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Irradiation of 20L LEU Uranyl Sulfate Solution for Production of Mo-99

Experimental Operations and Facilities Division

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ABBREVIATIONS

ABO	alpha-benzoin oxime
AC	activated charcoal
Ag/C	silver-coated charcoal
AMORE	Argonne Molybdenum Research Experiment
ASE	Accelerator Safety Envelope
DU	depleted uranium
EOB	end of bombardment
FEP	fluorinated ethylene propylene
FMI	Fluid Metering, Inc.
FWHM	Full Width at Half Maximum
GCS	gas collection system
GDH	gas distribution hub
HC	hazard category
HDPE	high-density polyethylene
HPGe	high-purity germanium
HZO	hydrous zirconia
ICPMS	Inductively Coupled Plasma Mass Spectrometry
LEAF	Low Energy Accelerator Facility
LEU	low-enriched uranium
linac	linear accelerator
LMC	LEU-Modified Cintichem
LN_2	liquid nitrogen
MDA	minimum detectable activity
PE	polyethylene
PEEK	polyether ether ketone
PP	polypropylene
PSI	pounds per square inch
RFW	raw fission waste
RGA	Residual Gas Analyzer
RRF	Relative Response Function
SAD	Safety Assessment Document
SOF	sum-of-fractions

TSV	target solution vessel
VDG	Van de Graaff
XRD	X-ray diffraction

1 INTRODUCTION

Argonne National Laboratory, with support from the National Nuclear Security Administration's Office of Material Management and Minimization, is assisting SHINE Medical Technologies in the development of accelerator-driven production of ^{99}Mo using low-enriched uranium (LEU). Technetium-99m (^{99m}Tc), a daughter of ^{99}Mo, is a workhorse of nuclear medicine, used in approximately 40,000 medical testing procedures each day just in the U.S.

In 2015–2016, as part of its Phase I irradiation campaign, Argonne successfully demonstrated accelerator-driven subcritical fission of an aqueous LEU uranyl sulfate solution using an electron linac accelerator. In Phase I, a tantalum converter was used as the fast-neutron source with a maximum beam power of 10 kW, and the solution volume was limited to 5 L. This configuration generated a peak fission power density of 0.05 kW/L and production of 60 mCi-^{99}Mo/kWh-kg-^{235}U. Separation methods applied to purify ^{99}Mo showed that the product met the British Pharmacopoeia purity specifications. ~1 Ci of ^{99}Mo was sent to GE Healthcare, where it was separately loaded onto a DRYTEC™ ^{99m}Tc Generator, and the ^{99m}Tc product was tested using GE Healthcare ^{99m}Tc-based products: Myoview™ (kit for the preparation of ^{99m}Tc - Tetrofosmin for injection) and Ceretec™ (kit for the preparation of ^{99m}Tc Exametazime for injection). Quality-control testing performed on the reconstituted kits indicated the viability of ^{99m}Tc radiopharmaceuticals prepared using ^{99}Mo product solution produced at Argonne [1, 2].

In the Phase II irradiation campaign, several major modifications were made to allow for increased production of ^{99}Mo. Among these changes, the tantalum target was replaced by a depleted-uranium (DU) disc target, allowing an increase in power up to 20 kW, while the volume of 140 g-U/L solution was increased up to 18 L. These changes led to an increased peak fission power density of 0.3 kW/L, and to production of ~87.5 mCi-^{99}Mo/kWh-kg-^{235}U.

Changes to the gas-handling system in Phase II included relocation of the gas analytical system from the main irradiation vault to an adjacent room, to minimize radiation damage to components and electronics. The relocation of the gas analytical system led to a 5-min delay in data analysis of hydrogen and oxygen production in the target solution vessel (TSV). Rapid changes in hydrogen production were mitigated by slowly ramping up the linac power to avoid spikes in hydrogen concentration. Monitoring of the analytical system was moved from a space adjacent to the linac vault to the linac control room to avoid the high radiation fields caused by the fission gas moving through the manifold in the analytical enclosure.

Further modifications were made to the target solution monitoring and ^{99}Mo recovery gloveboxes to accommodate the increase in target-solution volume and ^{99}Mo production. The solution-monitoring and ^{99}Mo-recovery gloveboxes were replaced by a single large "recovery glovebox" that incorporated the functions of both gloveboxes from Phase I. The Phase II glovebox was built using 2-in.-thick carbon steel to provide extra shielding, and, wherever feasible, liquid lines were made of ¼-in.-O.D. 316L stainless steel tubing to allow a higher flow rate than in Phase I. Shielding was also added to the effluent collection vessels, verification tank, and column. Several sensors from Phase I (pH, turbidity, conductivity) were removed since they did not provide usable measurements. Also, the Phase II glovebox recovery system split the

solution-handling system into base-side and acid-side sections to eliminate uranium precipitation in the lines due to changes in pH. Splitting the system into two sides also prevented cross-contamination of the feed and effluent and the unnecessary dilution/neutralization of the target solution. The recovery column size was increased to 40×100 mm. The increased column size was required to handle the larger volume of target solution used in Phase II.

For the hot-cell concentration and purification operations, changes were made to the concentration-column setup. The volumes and flow rates of the system were increased to accommodate the larger volumes of solution being processed, and the length of the concentration column was increased from 1 cm to 1.5 cm, while the diameter stayed at 1 cm. The column loading speed was increased from 16 mL/min to 50 mL/min and the column stripping was increased from 25 mL at 4 mL/min to 66 mL at 11 mL/min. Additional safety features were added to the system, including a ¼-in. gas collection line connected to the receiving vessel for pH adjustment and a liquid trap installed between the gas collection system (GCS) and the connections inside the hot cell. Further, a shielded effluent-storage system with a connection to the GCS was constructed under the hot cell to store the effluent from the concentration column for fission-product decay and long-term storage. No modifications were made to the LEU-Modified Cintichem (LMC) process between Phases I and II.

The processing steps following irradiation are briefly described here: irradiated LEU uranyl sulfate solution (~18 L of 140 g-U/L solution) at pH=1 was mixed for several hours[1] and then loaded on a titania (TiO_2) primary recovery column by remote operation in the recovery glovebox to separate ^{99}Mo from uranyl sulfate solution. After the solution was loaded, the titania column was washed with pH=1 sulfuric acid and water, and then ^{99}Mo, along with reduced but not negligible amounts of other fission products, was stripped using 1 M NaOH. The strip solution was transferred to hot-cell operations for concentration and further purification. Approximately 2 L of solution containing ^{99}Mo in 1 M NaOH was received in the hot cell and then acidified to pH=2 using HNO_3. The solution was then loaded onto a titania concentration column, and the column was washed with dilute acid and water. The ^{99}Mo was then stripped from the concentration column in ~70 mL of 1 M NaOH. The alkaline solution containing ^{99}Mo was then acidified to ~1 M HNO_3 solution, and the LMC process was used for final purification. The product from the LMC process contained purified ^{99}Mo in ~50 mL of 0.2 M NaOH.

Phase II experiments are summarized in Table 1.1, and experimental results obtained from these experiments are discussed later in this report.

[1] Mixing was done to ensure a constant feed composition to the column. There was no mechanical mixing in the irradiation tank during irradiation.

TABLE 1.1 Phase II experiments

Experiment	End of Bombardment (date, time)	Chemical Processing
Commissioning run, no irradiation	8/14/19	Yes
Irradiation #1	10/1/19, 20:00	Yes
Irradiation #2	11/11/19, 02:00	Yes
Irradiation #3	3/2/20	No
Irradiation #4	8/30/20, 07:00	Yes
Irradiation #5	1/18/21, 8:00	Yes

References

[1] Youker, A.J., Chemerisov, S.D., Tkac, P., Kalensky, M., Heltemes, T.A., Rotsch, D.A., Krebs, J.F., Makarashvili, V., Stepinski, D.C., Alford, K., Bailey, J., Byrnes, J., Gromov, R., Hafenrichter, L., Hebden, A., Jerden, J., Jonah, C., Micklich, B., Quigley, K., Schneider, J., Wesolowski, K., Vandegrift, G.F., and Sun, Z., *Compendium of Phase-I Mini-SHINE Experiments*, ANL/NE-16/39, Argonne National Laboratory, October 2016. Available at https://publications.anl.gov/anlpubs/2017/01/131828.pdf

[2] Youker, A.J., Chemerisov, S.D., Tkac, P., Kalensky, M., Heltemes, T.A., Rotsch, D.A., Vandegrift, G.F., Krebs, J.F., Makarashvili, V., and Stepinski, D.C.. Fission-Produced ^{99}Mo Without a Nuclear Reactor. *J. Nucl. Med.* 2017; 58:514–517.

2 EXPERIMENTAL

2.1 LINAC IRRADIATION HARDWARE AND SAFETY CONSIDERATIONS

Mini-SHINE experiments (5-L uranyl sulfate solution irradiation or Phase 1 irradiations) and the Argonne Molybdenum Research Experiment (AMORE or Phase 2 irradiations) were performed using the high-current electron linear accelerator (linac) at the Low Energy Accelerator Facility (LEAF) at Argonne. This linac can provide electron-beam energies of up to 50 MeV and deposited power on a target of up to 30 kW. Mini-SHINE experiments use an electron/X-ray/neutron converter to generate neutrons that produce fission in the target solution. In Phase 1, the target solution was 90–150 g-U/L LEU uranyl sulfate at pH 1. The converter was a water-cooled solid tantalum slug; the maximum beam power on the converter was limited to 10 kW, and the target solution volume was limited to 5 L. This configuration generated a peak fission power density of 0.05 W/mL. In Phase 2, the solution was 145 g-U/L LEU uranyl sulfate at pH 1. The X-ray converter/photo-neutron target was an array of water-cooled DU disks, with the maximum allowable power on the target limited to 20 kW, and the solution volume was 18 L. This configuration generated a fission power density of up to 0.3 W/mL. Several reports have been published on mini-SHINE developments for phases 1 and 2 [1, 2].

2.1.1 Linac and Beamline Configuration

AMORE irradiations were conducted at the Argonne linac, which is an L-band (1.3-GHz) RF accelerator operating at a maximum (no-load) energy of 53 MeV. The maximum average beam power for this machine can reach 25 kW at 30 MeV beam energy. The average beam power as a function of beam energy for the linac at nominal operation parameters (20 kW peak RF power per klystron) is shown in Figure 2.1.1.1. For AMORE irradiation, we have chosen 40-MeV beam energy, because at this energy we can achieve maximum neutron yield with heat deposition limitations imposed by the current design of the DU target.

In the AMORE experiment, the accelerated electron beam is delivered to a photo-neutron target through a beamline that consists of several beam elements. The shielded enclosure housing the AMORE experiment is located on the 10-degree beamline. To direct the beam to the target, it is first deflected by 10 degrees to the right, then 10 degrees to the left, creating an offset from the zero-degree beamline. This beamline has two pairs of quadrupole magnets (focusing elements) and a pair of correction dipole magnets. Drawings of the beamline and the location of the shielded box housing the target and the vessel are shown in Figure 2.1.1.2.

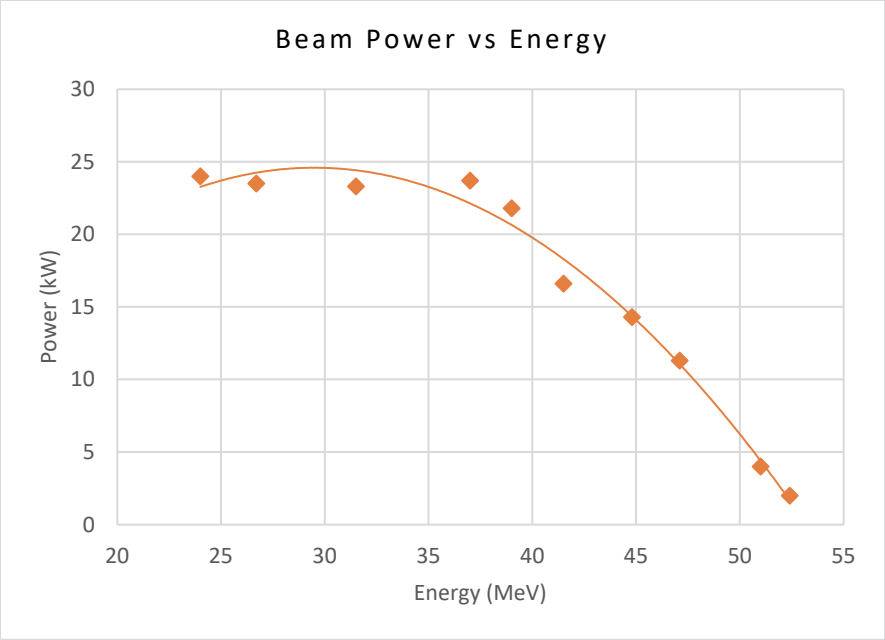

FIGURE 2.1.1.1 Average beam power versus beam energy for the linac. Measurements were taken at 20 kW RF power per modulator. The maximum average beam power, 25 kW, is achieved at a 30-MeV beam energy, 240-Hz pulse rate, and 5.5-μs pulse width.

