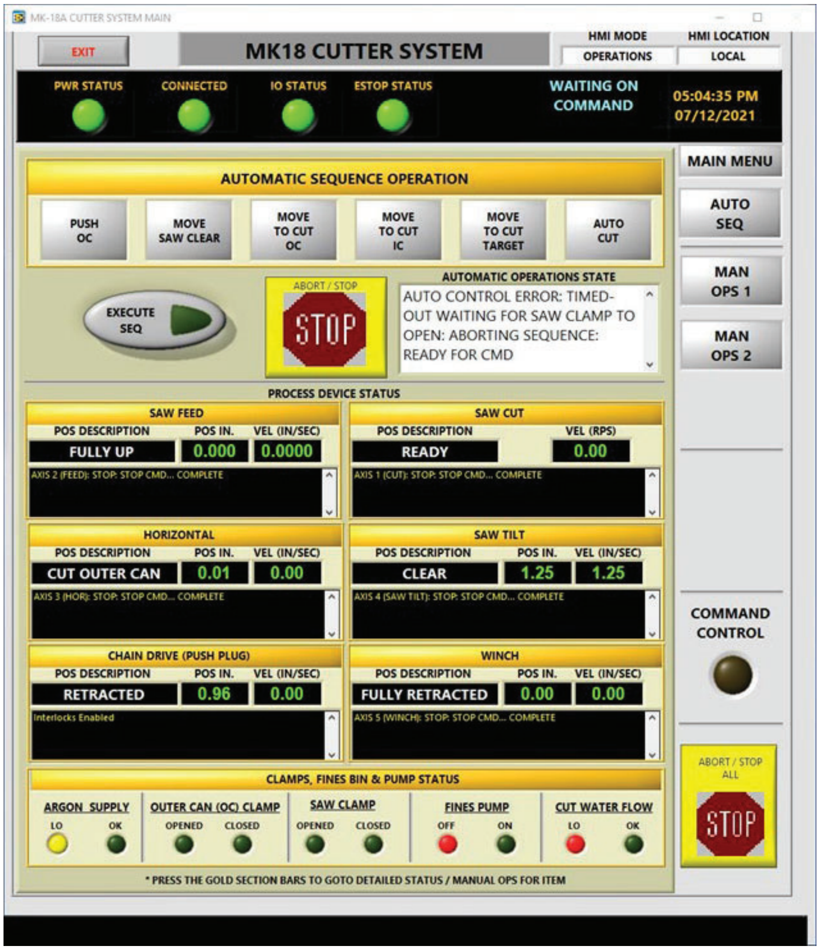
Figure 3-5. Cutter System HMI Display

Table 3-1. Mk-18A Equipment Issues During Prepare to Cut Phase

Issue	Status	Resolution
Containment Bags were very difficult to install/remove from the ends of the cask which increases time in high dose area.	Process was demonstrated successfully but raised ALARA and safety concerns.	Resolved: Use of containment bags has been eliminated. Procedure L30-5.10 has been updated to incorporate this change.
An argon leak in the manifold connected to the CITB leg.	Holds enough pressure to work for the cold run.	Enter E-079 and tighten the leaking item.
The Cask sensor, located on the CITB, is too far from the Cask to get the appropriate distanced reading.	Able to sense the cask for the cold run.	Resolved: Placed a spacer behind the sensor to move it closer to the Cask.
Mk-18A bundle was pushed too far into the cell.	Bundle was pulled back through the cell insert to the OC First Cut position to resume Cold Runs.	The reason this happened was because the bundle length in the software had been changed to accommodate an alignment test bundle that was shorter. The program had not been updated for the Cold Run dummy bundle. The software has been updated with the correct length. This will not happen during real operations as all the Mk-18A Assembly lengths are the same and there will be no changes to the software, without approvals, once all testing and demonstrations are complete and the software is locked down.
During a second demonstration the Sacrificial Plug bound as it entered Cell 7.	Able to use the manipulators in Cell 7 to realign the Sacrificial Plug.	Inspect Sacrificial Plugs and determine if additional deburring/smoothing of edges is needed. To further prevent re-occurrence, the Sacrificial Plug will be pushed further into the cell insert sphincter seal at the end of target resizing. This should center the plug to reduce the chances of binding when the target or dummy bundle pushes it into the cell.

3.1.4 Mk-18A Target Bundle Resizing

Once the Mk18-A Dummy Bundle was moved into position, The Mk-18A Cutter System was utilized to resize the simulated Mk-18A target for further processing. The inner and outer J-Cans were re-sized for disposal utilizing automated sequences initiated using the Cutter System HMI. The Automatic Sequence Operation controls and indicators are shown on the HMI Main window as seen in Figure 3-6.



HMI MODE: OPERATIONS

HMI LOCATION: LOCAL

WAITING ON COMMAND

05:04:35 PM
07/12/2021

AUTOMATIC OPERATIONS STATE

AUTO CONTROL ERROR: TIMED-OUT WAITING FOR SAW CLAMP TO OPEN: ABORTING SEQUENCE: READY FOR CMD

	POS DESCRIPTION	POS IN.	VEL (IN/SEC)	VEL (RPS)	
SAW FEED	FULLY UP	0.000	0.0000		AXIS 2 (FEED): STOP: STOP CMD... COMPLETE
SAW CUT	READY			0.00	AXIS 1 (CUT): STOP: STOP CMD... COMPLETE
HORIZONTAL	CUT OUTER CAN	0.01	0.00		AXIS 3 (HOR): STOP: STOP CMD... COMPLETE
SAW TILT	CLEAR	1.25	1.25		AXIS 4 (SAW TILT): STOP: STOP CMD... COMPLETE
CHAIN DRIVE (PUSH PLUG)	RETRACTED	0.96	0.00		Interlocks Enabled
WINCH	FULLY RETRACTED	0.00	0.00		AXIS 5 (WINCH): STOP: STOP CMD... COMPLETE

* PRESS THE GOLD SECTION BARS TO GOTO DETAILED STATUS / MANUAL OPS FOR ITEM

Figure 3-6. Cutter System HMI Automatic Sequence Operations

The automatic sequences used for Cutter System operations are described below.

- **PUSH OC** – this sequence allows the operator to enter the number of inches to push the Outer Can (OC) into the cell. The sequence performs the following actions:
    - ➢ Commands the CRT System to push the OC further into the cell by the number of inches entered by the operator.
    - ➢ Monitors interlocks to ensure the Cutter System state is OK to push to OC.
- **MOVE SAW CLEAR** – this sequence prepares the Cutter System for pushing the OC into the cell. The sequence performs the following actions:
    - ➢ Ensures the SAW is OFF.
    - ➢ Ensures the SAW FEED is at its FULLY UP position.
    - ➢ Ensures the SAW Clamp is OPEN.
    - ➢ Ensures the SAW is Tilted to its CLEAR position.
- **MOVE TO CUT OC** – this sequence prepares the Cutter System for cutting the Outer Can (OC). The sequence performs the following actions:
    - ➢ Ensures the SAW is OFF.
    - ➢ Ensures the SAW FEED is at its FULLY UP position.
    - ➢ Ensures the SAW Clamp is OPEN.

  - ➢ Ensures the SAW is Tilted to its CLEAR position
  - ➢ Ensures the SAW is moved horizontally to the CUT OUTER CAN position.
- **MOVE TO CUT IC** – this sequence prepares the Cutter System for cutting the Inner Can (IC). The sequence performs the following actions:
  - ➢ Ensures the SAW is OFF.
  - ➢ Ensures the SAW FEED is at its FULLY UP position.
  - ➢ Ensures the SAW Clamp is OPEN.
  - ➢ Ensures the SAW is Tilted to its CLEAR position
  - ➢ Ensures the SAW is moved horizontally to the CUT INNER CAN position.
- **MOVE TO CUT TARGET** – this sequence prepares the Cutter System for cutting the Target. The sequence performs the following actions:
  - ➢ Ensures the SAW is OFF.
  - ➢ Ensures the SAW FEED is at its FULLY UP position.
  - ➢ Ensures the SAW Clamp is OPEN.
  - ➢ Ensures the SAW is Tilted to its CLEAR position
  - ➢ Ensures the SAW is moved horizontally to the CUT TARGET position.
- **AUTO CUT** – this sequence automatically cuts a segment of the Mk-18A Assembly. The steps are calculated based on the current HORIZONTAL position. The sequence performs the following actions:
  - ➢ Ensures the SAW FEED is HOMED and at its FULLY UP position.
  - ➢ Ensures the OC Clamp is CLOSED.
  - ➢ Moves the SAW TILT to the CLAMP position and CLOSES the SAW CLAMP.
  - ➢ Moves the SAW FEED to the CUT START position.
  - ➢ Turns the FINES PUMP and SAW ON.
  - ➢ Moves the SAW FEED slowly down to cut the assembly.
  - ➢ Turns the SAW OFF once the cut is complete.
  - ➢ Returns the SAW FEED to its FULLY UP position.
  - ➢ Opens the OC Clamp.
  - ➢ Moves the SAW TILT to the Clear Position.

Using the sequences as called out by the procedure (L30-5.10), the Mk-18A dummy bundle assembly was resized. During the resizing operation, the auto sequences performed the required actions with only two problems. The first problem occurred during cutting of the IC lid. The IC was not gripped completely and rolled as the saw blade tried to engage. This problem was witnessed during mock-up testing and it is not uncommon to not get a good clamp every time. The lack of a good clamp is enhanced due to the IC just coming out of being submerged in water. When the IC rolls it will back itself off the proper cut location. The operator, with assistance of the Cutter System SME, was able to complete the cut utilizing manual controls available on the Cutter System HMI. To complete the cut the following steps were performed:

- The Auto Cut automated sequence was ABORTED
- The Saw Feed Axis was jogged up.
- The Saw Clamp was opened.
- The IC was repositioned to the proper location using the manipulator.
- The Auto Cut automated sequence was resumed and completed the cut.

The second problem occurred on the last two cuts of the simulated target. During these two cuts, the saw paused cutting near the end of the cut just prior to cutting completely through the target. In both instances, the operator, with the assistance of a Cutter System SME, was able to complete the cuts utilizing manual controls available on the Cutter System HMI. To complete each of these cuts, the following steps were performed:

- The Saw Feed Axis was jogged up.
- The Saw Clamp was opened.
- The Tilt Axis was jogged up to relieve some of the downward force holding the target in place.
- The Saw Clamp was closed.
- The Auto Cut automated sequence was resumed and completed the cut.

The Cutter System SME believes this issue will be resolved by installing a thicker spacer on the anvil type device that is used to secure the target for cutting. The need for the thicker spacer on the anvil is due to the height that the Target Assembly enters the cell. This height is slightly higher than the height the assembly entered the cell in the Mk-18A Mock-up Facility. A thicker spacer has been fabricated and will be installed and tested. In addition, information and notes will be added to the procedure to give the operator flexibility to utilize the HMI controls available to recover from events like the two described above.

3.1.5 Preparing the Empty Mk-18A Cask for Return Transfer

Once the Mk18-A Dummy Bundle was successfully cut up the cask was prepared for return transfer to L-Area. The sequence of events for this evolution is just the reverse process of bringing the cask into the building for processing. The [Prepare To Unload Cask] automatic sequence on the HMI was selected and executed. The following configuration was verified on the CRT and Cell Insert after completion of the automatic sequence:

- Plug Platform (Shelf) is in the STOWED position.
- Cradle Lift (Trough) is in the FULLY DOWN position.
- Cask Table is in the LOAD position.
- Chain Drive (Push Plug) is in the RETRACTED position.
- Shield Door Plug is in the FULLY DN position.
- South End Shield Plug Lift Table is in the DN position.
- Cask Seal is DEFLATED

Once the above items were verified, the South End Shield Plug was installed using the South End Plug Lift Table and manipulating the CRT as needed to engage the Shield Plug. The South End Shield Plug bolts were installed and torqued to 40-45 ft-lbs. Using the North End Plug Platform (shelf) the North End Shield Plug was installed and the bolts torqued to 40-45 ft-lbs. Next, the drain valves on the drain valve assemblies were verified to be closed to prepare to move the cask back to E-055 for placement on the trailer. RPD performed surveys during the reassembly of the cask to ensure it met requirements to move it to E-055.

The E-079 doors were opened, including the overhead crane doors, and the crane was moved into E-079 where the rigging from the cask was attached to the E-055 crane hook. The cask was lifted off the CRT and placed back in the cask frame on the trailer. The cask bag support structure was installed, and the cask bag was zipped up around the cask frame. Final surveys were taken of the cask bag and trailer. The E-055 door was opened, and the truck backed in and connected to the cask trailer. The cask trailer was removed from the building and staged in the SRNL limited area. No issues were identified during the cask reassembly and trailer loading evolutions.

3.1.6 Fines/Chip Collection and Removal

Once the Mk18-A Cask was removed from the building, the fines/chips generated during the cutting evolutions were vacuumed up into the hydrocyclone. The fines/chips are swept and/or moved using a water stream into the suction hole in the bottom of the catch pan which pulls them into a hydrocyclone to be captured for eventual transfer to the caustic dissolver. The fines were dumped from the hydrocyclone into a fines holder tube. The fines holder tube was simulated to be dumped into the Caustic Dissolver. This portion of the process was simulated because the Caustic Dissolver was not prepared with the proper chemistry to dissolve the fines and, therefore, would cause issues of the fines hardening up and clogging lines. The excess water in the catch pan was transferred to the HAD tank in Cell 8.

During the vacuuming operations, two issues were identified:

1) The water line to the saw came loose during cutting evolutions. The cutting evolution was stopped, and the hose temporarily reattached to continue Cold Runs. Once Cold Runs were completed, the fittings on the hose were tightened to ensure a good connection.
2) Low circulation flow was noticed during the vacuuming evolution. Investigation revealed that the suction line from the catch pan to the hydrocyclone had a kink in it which was allowing fines to build up and clog the line. The line was disconnected and cleared out which allowed Cold Runs to continue. However, the kink could not be removed from the line. Once Cold Runs were completed, the entire line and pump unit was replaced to eliminate the kink in the hose.

3.2 Nitric Acid Transfer into TK-107

The Mk-18A acid drum was transferred to the Engineering Development Laboratory (EDL) where the water heel was drained, and the drum was dried. Approximately 57 L of concentrated (67.53 wt. %) nitric acid was transferred into the drum. The filled drum was sealed and then transferred back to the Shielded Cells by forklift using the custom designed secondary containment vessel. Initial attempts to transfer acid into TK-107 were unsuccessful and the drum was transferred back to EDL for inspection. At EDL, it was discovered that the transfer line and vent connections had been inadvertently swapped. The connections were color-coded to avoid repeating this mistake in the future.

After changing the connections, approximately 52.5 L of acid were transferred into TK-107 with no issues. A water heel of approximately 3.3 L in TK-107 (based on Mark-18A LabView Level Elements) diluted the acid to near 64.45 wt. %, which corresponds to 14.21 M HNO_3. Since a mixer is not present in TK-107, the homogeneity of the water acid mixture at this point was unknown. It is possible that a lower density water layer was present on the top of the concentrated acid. Hopefully, transferring the large volume of nitric acid into the small volume of water in the tank resulted in some degree of mixing. Complete mixing of the solutions was assumed for all subsequent calculations. The concentrated nitric acid in TK-107 was used to prepare more dilute nitric acid reagents for use in the ion exchange process in tanks TK-106, TK-108, and TK-109, as discussed below. The assumed density of the concentrated acid in TK-107 was 1.458 kg/L.

3.3 Caustic Dissolution

3.3.1 Cold Chemical Preparation – Preparation of 2.19 M Sodium Nitrate

Sodium nitrate, as received in 2.5 kg containers, was found to be solidified and was unable to be easily transferred directly from the manufacturers bottle. Therefore, the 20.0 kg of $NaNO_3$ were prepared in bottles for Shielded Cell Operations (SCO) personnel to transfer the reagent into the cells. The process of breaking up the $NaNO_3$ and transferring it to smaller containers was cumbersome, resulting in 14x 1-L bottles and 2x 0.5-L bottles of $NaNO_3$ being prepared. Upon cell-transfer, per L33-0252, Section 5.1.2, and supplemental R&D directions, the $NaNO_3$ was placed in B-Block Shielded Cell 8.

The pre-job briefing for the cold-chemical preparations began on February 1, 2024, at 0900 hrs. The activity started at 0938 hours. The activity was initiated by Operations removal of PW system isolation from the Shielded Cells per UET procedure, TO-13-003.[5] Using the PW source, TK-101 water addition was initiated at ~ 4 L per minute per FE-020. Upon initiation of flow, the receipt of water was confirmed to TK-101 per LE-020. LE-020 increased to 80.55 L, and the PW addition was stopped with closure of solenoid valve, V-500, per R&D directions.

At a level reading of 80.30 L, per LE-020, the $NaNO_3$ solid reagent was added to TK-101 via a wide-mouth funnel. All 20.0 kg, per vendor weight, was transferred successfully from the 16x bottles. After addition of the $NaNO_3$, TK-101 mixer, MIX-500, was set to 150 rpm and turned on. Then the process water addition was restarted by opening V-500 per R&D directions, as well as other points of isolation. At 1136 hours, the LabView user interface communicated an error message and, by default, the LabView system shutdown operation of miscellaneous components. In this case, the TK-101 mixer, MIX-500, was the only component to be de-energized.

At 1146 hours, the PW addition was completed for a $NaNO_3$ Tank TK-101, level reading of 107.33 L, per LE-020. The final target of the addition was 107.4 L, reading was just shy of the target and likely within uncertainty. Per estimation, the resulting molarity of $NaNO_3$ in TK-101 is 2.19 M despite the small variation in volume.

3.3.2 Cold Chemical Preparation – Preparation of 10 M Sodium Hydroxide

Similar preparations were made for sodium hydroxide pellets (NaOH). 13x 1-L bottles of NaOH were prepared from the three 5-kg bottles as received vendor containers on January 30th, prior to Cold-Runs. It was noted during transfer outside of the cells, that transfer needed to be completed quickly due to the hydroscopic nature of the NaOH pellets which did begin to become damp after short exposures to the atmosphere. Upon cell-transfer, per L33-0252, Section 5.1.1, and supplemental R&D directions, the NaOH was placed in the B-Block Shielded Cell 7.

Caustic cold chemical preparation began on February 1, 2024, at 1148 hours. TK-102 water addition was initiated at ~ 4 liters per minute per FE-020. Upon initiation of flow, the receipt of water was confirmed to TK-102 per LE-030. LE-030 increased to 28.0 L and the PW addition was stopped with closure of solenoid valve, V-500, per R&D directions.

The NaOH was intended to be added per wide-mouth funnel to TK-102 in Shielded Cell-7. However, unlike TK-101, a cap was installed, obstructing the port for solid chemical additions to TK-102. The PI and SME were not aware of the modification, which was unobserved (and likely not present) in two prior Pre-Acceptance-Tests, prior to transport of the Mark-18A equipment to the technical area.[6] Prior to discovery of the condition, NaOH was added to the funnel. The systems were placed in a safe state, isolated, and planning was executed to ensure mitigation of the condition. Following the planning discussions, SCO personnel were able to return a minimum of 80% of the NaOH to the original bottle, although some fell to the containment of Cell 7. Later, troubleshooting and corrective maintenance on multiple systems discovered that the TK-102, MIX-510 motor casing was glazed with the residues of the NaOH pellets.

Following the discovery, the cap was opened, and the ~ 14.7 kg, 13x 1-L bottles were transferred to TK-102. After addition, TK-102 mixer, MIX-510, was set to 150 rpm and turned on. Then the PW addition was restarted by opening V-500 per R&D directions, as well as other points of isolation. At 1226, an error message was received, similar to the error message at 1136 hours. The error message resulted in both MIX-500 and MIX-510 not operating. Both mixers were restored to operating condition at 1320 hours.

At 1252 hours the PW addition was completed for a NaOH Tank, TK-102, level reading of 36.7 L, per LE-030. The final target of the addition was 36.8 L; so the reading was just shy of the target but likely within uncertainty. The final concentration per preliminary calculation was 10.0 M NaOH.

Mixing of TK-101 and TK-102 progressed for a duration greater than 2-hours from chemical additions. The mixing continued for both cold-chemical tanks until 1634 hours at a speed of 150 rpm. Intermittent breaks in operation occurred due to LabView functionality.

3.3.3 Caustic Dissolution

Caustic Dissolution of the Mark-18A cold quarter-target, an aluminum tube, was initiated at 1535 hours on February 1, 2024, following a pre-job briefing with SCOs. The Caustic Dissolution was performed per Manual L33, Procedure 0252, Rev. 0, Section 5.2.1, and supplemental R&D directions.

Caustic dissolution was initiated by transferring 2.19 M $NaNO_3$ from TK-101 into TK-215, the Caustic Dissolver. Transfer of 2.19 M $NaNO_3$ from TK-101 to TK-215 was initiated at 1608 hours, progressing at 300 $mL \cdot min^{-1}$, per FE-040. Motive force was provided by P-020. The cold quarter-target in the basket was lowered into TK-215 with $NaNO_3$ addition in progress. See Figure 3-7 for a photo of the simulated quarter target (aluminum tube) being placed into the basket prior to being lowered into TK-215. At 1632 hours, the TK-215 level indicator, LE-040, read 7.7 L, the PE-020 flow totalizer read 8.5 L in (relative) agreement with the TK-101 LE-020 reading of 98.9 L. Upon completion of the transfer, all components were de-energized, shut down, and the valves were closed, including the PW system isolation, until the restart of Caustic Dissolution on February 2, 2024.

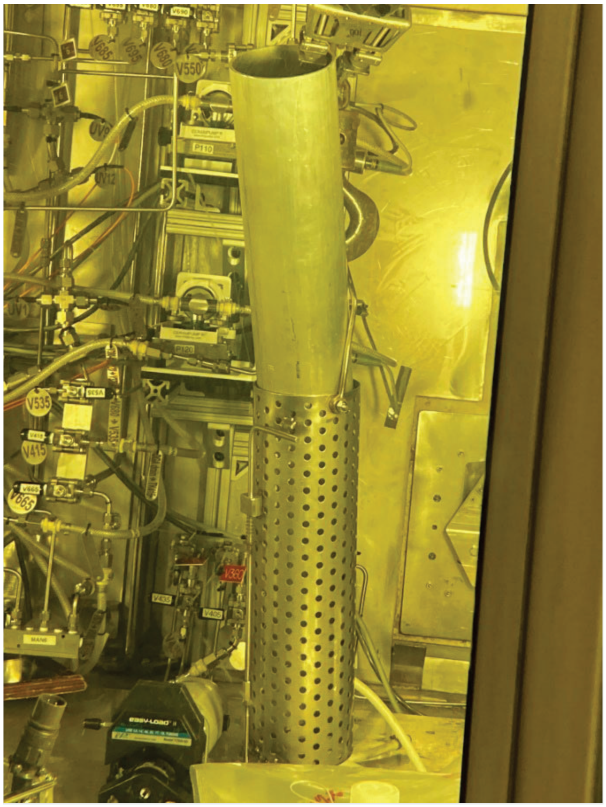


Figure 3-7. Surrogate quarter target (aluminum tube) being lowered into the basket for placement into the Caustic Dissolver (TK-215).