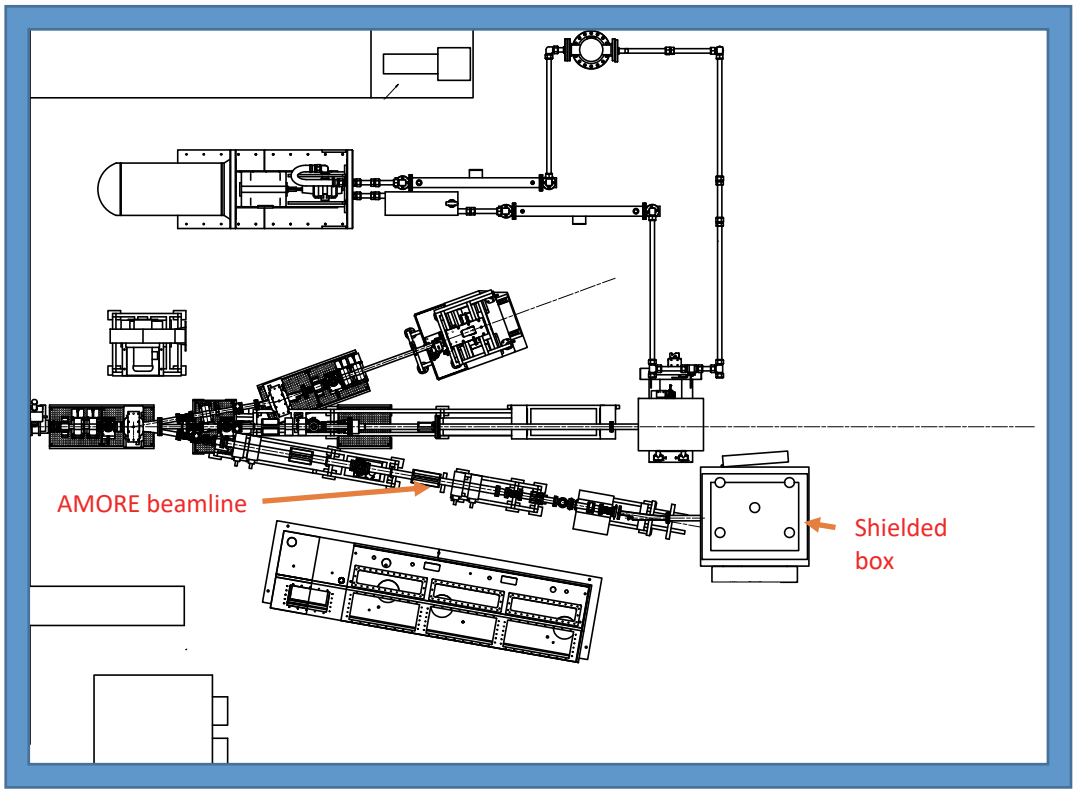

FIGURE 2.1.1.2 Beamline configuration for AMORE irradiations

Before each irradiation, the beam energy was tuned to 40 MeV; then the electron beam was transported through the first 10-degree magnet to the second, and beam losses in the beamline were minimized. After that, the second 10-degree magnet was turned on and beam was placed on the target window. With the beam on the target window, transport was optimized to achieve minimum loss and desired beam size on the target. The beamline is equipped with current monitors from Bergoz that allow us to measure the beam current leaving the accelerator, passing through the first 10-degree magnet and arriving at the second 10-degree magnet. Also, for initial tune-up, the second 10-degree magnet chamber is equipped with a through port with a window so beam can be brought out and beam shape and current can be measured at that point. We used optical transition radiation detection to observe the beam position and shape on the target. Generally, the minimum acceptable size of the beam on the target is limited by peak heat generation in the target disks and the coolant's ability to remove the heat without boiling on the surface. The target was designed to accept 20-mm by 20-mm Full Width at Half Maximum (FWHM) at 20-kW beam power. Most of the irradiations were limited to lower power, because of high hydrogen gas generation, so we used a smaller beam size for most irradiations.

2.1.2 DU Target Design

The target assembly (Figure 2.1.2.1) contains the DU target. The target consists of 21 Zircaloy-4-clad uranium disks, 2.1 in. in diameter, arranged in an array as shown in Figure 2.1.2.1. Of these disks, 11 are 0.232 in. thick, and 10 are 0.074 in. thick. The Zircaloy-4 cladding on the faces of the disks is 0.010 in. thick. The Zircaloy is metallurgically bonded to the DU; this bonding is critical in providing efficient heat transfer from the uranium to the coolant. The DU disks are arranged in a horizontal stack, with alternating spacers and flow orifices to facilitate water flow from the target cooling loop across and around the target disks to optimize cooling (Figure 2.1.2.2). The "stack" of DU disks and spacers is held in place and compressed by a spring housing at the back of the target, which also allows for thermal expansion of the disks during irradiation. The overall dimensions of the target assembly are 2.75 in. in diameter by 6.92 in. in length. The total quantity of DU in the target assembly is 6.01 lb. (2.73 kg). Several calculation notes on the target and cooling system designs are presented in Appendices 1–6.

The direction of coolant flow through the target assembly is shown in Figure 2.1.2.2. Coolant water enters at the bottom of the target housing from the cooling system at a design flow rate of nominally 40 GPM at a temperature of 65°F. The flow is directed across both faces of each disk by flow diverters, and disk spacing was designed to accommodate the calculated heat distribution throughout the target. The flow-channel gap between the disks is nominally 0.039 in. to provide a minimum average flow velocity of 22 ft/s in the gap for the worst-case (hottest) disk. This flow velocity is required to provide sufficient convective cooling at the full beam power of 20 kW while maintaining the temperature of the disk surfaces below the boiling point (212°F) of the coolant. The required flow for each disk is controlled by the fixed-orifice resistance at the inlet to the individual intra-disk channels. The coolant water exits the flow channels into the outlet manifold and then flows out of the target assembly back to the cooling system. The total required heat removal from the target disks at full beam power is 16.4 kW, which results in an ~3°F temperature rise of the coolant from inlet to outlet of the target assembly. Under these flow conditions, the average static hydraulic pressure in the target

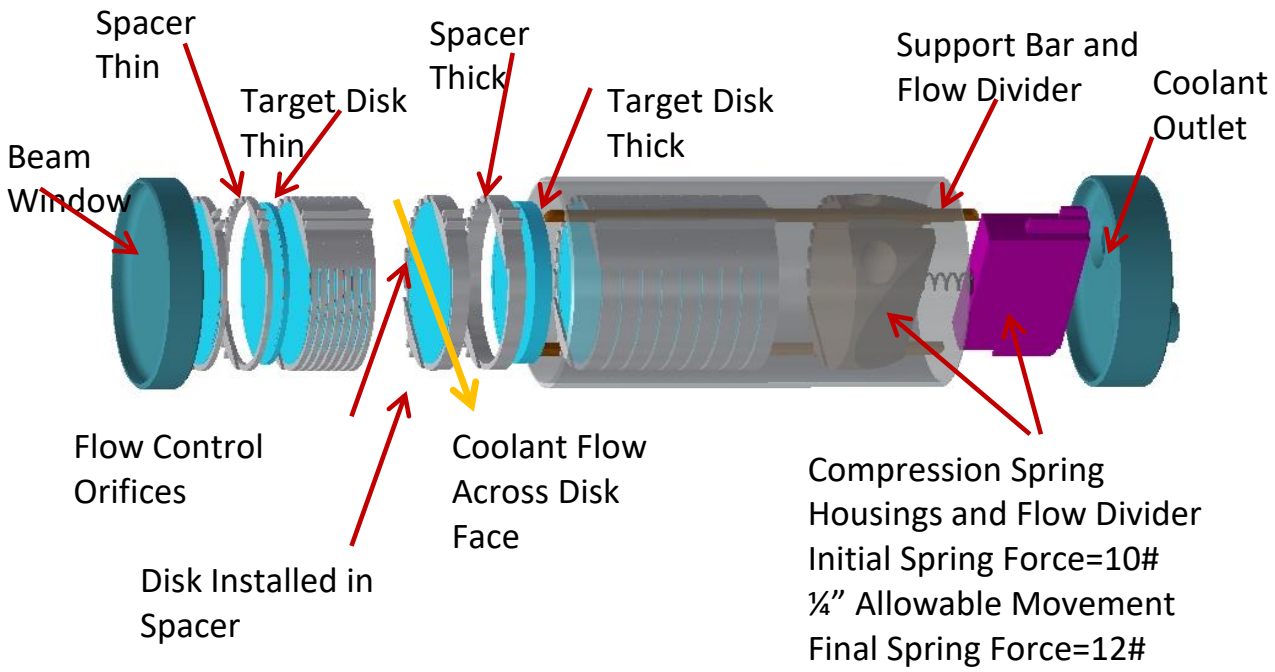

FIGURE 2.1.2.1 Depleted-uranium target assembly

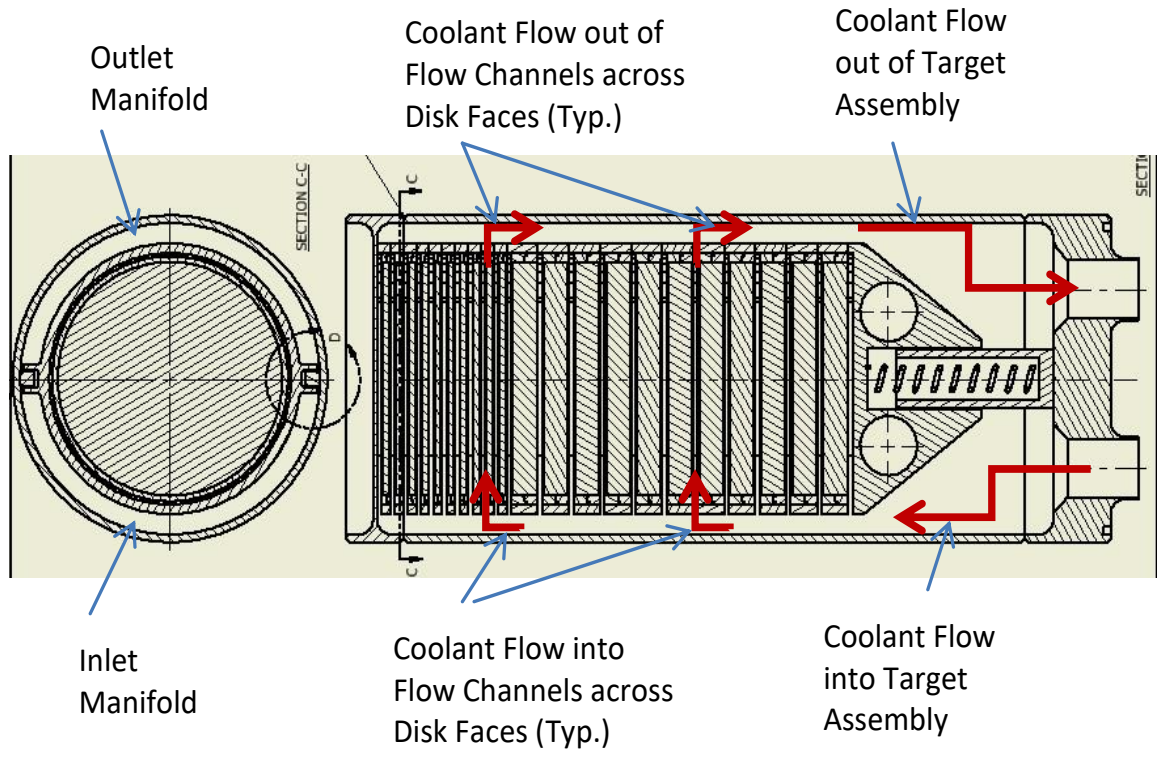

FIGURE 2.1.2.2 Coolant flow through the depleted-uranium target

assembly is 22 psig, with a pressure differential between inlet and outlet of 16 psig. At the maximum allowed disk surface temperature of 212°F, the maximum uranium temperature in the middle of the disks remains below 300°F, which prevents both significant grain growth in the uranium and excessive thermal stresses in the bonded Zircaloy-4 cladding. The general cooling-system piping and instrumentation diagram, which illustrates both the solution and target cooling loops, is shown in Figure 2.1.2.3.

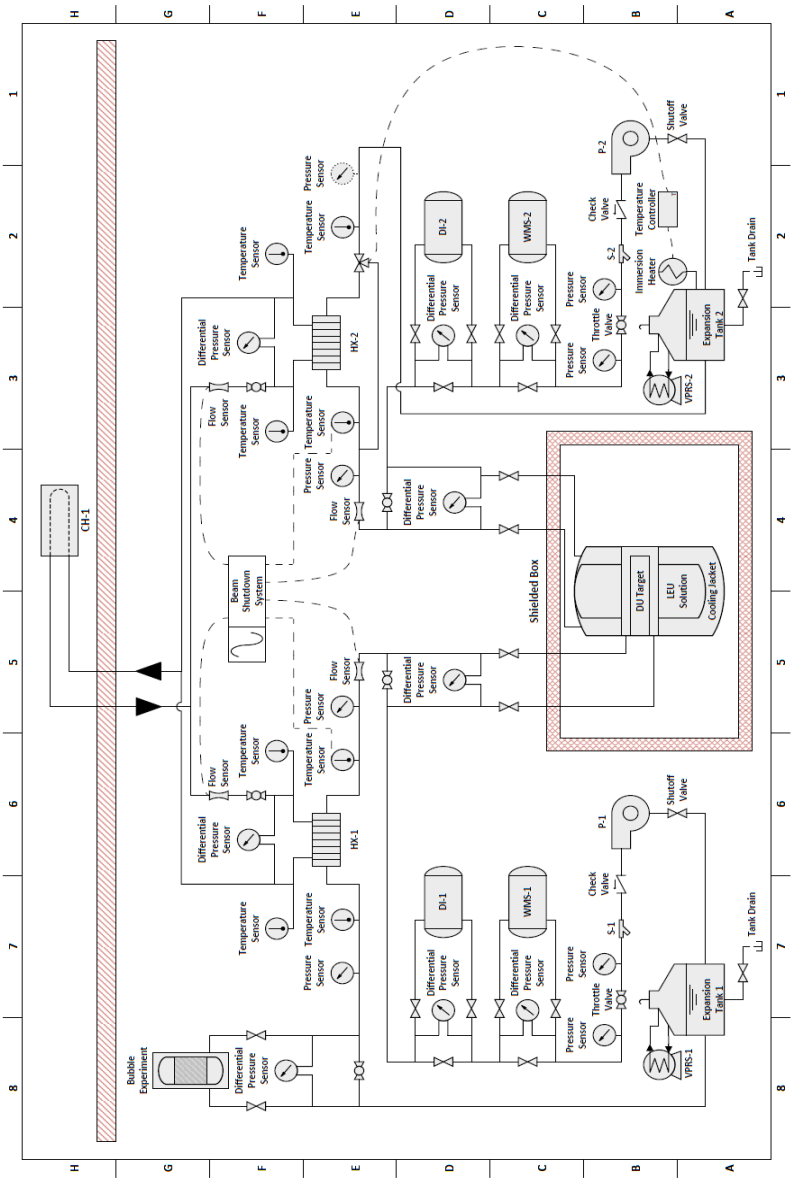

FIGURE 2.1.2.3 Overall cooling system piping and instrumentation diagram. CH1 is the chiller that supplies cooling. Water for the DU target and the vessel is cooled by exchange with this chilled water through Hx1 and Hx2, respectively

The installed cooling system is shown in Figure 2.1.2.4. The cooling system is placed inside an air-tight enclosure because of the possibility of fission products entering the cooling water if the Zirconium cladding on the DU disks is breached. The enclosure is connected to a HEPA/Silver Zeolite filtered exhaust system to prevent radioactive particulates and iodine from escaping.

FIGURE 2.1.2.4 Cooling system for target solution vessel and DU target

2.1.3 Target Solution Vessel Design

The TSV and all of its components are shown in Figure 2.1.3.1, and Figure 2.1.3.2 shows a cutaway view of the vessel and its position relative to the shielded box and DU target. Penetrations in the target vessel accommodate the incoming radiation beam, instrumentation (thermocouples), dry wells for inserting tubes containing smaller volumes of materials to be irradiated (mini-AMORE experiments), cooling loop connections, a drain tube extending to the bottom of the inner vessel for removal of the solution following irradiation, gas ports, and a 2-in. viewing port on top of the vessel. There are cooling loops for the target tube to remove the heat in the target generated by impingement of the accelerator beam, and for the outer water jacket. To reduce water losses during irradiation, gas ports inside the vessel are equipped with condensers. The vessel is positioned inside a hot cell. A helium cover gas is maintained over the LEU solution, and fission and radiolytic gases from the process are collected in a gas collection and analysis system connected to the vessel's gas ports. The completely installed TSV, DU target, and beamline are shown in Figure 2.1.3.3.

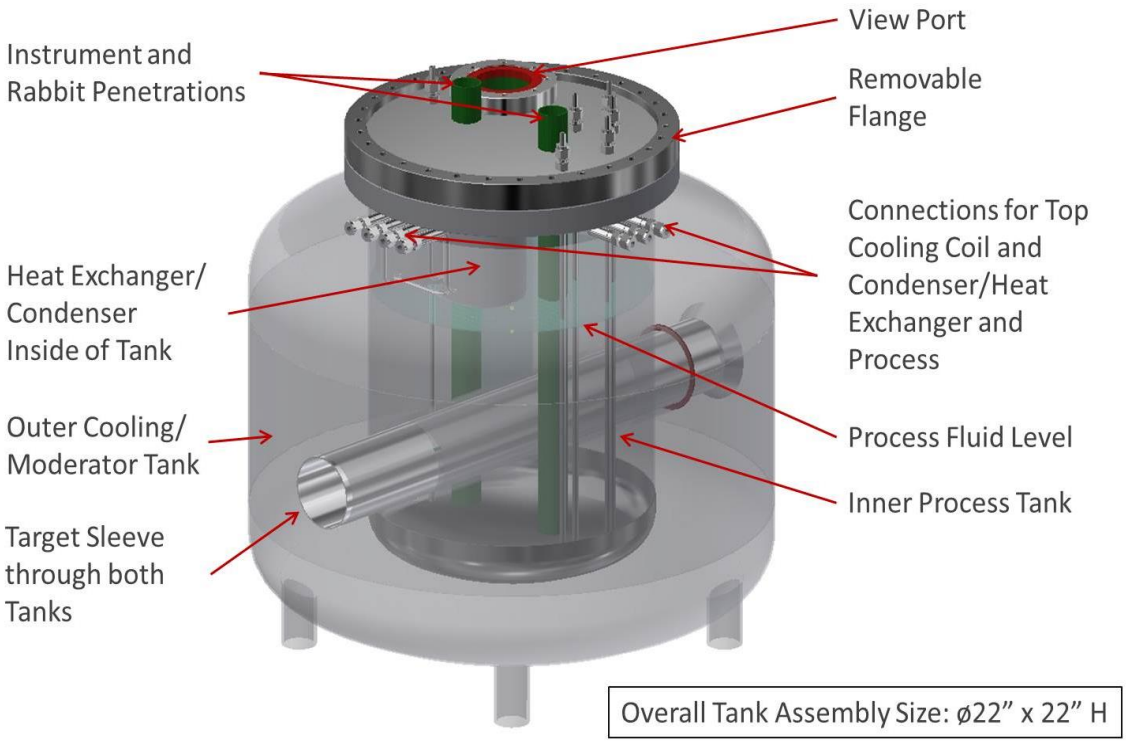

FIGURE 2.1.3.1 AMORE target solution vessel

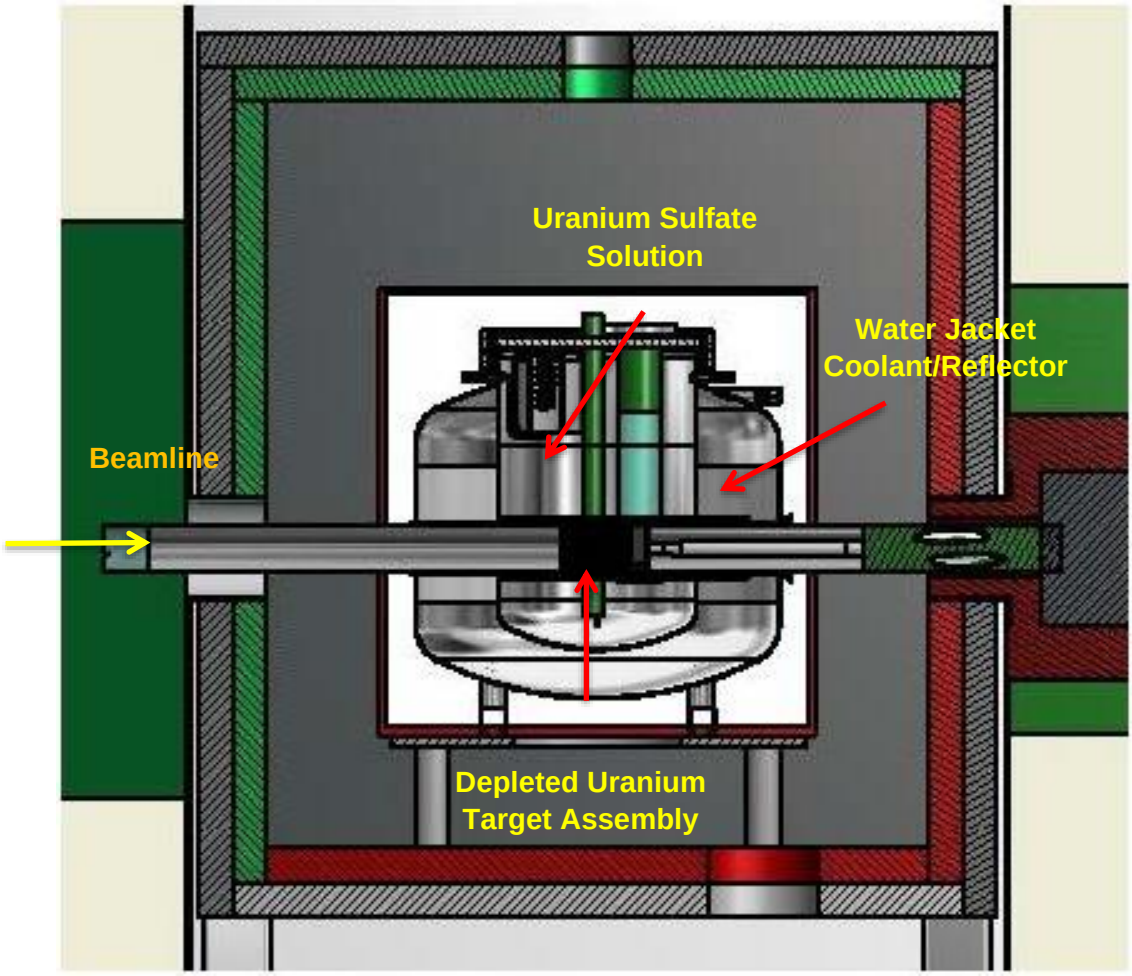

FIGURE 2.1.3.2 Target solution vessel cross-section inside the shielded box

FIGURE 2.1.3.3 Target solution vessel and depleted-uranium target installed inside the shielded cell

2.1.4 Monte Carlo simulations

To predict accumulation of radioisotopes in the AMORE experiments, Monte Carlo simulations using the MCNP code were performed (see Section 3.5). A 35-MeV beam energy and 20-kW beam power were used. It was predicted that one would need to irradiate 18 L of LEU uranyl sulfate solution for 19.3 hours to produce 20 Ci of ^{99}Mo (Figure 2.1.4.1). The proposed irradiation campaign was to consist of a maximum of 5 irradiations, separated by 4 weeks of cool-down time. Radionuclide inventories were calculated at the shutdown of each irradiation and for decay times out to one year following the final irradiation (as well as at intermediate times during the irradiation process). More details of the simulation results are described in Reference [1] and Appendix 7.

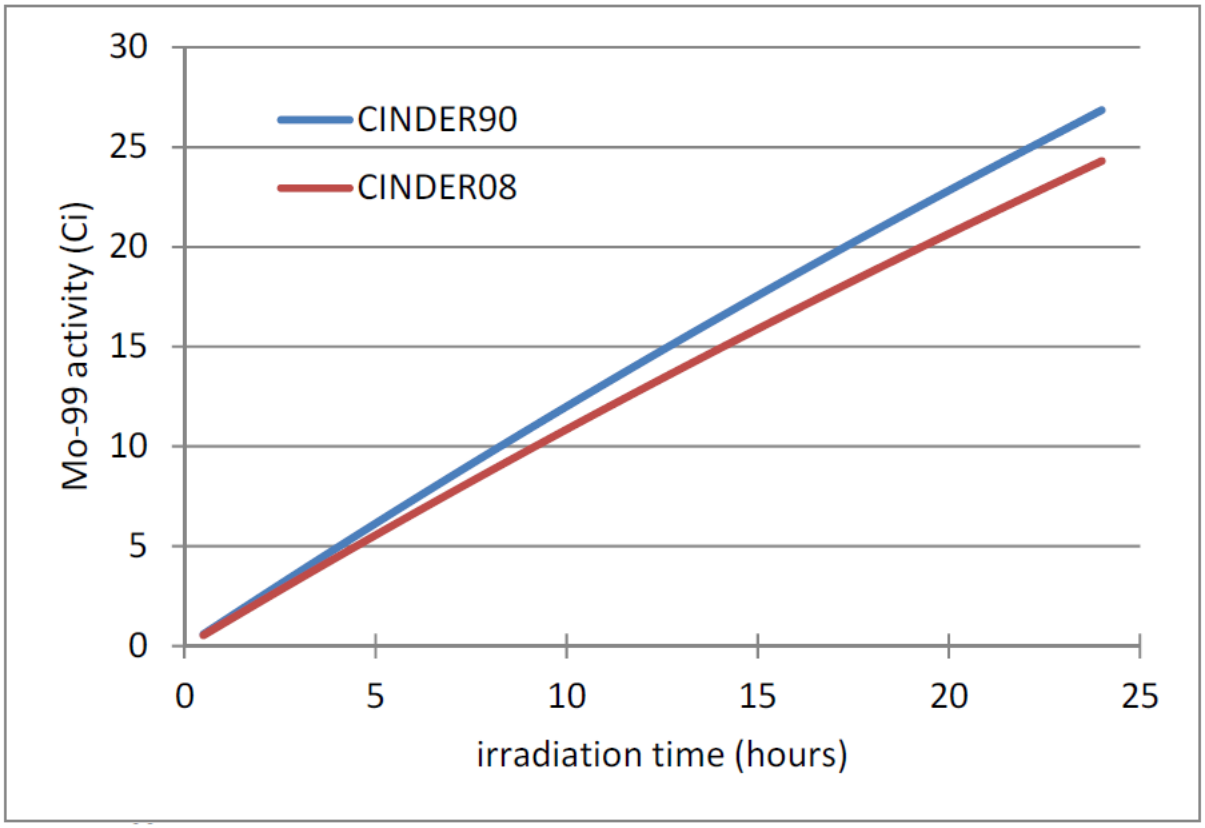

FIGURE 2.1.4.1 Buildup of ^{99}Mo in the 20-L uranyl nitrate solution

Figure 2.1.4.2 shows the hazard category 3 (HC-3) sum-of-fractions (SOF) in the entire irradiated volume, as well as in selected subsets, for the complete campaign of five 19.3-hour irradiations (to produce 20 Ci of ^{99}Mo each), with 4-week breaks between successive irradiations. The percentage of the SOF for the solution is about 91–92% of the total. Table 2.1.4.1 lists the top 41 contributors to the SOF, with their activities, at shutdown following the fifth irradiation. The contribution of the target varies between 8 and 9%, and only a small contribution to the SOF comes from the box and vessels. The SOF is dominated by the fission products ^{131}I and ^{133}I out to several months following the last irradiation, by which time the entire SOF is only about 0.01.