At 0700 hours on February 2, 2024, a pre-job briefing was completed, and at 0709 hours the transfer of $NaNO_3$ from TK-101 to TK-215 via P-020 was continued. At 0725 hours, the P-020 flow rate was increased from 300 $mL \cdot min^{-1}$ to 1500 $mL \cdot min^{-1}$ within performance tolerances. At 0739 hours, the transfer of 24.0 L of 2.19 M $NaNO_3$ was complete.

Following the $NaNO_3$ addition, aluminum fines,[b] chemical surrogates, and silicate were added to the TK-215 caustic dissolver to mimic insoluble target material. 31.7 g of aluminum fines were added to provide a total aluminum mass of 1185.6 g (i.e., including the Al target). The surrogates were neodymium oxide, Nd_2O_3 (surrogate for Am^{3+} expected to adsorb to DGA resin), and cerium oxide, CeO_2 (surrogate for Pu^{4+}, though adsorption to Reillex resin was not expected) added at 8.89 g and 4.19 g, respectively. The silica species added was sodium metasilicate nonahydrate, $Na_2SiO_3 \cdot 9H_2O$. Sodium metasilicate nonahydrate was added at 210.3 g. Masses of surrogates added to TK-215 are summarized in Table 3-2.

Table 3-2. TK-215 Solids Added – Aluminum Fines, Surrogates, and Silicate

Species	Formula	Mass	Molar Mass (g/mole)	Moles (mole)
Tubing & Fines				
Aluminum (fines)	Al(s)	31.7	26.98	1.17
Aluminum (tube)	Al(s)	1153.9	26.98	42.77
Aluminum (total)	Al(s)	1185.6	26.98	43.94
Surrogates				
Neodymium oxide	Nd_2O_3	8.89	336.48	2.64E-02
Cerium oxide	CeO_2	4.19	172.115	2.43E-02
Silicates				
Sodium Metasilicate Nonahydrate	$Na_2SiO_3 \cdot 9H_2O$	210.3	284.19	0.7400

Note: Solids added to mimic solids in radiological processing to extent practical.

The 43.94 moles of Al requires the addition of ~ 6.6 L of 10 M NaOH (i.e., 65.91 moles) to provide 1:1 stoichiometric equivalence for dissolution and a 50 percent buffer, based on similar Al-tubing dissolution.[7] Similarly, past testing dissolved 16” Al-tubing ranging from 1165 g to 1190 g (with an endcap).[7] The first of two 10 M NaOH additions was initiated at 200 $mL \cdot min^{-1}$, from TK-102 to TK-215 per P-030. P-030 was de-energized at 0851 hours and TE-120, the Caustic Dissolver Tank temperature element (TE), indicated exothermic reaction with the aluminum, as expected, per a significant increase in TK-215 bulk solution temperature. The bubbler was turned on to agitate the bulk solution following the addition with a flow of 5 scfh per FE-240. A volume of 1.1 L of 10 M NaOH was transferred from TK-102 by P-030 to TK-215. As previously stated, this was the first of two NaOH transfers. The second occurred upon increasing temperature of the TK-215 solution.

At 0908 hours, heating elements were energized at 100% power with a target/setpoint of 75 °C for TK-215 TE-120 (the bulk solution temperature of TK-215). However, upon command of P-060 to on, with intent to cool off-gas contents, a small decrease in TK-215 bulk solution temperature was observed. Furthermore, the solution approached 72 °C, but did not reach the setpoint despite feedback from TE-120. The temperature of cooling water in TK-123 was observed to be rising above the initial temperature, and it was concluded that the cooing water was not functional. Conclusion resulted in de-energizing P-060 and de-energizing the fan to test presumed conditions. Soon after, TE-120 approached the setpoint of 75 °C. Operations were stopped and the system placed in a safe state.

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[b] The caustic dissolution was performed prior to the target receipt and cutting described in Section 3.1, therefore, fines that were added here were obtained from previous cutting demonstrations performed in the mock-up facility.

It was determined that both chilled process water transfer paths to TK-215 from TK-123, the Chilled Process Water Tank, were misaligned. The issue was resolved by swapping the discharge lines on P-050 and P-060 to restore the system configuration to the design. This was completed by close of business.

On February 5, 2024, at 0754 hours, a pre-job briefing was complete, and operation of the Caustic Dissolver resumed. By 0758 hours, the chilling water system was cooling, iteratively, the TK-215 argon sparger flow was 5 scfh, and the temperature was increasing to the set point of 75 °C. Between 0807 hrs and 0838 hours, temperature of the TK-215 bulk liquid temperature, per TE-120, had increased from 14.8 °C to 59.3 °C at a rate of 1.48 $°C \cdot min^{-1}$ with the heater power at 100%.

From the initiation of the Chilled Water System operation, LE-050 of the TK-123 (Chilled Water Tank) was found to be inoperable. Chilled Water flow was confirmed by FE-100 and checked by redundant temperature changes during processing. By the end of Caustic Dissolution, LE-050 returned to an accurate indication of liquid level.

Heating continued, but heater power decreased to 71.3% at 73 °C, and to 30% at 74.7 °C to maintain steady-state near the 75 °C setpoint, at 0859 hours. The temperature continued to rise to 75 °C at 23.2% power, due to transient thermal lag, at 0920 hours.

At 0921 hours, the LabView software failed again resulting in shutdown of the operating components. The LabView program was reset by the SME but failed to resolve MIX-510 inoperability, foreshadowing the discovery of the NaOH on MIX-510 discussed earlier in Section 3.3.2.

At 1101 hours, feed of the second 5.5 L volume of 10 M NaOH from TK-102 to TK-215 was initiated at 22 $mL \cdot min^{-1}$. The selected rate of addition was based on past observations of the exothermic aluminum cladding dissolution.[7] Expectedly, the TK-215 heater decreased in power output and at 1158 hours de-energized (cycled), indicating the exothermic reaction had been initiated and was increasing to a desirable reaction rate. The temperature of the TK-215 bulk liquid was controlled at 90.1 °C and 1.266 L of the 5.5 L transfer volume of 10 M NaOH had been added to TK-215 per the P-030 flow totalizer. Figure 3-8 illustrates the level indications of the Caustic Dissolver (TK-215) and NaOH reagent tank (TK-102) across caustic dissolution and filtration on the primary axis. The secondary axis of Figure 3-8 provides the temperature reading for TE-120, the bulk liquid temperature of the Caustic Dissolver.

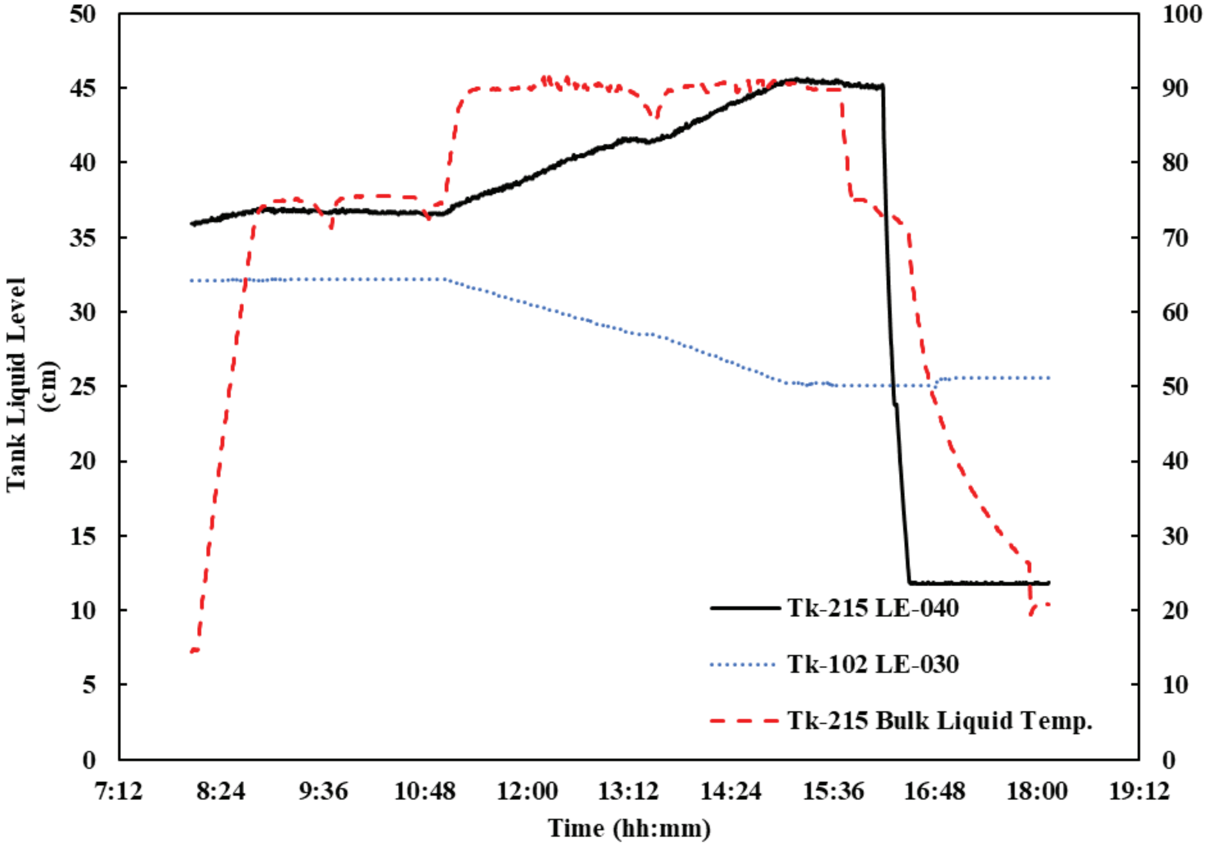

Figure 3-8. TK-215, Caustic Dissolver and TK-102 NaOH Tank Level Indication (cm) and Caustic Dissolver Bulk Temperature (°C) versus time.

At 1209 hours, TE-120 reached a temperature of 90.7 °C energizing P-050 to cool the TK-215 bulk liquid with TK-123 chilled water. Cooling again was observed to be sufficient returning the temperature reading of TE-120 to 90.3 °C by 1212 hours. Figure 3-9 provides the temperature profile of the Caustic Dissolver Tank Bulk Liquid Temperature per indication of TE-120 on the primary axis (right hand side). Figure 3-9 provides the normalized power of P-050 (the chilled water pump) which actively cools the Caustic Dissolver contents and the EHTR-010, EHTR-020, and EHTR-030 which collectively heat the Caustic Dissolver contents on the secondary axis (left hand side).

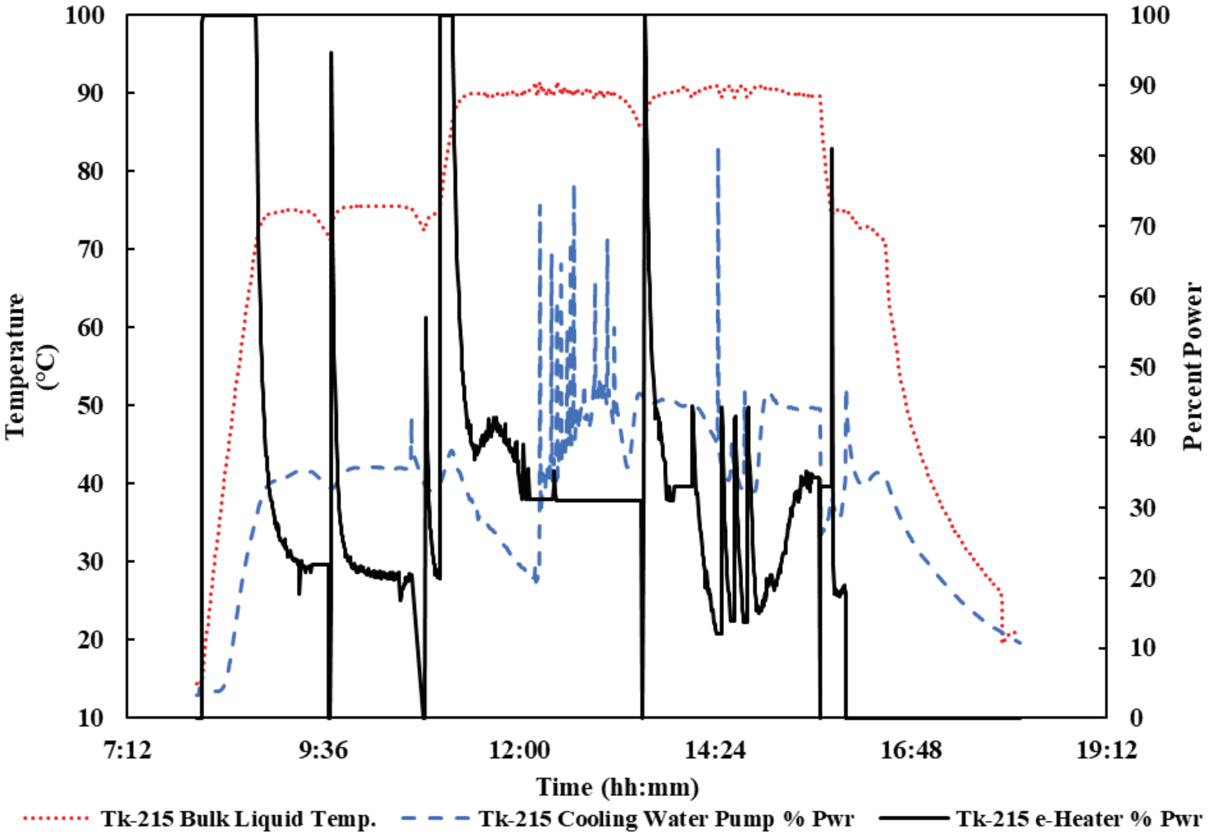

Figure 3-9. Temperature of Caustic Dissolver, Heating, and Cooling through Cladding Dissolution and Filtration

At 90 °C, the LabView program tripped off, under the similar error noted previously causing components to de-energize. The cooling was interrupted during the aluminum dissolution and under the conditions of the Cold Run, the exothermic reaction was insufficient to exceed the 90 °C threshold. However, the cooling system, LabView software, was required to be restarted promptly due to inadequacies. Under similar conditions, 90 °C may be exceeded despite SME or operator action.

Across the caustic dissolution, the cooling system demonstrated adequate capacity to control the temperature of the aluminum dissolution. P-050, which cools the bulk fluid of TK-215, never exceeded 50% of cooling capacity and the thermal reservoir, TK-123, bulk liquid temperature reached an observed maximum of 35.7 °C. This conclusion is limited to 10 M caustic addition rates of 22 $mL\cdot min^{-1}$ to 28 $mL\cdot min^{-1}$. Figure 3-10 provides the temperature of the bulk liquid of the Caustic Dissolver per indication of TE-120 and the temperatures of various thermal reservoirs from the Chilled Water system to ambient air, on the primary axis. On the secondary axis of Figure 3-10, on the left-hand side, the volumetric flow rate, indicated by FE-100, is provided.

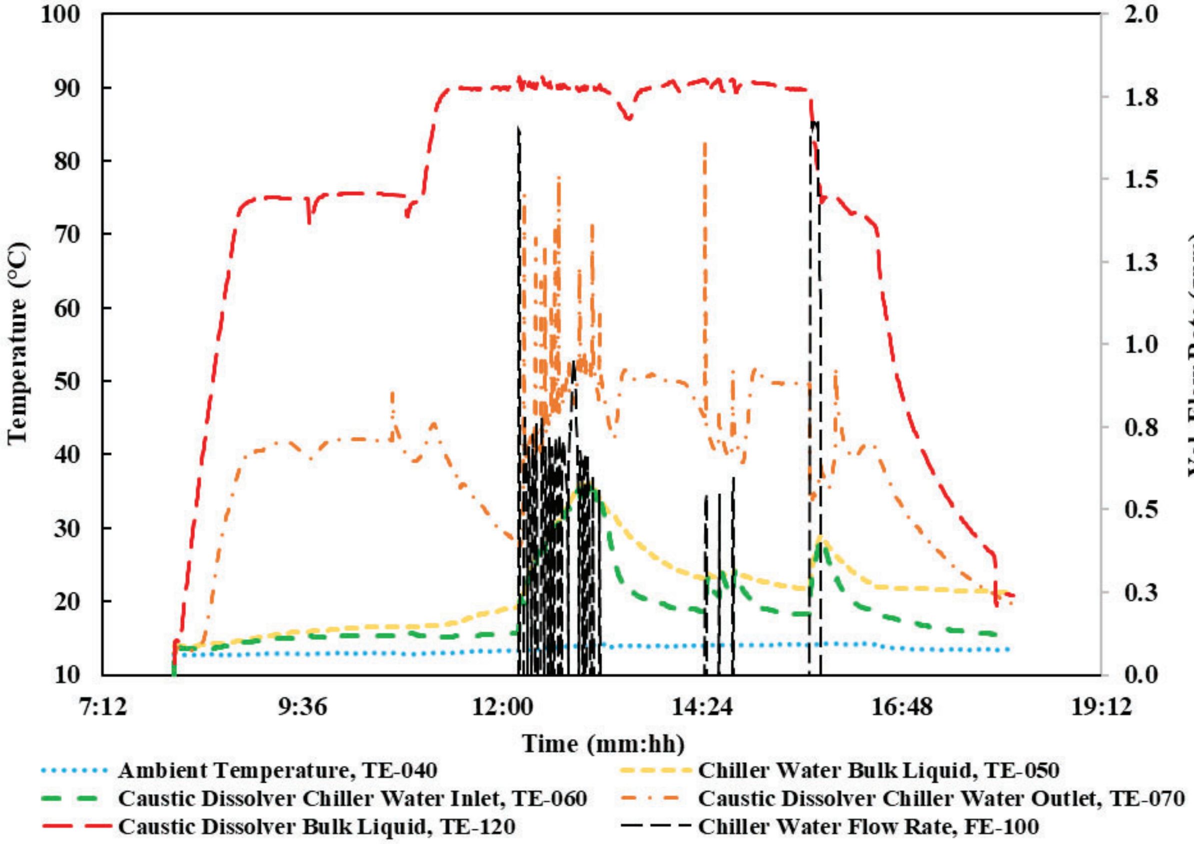

Figure 3-10. Bulk Temperature of Caustic Dissolver, Bulk Temperature of Thermal Reservoirs of Chilled Water System, and Active Cooling of Chilled Water per P-050 to Caustic Dissolver by FE-100

At 1505 hours, the flow totalizer of P-030 reached 5.5 L. TK-215 LE-040, TK-102 LE-030, and the P-030 flow totalizer were not in agreement with the volume transferred. Due to the discrepancy, an excess of 1.10 L of 10 M NaOH, may have been added. The excess was added for assurance of complete aluminum dissolution. Similar discrepancies between flow totalizer and level indications occurred with TK-101 level elements and P-020 flow totalizers. Checks are recommended to ensure agreement between flow totalizer indication (i.e. the volume transferred), receipt vessel level indication (to determine the change in level), and sending vessel level indication (to determine the decrease in level). In doing so, loss of (uncredited) containment may be confirmed for future operations. All three instruments, when providing indication, calibrated to be in agreement (i.e., within +/- 10%) is also recommended because radar level instrumentation has been integrated into the design, which fails to provide level indication at low liquid levels within TK-215, the Caustic Dissolver. The Caustic Dissolver, TK-215, provided a reading initially at 8.5 L of liquid, with inconsistent signal.

For confirmation of complete aluminum dissolution, an additional 0.05 L (50 mL) of 10 M NaOH was transferred at 28 $mL\cdot min^{-1}$, without an observed exothermic response from the temperature readings of the TK-215 bulk solution temperature element, TE-120. Following completion of successor evolutions, the Caustic Dissolver was inspected for residual target, undissolved aluminum tube, or any sign of suboptimal dissolution. Later, visual inspection confirmed near-complete dissolution. Minimal undissolved material remained in the Caustic Dissolver, TK-215. The undissolved material was two, thin, white-flakes no greater than 0.5 inches in diameter. Figure 3-11, illustrates similar undissolved material[c] from prior testing of the caustic dissolution processing, but in much larger quantity compared to the Mark-18A Caustic Cold-Run Dissolution.[7]

At 1538 hours, indications demonstrated complete dissolution of the aluminum cladding. The hydroxide was in excess due to no further reaction observed from addition of 10 M NaOH, and the TK-215 heater had returned to providing nearly the same power as before the exothermic reaction had been initiated by NaOH addition. Increase in ambient air temperature around the cell was providing some additional heat flux into TK-215 bulk liquid. Considering process conditions, the TK-215 heater setpoint was reduced to 75 °C and Caustic Dissolution was complete. A pre-job briefing for the Caustic Filtration and Back-pulse was completed to refresh personnel on the upcoming evolutions.

Figure 3-11. Similar Solids from Prior Caustic Dissolution of Aluminum Cladding – Test 6 of SRNL-STI-2020-00264.[7]

3.3.4 Caustic Filtration

At 1547 hours, Mark-18A Caustic Cold-Runs initiated Manual L33, Procedure 0252, Rev. 0, Section 5.2.2, “Sludge Removal,” and supplemental R&D directions which transfer the TK-215 contents, through the caustic filter, FLT-305.[8] Caustic Filtration is summarized by FLT-305 separating the liquid and the undissolved material mechanically from the product of Caustic Dissolution. The liquid, or effluent, contains the dissolved aluminum in the form of the anions. Under basic conditions, a majority of these anions exist as aluminate, $Al(OH)^-_4$. The effluent is routed to two of three HAD Collection Tanks, TK-103B or TK-103C. The undissolved materials are retained on FLT-305 for caustic back-pulse operations.[9, 10] A simplified pathway for the formation of the aluminate anion is below.[9] The first reaction provides the empirical reaction for aluminum dissolution.[10,11]

$$Al + 0.63 \cdot NaOH + 0.38 \cdot NaNO_3 + 0.26 \cdot H_2O \leftrightarrow NaAlO_2 + 0.37 \cdot NH_3 + 0.01 \cdot NaNO_2 + 0.02 \cdot H_2$$

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[c] Only two flakes, similar to the flake circled in red remained from the caustic dissolution executed here-in. The purpose of Figure 3-11 is to illustrate the properties of the material.

$$NaAlO_2 + 2H_2O \leftrightarrow NaOH + Al(OH)_3$$
$$Al(OH)_3 + Na^+ + OH^- \leftrightarrow Al(OH)_4^- + Na^+$$

SCO initiated removal of isolation from the PW system to the shielded cells per TO-13-003.[5] Once isolation was removed, the transfer path was aligned, per L33-0252, Section 5.2.2 and associated R&D directions.[8] The transfer path for Caustic Filtration is described below:

1. P-040 provides suction from the base of TK-215, and flow is directed from the base of TK-215 to 3-way valve V-100.
2. 3-way valve V-100 is aligned to direct flow from the base of TK-215 through P-040 to 3-way valve V-130. This isolates the TK-215 from the process water manifold.
3. 3-way valve V-130 is aligned to FLT-305, the caustic dissolution filter, and directs flow through V-130 to FLT-305.
4. FLT-305 redirects the liquid to V-120, aligned to one of two HAD Collection Tanks, TK-103B or TK-103C. FLT-305 is single-valve isolated from the unintended HAD Collection Tank, TK-121, the Back-Pulse Tank, and TK-235, the Acid Dissolver Tank, by V-120, V-140, and V-125, respectively. Double-valve isolation is applied, where possible.
5. The liquid continues from V-120 and is received by either HAD Collection TK-103B or TK-103C.