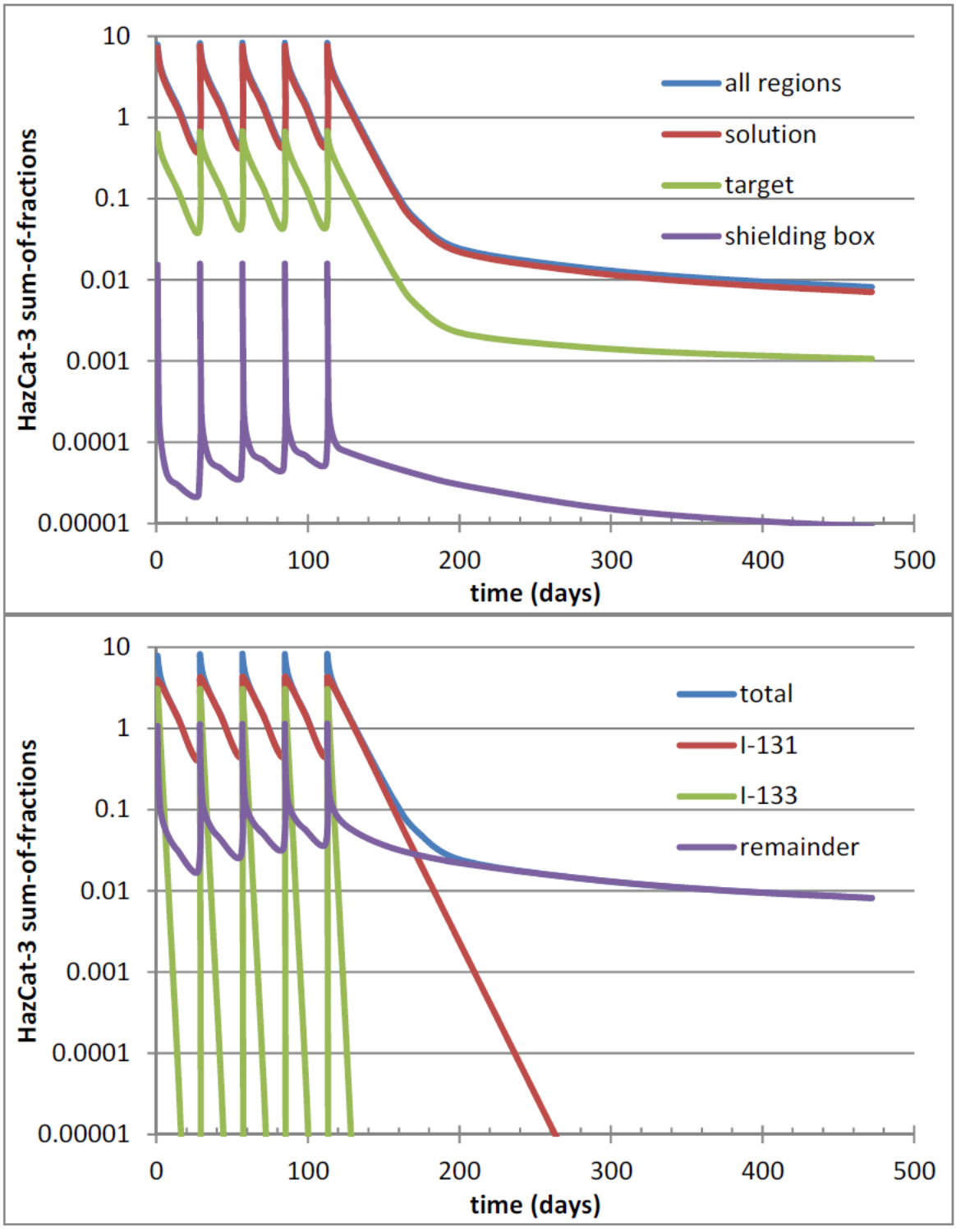

FIGURE 2.1.4.2 (Top) HazCat-3 SOF for the entire assembly, the uranyl sulfate solution, the target assembly, and the balance of the system. (Bottom) HazCat-3 SOF for the entire assembly, the nuclides ^{131}I and ^{133}I, and the remainder of the nuclides.

TABLE 2.1.4.1 Activities of the radionuclides with highest HC-3 SOF at the end of the fifth irradiation

Nuclide	Activity in Ci	Nuclide	Activity in Ci
^{131}I	6.84E-01	^{143}Ce	3.66E+00
^{133}I	5.71E+00	^{134}Te	1.79E+01
^{135}I	1.20E+01	^{92}Y	1.21E+01
^{88}Kr	8.59E+00	^{95}Zr	6.04E-01
^{138}Xe	1.61E+01	^{132}I	1.32E+00
^{87}Kr	6.68E+00	^{141}Ce	7.95E-01
^{91}Sr	1.47E+01	^{99}Mo	2.41E+00
^{97}Zr	6.38E+00	^{239}Np	5.46E+00
^{92}Sr	1.47E+01	^{85}Kr*	2.66E+00
^{134}I	2.01E+01	^{94}Y	1.65E+01
^{142}La	1.49E+01	^{32}P	6.83E-03
^{135}Xe	5.54E+00	^{129}Sb	1.23E+00
^{140}La	1.09E+00	^{105}Ru	2.16E+00
^{132}Te	1.55E+00	^{93}Y	9.45E+00
^{140}Ba	1.39E+00	^{89}Rb	1.21E+01
^{138}Cs	1.72E+01	^{147}Nd	5.21E-01
^{89}Sr	5.20E-01	^{97}Nb	5.71E+00
^{91}Y	5.18E-01	^{131}Te*	2.72E-01
^{144}Ce	1.40E-01	^{130}Sb	2.01E+00
^{135}Xe*	2.31E+00	^{131}Sb	6.54E+00
^{143}Pr	1.03E+00		

2.1.5 Safety Considerations

A comprehensive safety analysis for the linac facilities was conducted to evaluate the consequences of possible incidents due to the AMORE inventory. A full-facility fire was designated the "design basis" accident related to AMORE-generated radionuclides because it constitutes a credible, but extremely low-probability, event that produces the maximum set of consequences. The consequence determination was based on a maximum radionuclide inventory for the entirety of the AMORE irradiation campaign, resulting in maximum and "design-basis" consequences.

The calculated doses for this accident are documented in the consequence calculation.

Public consequence (at Argonne site boundary) = **0.32 mRem/year**

Co-located worker (100-m distance) = **26 mRem/year**.

2.1.6 References

[1] Chemerisov, S., Bailey, J., Makarashvili, V., Micklich, B., and Vandegrift, G.F. *Design of the Phase-2 Target for Mini-SHINE/MIPS Experiments.* ANL/CSE-14/9, Argonne National Laboratory, 2012.

[2] Chemerisov, S., and Vandegrift, G.F. *Mini-SHINE/MIPS Experiment*. ANL/CSE-14/2, Argonne National Laboratory, 2011.

2.2 GAS-HANDLING SYSTEM

The purpose of the gas-handling system is to collect all radioactive fission gases and to keep hydrogen concentration in the AMORE system below the flammability limit (4%). These goals are achieved in three ways: (1) Keep chemical processes under sub-atmospheric conditions, preventing the release of fission gas; (2) store fission gas for decay, to release at a later date; and (3) analyze for and recombine the hydrogen and oxygen generated from the radiolysis of water. The essential parts of the system are the GCS, the gas-distribution hub (GDH), the catalyst and pump, and the analytical system.

The GCS is the main feature, as it keeps all processes of the AMORE experiment sub-atmospheric and is used to store radioactive gases for decay. The GDH serves as a central connection point to the GCS. The catalyst pump recirculates the headspace gas of the TSV through a catalyst that recombines hydrogen and oxygen. The analytical system is used to monitor hydrogen and oxygen and includes safety interlocks that shut down the experiment at a 2% hydrogen level. It also generates an audible alarm at a 1% hydrogen level to notify the operator to reduce beam power by 50%.

2.2.1 Hydrogen and Oxygen Gas Generation

Hydrogen, hydrogen peroxide (H_2O_2), and oxygen are the molecular products generated by the radiolysis of water. The overall reactions are shown in Equations (1) and (2). The initial stage of the process is the formation of a solvated electron and the ionized and excited states of the water molecule created by incident radiation: $H_2O^{\bullet}$, H_2O^{+}, and $e^{-}(aq)$. Collisions generate radical fragments: $H+$, $H^{\bullet}$, $OH^{\bullet}$, OH^{-}, and others. These fragments combine to form molecular species: H_2, H_2O_2, or re-formed water [1]. Subsequent decomposition of H_2O_2 generates oxygen.

$$2H_2O + \gamma \rightarrow H_2 + H_2O_2 \quad (1)$$

$$2H_2O_2 + \gamma \rightarrow O_2 + 2H_2O \quad (2)$$

A steady-state hydrogen and oxygen concentration can be maintained in the TSV because the rate of gas generation is linear with respect to the linac beam power [2]. Since the beam power for 99Mo production needs to be as high as possible, maintaining hydrogen concentration in the vessel depends on the rate of hydrogen/oxygen recombination in the catalyst. Therefore,

the flow rate through the catalyst should be high enough to maintain hydrogen at a safe level. In these experiments, the length of the tubing to and from the pump and in the heat exchanger/condenser limited the flow of gas through the catalyst. This caused pressure in the return lines to increase, and the pressure in the supply lines and inside the TSV headspace to be reduced, thus increasing the differential pressure across the pump and reducing the gas flow through the catalyst. This result ultimately established an upper limit of approximately 18 kW of beam power with a KNF Neuberger Inc. NBR872 double-diaphragm pump, and 12 kW with a Senior Aerospace MB-151 Metal Bellows pump.

We had the ability to add oxygen to the TSV through a solenoid valve connected to a cylinder containing 40% oxygen. During previous experiments, the TSV was purged with helium before the irradiation [3]. Hydrogen is liberated into the headspace faster than oxygen in the initial stages of radiolysis. H_2O_2 is produced from the reaction of radicals and ions. Molecular oxygen is produced from the decomposition of H_2O_2 and has a greater solubility in the solution than hydrogen. The result is a delay in the occurrence of oxygen in the headspace gases. During the present set of experiments, the TSV was not purged prior to the irradiations.

2.2.2 Gas-handling System Setup

The uranyl sulfate solution resides inside the TSV, as shown in Figure 2.2.2.1. The headspace gas of the vessel was initially recirculated through a catalytic recombiner using a KNF Neuberger Inc. N186.1.2SN.12E double-diaphragm pump. After the March 2020 irradiation, that pump overheated, and it was replaced with the Senior Aerospace MB-151 Metal Bellows pump used in earlier irradiations. The catalyst, shown in Figure 2.2.2.2, is composed of platinum/palladium on alumina/cordierite and has a honeycomb configuration, which allows the gas to flow down the length of the bed. The catalytic material was commonly used to reduce emissions in diesel engine exhaust. Here, it is being used as a reactor to recombine the hydrogen and oxygen produced from the radiolysis of water. The purpose is to reduce the concentration of hydrogen in the headspace of the TSV to well below the flammability limit of 4%. Part of the safety basis for these experiments was to keep the concentration of hydrogen to less than 1% (see Appendix 8). The catalyst is heated to 130°C to facilitate the removal of water produced in the catalyst bed during the recombination reaction. This operating temperature also prevents condensation of water vapor on the catalyst surface caused by humidity in the headspace gas. It was found during sodium sulfate irradiations in April 2014 that condensation on the catalyst inhibits the active sites of the catalyst, causing a buildup of hydrogen in the system.

A condenser (Figure 2.2.2.3) is located inside the TSV, upstream of the point where the headspace gas flows to the recirculating pump, and serves to decrease the humidity of the headspace gas prior to entering the catalyst. The gas passes around the outside of a water-cooled coil. At the catalyst exhaust, a heat exchanger (Figure 2.2.2.4) recondenses the water vapor generated in the catalyst. In the heat exchanger, gas passes through the interior of a water-cooled tubing coil.

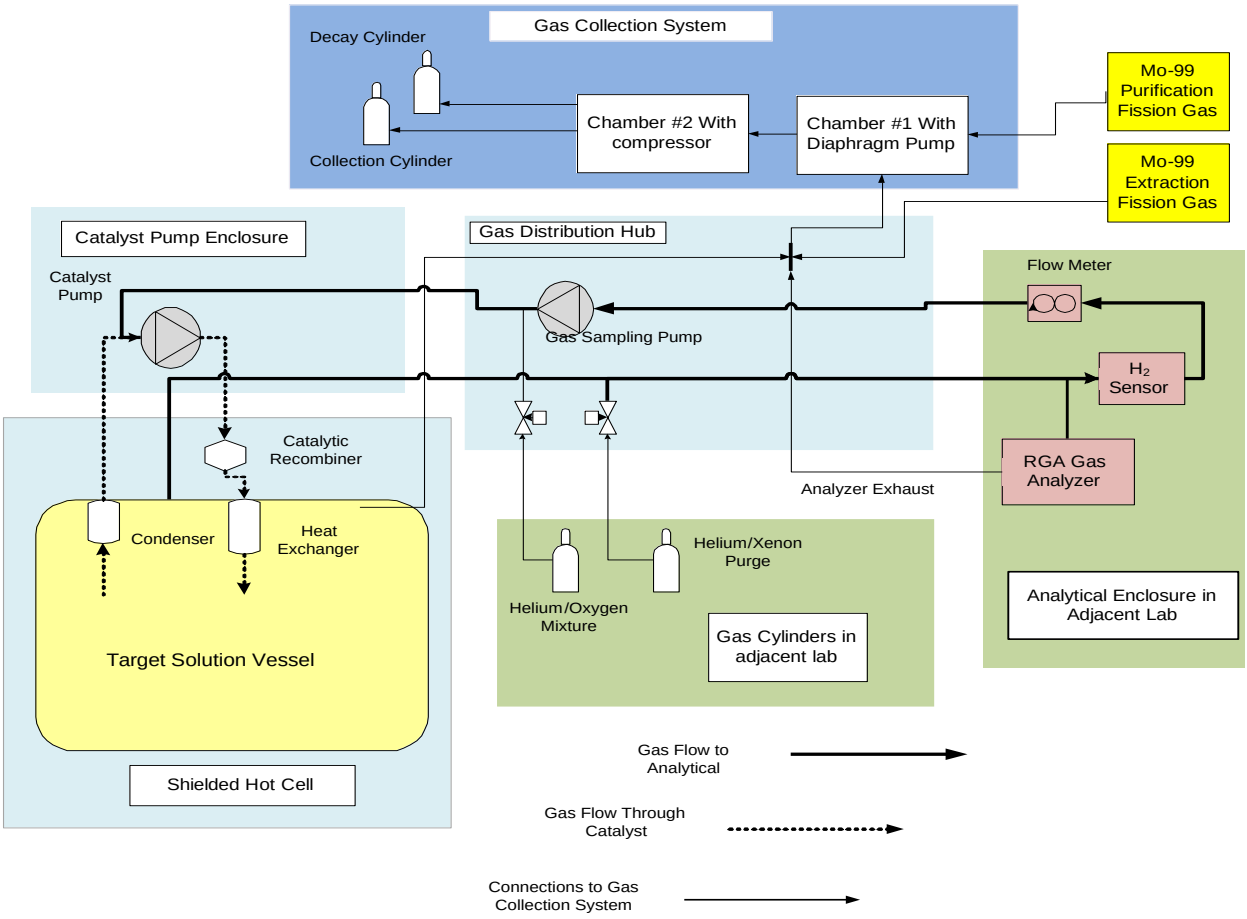

FIGURE 2.2.2.1 Diagram of the gas-handling system gas flow. All subsystems interconnect and lead to the GCS.

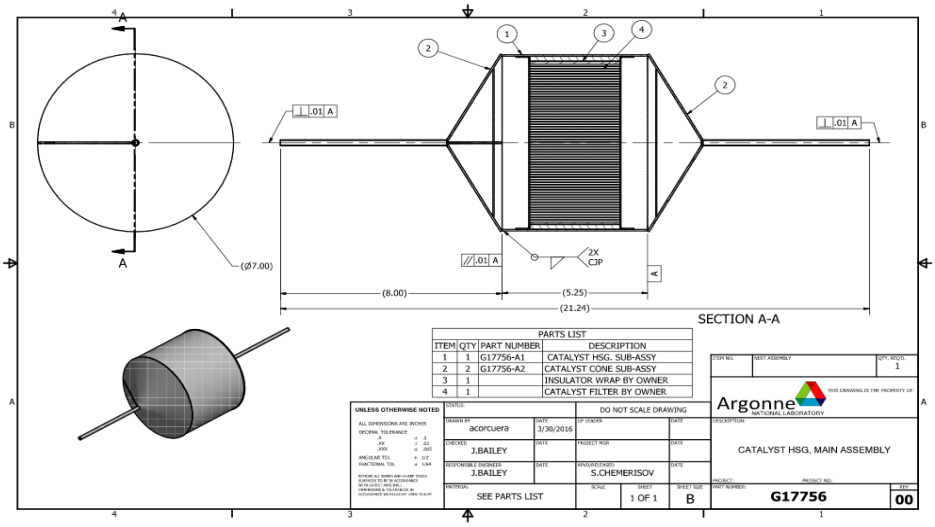

FIGURE 2.2.2.2 Catalytic recombiner design. (1) cylindrical part of the catalyst housing; (2) conical reducers; (3) fiberglass layer; (4) catalyst

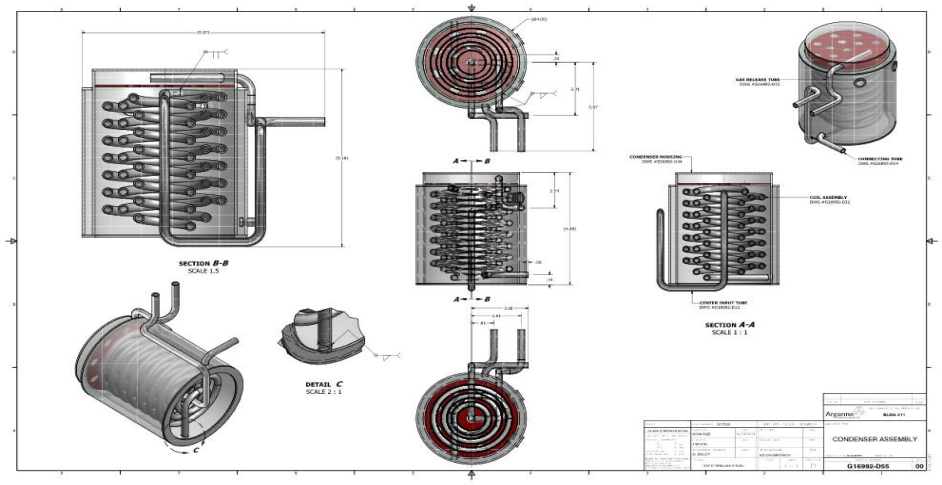

FIGURE 2.2.2.3 Condenser design

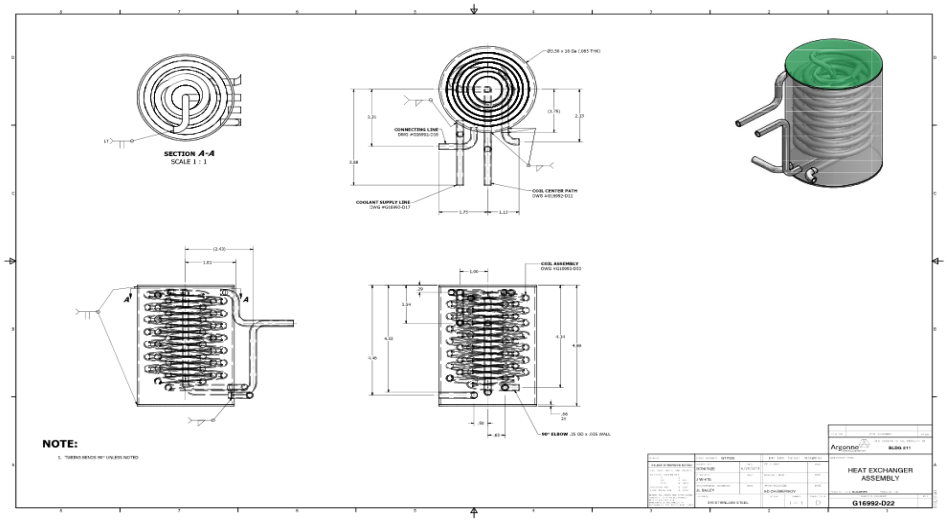

FIGURE 2.2.2.4 Heat exchanger design

Analytical instrumentation is connected to the vessel headspace by stainless steel tubing. The instruments are located inside an enclosure (Figure 2.2.2.5) in a room adjacent to the linac irradiation cell because the high radiation dose has been shown not only to interfere with gas measurement but also to cause total failure of any equipment containing sophisticated electronics. A metal bellows diaphragm pump (Senior Aerospace MB-41) is used to transport the gas from the headspace of the TSV to the analytical system. The gas is pulled from the headspace, analyzed, and then returned to the vessel. Headspace gases were analyzed using two instruments: an H_2 Scan hydrogen sensor and a Pfeiffer Prisma Plus QMG 220 Residual Gas Analyzer (RGA), which is equipped with a quadrupole mass filter and a secondary electron multiplier as the detector.

FIGURE 2.2.2.5 AMORE analytical enclosure

The GCS (Figures 2.2.2.6 and 2.2.2.7) connects to the headspace of the TSV by a single ¼-in. stainless steel tube via the GDH. The GCS maintains the TSV at sub-atmospheric pressure, preventing the release of volatile isotopes of iodine and the fission gases xenon and krypton. It also provides an outlet for purge gas and prevents over-pressurization of the vessel. Gas from the analytical instruments, as well as purge gas and fission gas released from the ^{99}Mo extraction process, exhaust to the GCS through the GDH. A single ¼-in. stainless steel tube connects the ^{99}Mo purification and recovery processes to collect fission gases released from those processes.

The GCS (Figure 2.2.2.6) consists of two chambers and two collection cylinders connected in series. Each chamber is equipped with a pressure transducer (OMEGA Engineering Model MMA030V10H3C0T3A6CE) to measure the pressure inside the chamber. The collection cylinders are also connected to a transducer (OMEGA Engineering Model MMG5.0KV10P2C0T3A6CE) to measure their pressure. Check valves (Parker Hannifin 4A-CAL-1/3-NE-SS) are installed between Chamber #1 and Chamber #2 and between Chamber #2 and the collection cylinders. The check valves prevent the backflow of gas between chambers and from the collection cylinders to Chamber #2. Later, we installed check valves with higher cracking pressure to reduce the rate of back-leak through the valves. The inlet of Chamber #1 is connected to the TSV, the GDH, and the ^{99}Mo purification process; the outlet connects to the inlet of Chamber #2. Inside Chamber #1, there is a diaphragm pump (GAST Manufacturing Corporation Model D0AP704AAEMD). The inlet of the pump is open to the chamber volume, and the outlet connects directly to Chamber #2. Chamber #2 has a compressor (NARDI Compressor Atlantic 100) inside. The compressor inlet is open to the chamber volume; the outlet is connected to the collection cylinders. At the inlet of the GCS, humidity is reduced by a

water-cooled condenser. A catalyst bed further reduces the concentration of hydrogen. A cartridge containing silver-impregnated zeolite is used to trap iodine. There is a solenoid valve between Chamber #2 and the collection cylinders. It is interlocked and automatically closes when a high pressure is detected in the chamber. A flow-limiting orifice is installed between Chamber #2 and the collection cylinders in case of a system failure that could lead to release of the gases into the enclosure. This orifice limits the rate of gas released into the enclosure, ensuring that it can be handled by the enclosure ventilation system (50 SCFM). There is a port on the collection cylinders for sampling and releasing the collected gas. A relief valve on Chamber #1 and a burst disc on Chamber #2 prevent over-pressurization of the chambers. Water-cooled fans inside each chamber cool the equipment. Power relays to the diaphragm pump and compressor are controlled with OMEGA Engineering Cni8 controllers. The controllers also have relays connected to interlocks that will shut down the linac if an over-pressurization occurs. Multiple procedures address the periodic maintenance and replacement of parts in the GHS (see Appendices 9–12).