For Mark-18A Cold-Runs, both transfer paths were utilized to test the containment and instrumentation for each HAD Collection Tank. TK-103B received 16.2 L of FLT-305 effluent, or filtrate. TK-103C received the remaining 15.8 L of filtrate. The LE-040 for TK-215 agreed with the LE-090 of TK-103B, as 16.2 L were received per indication and TK-215 decreased from 32.0 L to 15.8 L. LE-040 and LE-110 did not fully agree as LE-110 started at 1.8 cm. This level is near the bottom of the tank where uncertainty in the measurement is high. Some heel of water was expected in the tanks from the water runs, but the volume was not known with accuracy. Upon PE-020 indicating significant decrease in back pressure from FLT-305, LE-110 indicated a level of 21.9 L, having received 15.8 L from TK-215.

During filtration, pressures were close to the design limits. Solids obstructing the filter resulted in PE-020 reading no greater than 95 psig but did approach the limit with readings of ~ 93 psig. The filter design pressure is 100 psig. During filtration, monitoring of the back pressure per indication from PE-020 is necessary to not exceed design specifications.

After the contents of TK-215 were filtered through FLT-305 and received by HAD Collection Tanks B and C, flushes were executed to further dilute and potentially filter the residual contents of TK-215. PW flushes also serve to protect the transfer path and components. A minimum of 2 L of PW was used in this flush. The initial flush was completed at 1652 hours. The second flush was completed shortly thereafter. The flush and valve alignment checklist completed the Caustic Filtration evolution, per Manual L33, Procedure 0252, Rev. 0 and supplemental R&D directions at 1704 hours.[8]

3.3.5 Back-pulse Evolutions

Shortly after completion of the Caustic Filtration evolution, the Caustic Back-Pulse was initiated per Manual L33, Procedure 0252, Rev. 0, Section 5.2.3, "Regular Back-Pulsing." with supplemental R&D directions to the Technical Reference Procedure.[8] Acid Back-Pulse was performed in a similar manner, except the acid concentration was raised to 4 M nitric acid within the back-pulse tank, with additions from the Nitric Acid Tank, TK-107. The single Acid Back-Pulse was executed per Manual L33, Procedure 0252, Rev. 0 Section 5.2.4, "Acidic Back-Pulse," with supplemental R&D directions.[8] Back-Pulses were initiated by filling TK-121 with a volume of PW. PW system interface, the source of PW to the B-Wing shielded cells, remained open to facilitate the prior Caustic Filtration evolution, more specifically, flushes.

PW flows from the PW source of the SRNL technical area, through FE-020, through manifold-002 (MAN-002), to TK-121. The flow path includes miscellaneous isolation, solenoid, and directional flow control valves, components of the Mark-18A caustic system. After the water addition, concentrated caustic or nitric acid was added to TK-121 to achieve the desired concentration of solution. Four back-pulses were performed. The first two and final back-pulses were performed with 0.1 M NaOH prepared by syringe of 50 wt% NaOH through V-110 to PW in TK-121. The third back-pulse was performed as an acidic back-pulse with 4 M HNO_3, composed in TK-121 by transfer of concentrated nitric acid from TK-107 to TK-121, post PW addition.

Table 3-3. Summary of Back-Pulse Evolutions

No.	Time Complete (hhmm)	TK-121 Volume	Soln	TK-235 Volume Received
1st	1734	1.2 L	0.1 M NaOH	2.4 L
2nd	1807	1.2 L	0.1 M NaOH	1.2 L
3rd	1054	1.0 L	4 M HNO_3	1.2 L
4th	1202	1.2 L	0.1 M NaOH	2.2 L

With TK-121 containing a flush solution, TK-121 valve alignment is needed to be aligned to build pressure to ~ 95 psig from the high-pressure Mark-18A argon system. The argon is pressurized to TK-121 from an argon cylinder, to V-490, through PCV-210, V-160, and into TK-121, the Back-Pulse Tank for FLT-305. Pressure is isolated upstream of TK-121 by closing V-140, until reaching, but not exceeding, the desired back-pulse pressure level of 90 – 95 psig. Upon reaching a minimum of 90 psig, V-140 is opened, and the argon pressure drives the TK-121 and FLT-305 (or filtered) solids from FLT-305 to TK-235, the acid dissolver.

One key area for improvement was observed upon adding the NaOH to TK-121 for the second time, following the first back-pulse. When V-110 was opened, TK-121 had residual pressure source, much less than 95 psig, resulting in minute mist of TK-121 contents from the orifice of V-110. The mist was brief and a result of V-140 being closed prior to V-160 permitting residual pressure in TK-121. This was corrected in the 4th back-pulse. R&D directions will emphasize and direct the SCO to perform the closure of V-160 as a predecessor to closure of V-140. Performing the action in this sequence requires isolation of high pressure argon from Tk-121, before the liquid transfer path is isolated. This will result in Tk-121 being at atmospheric pressure prior to opening of V-110 for the next backpulse evolution.

Completion of the 2nd back-pulse at 1807 hours resulted in the final operation for the Mark-18A Caustic system on February 5. All systems were placed in a safe state, and the PW system was isolated from shielded cells per TO-13-003.[5]

Back-pulses resumed on the morning of February 6. However, the acid-back pulse would be delayed due to the LE-060 of TK-107 not providing a reading after prior indications exceeded 56 L. Immediately, researchers confirmed the level in TK-107, safe conditions, negated siphon potentials, and no leaks. The acid back-pulse was delayed for troubleshooting, inspection, and corrective maintenance on the LE-060. Upon inspection, the LE-060 surface had been corroded to a black layer. Furthermore, the memory issue with the LabView instrumentation continued throughout February 6th. This LabView memory issue was frequently observed.

LE-060 was restored to operable upon rinsing the corrosive residue from the instrument. The reading returned to the initial value following the external cell acid transfer. The life of the LE-060 was dramatically shortened by the corrosion of the instrument.

Following resolution of the LE-060 issue, SCO removed isolation of PW to the B-Wing Shielded Cells per TO-13-003, and 0.8 L of water was added to TK-121. 205 mL of 14.2 M HNO_3 was transferred to TK-121 increasing the level to 1.0 L per LE-070. TK-121 was pressurized and the back-pulse was completed at 1054 hours. 1.2 L of material was pulsed to TK-235 indicating 0.2 L of solids.

The 4th (and final) 0.1 M NaOH back-pulse followed the acid-back pulse to not leave 4 M HNO_3 within the respective transfer paths or components. 1.2 L of 0.1 M NaOH was back-pulsed from TK-121 and, unexpectedly, 2.2 L of material was received into TK-235 per LE-070. This higher than anticipated volume increased TK-235 total level to 7.0 L per LE-070. The 4th back-pulse was completed at 1202 hours.

The final evolution of the back-pulses, a flush of FLT-305 was completed at 1213 hours. This resulted in an observed reading of 70-77 psig across FLT-305, which was a reduced pressure drop compared to prior flushes. At 1222 hours Mark-18A Caustic System Cold-Runs were complete, with argon, process air, and Mark-18A systems being isolated and secured for Acid Dissolution. Furthermore, the limited volume of the Acid Dissolver and need to reach an acid concentration of 7-8 M for dissolution of target solids required the volume to be allocated to acid addition.

Figure 3-12 provides the associated pressure readings for the filtration of the Caustic Dissolver contents, at transfer and the back-pressure from flushes following caustic back-pulses from TK-121 to the Acid Dissolver, TK-235.

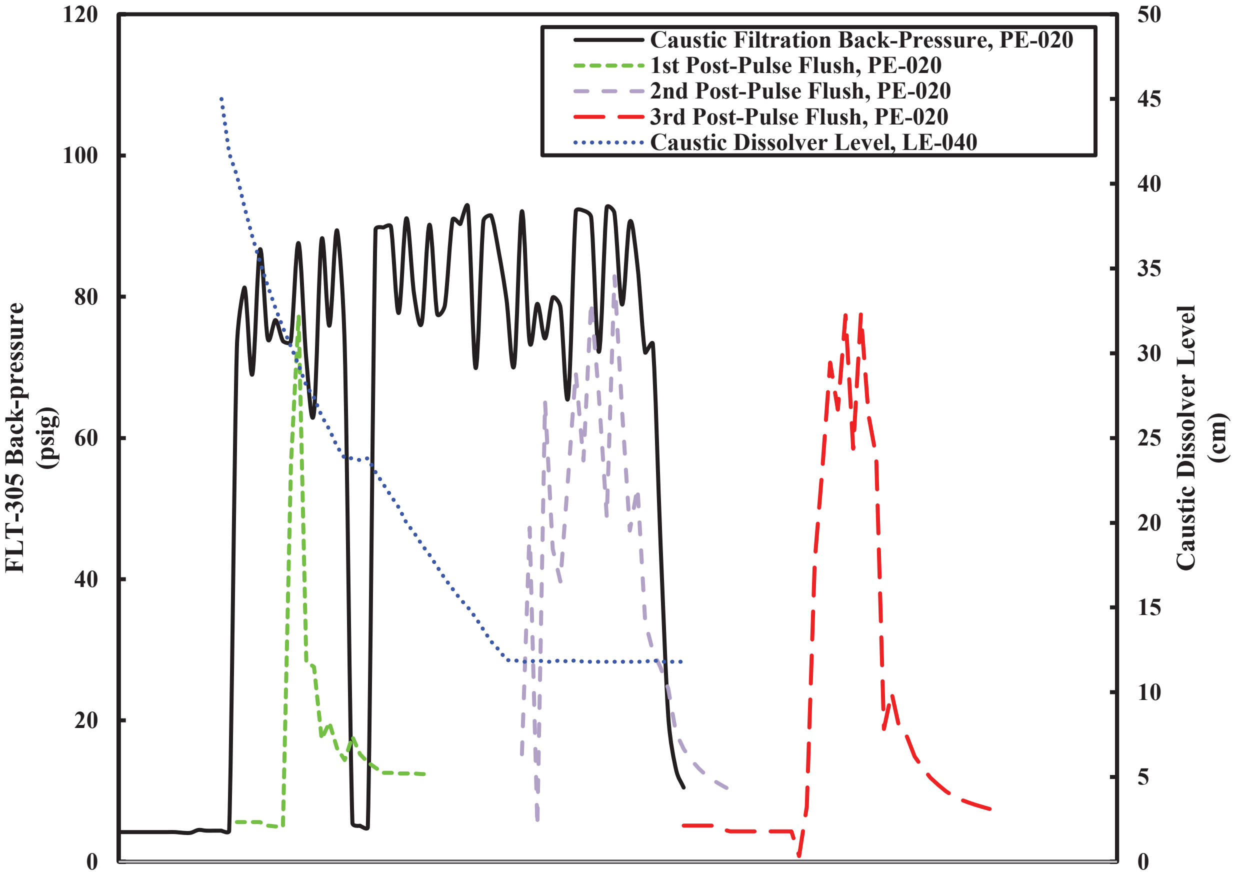

Figure 3-12: Comparison of Back-Pressure Readings from FLT-305 PE-020 following Caustic Filtration and Caustic Back-Pulses

3.3.6 Caustic System Samples - Analytical Results

Samples were pulled from TK-215(Caustic Dissolver), TK-103B (HAD Tank B), and TK-103C (HAD Tank C). Samples pulled are listed below in Table 3-4.

Table 3-4 Samples of Caustic Dissolution, Filtration, and Back-pulse Evolutions

Analytical Development Sample No.	Process Vessel	Method(s)	Temperature per TE-120 (°C)	Notes
LW34374	TK-215	ICP-ES	75.5	Filtered in cell; Before initiation of the second addition of 10 M NaOH to TK-215
LW34375	TK-215	ICP-ES	83.5	Filtered in cell; Initial Sample during addition of 10 M NaOH to TK-215. Sample 1 of 3 during addition.
LW34376	TK-215	ICP-ES	89.9	Aqua-Regia Digestion due to precipitate post-filtration. Sample 2 of 3 during addition.
LW34377	TK-215	ICP-ES	90.4	Aqua-Regia Digestion due to precipitate post-filtration; Sample 3 of 3 during addition.
LW34378	TK-215	ICP-ES	90.9	Filtered in cell; Sample to confirm complete aluminum dissolution at 90 °C setpoint.
LW34379	TK-215	ICP-ES	81.3	Filtered in cell; Sample pre-filtration through FLT-305.
LW34382	TK-103 B	Free-OH	N/A[d]	Pulled on 2/15/2024 at 1051 hours
LW34383	TK-103 C	Free-OH	N/A[d]	Pulled on 2/15/2024 at 1056 hours
LW34384	TK-103 B	ICP-MS	N/A[d]	Pulled on 2/15/2024 at 1051 hours
LW34385	TK-103 C	ICP-MS	N/A[d]	Pulled on 2/15/2024 at 1056 hours
LW34386	TK-103 B	ICP-ES	N/A[d]	Pulled on 2/15/2024 at 1051 hours
LW34387	TK-103 C	ICP-ES	N/A[d]	Pulled on 2/15/2024 at 1056 hours

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[d] Temperature not measured in HAD collection tanks. Ambient temperature.

The samples from TK-215 were allowed to cool and were filtered through syringe filters following Caustic Dissolution. During filtration, within the B-wing Shielded Cells, some samples were unable to be filtered. Precipitate build-up had accumulated on the filter. The precipitate was removed but could not be characterized effectively by ICP-ES due to the inability to distinguish between the insoluble and soluble components under operations conditions of the R&D system. Upon pulling the samples, the samples were clear. Although the values for the TK-215 samples are not presented due to the inaccuracy, being unrepresentative of the dissolution, the kinetic curve is illustrated by Figure 3-13. The kinetics of dissolution represent the anticipated progression of the dissolution of aluminum cladding.

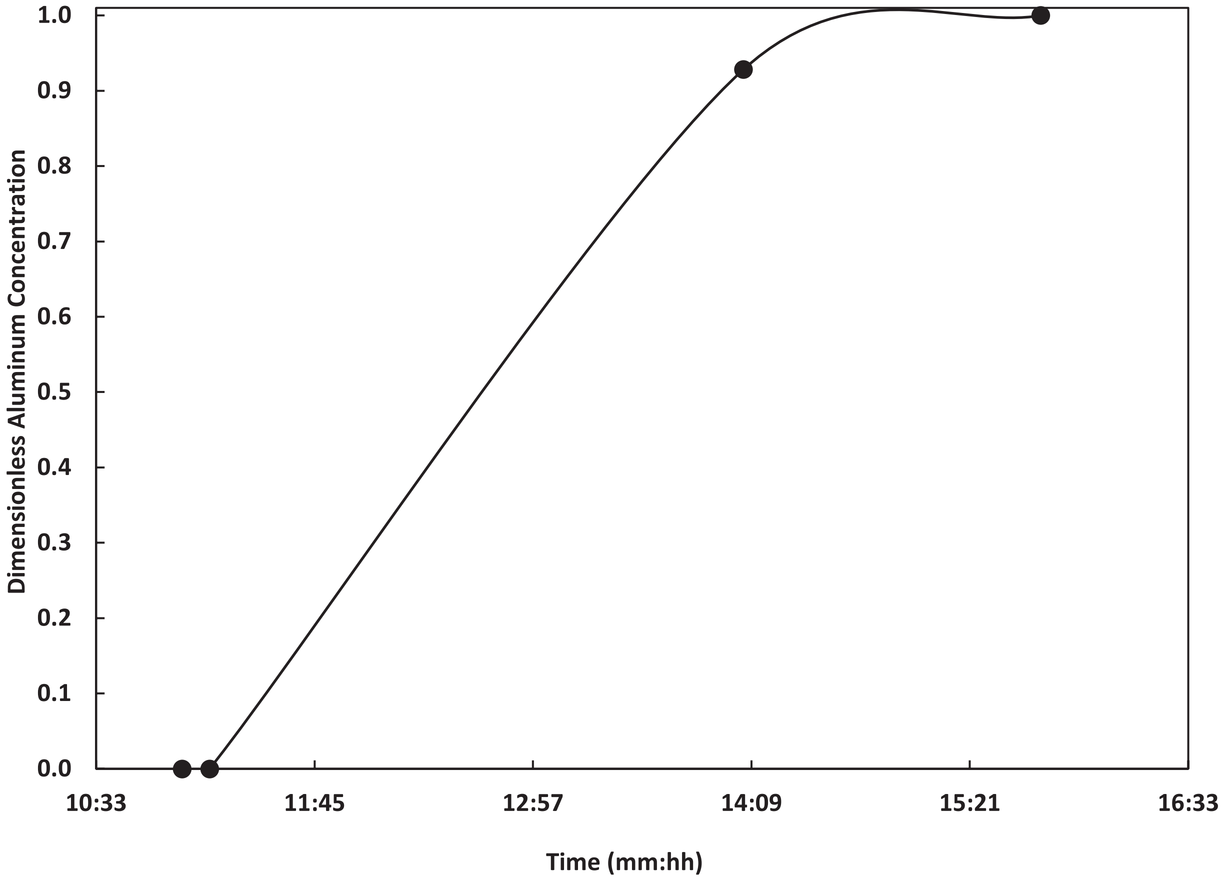

Figure 3-13: Dimensionless Aluminum Concentration versus Caustic Dissolution Sampling

Unlike the TK-215 samples, samples pulled from HAD Tanks B and C, TK-103B and TK-103C were not filtered outside the Caustic Dissolution System and did not require aqua-regia digestion for spectroscopic methods. These samples include LW34382 – LW 34387. The Analytical results for Free-OH and ICP-ES in TK-103B are in Table 3-5. Analytes below the limit of detection remain unlisted.

Table 3-5: Analytical Results for Caustic Filter Effluent to HAD Tank B, TK-103B

Analyte	Result	Units	RSD (%)	Method	Notes
Al	2.09E+04	mg·L^{-1}	5.03	ICP-ES	-
Fe	2.9	mg·L^{-1}	5.04	ICP-ES	-
Na	6.45E+04	mg·L^{-1}	5.	ICP-ES	-
Si	95.1	mg·L^{-1}	5.02	ICP-ES	-
Zn	11.8	mg·L^{-1}	5.02	ICP-ES	-
Free OH$^-$	2.13	M	5.	Free OH	Excludes carbonate anion
Other Base	0.137	M	5.	Free OH	Excludes carbonate anion

Analytical results for Free-OH and ICP-ES in TK-103 C are in Table 3-6. Analytes below the limit of detection remain unlisted.

Table 3-6: Analytical Results for Caustic Filter Effluent, FLT-305 to HAD Tank C, TK-103 C

Analyte	Result	Units	RSD (%)	Method	Notes
Al	3.11E+04	mg·L^{-1}	5.03	ICP-ES	-
Fe	4.79	mg·L^{-1}	5.04	ICP-ES	-
Na	9.54E+04	mg·L^{-1}	5.	ICP-ES	-
Si	143	mg·L^{-1}	5.02	ICP-ES	-
Zn	17.2	mg·L^{-1}	5.02	ICP-ES	-
Free OH$^-$	3.15	M	5.	Free OH	Excludes carbonate anion
Other Base	0.193	M	5	Free OH	Excludes carbonate anion

Differences in the concentrations of analytes measured in TK-103B versus TK-103C are due to additional dilutions made during post-backpulse flushing of TK-215. These flushes were received in TK-103B resulting in dilution of the filtrate collected in that tank.

From the analytical results provided per Table 3-5 and Table 3-6, ASTM B221 standard for 6061 aluminum alloy,[12] and the final level indications of HAD Tanks B and C, a material balance (Table 3-7) illustrates the aluminum mass balance from caustic dissolution. Results are well within uncertainties (e.g., Level elements, alloy composition, and analytical results).

Table 3-7: Mass Balance of Target Dissolved versus HAD Tank B and HAD Tank C Caustic Filter Effluent

Target	Input	Units	Effluent	Value	Units
Total Mass (g)	1185.6	g	TK-103 B Conc.	2.09E+04	g/L
6061Fraction Al	0.9680	-	TK-103 B Vol.	24.1	L
Al Mass (g)	**1147.7**	**g**	TK-103 C Conc.	3.11E+04	g/L
			TK-103 C Vol.	19.4	L
			Al Mass (g)	**1107.0**	**g**
Balance	**1147.7 g**				
	(1107.0 g)				
Difference	**40.7 g (3.5%)**				

From the mass balance of Table 3-7, one may conclude the aluminum cladding was sufficiently dissolved and filtered, fulfilling the design function of the Caustic System. The 40.7 g was within uncertainty and inclusive of the minimal, but observed, insoluble residue from the process. In agreement with the mass balance, minimal residues were observed during visual inspections, post-filtration.

3.4 Acid Dissolution

Mark-18A cold run material transferred from the caustic dissolver (TK-215) was available for acid dissolution in TK-235 on 2/7/24. It contained nominally 7.4 L of slurry including undissolved solids (UDS) from caustic dissolution and the contents of three caustic backpulses and one acid backpulse of the sintered filter (FLT-305). The filter was first backpulsed with two volumes of dilute caustic to knock solids into TK-235, followed by an acid backpulse as a demonstration, and a final caustic backpulse. TK-215 volumes after these backpulses are provided in Table 3-8.

Table 3-8. TK-235 backpulse volumes

Evolution	TK-235 volume (L)	Backpulse composition	Approximate backpulse volume (L)
1st caustic backpulse	2.4	~0.1 M NaOH	2.4 (a)
2nd caustic backpulse	3.6	~0.1 M NaOH	1.2
Acid backpulse	4.8	~4 M HNO_3	1.2
3rd caustic backpulse	7.4 (b)	~0.1 M NaOH	2.6

(a) Includes sludge with UDS.
(b) TK-235 volume after 3rd backpulse reported as 7.0 L at the completion of the evolution. On 2/7/24, prior to adding acid, 7.4 L was recorded.

Based on the above volumes and compositions, the initial bulk composition of TK-235 was estimated to be 0.6 M HNO_3 but was measured to be pH 11.8 (basic). It is likely that entrained NaOH in the sludge transferred from the caustic dissolver contributed to the difference in the estimated and measured pH prior to the addition of concentrated acid. The stock concentration of HNO_3 from TK-107 was estimated to be 14.2 M based on the vendor's specification of 67.53 wt.% (15.1 M) and TK-107 containing a water heel from water testing of 3.3 L; TK-107 was filled with stock acid to 56.1 L. The target acid concentration for dissolution is 8 M HNO_3 to provide a solution ready for anion exchange without acid adjustment for Pu recovery via INEX-001. This would have required ~8.9 L of stock acid and brought the total TK-235 volume to 16.3 L. TK-235 has a total volume of 14 L and ≤80% fill volume (11.2 L) is targeted to ensure that the tank is not overfilled. Thus, acid was added to TK-235 to a total volume of 11.2 L (~3.8 L of stock acid) for an estimated HNO_3 concentration of 4.8 M (based on measured pH of initial sludge and estimated stock HNO_3 concentration). The acid concentration was subsequently measured to be 3.48 M ± 5% RSD by free acid titration.

An effort should be made to minimize the backpulse volume of FLT-305 into TK-235 so that the target acid concentration can be reached. If more than ~5.2 L of 0.1 M NaOH is transferred to TK-235, the target acid concentration will not be achievable. The volume after caustic backpulse should be maintained well below this due to additional hydroxide entrained in the solids. It is suggested that if one caustic backpulse does not clear FLT-305, an acid backpulse should be done to clear the filter and knock the solids into TK-235. If this clears FLT-305, then a caustic rinse of the filter can be performed to neutralize the acid, but the solution should be transferred to HAD TK-103B/C rather than TK-235.

Ideally, there would be three indications of the volume of acid transferred to TK-235: the TK-235 level element (LE-080), the TK-107 level element (LE-060), and the acid pump (P-070) volume estimated based on the revolutions of the fixed volume FMI pump ("totalizer" feature). On 2/7/24, when acid was transferred to TK-235, LE-060 was not functioning. It is suspected by the team that the UT sensor in LE-060 failed due to concentrated nitric acid vapors and it is likely not suitable for this environment. Additionally, there was a significant discrepancy between the volume change indicated by LE-080 and the estimated volume of acid transferred from P-070. When LE-080 indicated a TK-235 volume of 11.2 L (volume change of 3.8 L), the P-070 volume estimate was 1,862.5 mL, representing a 68% difference. The reading of LE-080 was used, as this seemed most likely and was conservative to ensure that the tank was not overfilled. LE-060 should be returned to a functional state and the calibration of LE-080 and P-070 should be confirmed. It is noted that the output of the level elements in the data log is in centimeters, but the LabView display provides a volume in liters as well as fill height. Level element volume outputs in the data log are requested. Additionally, the P-070 output volume estimate is not recorded in the data log. This would be beneficial in the data log to cross check the calibration of tank level elements.

The contents of TK-235 were heated on 2/8/24 for dissolution. The setpoint (SP) dissolution temperature was initially 110 °C since the boiling point (BP) of 8 M HNO_3 (~40 wt.%) is ~111 °C.[13] The actual BP of the solution/slurry at 8 M HNO_3 will be higher due to the additional $NaNO_3$ formed from the neutralized caustic and from dissolved and UDS target components. However, since the target HNO_3 concentration was not achieved, the BP was lower. The TK-235 overtemperature SP was set to 105 °C from previous testing and mistakenly not adjusted. When this temperature was reached, power to the heaters was automatically shut down. This was noticed immediately and adjusted to restart heat. The temperature SP was changed to 105 °C but reduced to 100 °C shortly thereafter to ensure the solution was maintained below boiling. It took ~1.8 h to reach 105 °C. A dissolution time of ~7 h was targeted at temperature. The time was considered started when TK-235 reached 100 °C, which was 1.63 h from the start. The TK-235 temperature profile and heater power output are provided in Figure 3-14.

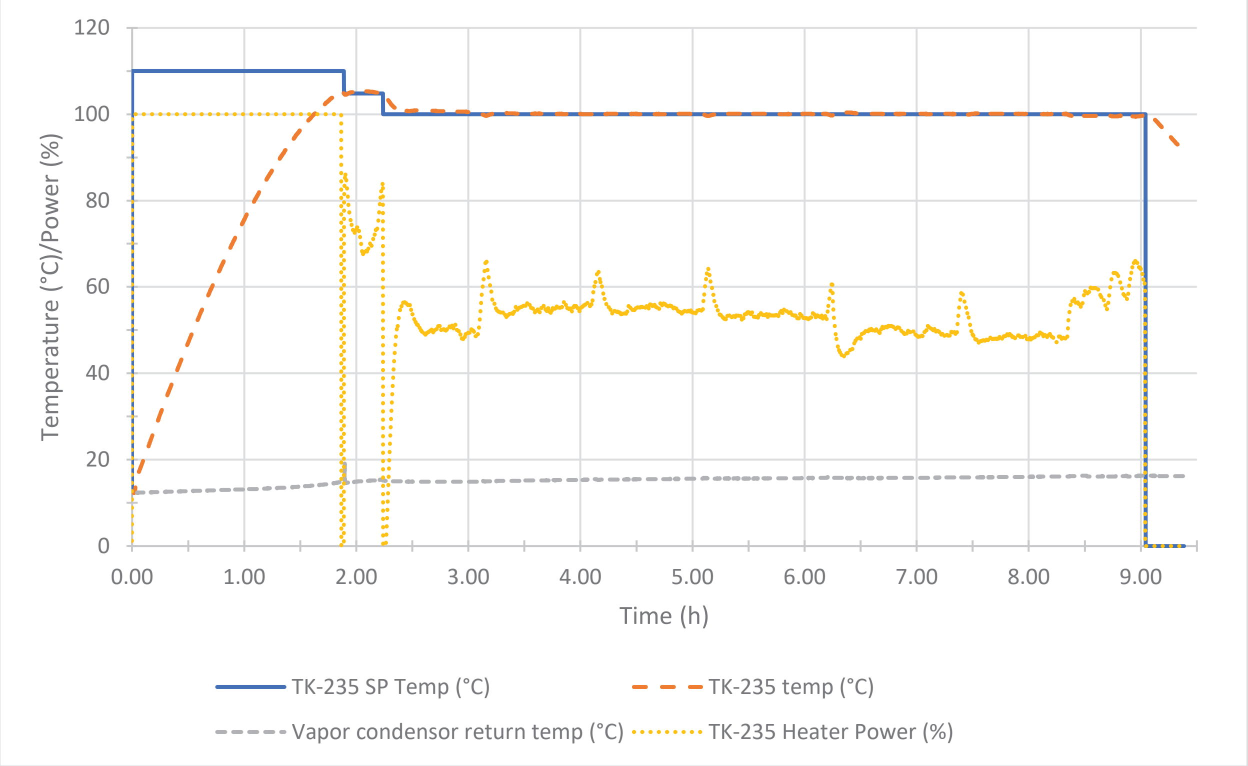

Figure 3-14. Cold Run Acid Dissolution Temperature Profile

The contents of TK-235 were sampled before and after acid addition, periodically at temperature, and after the gelatin strike (discussed below) / cooling to ambient temperature. Mixing was paused to collect samples during the dissolution, and the hot samples were put in intermediate vials to cool. All samples were filtered through 0.45-µm syringe filters at ambient temperature prior to analysis. The evolution of the dissolved Nd, Ce, and Fe concentrations in TK-235 during the dissolution are shown in Figure 3-15, Figure 3-16, and in Appendix A. Within analytical uncertainty, there is no statistically discernable change in the concentration of these analytes with respect to heating time. Thus, complete dissolution or solubility limits of these components may be achieved within the 8.5 h of heating. Plutonium as PuO_2 may be more resistant to dissolution so the baseline strategy targeting 7 h at temperature will initially be evaluated. With respect to the amounts of Nd_2O_3 and CeO_2 added to TK-215 for caustic dissolution, as listed in Table 3-2, 54% of the added Nd and 0.12% of the added Ce was dissolved in nitric acid in TK-235 after the gelatin strike. CeO_2 is resistant to dissolution in nitric acid so the Ce result is not surprising. Nd_2O_3 is highly soluble in nitric acid and the amount of Nd dissolved in TK-235 is lower than expected.

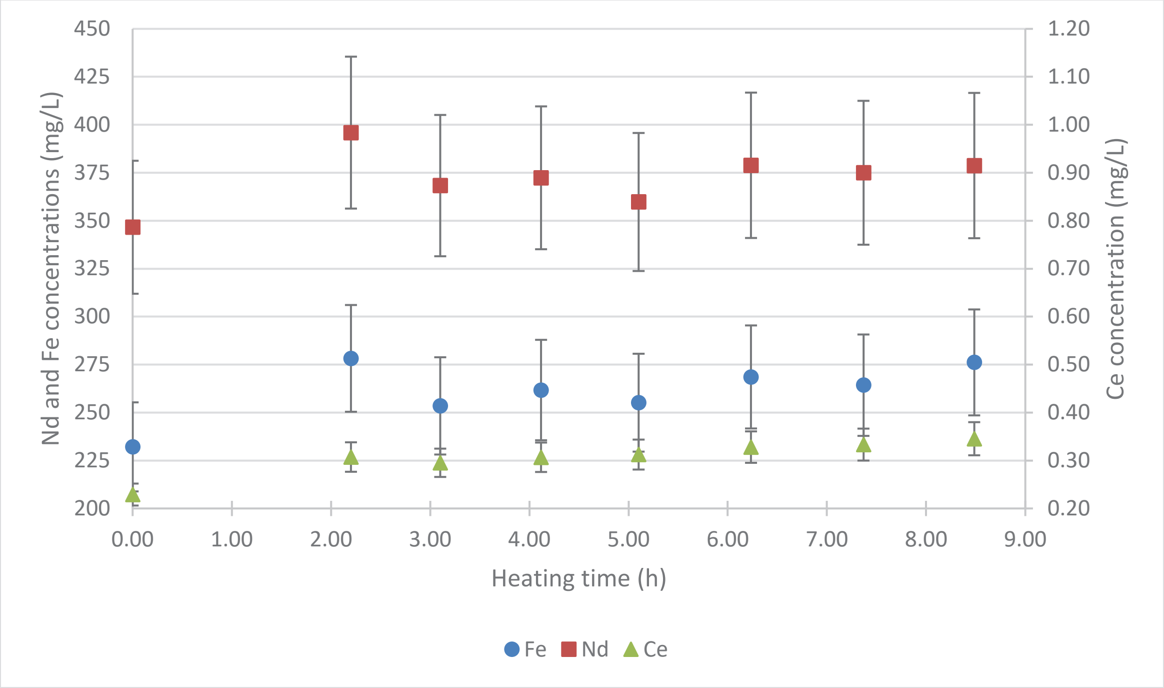

Figure 3-15. Dissolved concentrations of Nd, Ce, and Fe throughout dissolution

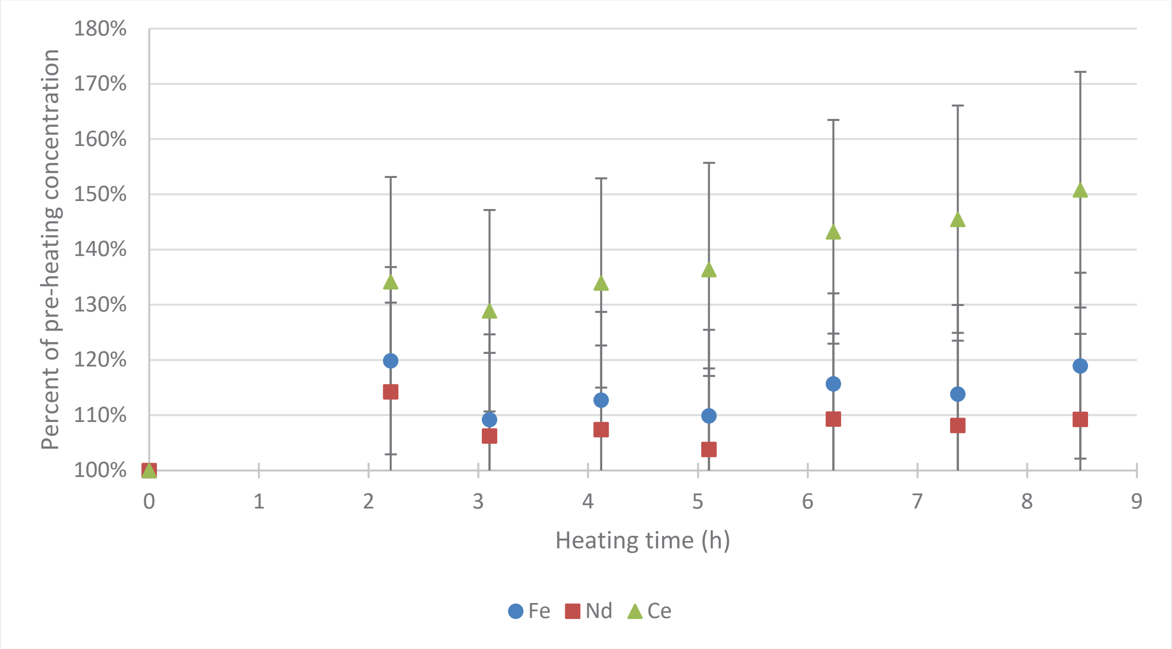

Figure 3-16. Percent change in Nd, Ce, and Fe concentrations throughout dissolution relative to pre-heating concentrations

Note that acid was added to TK-235 and the sample for initial concentrations was pulled the day prior to heating. Some dissolution may have occurred overnight at ambient temperature.

After 6.90 h at ≥100 °C, three 215 mL aliquots of ~1 wt.% aqueous gelatin solution were added to the dissolver at 10-minute intervals while mixing. The acid dissolution evolution occurred during a steam outage in 773-A and the ambient cell temperature was ~10.5 °C. To prevent the gelatin solution from setting up prior to addition to TK-235, the solutions were kept warm near a heater until they were placed in the cells several hours before addition. By the time the gelatin was added to the dissolver, it had begun to setup in the bottle. Ambient temperatures were unusually cold during this activity due to the steam outage. Previous experience with gelatin solutions prepared in the lab were that they did not setup overnight at typical room temperature (~20 °C). Nonetheless, it would be prudent to mitigate this in the future. Possible options include:

1. Preparing the gelatin solution in the shielded cell mockup the day of use and placing in the cells about an hour before use. However, this requires detailed coordination of operational resources and may not always be feasible.
2. Placing the gelatin solution in the cells up to 16 hours in advance and keeping it warm in the cells. This may not be feasible.
3. Development of a method for direct addition of gelatin solids to the dissolver. This is the preferred path forward, as it is the simplest processing approach but requires some development effort.
4. Prepare the gelatin solution in the cells, using a hot plate as needed for warming the solution.

The compositions of TK-235 prior to and after the gelatin strike are provided in Table 3-9. Despite an increase in total volume from the addition of ~645 mL gelatin solution, the post-gelatin strike Si concentration after cooling to ambient temperature increased. For the gelatin strike to be considered effective, a significant reduction in Si capable of passing through the 5-µm polypropylene bag filter is necessary. The results of this cold run indicated no effectiveness with the simulant tested. This could be in part due to the use of Na_2SiO_3 as a surrogate to supplement the Si contained in the unirradiated Al alloy since the Si content of the cladding is anticipated to increase due to transmutation to 515% of the pre-irradiation amount.[14] Si in Na_2SiO_3 represented about 75% of the Si in the cold runs. Lab testing has indicated the Na_2SiO_3 may not be a representative form of Si from the caustic dissolution of the Al cladding followed by acid dissolution of the UDS. Thus, the efficacy of the gelatin strike for a real target is inconclusive with this data.

Table 3-9. TK-235 compositions prior to and after gelatin strike

	Prior to gelatin strike	After gelatin strike
Volume (L)	~11.2 [(a)]	11.5 – 11.6
Nd (mg/L)	379 ± 10% RSD	353 ± 10% RSD
Ce (mg/L)	0.345 ± 10% RSD	0.349 ± 10% RSD
Si (mg/L)	8.74 ± 5.1% RSD	10.9 ± 5.1% RSD
HNO_3 (M)	3.48 ± 5% RSD [(b)]	3.11 ± 5% RSD

(a) Volume prior to heating and neglects losses by evaporation. While heating and mixing, volumes up to 11.7 L were observed, possibly due to eddies in the tank while mixing and thermal expansion.
(b) HNO_3 concentration prior to heating.

The contents of TK-235 were allowed to cool to ambient temperature and transferred to the anion exchange feed tank, TK-106, via the process sock filter, FLT-315, on 2/11/24. The intent was to transfer the slurry through FLT-310 based on the labels in LabView for the filter housing inlet solenoid valve, V-300, where "off" indicated flow through FLT-310 and "on" indicated flow through FLT-315. These labels were assumed to be correct when the corresponding valve positions for the three-way valve after the filter housing, V-455, were determined. However, upon inspection of the filters, it was discovered that FLT-310 contained only a small amount of debris and FLT-315 contained significant solids. Thus, the LabView labels for solenoid valve V-300 need to be corrected such that "off" indicates flow through FLT-315 and "on" indicates flow through FLT-310. Similarly, the filtration R&D directions require updating such that the V-455 is set to the 9 o'clock position to provide flow through FLT-310 and the 3 o'clock position to provide flow through FLT-315. The contents of FLT-315 after the filtration are shown in Figure 3-17.

Figure 3-17. Contents of FLT-315 after cold run filtration

The TK-235 and TK-106 volume changes and PE-010 (pressure prior to FLT-310/315) measurements are shown in Figure 3-18. It is noted that the LabView data log does not indicate if P-100 is on or off. It was requested that this be recorded in the data log. The contents of TK-235 were easily filtered and transferred to TK-106. This is encouraging as this was the first time that the filtration had been tested with silica/coagulated solids. Only one of the two parallel filters was necessary for the filtration. The filters are rated for 200 psi but the process targets pressures less than 100 psi. The maximum pressure measured during the transfer was 46 psi. Most of the volume contained in TK-235 transferred rapidly to TK-106 (within 5 minutes), but the liquid height in TK-106 continued to slowly increase until 35 minutes after the start of the transfer. As previously discussed, tank volumes are not recorded in the data log (recommend adding this data). However, volume data were recorded from the LabView interface during the evolution. The initial volume in TK-106 was <1.9 L (minimum reading). TK-106 contained a heel from water testing. With a TK-235 volume after gelatin strike of 11.5 to 11.6 L, the expected volume of TK-106 was 11.5 to 13.2 L after the transfer. The measured volume after the transfer was 12.5 L and within range of the expected value. Although significant solids were captured on FLT-315, there is evidence of possible solids breakthrough or bypass to TK-106 due to fouling issues when attempting loading operations of INEX-003. If this was a result of inadequate filtration, it may have been due to slurry bypassing the bag filter when the housing was filled above the bag filter ring during the highest rate of transfer in the first 4 minutes.

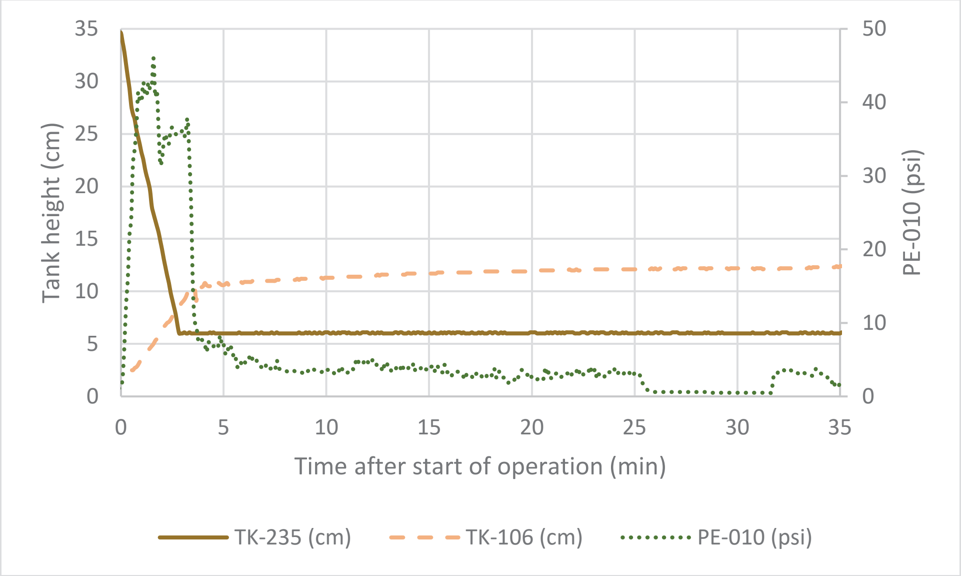

Figure 3-18. Transfer of solution from TK-235 to TK-106 via FLT-310

The liquid level heights in TK-235 and TK-106 show at least the minimum height measurements of 6 cm and 2.4 cm, respectively.

Samples were pulled periodically before, during, and after dissolution and placed in intermediate vials to cool to ambient temperature. Filtration of the samples through 0.45-µm disk syringe filters was attempted in the Shielded Cells. One sample was filtered with significant effort. Another was attempted but could not be successfully filtered with the manipulators. These samples were removed from the cells and filtered by hand with relative ease in a radiological hood. While this was fine for cold testing, due to the high activity of the Mk-18A material, this will not be practical for process samples. Thus, a filtering approach needs to be developed. One option may be a centrifuge with filtered tubes, which would allow processing of multiple samples at once. Another option recommended by SCO is a screw-driven piston to apply pressure on syringes pistons, which they have used with success in the past.

3.5 Ion Exchange Operations

3.5.1 Reillex Column Preparation and Installation[e]

This activity was performed prior to water runs. In November 2023, the acrylic-jacketed glass Mark-18A Reillex column was washed, assembled, and packed with Reillex resin beads in a non-radioactive laboratory (735-11A Lab 126) in preparation for water evaluations and simulant testing inside Cell #9 as follows. A pump was used to transfer deionized water into the assembled column by upflow through the bottom valve and screen. Water was introduced into the column in such a way as to expel any gas bubbles through the resin support screen at the bottom of the column. The column was filled approximately halfway with water and a funnel was placed on the top.