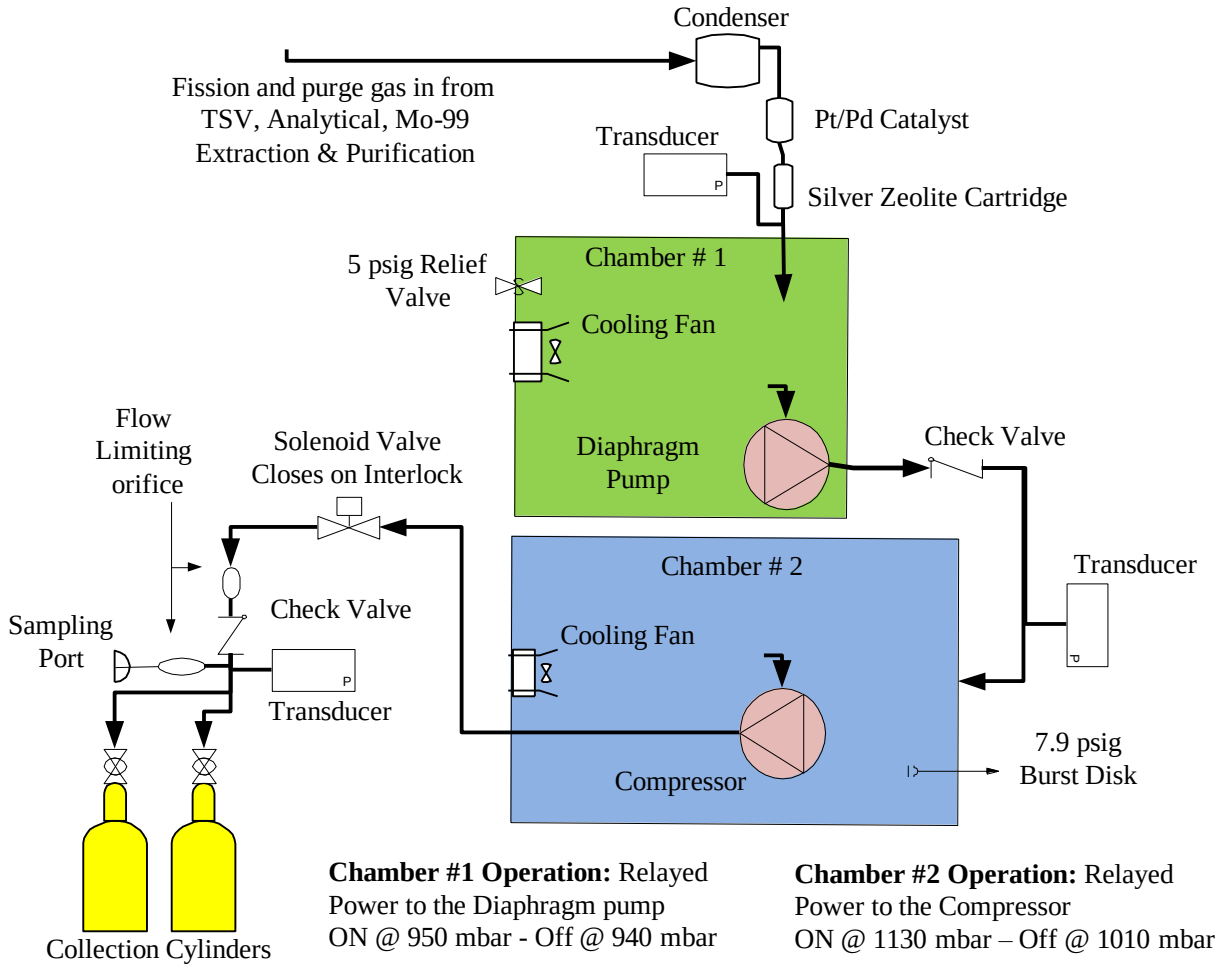

FIGURE 2.2.2.6 Diagram and function of the gas collection system

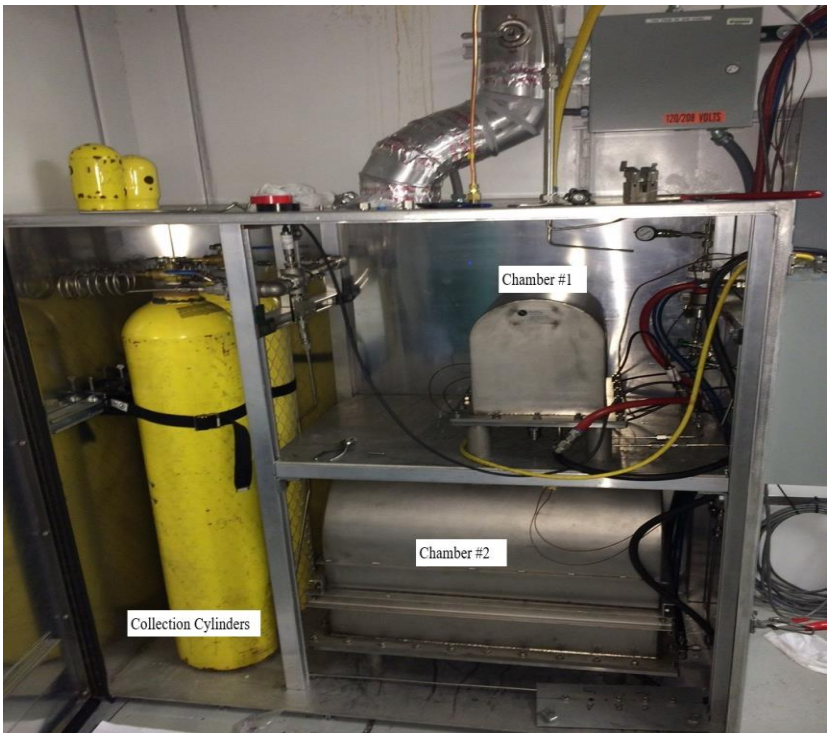

FIGURE 2.2.2.7 Photograph of the gas collection system inside the enclosure

The GCS functions to maintain all AMORE processes at sub-atmospheric pressure and to store fission gas for decay. As gas enters the AMORE system, the pressure rises in Chamber #1. At 950 mbar, the diaphragm pump actuates and reduces the pressure in the system and pressurizes Chamber #2. The pump turns off at 940 mbar. When the pressure inside Chamber #2 reaches 1130 mbar, the compressor actuates and transfers gas to the collection cylinders. The compressor turns off at 1010 mbar. Check valves prevent backflow of the gas. This arrangement effectively maintains the entire AMORE system at sub-atmospheric pressure and within a narrow pressure range.

Before each experiment, the performance and configuration of the GHS is verified (see Appendices 13 and 14). The catalyst pump flow is verified, along with associated alarms and interlocks. A calibration check is performed on the analytical instruments. Interlocks and alarms associated with hydrogen concentration are verified. Instrument calibration is performed if necessary. The GCS is tested by adding helium into the system to ensure that the pumps are functioning and turn on and off at specified pressures. The alarms and interlocks associated with over-pressurization are also verified.

During the AMORE experiment, gas is collected and stored in one of the collection cylinders while the other is kept closed. The makeup of the gas is predominantly helium, nitrogen and oxygen with small amounts of fission gas. Some of this fission gas is from a post-irradiation purge of the analytical lines. The gas lines in the analytical enclosure are purged with helium immediately after each irradiation (see Appendix 8). Since the enclosure is located in a lab adjacent to the irradiation cell and has co-located AMORE activities, the lines are purged to reduce the radiation field to allow entry into the space. Specifically, the lines are purged of short-lived isotopes of xenon, which cause a significant radiation field (>500 mR) in the lab. After an irradiation, chemical processing of the solution occurs. This step accounts for most of the atmospheric leakage into the GHS. Solution transfer, vacuum pump operation, and solution sampling all contribute to the gas collected in the GCS.

Inside the collection cylinders, the short-lived isotopes of xenon quickly decay, leaving ^{133}Xe (half-life, 5.25 days) and ^{85}Kr (half-life, 10.8 years). Approximately 60 days (10 half-lives of ^{133}Xe) after a cylinder is filled, a sample is taken and analyzed by gamma spectroscopy to determine the concentration of radioactive isotopes (see Appendix 15). The gas is then carefully released to the atmosphere (see Appendix 16).

2.2.3 References

[1] Spinks, J.W.T., and Wood, R.J, *An Introduction to Radiation Chemistry*. John Wiley & Sons, Inc., New York, 1990.

[2] Kalensky, M., Youker, A., Chemerisov, S., and Brossard, T. *Analysis of Radiolytically Generated Gases in Mini-AMORE Experiment*, ANL/CFC-18/3, Argonne National Laboratory,2018.

[3] Youker, A.J., Chemerisov, S.D., Tkac, P., Kalensky, M., Heltemes, T.A., Rotsch, D.A., Krebs, J.F., Makarashvili, V., Stepinski, D.C., Alford, K., Bailey, J., Byrnes, J., Gromov, R., Hafenrichter, L., Hebden, A., Jerden, J., Jonah, C., Micklich, B., Quigley, K., Schneider, J., Wesolowski, K., Vandegrift, G.F., and Sun, Z., *Compendium of Phase-I Mini-SHINE Experiments*, ANL/NE-16/39, Argonne National Laboratory, October 2016. Available at https://publications.anl.gov/anlpubs/2017/01/131828.pdf

2.3 RECOVERY GLOVEBOX

2.3.1 Introduction

In Phase I of the AMORE project, initial processing of the irradiated target solution was carried out in the Target Solution Monitoring Glovebox and the Molybdenum Recovery Glovebox.[1] In Phase II, these two gloveboxes were combined into the "Recovery Glovebox" discussed here. The recovery glovebox was operated according to LEAF-PROC-024 (see Appendix 17). The primary purpose of recovery glovebox operations was to load freshly irradiated LEU uranyl sulfate solution (140 g-U/L) on a chromatographic column packed with titania sorbent and to carry out the initial separation of ^{99}Mo from the bulk uranyl sulfate target solution containing fission products. Once the initial separation was completed, the ^{99}Mo product was pumped directly to the hot cell, where it was further processed using the concentration column and LMC processes discussed in Sections 2.4.1 and 2.4.2 below.

In addition to combining the two gloveboxes from Phase I, the recovery glovebox implemented several changes based on observations and experience from operating the Phase I system and requirements from scaling up the operation. Owing to the inability to obtain measurements of pH, turbidity, and conductivity during Phase I, these sensors were removed from the recovery glovebox. Another lesson learned from Phase I was that the system needed to be split so that acid and base solutions stayed in separate systems to prevent cross-contamination of the feed and effluent and to prevent unnecessary dilution/neutralization of the target solution. As a result, the Phase II system had an acid side and a base side, each with its own feed pump, sampling ladders, feed vessels, effluent vessels, and piping. The only point where the acid and base lines overlapped was the recovery column. Finally, owing to the much larger volume of solution being processed (20 L in Phase II vs 5 L in Phase I) and the much higher activities generated during these irradiations, the recovery glovebox was built using 2-in.-thick carbon steel, and liquid lines, wherever possible, were made of ¼ in. O.D. 316L stainless steel tubing to provide extra shielding. In the areas where flexible tubing was required, fluorinated ethylene propylene (FEP) tubing of similar O.D. was used. The effluent collection vessels, verification tank, and column also required additional shielding because of the scale-up.

2.3.2 Experimental Setup

2.3.2.1 Operation of the Glovebox

The operation of the recovery glovebox is extremely complicated and is described in its entirety in LEAF-PROC-024 (see Appendix 17). A brief description of glovebox operations is included here to give an idea of the process steps involved. Several steps had to be undertaken before an irradiation could start. The first of these was removal of the spent column. During the previous irradiation and processing, fission and activation products built up in the column, so it was removed—inside its shielded pot—from the system and stored until radiation levels abated. Once the column was removed, the verification tank was put in its place and the uranyl sulfate

target solution was pumped to it to verify its mass and take a sample. By measuring the mass of the entire target solution when it was pumped into the verification tank and the mass of the target solution sample taken from the verification tan can determine the total volume of solution in the system via the density of the sample, which could be measured directly. This information was important, as the total volume of target solution was one of the parameters bound by the Accelerator Safety Envelope (ASE). After analyzing the sample taken from the verification tank, adjustments were made to the target solution to ensure that it adhered to all the ASE parameters (discussed in Section 2.3.4). This is also the time when stable Mo and Fe were introduced into the system. Stable Mo was required as a carrier for the LMC process (discussed in Section 2.4.2), and Fe was required to prevent the formation of uranyl peroxide precipitate during and after irradiation. Once the target solution was within the ASE parameters and the Mo and Fe had been added, the target solution was pumped into the TS, where it would stay until irradiation. Next, the verification tank was removed and replaced with a new packed, shielded column. At this point, the processing feed bottles were refilled with acid, water, and base solutions, and the effluent cart containing seven separate empty bottles to receive waste and raffinate was installed. Finally, the lines in the glovebox were primed with solution and the column was leak-checked before an irradiation could commence.

During irradiation, the system was monitored remotely. Initially, the target solution was circulated from the TSV through a mixing pathway that did not go through the column, then returned to the TSV during irradiation. This practice was terminated when it was realized that it would not be possible to re-engage the pump if it was stopped by a tripped leak sensor or pressure interlock until the linac was cleared of the radiation hazard (requiring several hours). Instead, solution was circulated for three hours after the irradiation was complete to ensure that a homogenous solution was loaded on the column. Some mixing was achieved during the irradiation through convection and bubble formation caused by radiolysis.[2]

Following irradiation and mixing, the circulation flow path was cleared of target solution by pumping it into the TSV while pulling in glovebox atmosphere from the surge vessel. Once this step was completed, the titania column was conditioned with pH 1 H_2SO_4 and the heaters serving the acid line and column were turned on. Once the acid line and column were at temperature, the column was loaded by pumping the target solution from the TSV through the column with the column effluent directed to the dump tank, where it was stored for the remainder of the process. After loading, the column was washed with additional acid and then water to remove weakly retained contaminants, with both washes directed to their respective bottles (post-load acid wash and post-load water wash) in the effluent cart. Following these steps, the base-side lines were primed, the base line heater was turned on, and the acid line heater was turned off. At this point, the column was stripped using 1 M NaOH. This solution was sent directly to the hot cell used for concentration column operations. The column-stripping step was followed by rinsing the column and base lines with water, then rinsing the acid lines with acid while bypassing the column. When these steps were complete, the glovebox portion of solution processing ended.