A bottle of Reillex resin stored in water was sub-sampled by dipping a 50 mL beaker into the resin and transferring the resin into a 1 L glass graduated cylinder. Approximately 6 resin sub-samples were collected in this manner and the composite volume was measured to isolate 220±3 mL of loosely packed beads. The resin was subsequently poured in portions from the cylinder into the column through the funnel allowing the beads to settle to the bottom. When approximately half of the resin beads had been transferred into the

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[e] These activities were performed prior to water runs.

column, the column was filled to the top with water and beads. The settled bed filled ~40% of the column. 300 mL of water were drained from the column by turning the bottom valve leaving a 1-inch head of liquid above the resin bed. This was repeated to transfer the remaining resin into the column and form a packed resin bed. This process left ~3/4 of an inch of visible liquid head above the packed resin bed.

The funnel was removed from the top of the column and a second glass elutriation column was attached using the stopper. A pump was attached to the bottom of the column and deionized water was pumped upflow into the column to fluidize the resin bed and elutriate fines from the bed through the upper elutriation column. An overflow tube was attached to the top of the elutriation column, and the other end of the tubing was placed in a beaker to collect excess water and elutriated resin fines. The initial water flowrate was 50-100 mL/min. This resulted in the expansion of the bed into the upper elutriation column to near the overflow line. A few fine particles were observed to exit the top of the upper column which were collected in a beaker. A total of ~500 mL of deionized water was used to elutriate the fines from the column. A small quantity of fines (≤100 mg, ~100 particles) were isolated in the beaker. Additional water was drained from the lower column by opening the bottom valve while tapping the column. This resulted in the settling of the remaining beads from the upper elutriation column into the lower Mark-18A column during liquid downflow. As shown in Figure 3-19, the visible portion of the final settled Reillex bed was 17 and 7/8 inches tall at this point (not including the bed behind the lower fitting, which was not visible). The water level was raised to the top of the lower column connection by upflow. Next, the upper column was removed, and an upper bed screen was inserted and immersed in the liquid in such a way as to remove entrapped air below the screen. The upper valve was reattached and filled with water by slow upflow through the column prior to closing the upper valve.

The Reillex column was installed in the INEX-003 position of the Mark-18A equipment in Cell 9 and used for water evaluations, discussed above, and cold simulant testing as discussed below.

Figure 3-19. Final Elutriated, Settled, and Packed Reillex Column Used for Mark-18A Water Evaluations and Cold Simulant Testing.

3.5.2 Preparation of Acid Reagents

TK-106 contained 12.5 L of 4.65 M HNO_3 (based on calculations) prepared from the acid dissolution and filtration process. This is the feed tank for the Reillex ion exchange process. The target acid concentration for this tank is 8 M, which corresponds to 40.65 wt. % HNO_3. 6.2 L of concentrated acid from TK-107 was transferred into TK-106 to give a final volume of 18.7 L. The solution was mixed for at least 5 minutes at ~150 rpm using the installed impellor blade mixer and the LabView software.

8 M HNO_3 was prepared in TK-108 by first transferring 7.1 L of process water into the tank. 8.3 L of concentrated acid from TK-107 was subsequently transferred into the tank with continual mixing at 150 rpm to give a final volume of 15.2 L. 15.2 L corresponds to 80% of the working tank volume.

0.35 M HNO_3 was prepared in TK-109 by transferring 350 mL of concentrated nitric acid into the tank (based on the TK-109 level element) with continuous mixing at 150 rpm. Process water was subsequently added to the tank with mixing to give a total volume of 15.2 L.

3.5.3 Reillex Column Nitric Acid Simulant Processing

On February 12th, 2024, nitric acid was transferred into Reillex column INEX-003 from TK-108 to adjust the nitric acid concentration in the column to near 8 M. To prepare the Reillex feed, TK-106 was mixed for at least 10 minutes and 1 mL of ferrous sulfamate (FS) solution (42 wt%) diluted to 20 mL with water was subsequently transferred via syringe into TK-106 to mimic valence adjustment of the dissolved Pu from a Mk-18A quarter target. This FS dose is based on a targeted addition of 2 moles of Fe^{2+} from FS per mole of Pu, but the FS addition amount was 10% of the total anticipated Pu amount for a typical dissolved target accounting for the fraction of Pu requiring valence adjustment. The FS reagent concentration was assumed to be 2 M to account for decomposition of this aged reagent sample. The 20 mL FS sample was transferred to TK-106 with air flushing of the lines and the tank was stirred for greater than 5 minutes. Over the next two days, nitric acid feed solution from TK-106, acid wash solution from TK-108, and dilute acid eluate solution from TK-109 were processed through the column (INEX-003). The column effluent solutions were collected during washing and feed processing in TK-124. When processing dilute acid from TK-109, the eluate solution was collected in tanks TK-111 and TK-112. Measured and targeted flow rates and measured solution volumes during processing are summarized in Table 3-10. The processing periods identified in Table 3-10 represent times during which the average flow rate was calculated. The total volume in TK-124 in the table represents the cumulative volume in TK-124 at the conclusion of each processing period (includes water heel volume present prior to processing acid).

During initial column preconditioning with ~1 L of acid (Processing Period 1) from TK-108, an average flow rate of 36 mL/min was achieved, which was near the target value. Some moderate mixing of water and acid was observed in the column headspace during initial acid transfer into the column due to density differences between these solutions, as indicated by some Reillex resin bead suspension and movement. This resulted in a brief disturbance of beads in the upper 1-cm portion of the packed bed, but the beads subsequently settled back down to reform the bed once mixing was complete. The preconditioning solution was drained from the standpipe to TK-106 prior to processing feed solution to give a total volume of 19.7 L in TK-106 at the start of loading. As indicated in Table 2-4, the green color disappeared from the top of the Reillex column when nitric acid was introduced. Note that the estimate of 1 L of acid from TK-108 was calculated as the difference between the TK-124 level element reading of 3.1 L and the minimum pump out volume for this tank of 2.0 L since the TK-108 Level Element was not working.

During processing of TK-106 feed solution containing cerium and neodymium, it was not possible to achieve the target flow rate of 60 mL/min. Average flow rates measured during the various processing periods for the feed solution are provided in Table 3-10. As indicated by trends in the flow rates over Periods 2-6, the acid flow rate through the column continually decreased as the first ~7 L of feed solution were processed. The initial flow rate was 50 mL/min (Period 2) and the final flow rate during this time (Period 6) was 11 mL/min. Changes in the plumbing downstream of the column were made during Periods 3-5 to attempt to maximize the flow of liquid through the column. Specifically, the U-tube and vent which were originally in place to eliminate the possibility of inadvertently draining the column were removed to minimize the pressure drop across the system. Initially this appeared to allow for faster flow though the column, but the average flow rate continued to decrease over time. During most of the column processing during this time, the standpipe was completely filled and there was continual overflow of liquid back into TK-106. At the conclusion of Period 6, the remaining volume of solution in TK-106 was 12.3 L and 7.4 L of feed had been removed from the tank.

Due to the difficulties in processing feed solution through column INEX-003, upflow processing of nitric acid from TK-108 was conducted to restore the column porosity and flow characteristics (Period 7). The flowrate used for acid upflow varied from 15-100 mL/min depending on observations. The Reillex bed was observed to rise to the top of the column during this time to form an inverted packed bed. Suspended white solids were observed below the column during upflow. Upflow processing was ceased, and the bed was observed to fall to the bottom of the column. However, the top of the column was not visible as it had been prior to upflow processing, indicating that the bed volume had expanded. These observations were consistent with bed fouling, presumably from precipitation or transfer of solids into the column. Downflow processing of feed solution from TK-106 was resumed after draining the standpipe to TK-106 again. A total of 1.7 L of acid were transferred to TK-106 from TK-108 during upflow operations resulting in further dilution of the feed solution to give a total volume of 14.0 L in TK-106 at this point.

The average feed flowrate for the next downflow processing period (Period 8) was 53.3 mL/min which was near the initial flowrate achieved in Period 2. The end of the workday shift had been reached at this point, so the pump was stopped, and the column valves were closed for overnight storage. The following day, flow was resumed (Period 9) through column INEX-003, and a decrease in the flowrate to 36.7 mL/min was observed relative to Period 8. The standpipe was subsequently drained back to TK-106 to give a final volume of 8.3 L of unprocessed and diluted feed solution. The volume of solution in TK-124 at this point was 13.6 L.

Table 3-10. Reillex Column Processing Summary

Processing Period	Processing Day	Activity	Average Flow Rate (mL/min)	Target Flow Rate (mL/min)	TK-124 Volume or other tanks as indicated (L)
1	1	preconditioning from TK-108	36.0	35.0	3.1
2		loading from TK-106 (U-tube configuration)	50.0	60.0	4.0
3		loading from TK-106 (U-tube removed)	44.0		5.5
4			30.0		6.6
5			14.0		7.9
6		loading from TK-106 (U-tube configuration)	11.0		9.1
7		upflow processing 1.7 L acid from TK-108 to TK-106	15-100	NA	NA
8		resume loading from TK-106 (U-tube configuration)	53.3	60.0	10.7
9	2	resume loading on Day 2 from TK-106 (U-tube configuration)	36.7	60.0	13.2
10		post-loading wash from TK-108	20.0	35.0	14.9
11		dilute acid elution/displacement to TK-124	37.5	5.0	15.6
12		eluate hearts collection in TK-111	26.3		1.0 L (TK-111)
13		eluate tails collection in TK-112	N/A		no volume collected (TK-112)

* N/A = Not Applicable.

In Processing Period 10, acid wash from TK-108 was processed through the column in a downflow direction to displace the feed, resulting in a final volume in TK-124 of 14.9 L. The average flow rate achieved during this period of 20.0 mL/min was even lower than was observed during Period 9. The data trends in Periods 8-10 indicate that although acid upflow conditioning initially resulted in maximum downward flowrates near the initial value, the column porosity continued to degrade (decrease) resulting in lower average flow rates during continued processing. The standpipe was subsequently drained back to TK-106 prior to column elution.

In Processing Period 11, dilute acid from TK-109 was pumped into the Reillex standpipe. Then, 600 mL of this solution was transferred through the column into TK-124. The average measured flow rate was 37.5 mL/min, although the pump flow rate used for this period was 5 mL/min. During this processing period, the standpipe liquid level was steadily decreasing as the system equilibrated resulting in a much faster effective flowrate than the pump rate. The final volume of liquid in TK-124 was 15.6 L.

In Processing Period 12, dilute acid from TK-109 was pumped through the Reillex column at a rate of 5 mL/min while collecting the column effluent in TK-111. 1,000 mL of eluate solution were collected to simulate the volume of Pu hearts that might be recovered during the Mark-18A process. The average flow rate during this period was 26.3 mL/min, which was faster than the target value. To achieve the target flow rates throughout the process, the standpipe liquid heights required for the various processing solutions will have to be determined, and the standpipe height will need to be reset prior to processing the solution through the column. The required standpipe heights are still being determined for the various acid streams. While processing dilute acid eluate solution from TK-109, some cracks and voids were observed near the middle and top of the column. It was unclear whether these cracks resulted from air pockets or from bed shrinkage in more dilute acid.

In Processing Period 13, dilute acid processing from TK-109 was continued at the same pump flow rate (5 mL/min), but the LabView valve solenoids were set to transfer the column eluate solution to TK-112 (Pu Tails tank). Even after processing for several minutes, the TK-112 liquid level was not observed to increase. It was unclear whether there was an issue with the solenoid valve, or the level element, or if the column porosity had further decreased making it more difficult to process fluid through the column. The pump was stopped, the column valves were closed, and the Reillex column was left in dilute nitric acid.

Of the total volume of 19.7 L of acid feed in TK-106 at the beginning of Reillex column loading (including the acid drained from the standpipe following preconditioning), it is estimated that 63.0% was processed through Reillex column INEX-003. The original acid adjusted feed solution was diluted three times during processing by acid from TK-108. This occurred when the standpipe was drained following column preconditioning or washing and during upflow operations. The volume of effluent solution in TK-124 at the conclusion of Reillex processing was 15.7 L. This volume was available for processing though DGA column INEX-001.

3.5.4 DGA Column Preparation and Installation

8 M HNO_3 was prepared to wet a sample of DGA resin prior to resin transfer into the shielded cells in preparation for column loading. Two 125-g samples of DGA resin were placed in separate polymer bottles. 8 M nitric acid was transferred into the bottles to wet the resin samples. The resin beads were observed to float on the tops of solutions even after mixing. The appearance of the samples did not change significantly after overnight soaking. Deionized water was transferred into the bottles to dilute the acid and promote resin wetting and settling. The resin beads were observed to sink at an estimated acid concentration range of 2.5-3.5 M. This process was reversible. Adding acid to increase the concentration above ~3 M caused the beads to begin to float, and adding water to decrease the acid concentration to below ~3 M resulted in bead settling. Near an acid concentration of 3 M, portions of the beads would float while others would sink. The acid concentrations of the slurries were adjusted until most of the beads settled to the bottoms of the bottles. The resin slurries were then transferred into the shielded cells for column preparation.

Inside Cell 9, a DGA column with attached upper and lower valves was placed in a stand and an additional valve and hoses were attached to the bottom of the column. One of the hoses was attached to the Mark-18A equipment to upflow acid into the column. The top of the column was removed and a customized funnel with an overflow line was attached. The overflow line was attached to a polymer bottle. The bottom of the DGA column was filled with dilute nitric acid from TK-109 by upflow to promote the removal of air bubbles below the screen at the bottom of the column. The resin slurries were transferred in portions into the funnel with periodic reduction of the liquid head by downflow, taking care not to drain the liquid level below the packed resin bed. All the resin beads from both bottles were quantitatively transferred into the column in this manner. The beads settled into the column and formed a packed DGA bed. Toward the end of this process, the liquid level was inadvertently allowed to fall below the upper ~2 inches of the bed by the accidental opening of the lower valve. As a result, upflow of dilute acid was conducted to remove air bubbles from the upper portion of the bed. Following bed rewetting, the upper screen was placed in the liquid above the top of the bed in a manner to remove air bubbles from below the screen and position the screen directly above the resin bed. Upon attempting to attach the top of the column, it was discovered that the screen was too high because the resin volume was slightly too large. The upper screen was removed and approximately 20 mL of resin were subsequently removed from the top of the bed using a disposable slurry pipette. The upper screen was reinserted and the column top was reattached. The column was removed from the stand and attached to the Mark-18A processing equipment in the INEX-001 position.

3.5.5 DGA Column Nitric Acid Simulant Processing

Measured and targeted flow rates and measured solution volumes during DGA column simulant processing are summarized in Table 3-11. The processing periods identified in Table 3-11 represent times during which the average flowrate was calculated. The total volume in TK-110 in the table represents the cumulative solution volume collected in TK-110 at the conclusion of that processing period plus the initial water heel volume. During Processing Period 1, the DGA column was preconditioned with 3 L of acid from TK-108 at a flow rate of 120 mL/min. This flowrate was faster than the targeted value because the standpipe liquid level was decreasing throughout preconditioning. During Processing Period 2, ~8 L of Reillex raffinate solution from TK-124 was processed through the DGA column into TK-110 at an average flow rate of 35.7 mL/min. The flowrate and the standpipe liquid level height were stable throughout most of this processing period and the flowrate was near the target. Due to a cells outage, the pump was stopped and the column valves were closed for several hours.

Later, the valves were reopened and flow was resumed. At the conclusion of Period 3, the DGA column standpipe (TK-300) was drained back to TK-124, and 4.8 L of solution remained in TK-124. 725 mL of dilute nitric acid from TK-109 was subsequently pumped into the DGA standpipe (TK-300). The standpipe was then drained into TK-110, resulting in a final tank TK-110 volume of 16.6 L. The DGA column was stored in this condition overnight. The following day, the DGA column was removed from the Mark-18A test apparatus and placed in the stand. The free-flowing liquid was then drained from the column by gravity and collected in two separate bottles. Bottle 1 contained 190 mL and Bottle 2 contained 178 mL.

Of the total volume of 19.7 L of acid feed in TK-106 at the beginning of Reillex column loading, it is estimated that 43.7% was processed through both the Reillex and DGA columns and was transferred into TK-110. The product solution included a portion of the feed solution from TK-106 as well as nitric acid preconditioning and feed displacement solutions used during column operations. 8.9 g of neodymium oxide was added to the original feed solution, and as discussed in Section 3.4 above, approximately 54%of the added Nd_2O_3 was dissolved in the acid dissolver. Based on these amounts, if all the neodymium reagent was absorbed to the DGA resin, then the theoretical yield of neodymium oxide to be recovered from the DGA resin would be 2.1 g.

Table 3-11. DGA Column Processing Summary

Processing Period	Processing Day	Activity	Average Flow Rate (mL/min)	Target Flow Rate (mL/min)	TK-110 Volume (L)
1	1	nitric acid preconditioning from TK-108	120.0	75	3.0
2		loading from TK-124	35.7	37.5	10.7
3		loading continued from TK-124 followed by 725 mL TK-109 wash	not measured		16.6

3.5.6 Post-Processing Analysis Results for Ion Exchange

Sub-samples of various tanks and bottles used for ion exchange processing were collected at the conclusion of testing for analysis. Results are provided in Table 3-12. The Free H^+ results for most samples were lower than expected. The nitric acid concentration target for the ion exchange feed solution in TK-106 was 8 M HNO_3. The target concentration for TK-108, which was used for washing, upflow, and feed displacement, was also 8 M HNO_3. Therefore, the acid concentrations expected for tanks TK-124 and TK-110 (Reillex and DGA effluent collection tanks, respectively) were also 8 M.

The reported Free H^+ concentration for TK-106 was 5.9 M. The nitrate concentration reported for TK-106 of 7.9 M is generally consistent with the acid addition target of 8 M. The reported sodium concentration of 0.2 M for TK-106 corresponds to the combined sodium from both NaOH and $NaNO_3$. Sodium carryover into the acid dissolution step used to produce the ion exchange feed solution was not large. The aluminum concentration in TK-106 of 0.11 M was also low, and some nitrate complexation with Al may be occurring in this solution.

Based on these results, nitric acid additions to the feed solution may have been insufficient to reach the target nitric acid concentration of 8 M. However, the results are internally inconsistent since the reported free acid and nitrate concentrations differ by almost 2 M but only 0.2 M Na^+ and 0.1 M Al are present. No nitrate sources other than HNO_3 and $NaNO_3$ existed for the feed solution, but some nitrate complexation with Al may be occurring in this solution. It appears that either the measured nitrate concentration is too high or the measured free acid concentration is too low. In future operations, the free acid concentration will be confirmed for the feed solution prior to processing. In addition, it may be necessary to evaluate the accuracy of the free acid analytical method for these solutions prior to Mark-18A processing.

The free acid concentrations reported for TK-124 and TK-110 were 4.8 and 5.4 M, respectively, while the highest free acid concentration reported for TK-108 was only 6.5 M. Similar to the results reported for TK-106, the nitrate concentration for TK-108 of 7.9 M was near the target value. In addition, a sample of nitric acid was prepared from a separate concentrated nitric acid source targeting 8 M HNO_3 and a sub-sample was submitted for analysis to evaluate the analytical methods. The nitrate and free acid concentrations reported for this sample were consistent, 8.8 M.

Analysis results for the eluate solution in TK-109 which was prepared targeting 0.35 M HNO_3 are also provided in Table 3-12. Similar to results for the tanks containing more concentrated acids, the reported free acid concentration of 0.26 M was below the target value, while the reported nitrate result of 0.31 M was closer to the target. The reported free acid concentration for the Reillex eluate “hearts” solution in TK-111 of 0.33 M is near the target value. A separate sample of elution acid was prepared outside of the shielded cells targeting 0.35 M HNO_3 and a sub-sample was submitted for analysis. The reported nitrate concentration for this sample was 0.38 M, which is near the target value, while the free acid concentration was slightly lower at 0.29 M. The Pu hearts solution for Reillex columns is typically closer to 1 M H^+, but for cold testing effluent collection in TK-111 was conducted for an arbitrary time period since there were no spectroscopically-active absorbed species present in solution to indicate the onset of elution. The average free acid concentration for the DGA wash solution reported to be 1.8 M was expected to be between 0.35 and 8 M since this solution was collected during the transition from TK-108 solution from and TK-109 eluate solution.

Based on the analyses, the cerium oxide did not dissolve, as expected, and no significant amount of cerium was present in the ion exchange feed solution.

Neodymium (Nd) was observed in the original TK-106 feed solution at a concentration of 1.0 E-03 M and in the Reillex effluent solution in TK-124 at a slightly diluted concentration of 8.1 E-04 M, as expected since wash solution from TK-108 was also transferred to this tank. A much lower Nd concentration of 8.5 E-07 M was observed in the DGA column effluent solution in TK-110 due to sorption of Nd to the resin. The concentration of Nd in TK-110 was 0.10% of the concentration in TK-124 and 99.8% of the Nd processed through the DGA column was removed from the solution and absorbed to the resin. The Nd concentration in the DGA wash solution was 1.7 E-05 M, which corresponds to 2.1% of the feed concentration. Nd losses to the wash corresponded to only 0.07% of the total Nd loaded on the column. Greater Nd losses have been observed in previous studies,[15] but the loss percentage is a function of resin loading levels.

Sub-samples of the upper two inches of the Reillex column were collected and submitted for analysis in order to try to identify potential resin fouling agents. The Reillex beads were digested by alkali fusion and the resulting liquid was analyzed by ICP-ES and ICP-MS. Low levels of Pd (~20 µg/g) were observed in the solution. No other metals were observed at concentrations significantly above those observed in the sample blank.

Table 3-12. Analysis Results for Various Reagent and Tank Sub-Samples Collected During Mark 18 Ion Exchange Simulant Testing

Sub-Sample	Collection Date	Volume at Collection Time (L)	Molarity					
			Free H^+	NO_3^-	Na^+	Al	Ce	Nd
TK-106	2/14/24	11.0	5.90	7.85	0.20	0.11	<2.9E-06	1.0E-03
TK-108	May 2024	---	6.52	7.87	--	--	--	--
TK-109	May 2024	---	0.26	0.31	--	--	--	--
TK-124	2/14/24	4.8	4.78	---	0.15	0.08	<2.9E-06	8.1E-04
TK-110	2/15/24	16.6	5.37	---	0.13	0.06	<2.9E-06	8.5E-07
TK-111	2/14/24	1.0	0.33	---	---	---	<2.9E-06	1.2E-05
DGA Wash	2/15/24	0.368	1.76	---	---	---	<2.9E-06	1.7E-05
8 M HNO_3*	---	---	8.75	8.78	---	---	---	---
0.35 M HNO_3*	---	---	0.29	0.38	---	---	---	---

* Reagents prepared from separate concentrated acid source.