During any part of the irradiation, mixing, and processing steps, samples could be taken using in-line remote sampling ladders. Once the residual radiation in the irradiation cell had abated, these samples were retrieved from the glovebox and sent for analysis. Over the course of

the project, these sample ladders began to malfunction, and an alternate sample loop was installed and used for the final irradiation (discussed in Section 2.3.3.2). Once samples were recovered, the target solution could remain in the dump tank or be pumped back up to the TSV for storage until its properties needed to be measured/verified prior to the next irradiation. After these steps were complete, the acid side of the system was washed with acid and the base side of the system was washed with water to ensure that any components of the irradiated solution were removed from the system. This step was followed by purging the system with N_2 to ensure that all the acid and water from the rinse solutions had been removed from the system as well. Following the final wash and N_2 purge, the glovebox system was considered reset and was left idle until preparations for the next irradiation began.

2.3.2.2 Glovebox Description

The glovebox as a whole, seen in FIGURE 2.3.2.2.1 Image of the recovery glovebox with access port on the left. The three main windows and three cabinets discussed are to the right of the switch panel in the image., is composed of a shielded access port with a small window on the far left, with an electric-motor-powered door on the inside separating the main glovebox cavity, which stretches across the remainder of the box (to the right of the switch panel in Figure 2.3.2.2.1. The main cavity of the glovebox can be roughly divided into thirds, according to the placement of the three large windows and cabinets. Figure 2.3.2.2.2 is a detailed schematic of the glovebox main cavity and the cabinets below it. Furthest to the left is cabinet #1, which contains the recovery column or the verification tank, depending on the operational step (illustrated with column in place). In the glovebox above cabinet #1 are the column pre-heaters, as well as the connections for the verification tank and for the column itself, along with associated solenoid valves. In the middle section is cabinet #2, which contains the feed cart with all the feed solution containers and a balance to monitor solution levels. In the glovebox above cabinet #2 are both Fluid Metering, Inc. (FMI) pumps used to move solution, pressure and flow sensors for each side of the system, and the surge tank (used to catch the target solution if the system malfunctioned). Furthest to the right is cabinet #3, which houses the shielded effluent cart with all seven of the effluent containers and a balance to monitor solution levels. In the glovebox above cabinet #3 are the inlet and outlet connections to the TSV, the outlets to the various effluent bottles, the outlet to the dump tank, the three sets of sample ladders (one each for target mixing, column loading, and column stripping), and all of the sample pots. The engineering drawing in Figure 2.3.2.2.2 does not illustrate the surge tank or the sample ladders, as these were behind a shielded lead curtain in this drawing. It was later determined that this shielding was not necessary, and it was therefore not installed. These structures are shown in the top-view engineering diagram found in Figure 2.3.2.2.3. A piping and instrumentation diagram illustrating how the entire system is connected is found in Figure 2.3.2.2.4. The shielded verification tank replaced the column in cabinet #1 prior to each irradiation while the target solution parameters were obtained, as discussed above and in Section 2.3.4 The external nitrogen tank was connected to the system to purge the liquid lines during the washout process, as described above. When utilized, the external N_2 tank was attached to the glovebox through valve V-2038. Pipe P-305 was then attached to either valve V-2001 or V-2033 to provide gas for purging the acid lines or base lines, respectively (see Figure 2.3.2.2.4).

FIGURE 2.3.2.2.1 Image of the recovery glovebox with access port on the left. The three main windows and three cabinets discussed are to the right of the switch panel in the image.

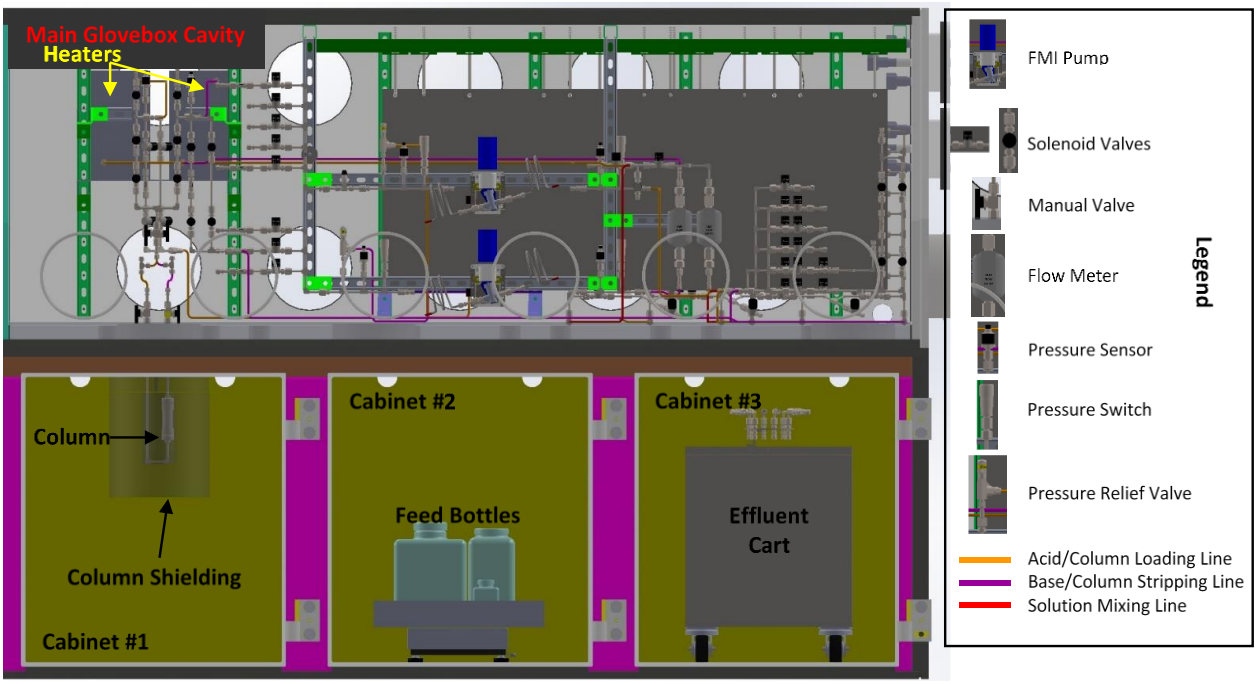

FIGURE 2.3.2.2.2 Engineering diagram of the glovebox main cavity and cabinets below

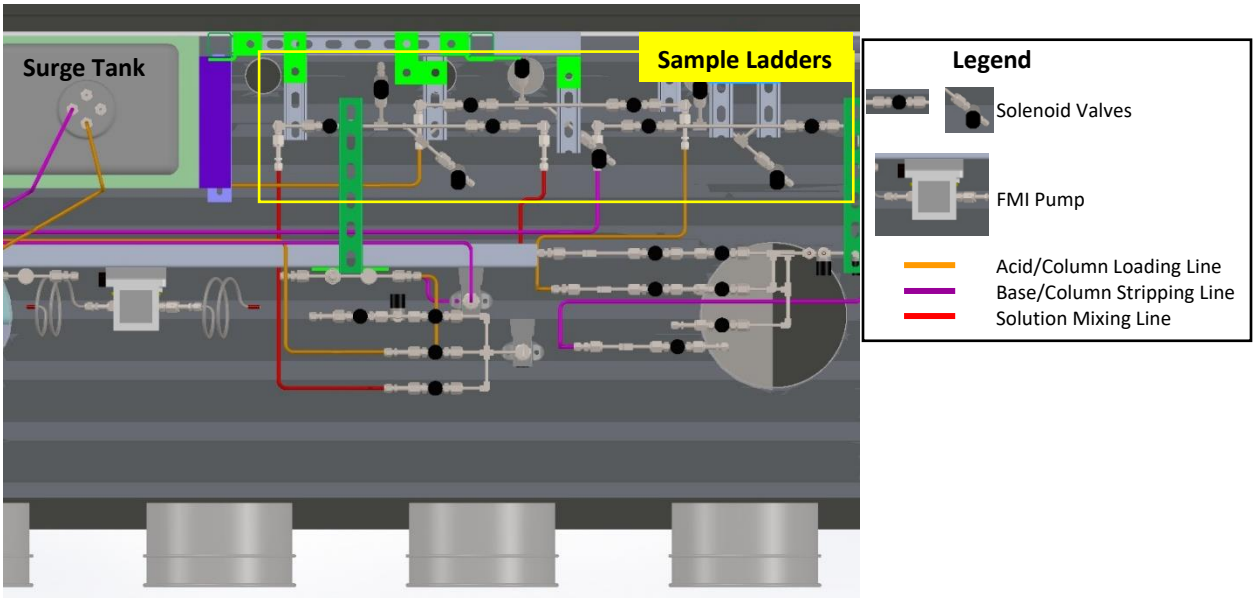

FIGURE 2.3.2.2.3 Engineering diagram top view above cabinet #2 and cabinet #3, showing the locations of the surge tank and sample collection ladders

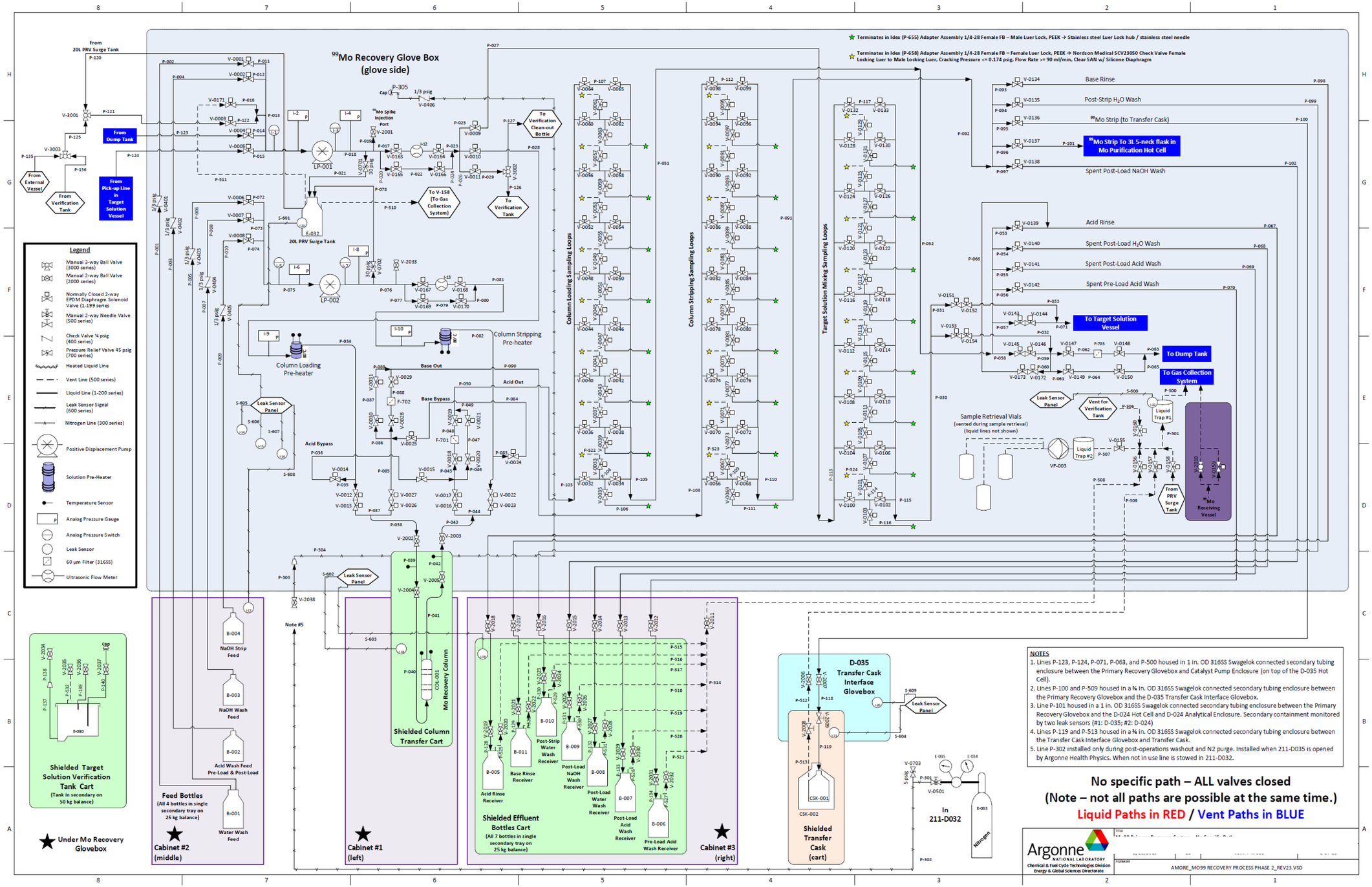

FIGURE 2.3.2.2.4 Piping and instrumentation diagram for the recovery glovebox

2.3.2.3 Recovery Column

Like the rest of the recovery glovebox, the primary separation column itself was also scaled up to handle the larger volume of solution and additional ^{99}Mo that needed to be retained.[2] The column is a 40 mm I.D. x 100 mm long Macherey-Nagel VarioPrep constructed of 316 SS and using Viton O-rings and 40 µm 316 SS frits. The column was packed with titania resin from ZirChrom (Sachtopore-NP, 60 Å, 110 µm size), which was acid washed with Ph 1 H_2SO_4 at room temperature prior to use to remove fines and otherwise leachable contaminants, following the method developed in Section 5.5 of Reference [3].