3.5.7 Spectroscopic Analysis During Mark 18 Simulant Testing

Real time monitoring of the Reillex and DGA column effluent was performed using UV-Visible (UV-vis) spectroscopy. Transmitted light intensities delivered from a Tungsten-Halogen and Xenon Arc lamp via fiber optic cable through ½” COTS stainless steel Swagelok flow cells was employed to monitor analytes present in the chemical flow streams. The flow cells were positioned at sampling points upstream of the

Reillex column (AP-010), downstream of the Reillex column (AP-030), and downstream of the DGA column (AP-040). Absorption spectra of the effluent from measured transmitted light intensity data collected over the instrument sampling time are shown in Figure 3-20 and Figure 3-21. Figure 3-20 shows the real time effluent absorption spectral evolution of the Reillex column. Absorption bands centered at *ca* 393 nm, 510 nm, 523 nm, 578 nm, 739 nm, 791 nm, and 867 nm are observed in the spectra. The 393 nm absorption band is assigned to Fe (present in the aluminum alloy used as a target surrogate), while the other bands are assigned to Nd. This is consistent with absorption spectra of standard solutions recorded for Fe, Nd, and admixtures from laboratory benchtop experiments. The Fe absorption band at *ca* 393 nm recorded the most intense absorbance. As sampling proceeds, this absorption intensity is observed to gradually decrease. This trend contrasts with the Nd absorption bands, which begin with a broad continuum band at *ca* 481 – 650 nm and 651 – 870 nm. As sampling proceeds, the different electronic transition bands of the Nd are perceptibly apparent in the spectra. The absorption bands increase in intensity over the sampling time.

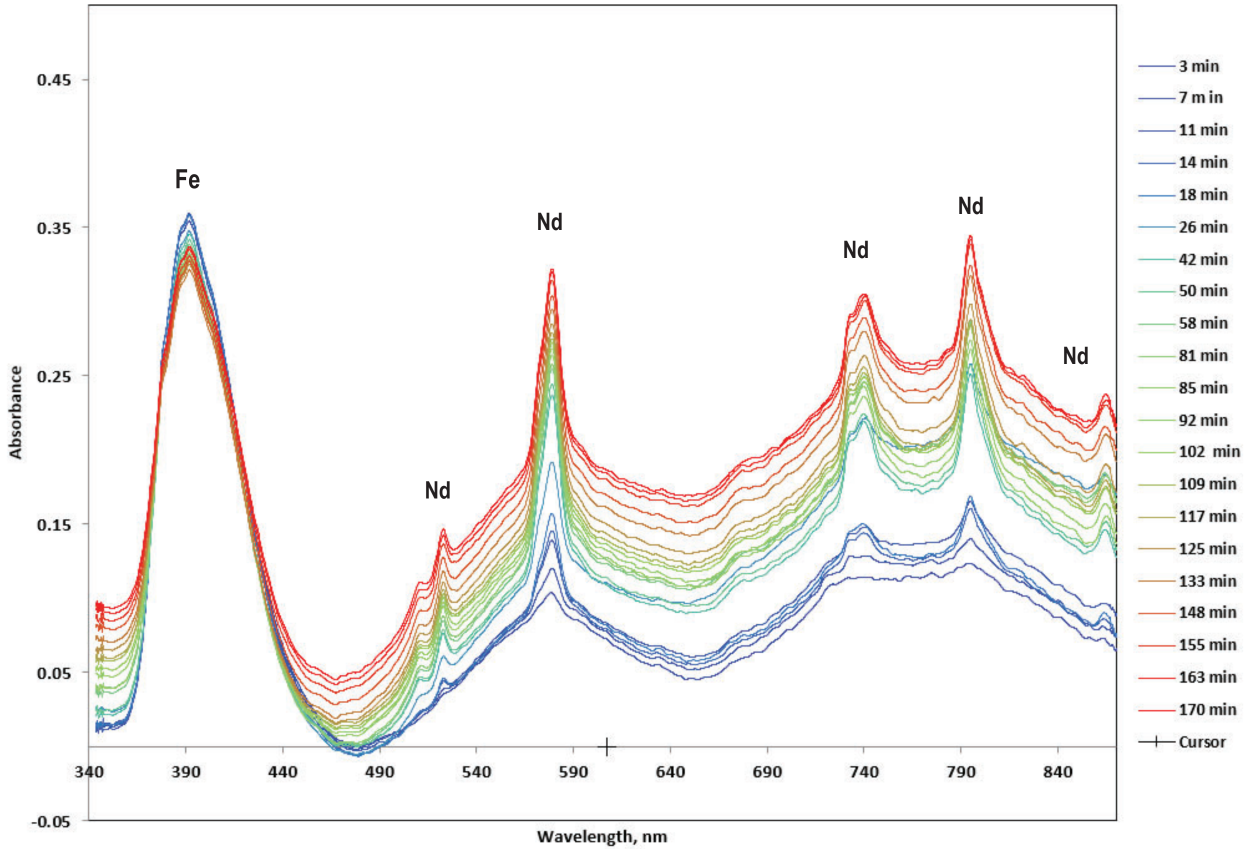


Figure 3-20. Absorption spectral evolution in Reillex Column effluents (AP-030)

Figure 3-21 shows the real time effluent absorption spectral evolution of the DGA column. Several absorption features are observed to increase as sampling and processing proceed. Absorption bands are observed at *ca* 372 nm, 381 nm, 397nm, and 402 nm. A broad continuum absorption band is also observed at *ca* 491 – 650 nm and 651 – 870 nm. The absorption bands centered near *ca* 372 nm and 397 nm are assigned to Cr(III) and Cr(VI).[16] The Fe absorption bands centered at *ca* 381 and 402 nm showed the most intense absorption intensity amongst other bands present in the spectra. The broad continuum bands ranging from 491 to 870 nm are assigned to Nd absorption. There is a gradual increase in the absorption intensities initially followed by an eventual stability in intensities as sampling proceeds. The unresolved continuum Nd absorption bands can be attributed to low concentration of analytes present in the DGA raffinate solution. This is different from perceptibly resolved absorption band features observed for Nd in Reillex raffinate absorption spectra shown in Figure 3-20, due to an increase in Nd concentration in the solution relative to the DGA raffinate solution.

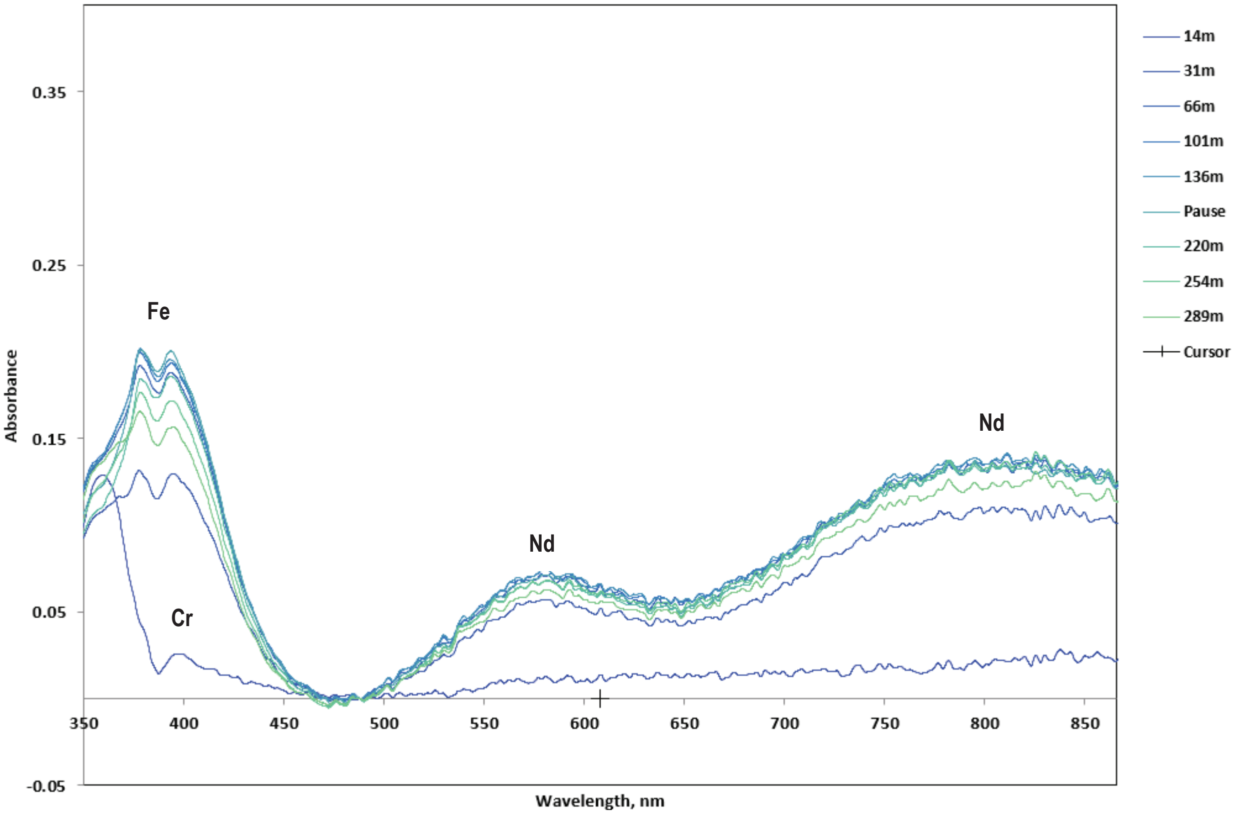


Figure 3-21. Absorption spectral evolution in DGA column effluents (AP-040)

3.5.8 Ion Exchange and Associated Equipment Cold Run Testing Issues and Status

Issues identified during acid simulant testing are summarized in Table 3-13. The connections on the nitric acid drum were inadvertently swapped. The connections were subsequently color coded and may eventually be modified to ensure that they cannot be swapped. Free H^+ analyses for tank acid concentrations near 8 M HNO_3 were low. The TK-107 level element initially did not work on several occasions, but it was discovered that wiping the sensor with a non-combustible wipe to remove black corrosion from nitric acid resulted in improved performance. Measures should be taken to minimize exposure of the current level element probe to the atmosphere above the concentrated nitric acid or the probe should be replaced with a different type. Degradation in the maximum achievable flowrate for the Reillex column was observed that is believed to have resulted from resin fouling. Physical properties observed in the bed during processing were also consistent with fouling by precipitation or solid deposition within the bed. The reason for the fouling remains to be determined. It may be necessary to optimize or remove the gelatin strike conducted prior to column processing. In addition, it may not be possible to achieve the target flowrate for Reillex processing of 60 mL/min (maximum observed flowrate with nitric acid to date is 50 mL/min). There was no indication of fouling issues for the DGA column. A set of issues persisted utilizing the spectroscopic instrument from real time monitoring of the chemical flow streams. Chiefly amongst them was the low transmitted raw light intensities through flow cells. Other issues such as fluctuating baseline levels and periodic loss of xenon light to the reference spectrometer were persistent. The low signal intensities observed through the flow cells can be improved by optimizing the optical instrument set up and arrangement. This, in turn, will resolve some of the issues associated with shifting baseline levels. The periodical loss of the xenon light observed in the raw data signal can be attributed majorly to instrument software real time processes. This issue has been evaluated to correct for any contribution associated with the instrument hardware components. Further testing of the instrument hardware and software are being explored to resolve this issue.

Table 3-13. Mark-18A Ion Exchange and Associated Equipment Issues during Cold Acid Simulant Testing and Status/Resolution

Issue	Status/Resolution
swapped nitric acid drum connections	connections color coded
accurate nitric acid adjustment to 8 M and Free H^+ measurement of 8 M HNO_3	evaluate analytical methods, review calculations
TK-107 level element corrosion and failure	prepare tank plug and remove level element when not in use; as needed, consider radar probe to monitor level
Reillex column fouling	sample and analyze Reillex resin in column, inspect column screens and pre-filters, further evaluate and optimize gelatin strike, as needed
Spectroscopic Monitoring	Improve light transmission through upstream Reillex column flow cells and downstream DGA column flow cells; further evaluation of optical design system is needed to optimize light signal. Further troubleshooting of instrument hardware and software are being explored to correct for periodic xenon light loss during real time monitoring of chemical flow stream.

3.6 DGA Drying, Volume Reduction, and Calcination

3.6.1 Column and Furnace Setup

The first step in preparation for DGA resin drying and calcination is the draining of the column. During hot testing, the draining will need to be performed in quick succession with the following drying and volume reduction steps; however, with the cold testing, we took advantage of the lack of radiolytic heating to examine the speed and efficiency of draining. The column was drained for approximately 10 minutes, until the flow from the bottom drain port was insignificant. The bottom drain port was closed, and the column was allowed to sit in the rack overnight. After sitting, the bottom drain port was opened again to see what residual material remained; an insignificant amount of liquid (<15 mL) was observed. Therefore, it was determined that the original draining duration had been sufficient.

With the column sufficiently drained, the top and bottom caps utilized for liquid operations were removed along with an internal screen designed to keep the DGA media in place. A new top cap designed with an embedded screen within an up-flow extension and a new bottom cap designed with an argon gas connection were installed. The removal and installation of the column endcaps was accomplished without significant incident and proved the design features implemented to aide in these tasks (flat spots for gripping of the caps and a loop on the internal screen for lifting) were sufficient.

The argon up-flow gas connection was the next component inspected; two issues arose during preparation. First, an issue arose with the rotameter affixed to the component panel on the left side of Cell 9. Flow through the rotameter was observed to spike and then drop to zero when the argon regulator and valve were turned on. This suggested a faulty or leaky rotameter. During examination of each of the associated components, the control valve for the argon leading to the rotameter set in the same component panel broke; the handle critically failed and fell off. This left the flow to the questionable rotameter in an uncertain state as it was not determinable whether the valve was in the open, closed, or partially-opened state when the handle fell off. With this complete failure of the argon delivery system, a replacement rotameter and set of tubing was installed between the KAPL sleeve and inlet to the DGA column up-flow piping. The installation of new tubing and rotameter resolved the argon flow issue, and furnace operations were able to proceed.

Following the installation of the new caps and the argon gas line attachment to the bottom port, the column was installed in the furnace on internal guiderails. Guiderails were an integral part of the tube furnace equipment. Alignment of the tabs on the column with the furnace guiderails proved little challenge, and the column was installed in the furnace without incident. The installation of the catalytic converter bucket on top of the furnace and column was more challenging due to the asymmetrical weight distribution of the bucket and misalignment of the catalytic converter within. The external bucket was installed on the tube furnace alignment pins using the crane and manipulators from Cells 8 and 9, but the internal catalytic converter alignment proved more difficult to coordinate between the alignment pins within the bucket and the power leads with the hole in the side of the bucket. Due to the attachment of the exhaust gas line from the catalytic converter to the outside of the bucket, readjustment of the catalytic converter within the bucket was restricted once it had shifted. The bucket assembly had to be removed from the top of the furnace and the internal catalytic converter handled rather roughly to improve alignment on the pins and with the hole in the bucket. Complete alignment with the internal pins could not be positively confirmed due to the inability to see well inside the bucket, but the alignment of the electrical leads with the bucket hole was sufficient to reduce the chance of incidental contact between the leads and the bucket. Additionally, due to the difficulty in aligning the catalytic converter components, the tube furnace is not sitting plumb. The tilt is not significant enough to alter operations, but it is visually noticeable and should be monitored to ensure that it does not progress.

After the internal catalytic converter was aligned, insulator padding was inserted around the electrical leads from the outside to further reduce the chance for incidental contact between the electrical leads and any metal, either from the bucket or other implement within the Shielded Cells. Installation of a 3D printed alumina cover was initially attempted, but the component was broken during installation, so flexible ceramic wool batting was utilized instead.

The final task associated with setup involved the exhaust vacuum pump. One issue was isolated with the operation of the vacuum pump, but it is a condition of the Shielded Cells and not an impact from the Mk-18A tasks. The quad-plug that powers the vacuum pump was mis-labelled. The quad-plug color-coding was reversed between the left and right switches. Therefore, to turn the pump on, the alternate switch must be flipped from what it appeared. With the correct switch flipped, the rotameter registered the desired flow from the outlet of the pump. With accurate flow verified, a diverter valve was turned to direct pump exhaust flow away from the rotameter and directly to exhaust to avoid potentially contaminating the rotameter with any particulates that may breakthrough the exhaust filtration. During operations, the temperature of the gas entering the vacuum pump never exceeded 33 °C, demonstrating that the cooling air input to the offgas stream was sufficient to protect the equipment below the process limit of 40 °C.

3.6.2 Tube Furnace Operations

The manipulation of the tube furnace was all performed by the control computer. Once initial communication issues were resolved, two faults were discovered: one of the three heated zones of the tube furnace failed and the over-temp controller was not functioning. As the tube furnace is configured, there are two 6-inch zones (the bottom and top zones) and one 24-inch zone (the middle zone). Based on programming feedback and impedance measurements, it was discovered that the top zone became inoperable during equipment transport and installation. Based on the load position of the DGA column, the top zone was predominantly responsible for heating the column. With the loss of the top zone, it was suspected that full drying and volume reduction of the DGA resin media would not be achieved following the same heating profile. However, since the column extended past the top zone and into the middle zone, it was decided that the heating profile would not be modified for the first cold run, and the final product would be evaluated for reaction completeness. As for the over-temp controller, since the tube furnace will not be operated unattended, the controller was bypassed to allow attended operations only.

The heating steps for the tube furnace (Table 3-14) were performed as directed by the control computer with no errors, and the drying and volume reduction reactions were observed to have occurred based on visual smoke exiting between the catalytic converter and the bucket and according to the temperature readout between the top of the DGA column exhaust tube and the catalytic converter inlet. The visual smoke is an indication that the catalytic converter was likely still not flush to the top of the furnace, as was suspected due to the difficulty during installation. This will feed into modifications of the catalytic converter assembly in future designs. As an immediate precaution, the argon flow through the DGA column was increased from 15 scfh to 20 scfh.

Table 3-14. Tube Furnace Heating Profile

Sequence	Step	Duration (minutes)	Cumulative Hours
1	Heat to 185°C at 5°C/min	33	0.55
2	Hold at 185°C	30	1.05
3	Heat to 275°C at 1°C/min	90	2.55
4	Hold at 275°C	45	3.30
5	Heat to 320°C at 1°C/min	45	4.05
6	Hold at 320°C	60	5.05
7	Heat to 350°C at 1°C/min	30	5.55
8	Hold at 350°C	45	6.30
9	Heat to 375°C at 1°C/min	25	6.72
10	Hold at 375°C	60	7.72
11	Heat to 385°C at 1°C/min	10	7.88
12	Hold at 385°C	120	9.88
13	Cool to ambient room temperature	-----	-----

As with previous experiments, in addition to the appearance of smoke, the temperature between the column exhaust tube and the catalytic converter spiked to over 730 °C once the DGA resin began to exothermically react. The difference experienced during this test was that the initiation of this exotherm was delayed until the furnace had reached 350 °C whereas previously this reaction started when the furnace was registering closer to 325 °C. This delay is likely due to the column being slightly cooler as the top zone of the furnace was not supplying additional heat to the upper portion of the column. The top zone remained approximately 100 °C below the controlled middle zone during the entire evolution. The elevated temperature in the space above the column lasted approximately 30 minutes to 1 hour before dropping back down to approximately match the internal temperature of the tube furnace. At peak reaction, the energy output was enough to offset the power draw to the catalytic converter band heater by approximately half of what had been required to maintain the 450 °C setpoint. This suggests that the majority of the exotherm was complete; however, this can only be confirmed after the material is removed from the column and weighed.

One interruption to heating occurred during the final hold step at 385 °C due to an attempt to initiate communication between the control computer and the muffle furnace. Due to the way the tube furnace and muffle furnace are connected to the control computer (via a "daisy-chain" type connection), initiating communication with one while the other is running causes an error in the response from both. The interruption caused the tube furnace to temporarily stop heating until communications could be ceased with the muffle furnace and the E-stop disengaged. The error lasted only a couple of minutes and the temperature of the controlled zone (the middle zone) only dropped approximately 30 °C; the temperature recovered in less than 10 minutes. Though an interruption was produced, this led to a better understanding of the communications between the furnaces and control computer. There is no instance during hot runs where both furnaces will need to be operating at the same time; therefore, this specific condition will not be reproduced.

Once the heating profile was complete, the furnace was set to 0 °C and monitored to below an observed temperature of 200 °C before being de-energized. With the column in place and argon still flowing, the temperature dropped to below 200 °C in less than 1 hour. The furnace was completely de-energized at this point, and the argon was turned off. The column was left in the furnace to cool overnight.

3.6.3 Calcination

The cooled column was removed from the tube furnace with some difficulty. It appeared that the column guide fins were becoming stuck somewhere along the internal guiderails after the column had been lifted only an inch or two. After some maneuvering, the column was able to be lifted out, but the exact cause of the difficulty could not be immediately determined. It was suggested that the guide fins may not be exactly 180 degrees apart and aligned vertically between the upper and lower fin positions. Therefore, more control may need to be applied during the fabrication of future columns.

The top cap from the column was removed with ease and the DGA residue was transferred to an alumina tray. The material was black-gray in color and flowed easily out of the column, suggesting good reaction occurred. However, the material overflowed the tray, suggesting that the volume reduction was not complete. See Figure 3-22 for a photo of the material in the crucible tray. Some material was lost on the deck plate of the Cells. To reduce the loss of additional material, a tray cover fabricated from a steel frame and Hastelloy mesh was placed on top. Due to the volume of material, the mesh contacted the residue. This is not ideal. However, this was deemed a worse-case scenario for evaluation of behavior of the material. The actual mass of material was nearly double what was anticipated if the volume reduction reaction had been complete. It is expected that in future runs further volume reduction will be achieved. If the volume is too great for the crucible tray, the final calcination will be performed in batches.

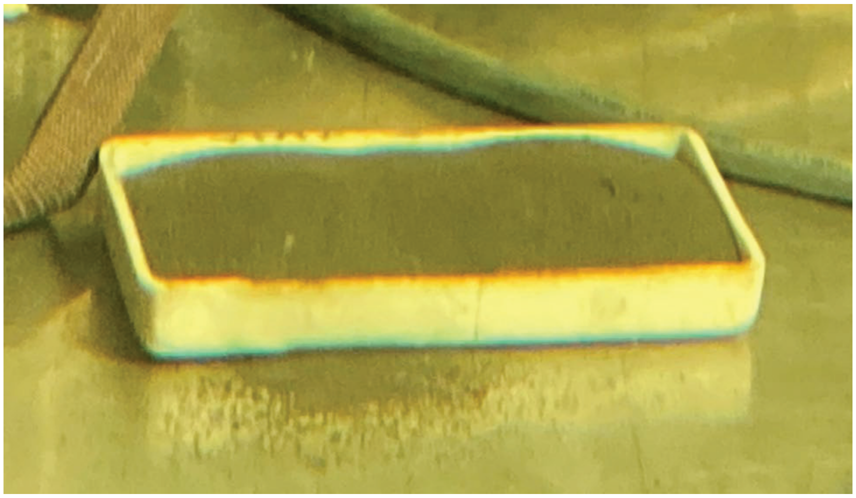


Figure 3-22. Dried and volume reduced resin after being poured from the column after tube furnace operations; prior to calcination in the muffle furnace.

After everything had been weighed, the covered tray was loaded into the muffle furnace. Due to the placement of the furnace between Cells 9 and 10 it was not required to move the furnace for operations, eliminating one risk of potential instrument damage. The furnace heating profile was operated manually as communications issues were still present, as discovered during the tube furnace operations. It was possible to program one ramp and hold step at a time; therefore, at the end of each hold phase, the next setpoint, heating rate, and hold time had to be manually entered. The muffle furnace heating operations performed as programmed, demonstrating full functionality of the muffle furnace after being moved into the Shielded Cells.

During the calcination (see Table 3-15), significant smoke was observed coming from the door of the muffle furnace and there was some black soot residue on interior portions of the door insulation. This was likely due to the incomplete volume reduction in the tube furnace, leaving remnant organic components of the resin to be decomposed at the higher temperatures of the muffle furnace heating profile. The smoke was no longer visible after the first hold step, suggesting the remaining organic material had reacted. Following the final hold step at 1020 °C, the furnace was set to 0 °C and allowed to cool. The muffle furnace took longer than the tube furnace to cool to a point where the furnace could be de-energized to cool overnight; the tray was left in the furnace to cool overnight.

Table 3-15. Muffle Furnace Heating Profile

Sequence	Step	Duration (minutes)	Cumulative Hours
1	Heat to 550°C at 15°C/min	35.3	0.59
2	Hold at 550°C	90	2.09
3	Heat to 700°C at 10°C/min	15	2.34
4	Hold at 700°C	60	3.34
5	Heat to 1020°C at 10°C/min	32	3.87
6	Hold at 1020°C	180	6.87
7	Cool to ambient room temperature	-----	-----

After cooling, the tray was removed from the furnace and weighed to evaluate effectiveness of the calcination process. Two issues were observed after the first calcination process was completed. First, the mass of the residue was approximately twice what was anticipated based on the amount of surrogate material that was run through the column during the loading portion of the cold run. This discrepancy was potentially related to the second issue that was observed: the Hastelloy mesh in the tray cover was completely degraded to the point there was no visible mesh in the cover frame. There is a high likelihood that residue from the mesh combined with resin residue to add to the total weight of the calcined material.

3.6.4 Loss-on-Ignition (LOI)

After weighing, the tray was returned to the muffle furnace without the cover and run through the LOI heating profile (see Table 3-16). After completing the LOI profile, the weight of the material had decreased by approximately 16.5%, much greater than the 1% weight loss criteria. This indicated incomplete calcination during the original calcination cycle in the muffle furnace. The tray was returned to the muffle furnace for a second LOI cycle. Unfortunately, when removing the tray from the muffle furnace after the second LOI the tray came in contact with a piece of plastic tubing when placed on the deck plate while still hot enough to melt the plastic. This resulted in plastic residue being present on the tray, altering the measured mass. The material was then transferred to a beaker to obtain a weight, but some sample was lost during the transfer, and therefore an accurate weight could not be determined.

Table 3-16. Loss-On-Ignition Heating Profile

Sequence	Step	Duration (minutes)	Cumulative Hours
1	Heat to 1000°C at 15°C/min	65	1.08
2	Hold at 1000°C	120	3.08
3	Cool to ambient room temperature	-----	-----

3.6.5 Loading the Mk-Vial

The calcined material was determined to be sufficiently processed to be used in demonstration of the next phase of the cold run. Approximately 200 mg of the final product was submitted for digestion and analysis. The remaining material was poured from the beaker used for the final weighing into a Mk-vial in preparation for demonstration of the bagless transfer system with a loaded vial. Again, this was considered a worst-case scenario as the material may contain additional residues beyond the calcined oxides of the added surrogates. There was little difficulty transferring the calcined residue from the beaker to the vial. The material fit nicely in the vial without the use of the extension tube and packing rod. The loaded vial was placed in the desiccator to await the bagless transfer process.

3.6.6 Hot Run Recommendations

Based on the observed complications and determined inadequacies, some modifications were made to the Mk-18A setup during the cold run, and some additional modifications before hot runs are performed are recommended.

- The column guide fins are recommended to be redesigned to allow for lower placement within the tube furnace, further extending the column into the controlled heating middle zone.; the column top extension cap, bottom argon up-flow cap, and rack will require modification to accommodate any changes made to the column guide fins. Additionally, greater oversight over the precise placement of the guide fins is recommended to reduce potential complications with insertion and removal from the tube furnace guide rails.
- The tube furnace heating profile is recommended to be adjusted up in temperature to accommodate the lack of heating from the inactive top heating zone.
- The catalytic converter bucket assembly is recommended to be redesigned such that the internal catalytic converter is secured to the bucket assembly so that the internal catalytic converter cannot be moved independent of the bucket.
- The argon flow control rotameter and tubing were replaced. It is recommended that the new rotameter be secured in such a way as to ensure it remains vertical and the tubing remains away from potential hot surfaces during operations.
- The tube furnace and muffle furnace heating profiles were controlled manually, either at the control computer or at the instrument controller directly. It is recommended that automated programs be completed in the control computer software to ensure consistency between operations and eliminate the potential human error of manually entering temperatures, times, and rates for each step.
- Insulating ceramic batting and administrative controls (Cell 8 right manipulator was tagged out) were utilized to reduce the risk of contacting the energized leads to the catalytic converter band heater. It is recommended that a more secure insulating cover be developed to further reduce the risk of contacting the energized leads and remove the administrative control requirement.
- The calcination tray will be placed in secondary containment so that any material that escapes the tray may be recovered easily.

3.7 Bagless Transfer and Packaging System

The final unit operation demonstrated during cold runs was the bagless transfer and packaging system (BTPS). The calcined oxide product obtained from calcination of the Nd loaded DGA resin was packaged in a MK-vial and removed from Cell 9 using the BTPS. Procedure L30-5.11, Mk-18A BTC and SFC Packaging Operations was utilized for performing the bagless transfer and packaging operations. This procedure includes steps related to implementing a combined exclusion zone and standoff distance around the Mk-18A robotic arm behind Cells 9 and 10, prohibiting all transient combustibles to satisfy the TSR requirements.

The bagless transfer process involves the following steps:

- Insertion of the bagless transfer can into Cell 9 through a sphincter seal
- Loading of the MK-vial into the bagless transfer can
- Insertion of a hollow plug into the bagless transfer can
- Brazing of the plug into the can
- Cutting of brazed plug

The packaging process involves the following steps:

- Loading the brazed bagless transfer container into a special forms capsule (SFC)
- Closure of the SFC
- Placement of the SFC into a Type A drum
- Radiological surveys at points within the process.

Many of these activities are performed remotely by the robot without personnel in the room due to the expected dose rate of the actual product. The remote operations are performed utilizing automated sequences that are initiated from the Mk-18A Bagless Transfer and Packaging HMI located in E-004 in front of the shielded cells. Networked cameras are utilized to remotely monitor system operation. Figure 3-23 shows the BTPS HMI main display.

HMI MODE: OPERATIONS
HMI LOCATION: LOCAL
WAITING ON COMMAND
01:07:20 PM 06/08/2023
AUTOMATIC OPERATIONS STATE: SYSTEM HAS BEEN INITIALIZED - WAITING FOR COMMAND
PROGRAM NAME & DESCRIPTION:
STATUS: Initialization successful.
POS DESCRIPTION: NOT HOMED
POS INCHES: 0.00
VEL IN./SEC: 0.00
STATUS:
BJIC BJ (PSIG): 0.000
BJIC SEAL (PSIG): 0.000
BJOC BJ (PSIG): -1.000
BJOC SEAL (PSIG): -1.000
BTC STATE: IN SFC
LOAD MODULE STATE: HOME
BRAZE MODULE STATE: HOME
CUT MODULE STATE: HOME
ROBOT ITEM STATE: NONE
ROBOT TOOL STATE: NONE
LAD MOD STATE: NONE
SFC STATE: CAN IN
DRUM STATE: RDY FOR PKG
ROBOT SKID TABLE STATE: OPEN
CYL POS (IN.): 0.000
SYSTEM HAS BEEN INITIALIZED - WAITING FOR COMMAND

Figure 3-23. BTPS HMI Main Display

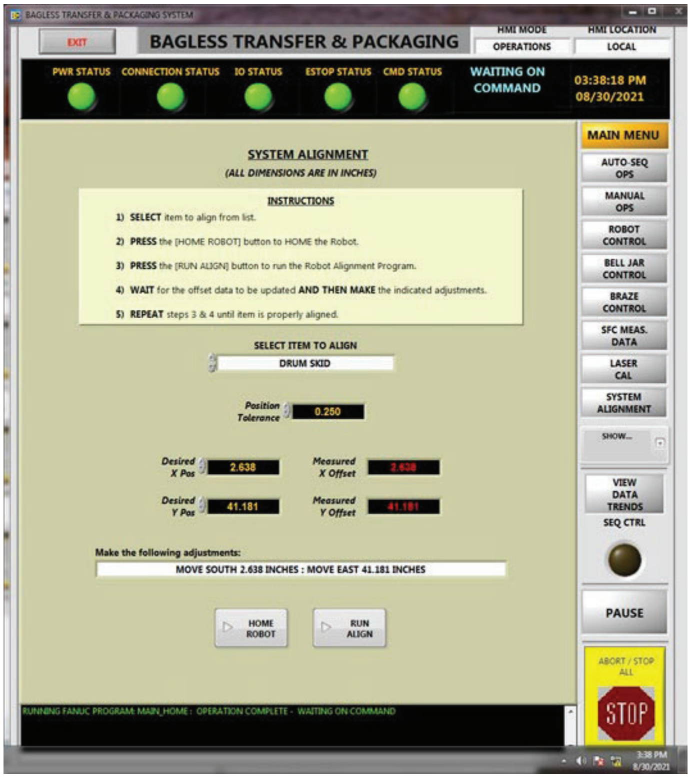
3.7.1 Field Preparations

Prior to performing the bagless transfer and packaging operations several items must be prepared as called out in procedure L30-5.11. For the cold run, the following field preparations were completed:

- The SFC was inspected, assembled and laser distance measurements performed. The required documentation was completed.
- Argon gas was turned on and regulators adjusted for the specified operating pressures.
- A Mk-Vial was introduced into Cell 9 and the serial number verified.
- The required items to prepare room E-081 for operation were gathered.
- Entry was made into E-081 where the BTPS equipment is located. The following preparations were completed:
    - RPD survey equipment was staged per RPD direction.
    - BTC plug and plug loading fixture were placed in a transfer port for supply entry into the cells.
    - All required equipment was powered-up as required.
    - System component locations and orientations were verified.
    - System Argon Pressures were verified.
    - The SFC without cap and plug was placed into its holder inside the shield box.
    - The SFC plug with the larger end up was placed in its holder on the Robot Skid Tabletop.
    - The previous SFC Cap knob was removed from the Reaction Bar.
    - Lubricant was applied to the SFC Cap and the SFC Cap was secured to the Reaction Bar.
    - The Reaction Bar was placed in its holder on the Robot Skid Tabletop.
    - New Swipes were placed on the Swipe Tools.
    - The BTC was placed in position in the Can Loading Module.
    - A new blade was installed on the Can Cutting Module.

3.7.2 Location of the Mk-18A Type A Drum

The final field preparation required for the BTPS operation while personnel are in E-081 is the location of the Mk-18A Type A Drum. For the Cold Run, the drum was already located in-place. The drum location was verified by a remote operator utilizing the BTPS HMI located in E-004. First, the remote operator ensured that the BTPS HMI was operational and ready for BTPS operations. The BTPS HMI contains a utility shown in Figure 3-24 that uses the BTPS Robot and 3D camera to determine if any adjustments of the drum's location are required for remote operation. The operator used the provided utility to verify the drum's location. No adjustments were required.



HMI MODE: OPERATIONS
HMI LOCATION: LOCAL

WAITING ON COMMAND
03:38:18 PM
08/30/2021

SYSTEM ALIGNMENT
(ALL DIMENSIONS ARE IN INCHES)

INSTRUCTIONS
1) **SELECT** item to align from list.
2) **PRESS** the [HOME ROBOT] button to HOME the Robot.
3) **PRESS** the [RUN ALIGN] button to run the Robot Alignment Program.
4) **WAIT** for the offset data to be updated **AND THEN MAKE** the indicated adjustments.
5) **REPEAT** steps 3 & 4 until item is properly aligned.

SELECT ITEM TO ALIGN: DRUM SKID

Position Tolerance: 0.250

Desired X Pos	2.638	Measured X Offset	2.638
Desired Y Pos	41.181	Measured Y Offset	41.181

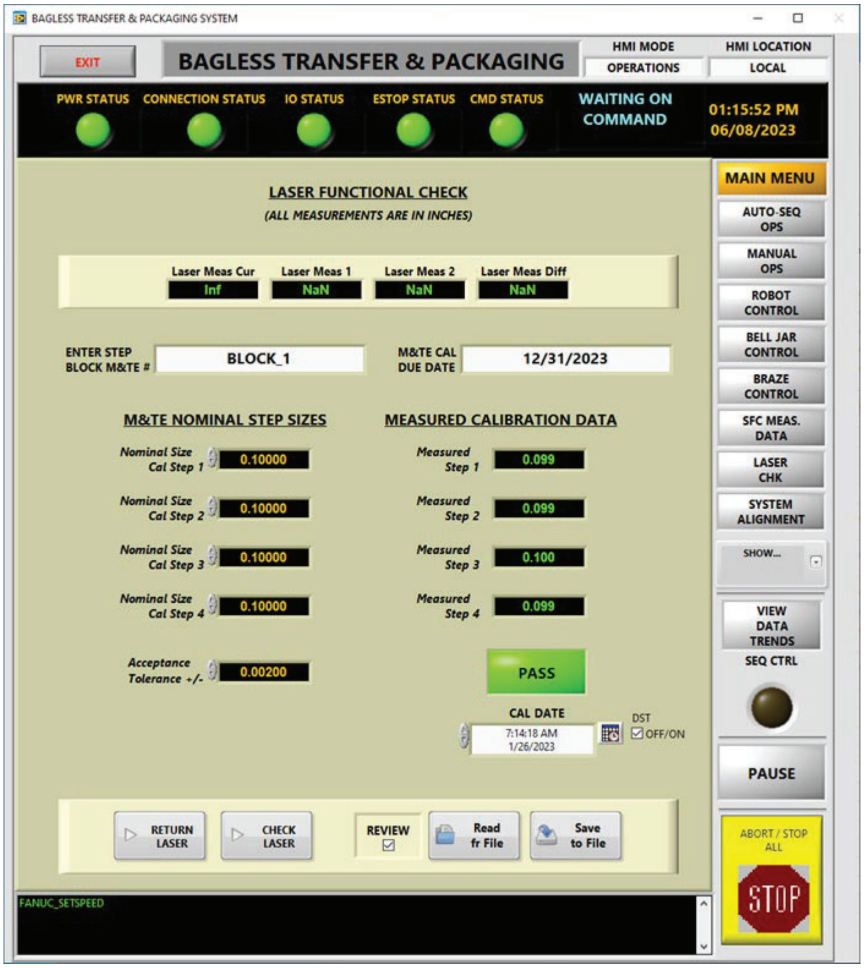
Make the following adjustments:
MOVE SOUTH 2.638 INCHES : MOVE EAST 41.181 INCHES

```
RUNNING FANUC PROGRAM: MAIN_HOME :  OPERATION COMPLETE -  WAITING ON COMMAND
```

Figure 3-24. BTPS HMI System Alignment Utility

3.7.3 Laser Function Check

The final field preparation for the BTPS operation is to perform a Laser Functional Check. The laser is used to make quality assurance measurements of the SFC once the BTC and SFC Plug are placed in the SFC and the SFC Cap torqued into place to close the SFC. An M&TE calibrated step block is located on the Robot Skid shielded enclosure. Step measurements of the calibrated block are taken to verify proper operation of the laser prior to closure of each SFC. The BTPS HMI contains a utility shown in Figure 3-25 that the BTPS operator utilizes to verify that the laser's performance is maintained within the required tolerance. For the Cold Run, the operator performed the Laser Functional Check and found the laser to be within the required tolerance.



BAGLESS TRANSFER & PACKAGING SYSTEM

HMI MODE: OPERATIONS
HMI LOCATION: LOCAL

WAITING ON COMMAND
01:15:52 PM
06/08/2023

LASER FUNCTIONAL CHECK
(ALL MEASUREMENTS ARE IN INCHES)

Laser Meas Cur	Laser Meas 1	Laser Meas 2	Laser Meas Diff
Inf	NaN	NaN	NaN

ENTER STEP BLOCK M&TE #: BLOCK_1
M&TE CAL DUE DATE: 12/31/2023

M&TE NOMINAL STEP SIZES		MEASURED CALIBRATION DATA	
Nominal Size Cal Step 1	0.10000	Measured Step 1	0.099
Nominal Size Cal Step 2	0.10000	Measured Step 2	0.099
Nominal Size Cal Step 3	0.10000	Measured Step 3	0.100
Nominal Size Cal Step 4	0.10000	Measured Step 4	0.099

Acceptance Tolerance +/-: 0.00200

PASS

CAL DATE: 7:14:18 AM 1/26/2023
DST ☑ OFF/ON

REVIEW ☑

FANUC_SETSPEED

Figure 3-25. BTPS HMI System Laser Functional Check Utility

3.7.4 Loading the Bagless Transfer Can

The first step of the bagless transfer process was to ensure the bagless transfer can (BTC) Plug with braze rings, the Plug Loading Fixture, and the MK-Vial with the correct serial number were all located in Cell 9. After verifying these, the new BTC was loaded into the sphincter seal port at the back of Cell 9. This was done by executing the LOAD LOAD-MOD on the BAGLESS TRANSFER & PACKAGING HMI. This initiated the FANUC robot to place the can loading module (CLM) onto the Linear Actuator Drive (LAD), and the LAD drove the module to insert the BTC into the sphincter seal, pushing out the previous BTC's remnant. It was necessary to use the manipulator to catch the remnant from falling below the cell tabletop when pushed out by the new can.

The MK-Vial was then placed into the BTC with the cap end first (toward the rear of the cell) and pushed to the end of the BTC using a ruler as a poker. The plug loading fixture, which holds the BTC plug, was placed into the BJIC stand which holds the bell jar. Using the HMI, the pneumatic actuator on the bell was extended. The HMI will display that it is waiting for a command when the sequence completes, and it is good to move on to the next step. The plug loading fixture was then removed, and the plug remained attached to the bell jar. Due to previous tests showing that the removal of the plug loading fixture can cause the plug to become skewed, a second extend step was added to the procedure. This step had the same wording as the previous extend step and was not being performed due to confusion on it already being performed. Due to this, the second extend step in the procedure was reworded and made clear that it was for a second time. The plug was then retracted using the HMI and observed to stay properly oriented.

The bell jar was lifted out of the BJIC stand, placed into position for brazing, and secured using the DESTACO clamps. Originally, to move the bell jar around the cell, it was required to use the lift assist. After observing how the crane worked in the cell, it was decided to allow use of the crane as well. This created less weight being held up by the manipulators and more control, which is crucial to ensure the plug does not become skewed. The STORE LOAD-MOD was executed using the HMI once the bell jar was secured in place. While this sequence was executed, the radial seal pressure and BJIC vacuum pressure did not change. Initially it was thought that the there was an issue with the Argon supply, but since the HMI displays stayed steady without any fluctuation due to signal noise, it was determined to be a communication error between the gauges and the HMI. To fix this, the I/O reset on the WMP & REM IO CTRL PNL in E-023 was pressed. This fixed the communication error but reset the pressure being supplied to the radial seal. If the radial seal was repressurized without one of the modules supporting it, there is a chance that the BTC could become skewed. Therefore, the loading module was reloaded and the process to store the load module was started again. To not run into this problem in the middle of a run, an I/O reset was added to the beginning of the loading operations.

3.7.5 Brazing the Bagless Transfer Can

Loading the braze module is the next step of the sequence after the load module is stored and the BJIC bell jar pressure indicates less than 600 mTorr. It was executed on the HMI under the LOAD BRAZE-MOD sequence. This initiated the FANUC robot to place the braze module onto the LAD which then drove the module to the cell wall. It is necessary to ensure the braze module is located over the can correctly to ensure a proper braze. This was previously done by ensuring the springs on the braze module compressed slightly using the cell insert cameras, but the springs are no longer visible. Rather, the braze module was confirmed to be in the right position by checking the LAD position on the HMI.

The next sequence is prep for braze in which the BJIC and BJOC go through 5 cycles of evacuation and argon backfilling. At the end of these cycles, the BJIC pressure needs to be under 200 mTorr. A few things were noticed when running the cycles in the cells. Since the environment was different than the mock-up in 716-4A, the vacuum takes a little longer to reach the correct pressure. A note was added to the procedure saying that there may be a wait time for the BJIC pressure to reach under 200 mTorr, and the time required to start the braze in the programming was extended to an hour. Another thing of note was when running with the different simulant it was difficult to reach 200 mTorr. The process continued to the next step with SME approval, and the braze was evaluated at the end. It was determined that if the pressures were below 300 mTorr, the procedure could continue.

At the end of prep for braze, the braze can sequence was executed. This started the braze module and heated the BTC. In one of the runs, the HMI skipped straight to the cool down count down which happens at the end of the braze, but no braze occurred. It was noticed at this point that the Braze Can screen showed that there was a fault for the brazing unit. Looking further into the fault, the brazing unit control screen showed that the bus voltage for the brazing unit was low. It was necessary at this point to look at the braze unit to see if the breaker was tripped; upon further investigation, that ended up being the problem. It is unknown if the breaker was tripped to begin with or if something caused the breaker to trip. One way to prevent this problem is to check the braze unit's faults before starting the braze sequence.

After the cool down at the end of the braze can sequence completed, the bell jar was removed. The in-cell camera was then used to view the inside of the BTC to confirm a braze puddle was present. All the runs that brazed produced a puddle and therefore a successful braze. The bell jar was then placed back into the position for brazing operations, so the braze module could be stored. The E-081 cameras were used to ensure all the modules were located back on the brazing cart. The cell insert cameras were used to verify the BTC was not melted.

3.7.6 Cutting the Bagless Transfer Can

The step following the brazing operations was to load the can cutting module (CCM). This was executed on the HMI, and the FANUC robot put the CCM onto the LAD which then positioned the CCM over the BTC for cutting. The cell insert cameras were used to ensure the CCM was in the correct position for cutting. The cut can sequence was then executed which starts the rotary chuck that holds the cutter. The chuck moves into the can for 10 minutes to cut and then back out of the can for the same amount of time to get out of the way. During one of the runs, the rotary chuck stopped moving towards the can while it was in the middle of a cut. To finish fully cutting the can, the rotary chuck was manually backed out a few rotations through the HMI. The cut can sequence was restarted and was able to complete without stopping. Once the can was cut and confirmed cut through using the cell insert cameras, the STORE CUT-MOD sequence was executed. This backs the CCM up a bit to see the can separate from the remnant. The BTC state was then changed to CUT on the HMI, the sequence completed, and the CCM was placed back onto the brazing cart.

3.7.7 Packaging the Bagless Transfer Can

After the brazed BTC was cut and the CCM was placed back in its home position with the cut can inside, the system was ready for packaging the can. The PKG CAN sequence was executed from the HMI, and the automatic sequence was started. The FANUC robot was observed retrieving the BTC from the cutting module and placing the BTC inside the SFC. The SFC plug was then placed inside the SFC, and the robot used the 3D vision sensor to verify that the plug was placed inside the SFC properly. The "PLUG VERIFIED" indication that the plug was properly placed inside the SFC was shown on the SFC MEASUREMENT DATA screen on the HMI.

The robot was then observed placing the SFC cap onto the SFC to thread the cap on until it torqued off. After the SFC finished rotating, the robot paused due to an excessive disturbance alarm. Initially it was unclear what caused the excessive disturbance, but the cause was determined to likely be due to the SFC cap not starting to thread into the SFC quick enough. As the SFC was rotating, the SFC cap was noticed to be moving up and down. This motion is normal until the SFC cap begins to thread into the SFC. The robot moves down at a very slow speed to accommodate the threading of the SFC cap, but if the SFC cap takes too long to start to thread, then it is possible for the robot to run into the top of the cap and experience too much force. This has also happened while testing if the SFC cap was not lubricated enough and the threads get crossed before threading down all the way. This causes the cap to be too high and even though the cap may torque off, the robot continues to move down and run into the SFC cap. While the system was in a paused state, it was unclear whether the SFC cap was fully torqued off or if the cap was threaded into the SFC and still connected to the cap holder held by the robot. To determine if the cap was torqued off, the SFC was rotated manually by jogging the Lid Shear Drive counter-clockwise. This was determined to be the best action because the cap holder was still supported by the lead shielding. While the SFC was rotated manually, the cameras were used to watch the SFC cap to determine if the cap was spinning with respect to the reaction bar. If the SFC cap spins with respect to the reaction bar, then the cap can be determined to be torqued off. The sequence was then aborted and the PKG CAN sequence was executed again while making sure to adjust the system states to indicate that the ROBOT ITEM STATE was 'SFC CAP HOLDER' and the SFC CAN STATE was 'CAP ON.' Making these changes to the system states was necessary because the sequence was allowed to continue in the correct order which was returning the cap holder.

After restarting the PKG CAN sequence with the correct system states, the robot was observed returning the SFC cap holder to its home position. Using the overview cameras, the SFC cap was observed to be sheared off and looked normal. The robot then retrieved the laser and positioned it above the SFC to begin the SFC cap measurement steps. The HMI showed that the system was paused, and the operator was directed to rotate the Lid Shear Drive manually so that the red dot from the laser was aligned with the scribe mark on the SFC rim. After rotating the SFC and aligning the laser with the scribe mark, the sequence was resumed and the system state for SFC CAN STATE was changed to 'RIM ALIGNED.' The robot was then observed moving lower to take the first laser measurement of the SFC rim. This process was then repeated for the measurement of the SFC cap scribe mark.

After making the laser measurements on the SFC rim and SFC cap, the robot closed the table so that the SFC was shielded and the cap measurements could be compared to the measurements recorded on the SFC Traveler Sheet. The SFC cap measurement data was recorded on the SFC MEAS DATA screen on the BTPS HMI, and the data was saved. The gap measurements were verified to meet the necessary criteria, and the sequence was resumed with the SFC CAN STATE changed to 'CAP VERIFIED.'

When the sequence resumed, the robot was observed opening the table, and the SFC top, side, and bottom were swiped for contamination. The table was then closed again by the robot, and the swipe tray was positioned behind the robot for removal of the swipes. At this point, RPD entered E-081 and pressed the emergency stop button on the FANUC robot controller so that the surveys of the swipes could be performed. The top, bottom, and side swipes were removed from the swipe tray and the swipes were discarded. The emergency stop button was then released before RPD exited the area, and the sequence was resumed with the SFC CAN STATE changed to 'SWIPE VERIFIED.'

Once the sequence was resumed, the robot then returned the swipe tray and opened the robot skid table. The robot attempted to remove the shield plug from the Type A drum, but the sequence paused with an indication that the robot could not find the item it was looking for. The 3D vision system was unable to find the shield plug due to lighting discrepancies with the taught reference image. The solution was determined to be re-teaching the vision program for the shield plug. Once this was complete, the sequence was resumed, and the robot was observed retrieving the shield plug from the Type A drum and placing it on the robot skid table. The robot then picked up the SFC, placed it inside the drum, and returned the shield plug to the drum. While the robot was returning the shield plug to the drum, the dose readings near the drum were recorded using the remote monitoring devices placed near the drum. The robot was then observed returning the 4-jaw gripper and moving to the home position. At this point, the packaging process was complete and the drum was ready for reassembly.

3.8 Type A Drum Loading

The Mk-18A Type A Drum is what is used to ship the SFC to ORNL. The procedure used for the Type A Drum, L30-5.12, is integrated with the BTPS procedure L30-5.11 since the BTPS loads the drum with material. L30-5.12 is used in conjunction with the Handling and Operating Instructions for the Type A Drum, G-HOI-G-00021. The Type A drum was disassembled by hand and inspected as required by L30-5.12 and G-HOI-G-00021. The markings on the drum were verified to be correct and the PO and Serial number were recorded in L30-5.12. The securement ring was verified to be snug against the sidewall of the drum. The turnbuckles and associated hardware were verified to be present and snug tight. The drum body was inspected for damage and rust/corrosion. The drum closure ring parts were verified to be in place and in good condition. The removal of the TID seal was simulated since there was no TID seal on this drum. This drum was a new and radiologically clean drum when sent to SRNL to use for testing/demonstrations, so no TID had been applied yet. RPD was requested to survey the drum during the disassembly process. The closure ring bolt and jam nut were removed and inspected for integrity. The closure ring was removed and then the closure lid was removed. The lid gasket was inspected for cuts, tears, rips, fold over or deep creases and that it was correctly installed on the drum outer lid curl. The

HDPE top shield was removed and inspected followed by the plywood spacer. The 12 pipe lid closure bolts were removed and inspected for any damage. The pipe component lid was removed and inspection of the Nuclear Filter Vent (NFV) filter and the O-ring were performed. The HDPE Upper Inner Shield was removed and inspected. The lead shield plug was removed for inspection and to verify the HDPE inner shield and lift basked were installed in the drum and showed no signs of damage. The lead shield plug was re-inserted in the drum. Once the disassembly and inspections were completed, the Type A drum was moved to E-081 and staged next to the BTPS robot skid. A special lifting adapter was attached the lead shield plug eyebolt to allow the BTPS robot to interface with the lead shield plug for removal and installation.

At this point, Procedure L30-5.11 was initiated to prepare a SFC for placement in the drum. Once the SFC was placed into the Type A drum and the lead shield plug installed by the robot, Procedure L30-5.12 was resumed to assemble the remaining components of the drum by hand. Following L30-5.12 and G-HOI-G-00021 the drum was reassembled in the reverse order it was taken apart. First, the HDPE Upper Inner Shield was installed followed by the pipe component lid. The pipe component lid bolts were lubricated with Krytox lubricant and torqued to 65 ft-lbs in the pattern specified in the HOI. Next, the top plywood spacer was installed followed by the HDPE top shield. The drum lid was installed, being careful of the placement and condition of the lid gasket. The lid closure ring was then installed, and the closure ring bolt torqued to between 55-60 ft-lbs. The jam nut was then threaded snug tight against the smooth bore lug, and a TID was simulated to be installed in the hole in the bolt. The TID was simulated to prevent using and destroying a TID seal just for this Cold Run. RPD performed surveys during the assembly of the drum and provided final survey data to the HMTR for a simulated shipment. The only issue identified during this evolution was the difficulty of trying to work from two procedures to complete the operations. Moving from one procedure to the other to perform steps was cumbersome and created some confusion of which step the operator was trying to complete. It was recommended to move the HOI steps into L30-5.12 which would eliminate having to work from two procedures, and it would help streamline the process.

4.0 Conclusions and Path Forward

All equipment necessary to carry out the Mk-18A operations was successfully installed in the shielded cells facility in 773-A and tested using surrogates and cold chemicals. Issues were identified during the cold runs, including equipment issues as well as technical challenges. Many of the issues were rectified during performance of the cold runs, and the remaining have a resolution identified. Table 4-1 provides a summary of all issues identified during the cold run operations, the status, and identified resolutions to outstanding issues.

Table 4-1. Summary of Issues Identified During Cold Run Operations

Issue	Status	Resolution
Equipment Checkout/Water Runs		
Quick connect for PW addition to TK-101 not allowing flow.	Able to add water through chemical addition port to complete water runs.	Quick connect to be replaced
Level Elements LE-060 (TK-107), LE-090 (TK-103B), LE-160 (TK-110) failure	Repaired by remaking connection at cable box.	Resolved
TK-108 Level Element LE-150 failure	Replaced prior to acid simulant processing.	Resolved
P-040 leak	Pump replaced	Resolved

Issue	Status	Resolution
Green deposition on Reillex column	Disappeared at beginning of acid contact.	Resolved
TK-300 supply line leak	Repaired prior to acid simulant processing.	Resolved
Cask Receipt and Target Resizing		
Containment Bags were very difficult to install/remove from the ends of the cask which increases time in high dose area.	Process was demonstrated successfully but raised ALARA and safety concerns.	Resolved: Use of containment bags has been eliminated. Procedure L30-5.10 has been updated to incorporate this change.
An argon leak in the manifold connected to the CITB leg.	Held enough pressure to work for the cold run.	Enter E-079 and perform troubleshooting to determine if the leak is in the manifold or the cell insert seal. Repair or replace as needed.
The Cask sensor, located on the CITB, is too far from the Cask to get the appropriate distance reading.	Able to sense the cask for the cold run	Resolved: Placed a spacer behind the sensor to move it closer to the Cask.
Mk-18A bundle was pushed too far into the cell.	Bundle was pulled back through the cell insert to the OC First Cut position to resume Cold Runs	The bundle length in the software had been changed to accommodate an alignment test bundle that was shorter. The program had not been updated for the Cold Run dummy bundle. The software has been updated with the correct length. This will not happen during actual operations as all the Mk-18A assembly lengths are the same and there will be no changes to the software, without approvals, once all testing and demonstrations are complete and the software is locked down.
During a second demonstration the Sacrificial Plug bound as it entered Cell 7.	Able to use the manipulators in Cell 7 to realign the Sacrificial Plug.	Inspect Sacrificial Plugs and determine if additional deburring/smoothing of edges is needed. To further prevent re-occurrence, the Sacrificial Plug will be pushed further into the cell insert sphincter seal at the end of target resizing. This should center the plug to reduce the chances of binding when the target or dummy bundle pushes it into the cell.
During cutting of the IC bail end, the bail end was not gripped completely and rolled as the saw blade tried to engage.	The operator and SME were able to complete the cut using manual controls on the Cutter System HMI.	Information and notes have been added to the procedure to give the operator flexibility to utilize the HMI controls available to recover from events like this.
During last two cuts of the simulated target, the saw paused cutting near the end of the cut just prior to cutting completely through the target.	The operator and SME were able to complete the cut using manual controls on the Cutter System HMI.	Resolved: A thicker spacer on the anvil type device that is used to secure the target for cutting has been fabricated, installed and tested.
Water line to the saw came loose during cutting evolutions.	Hose was temporarily reattached to continue the cold run.	Resolved: After completion of the cold run fittings were tightened down to ensure a good connection.

Issue	Status	Resolution
Low circulation flow was noticed during the fines vacuuming evolution. A kink in the suction line from the catch pan to the hydrocyclone was discovered, causing the accumulation of fines in the line.	The line was disconnected and cleared to allow cold runs to continue.	Resolved: The entire line and pump unit was replaced after cold runs completed to eliminate the kink.
Caustic Dissolution		
Transfer paths of chilled water from TK-123 to TK-215 were found to be swapped.	Discharge lines on P-050 and P-060 were changed to the correct configuration prior to proceeding with caustic dissolution.	Resolved
Pump flow totalizers not in agreement with level element indication of volume transferred.	Level elements were used as the basis for volume transferred.	Issue may have been due to communications issues with the software. The communication issues have been resolved, and accuracy of the pump flow totalizer functions will be monitored on future runs.
Release of small amount of liquid/mist from V-110.	Change order of valve closing post-backpulse to avoid residual pressure in TK-121.	Resolved: Changed order of valve closing in R&D directions.
Acid Dissolution		
Unable to achieve target [HNO_3] concentration in the acid dissolver.	Due to the number of backpulses performed, the starting volume in TK-235 was 7.4 L leaving room for only 3.8 L of concentrated acid to be added to the dissolver.	Resolved: In future runs, only a single caustic backpulse will be performed prior to moving to an acidic backpulse if required. Caustic rinse of the filter post-acid backpulse will be transferred to the HAD collection tanks rather than TK-235.
Pump flow totalizer not in agreement with level element indication of volume transferred.	Level element was used as the basis for volume transferred.	Issue may have been due to communications issues with the software. The communication issues have been resolved, and accuracy of the pump flow totalizer functions will be monitored on future runs.
Gelatin solutions began to set up prior to introduction into the acid dissolver due to unusually low temperatures.	Unclear of impact this may have on filtration.	Additional R&D being performed to optimize gelatin addition, including examining the possibility of adding gelatin as a solid.
Effectiveness of gelatin strike unclear.	Sodium metasilicate added as a surrogate for additional Si expected in the irradiated targets. No change in soluble [Si] observed before and after gelatin strike.	Additional R&D being performed to optimize gelatin addition.
LabView labels for solenoid valve V-300 were found to be incorrect.	Solids collected on FLT-315 versus the expected FLT-310.	Resolved. Labels have been corrected in LabView.
Potential solids breakthrough or bypass of FLT-315.	Downstream fouling issues indicated potential solids present after FLT-315.	Alternate filters and/or changes to the filter housing are being evaluated to avoid possibility of solids bypassing filter.

Issue	Status	Resolution
Difficulty in filtering samples in-cell.	Samples filtered in hood.	Evaluating alternate filtering options for in-cell filtrations (filtered centrifuge tubes or screw-driven piston for syringe filters).
Nitric Acid Transfer into Cell		
Initial attempt to transfer acid through the KAPL sleeve into TK-107 was unsuccessful. It was determined the transfer line and vent connections had been inadvertently swapped.	Connections were color coded to prevent reoccurrence.	Resolved
TK-107 level element (LE-060) stopped working due to corrosion from concentrated acid fumes.	The functioning was restored a couple of times during cold runs by cleaning the UT sensor.	Resolved: The sensor has been replaced with a new holder design that allows for rotating the level sensor off the tank (replacing with a cap) when measurements are not actively being taken. This will extend the life of the sensor by minimizing exposure.
Ion Exchange Operations		
Target flowrates unachievable for Reillex INEX column.	Upflow operations temporarily improved flowrates but were observed to decrease with time as additional volume of feed was processed.	Additional characterization of the resin is being performed to determine the source of fouling, but it is likely a result of filtration issues noted above (solids breakthrough or bypass of FLT-315). This issue expected to be resolved with resolution of the filter bypass issue.
Measured acid concentrations lower than expected.	TK-106 target nitric acid concentration was 8 M. Free acid titration result indicated 5.9 M. Ion chromatography analysis indicated 7.9 M nitrate.	A lab prepared standard of known acid concentration will be submitted along with all future sample sets. Additional troubleshooting of the method is needed to determine the cause for discrepancy in some samples.
Low transmitted raw light intensities through UV-vis flowcells.	Sufficient light transmission for cold runs.	Potential to improve light transmission through optimization of instrument set up and arrangement.
Periodic xenon light loss during UV-vis measurements.	Attributed mainly to instrument software real time issues.	Further testing of the instrument hardware and software are being explored to resolve this issue.
DGA Drying, Volume Reduction, and Calcination		
Upon initiation of argon flow to the rotameter, the flow was observed to spike and then drop to zero when the regulator and valve were opened. This suggests a faulty or leaking rotameter.	Rotameter was replaced.	New rotameter needs to be secured in such a way that it remains vertical, and tubing remains clear of potential hot surfaces during operations.
Control valve for argon into the rotameter failed (handle came off).	Resolved with replacement of the rotameter and tubing between the KAPL sleeve and inlet to the DGA column upflow piping.	Resolved

Issue	Status	Resolution
Difficulty aligning catalytic converter within the bucket assembly.	Assembly removed from the top of the furnace and internal alignment of the catalytic converter in the bucket was achieved.	Upon future equipment replacement, the catalytic converter should be attached to the bucket so that it is one integrated piece and does not require alignment of the catalytic converter in the bucket in the cells.
Quad-plug that powers the vacuum pump was found to be mislabeled.	Alternate switch from what is indicated by the color coding was flipped and rotameter on the vacuum pump was used to verify operation.	Quad-plug to be relabeled.
Failure of top heating zone of tube furnace.	Proceeded with middle and lower zones operating.	Based on results of the cold run, modifications have been made to the DGA column to allow it to sit further into the middle zone of the furnace during heating. The heating profile may also be adjusted on subsequent runs to optimize the volume reduction.
Failure of tube furnace over-temperature controller.	Controller was bypassed to allow attended operations only.	Resolved: No unattended operation of the furnace permitted. This restriction is noted in the procedure and R&D Directions.
Incomplete volume reduction of DGA resin.	Slightly overflowed crucible for final calcination resulting in a small amount of loss.	Resolved: Redesign of column to allow it to sit lower in the furnace and adjustment of heating profile (due to failure of top heating zone) expected to resolve this issue. If volume is still too great for calcination tray, final calcination will be performed in batches. Calcination tray will also be placed in secondary container during transfer to allow for recovery of any material that escapes the tray.
Mesh screen completely degraded during calcination.	Likely contributed to higher-than-expected mass of product.	Resolved: Alternate screen designs have been fabricated and will be tested.
Insulating cover for catalytic converter band heater needed.	During cold runs, insulating ceramic batting was used and the Cell 8 right manipulator was tagged out to avoid risk of contacting the exposed leads.	Resolved: An insulating cover has been fabricated and will be installed prior to the next furnace run.
Bagless Transfer and Packaging System		
Difficulty achieving <200 mTorr in the BJIC.	Approval obtained from SME to continue.	Resolved: Braze was evaluated, and it was determined that if the pressure reaches <300 mTorr, the process can continue.
Breaker trip on the brazing unit.	Reset and braze performed.	Resolved: Determine if there is a braze unit fault prior to initiating braze sequence.

Issue	Status	Resolution
Rotary chuck stopped moving towards the can during a cut.	The rotary chuck was manually backed out a few rotations through the HMI and the cut sequence was restarted.	Resolved: The cutting speed has been reduced from 0.5 rps to 0.4 rps to prevent recurrence. In the event of recurrence the cutter will be backed out 10 revolutions and the sequence restarted.
Robot experienced an "Excessive Disturbance Alarm" and stopped moving while torquing off the SFC cap. The system paused during the 'INSTALL SFC CAP' sequence.	This is normally due to the cap threads not starting to thread quickly enough while the robot moves the cap down.	Resolved; The Lid Shear Drive was manually rotated in the counter-clockwise direction to verify the cap had already been torqued off. The sequence was restarted after updating the SFC state to 'CAP ON'.
Upon attempting to remove the shield plug from the Type A drum, the sequence paused with an indication the robot could not find the item it was looking for.	The 3D vision system was unable to find the shield plug due to lighting discrepancies with the taught reference image. The solution was determined to be re-teaching the vision program for the shield plug.	Resolved
Working from two procedures for disassembly/reassembly of the Type A drum was found to be cumbersome and created confusion.	Recommended to move the HOI steps into the procedure L30-5.12.	Resolved: Revision of the procedure, L30-5.12.

Despite the issues identified above, the cold runs provided the opportunity to perform each of the unit operations necessary for processing of a Mk-18A target and identified areas of improvement that will be implemented prior to receipt of the first target. A dummy target bundle was successfully transferred from L-area and into the SRNL shielded cells, including resizing. A surrogate quarter target (aluminum tube) was successfully dissolved, demonstrating dissolution of the aluminum cladding. Target material surrogates were successfully filtered from the caustic dissolver and backpulsed into the acid dissolver. Neodymium oxide was used as a surrogate of the target material, and this was successfully dissolved in the acid dissolver. Cerium oxide was also included as a surrogate, and as expected, did not dissolve in the acid dissolver, but provide insoluble solids to collect on the filter after acid dissolution. Issues were identified with the filter and housing post-acid dissolution and these are being addressed with modifications to the filter housing. The dissolved Nd^{3+} was partially processed through the Reillex column, but fouling of the column occurred due to inadequate filtration of the feed coming from the acid dissolution process as noted above. The Nd^{3+} was successfully loaded onto the DGA column, and calcination of the DGA provided a surrogate oxide product. This material was loaded in an Mk-vial and successfully transferred out the shielded cells using the bagless transfer process, and then loaded into a special forms capsule and Type A drum to conclude the evolution.

5.0 References

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Appendix A. Supplemental Data

Table A-1. Concentrations of TK-235 throughout dissolution

Sample	Hours after heating start	Hours after reaching 100 °C	Fe (mg/L)	Nd (mg/L)	Ce (mg/L)
Prior to heating	Day prior	N/A	232	347	0.23
Dissolution 1	2.20	0.58	278	396	0.31
Dissolution 2	3.10	1.48	253	368	0.30
Dissolution 3	4.12	2.50	262	372	0.31
Dissolution 4	5.10	3.48	255	360	0.31
Dissolution 5	6.23	4.61	269	379	0.33
Dissolution 6	7.37	5.75	264	375	0.33
Dissolution 7/Pre-gelatin strike	8.48	6.86	276	379	0.35

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