To pack the column, the bottom half was assembled to the specified dimensions (see Appendix 18) and clamped in a ring stand. A beaker was placed under the column to collect water as it drained through, and a mark was placed on the inside of the column at the desired bed height (4.25 in. tall). The previously washed resin was slurried with water and poured into the column, allowing excess water to drain out the bottom while keeping a small head of water over the resin bed. Once the bed reached the desired bed height, the column bottom was capped and the top frit and collar were installed and hand-tightened. Once assembled, the column was moved to a vise and the top collar was further tightened using a pipe-wrench, ensuring that the top cap was removed to allow for water displacement and that the column inlet and outlet were pointing in the same direction during tightening. The overall length of the column from collar end to collar end when tightened was 6.75 in. Images of a fresh column are found in Figure 2.3.2.3.1. Once assembled, water was circulated through the column in each direction for 1-2 hours to ensure that any remaining fines were removed. As a leak check, the column was capped and the pressure was raised to approximately 35 PSI for 1 hour, checking periodically for pressure decreases and minor leaks. Once the column was determined to be leak-tight, the remaining piping, valves, thermocouples, heat tape, and insulation were added to the column prior to installation. A completely assembled column, ready for installation, is shown in Figure 2.3.2.3.2 along with the carbon-steel-encased lead-shielded pot it was placed in.

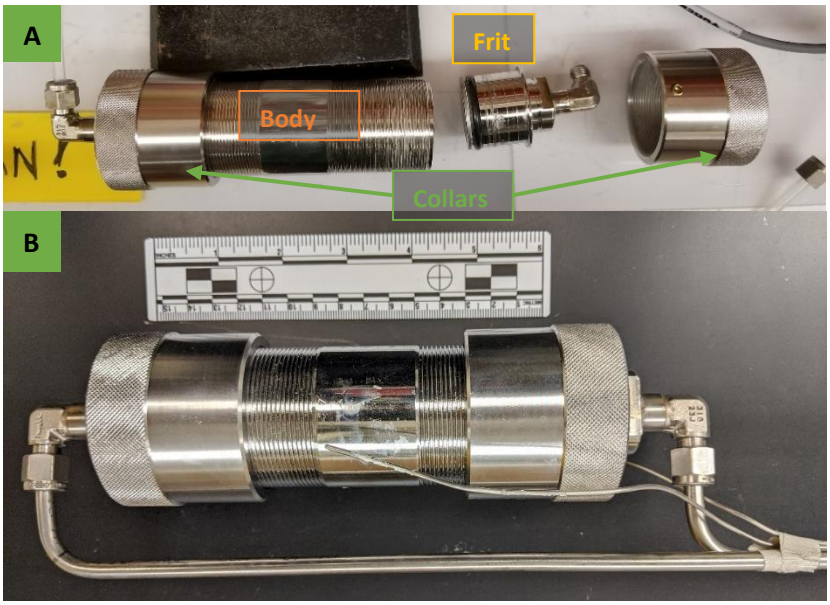

FIGURE 2.3.2.3.1 Images of the recovery column A) prior to assembly with frit and top collar removed and B) after filling and assembly, prior to installation in the recovery glovebox

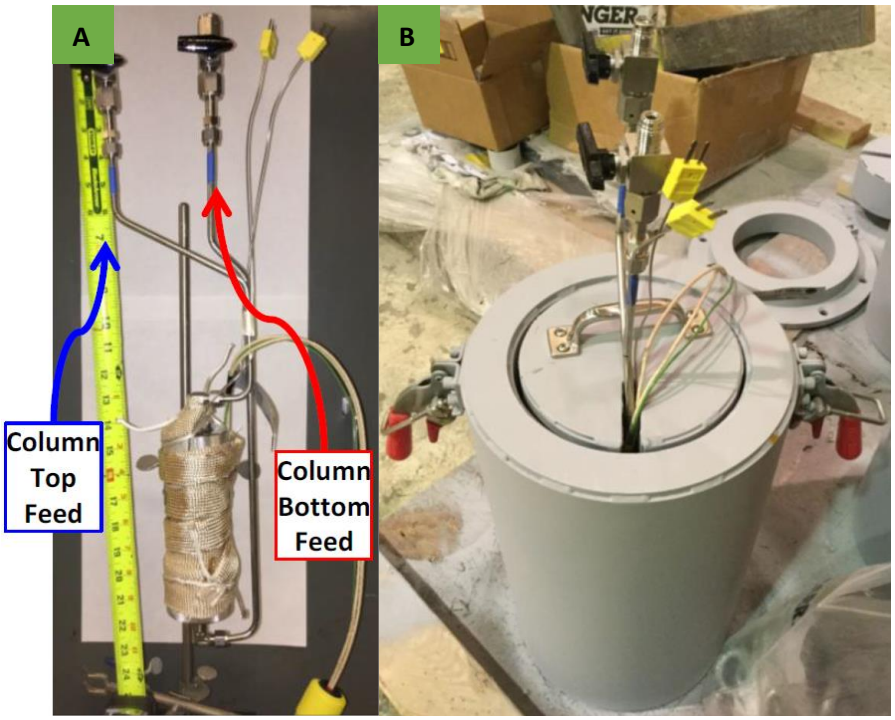

FIGURE 2.3.2.3.2 Images of the recovery column (A) wrapped in heat tape and insulation and (B) installed in the lead-shielded pot

The column and carbon-steel-encased lead-shielded pot were placed in shielded cabinet #1 under the glovebox, where the column was coupled to the system through a port in the glovebox floor. Images of the column attached to the rest of the system can be seen in Figure 2.3.2.3.3. To ensure minimal delay between irradiations, multiple column pots were kept on hand so a spent column could be removed and fission/activation products allowed to decay in its pot while a new column was put in place. During normal operations, all column activities using the acid-side system (pre-load acid washing, column loading, post-load acid washing, and post-load water washing) were carried out in the upflow direction (solution flowing from bottom to top of the column bed), while all base-side system column activities (column stripping and post-strip water wash) were carried out in the downflow direction (from top to bottom of the column bed). All acid solutions were pH 1 H_2SO_4 (including the uranyl sulfate target solution) and all base solutions were 1 M NaOH. Adsorption of Mo on the titania column was performed at 80°C with the temperature monitored using K-type thermocouples at the inlet and outlet of the column, as well as on the column body itself. The optimal temperature in which the highest K_d(Mo) values would occur was previously determined to be 80°C.[4]

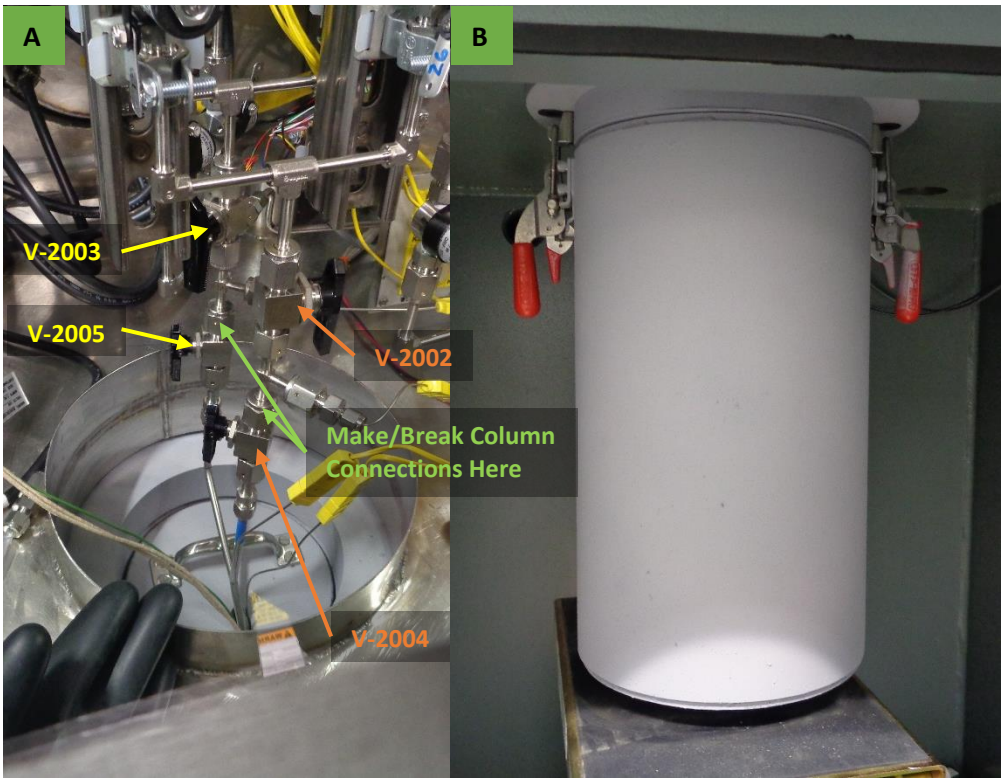

FIGURE 2.3.2.3.3 Recovery column in lead-shielded pot attached to the rest of the system, viewed (A) inside glovebox and (B) inside cabinet #1

2.3.2.4 Fluid Handling

Solutions introduced to the system were held in one of four feed bottles in Cabinet #2 under the glovebox. The acid feed bottle contained pH 1 H_2SO_4 used in conditioning the column, post-load acid washing of the column, and washing of the acid lines in the system after processing. The water-wash feed bottle contained 18 MΩ·cm deionized water, which was used in the post-load and post-strip water washes of the column and in washing the base lines after processing. The NaOH strip feed and NaOH wash feed bottles both contained 1 M NaOH. The NaOH wash feed bottle would have been used during an optional post-load NaOH wash of the column, but this step was never opted for. The NaOH strip feed was used for priming the base lines and stripping Mo product from the column.

Solutions were transferred within the glovebox and between the glovebox and other components (TSV, dump tank, or hot cell) using positive displacement pumps (model QV-50) from FMI, which were fitted with ceramic-lined pump heads employing ⅜ in. O.D. ceramic pistons in 316SS housings (model Q2-CSC) and Rulon® AR (fluorocarbon filled PTFE) lip seals. The individual pumps for the acid and base sides of the system were controlled by independent FMI V-300 controllers. All liquid lines in the system were made of either 316L stainless steel tubing or FEP tubing. The only lines in the system that were not ¼ in. O.D. were associated with the gas handling system and the sample retrieval system (discussed in Sections 2.2.2 and 2.3.3, respectively).

2.3.2.5 Dump Tank

After the target solution was passed through the column, it was sent to the shielded dump tank, which was located directly below the TSV. Figure 2.3.2.5.1 shows this arrangement, with lead bricks between the dump tank and TSV hot cell to shield workers from the lines connecting the two. These were arranged in an “L” shape to help reduce shine from either vessel. The TSV and dump tank were located outside the far-right end of the glovebox. The very top of the TSV hot cell can be seen on the right in Figure 2.3.2.2, behind the glovebox.

FIGURE 2.3.2.5.1 Image showing the relationship between the Dump Tank and TSV Hot Cell. These were connected via L-shaped transfer lines shielded by lead bricks (seen in picture). Image showing the relationship between the Dump Tank and TSV Hot Cell. These were connected via L-shaped transfer lines shielded by lead bricks (seen in picture).

2.3.2.6 Effluent Cart

The effluent cart was a thick steel box (with 1-in.-thick sides) on wheels, which housed the seven effluent bottles that collected waste from processing the target solution. Four different sizes of bottles were used to collect effluent: 64 oz, 1 gal, 2 gal, and 5 gal. The three smaller bottles were made of polyethylene (PE) with silicone seals at the caps, while the 5-gal containers were composed of high-density polyethylene (HDPE). The seven effluent bottles were contained within a PE tray which was placed on a 25-kg balance (Ohaus, model D25QRUS, 0.002 kg resolution) inside the cart. A rotating cam mechanism was installed in the bottom of the steel box to lift the effluent-bottle secondary tray off the balance during transport, when re-connecting the balance cables and establishing a connection between the balance and its indicator, and when taring the balance once the cart was positioned in its cabinet. Two scissor jacks were used to level the effluent cart once it was positioned in its cabinet, to ensure accurate balance readings. The lid of the steel shielding box was split, requiring only half the lid to be removed to replace effluent bottles. To the half of the lid that remained attached to the box, attached were two manifolds: one to connect the liquid lines from each effluent bottle to the associated effluent line in the glovebox and the second to connect the vent lines from each effluent bottle to the line running to the gas collection system (GCS). An image of the effluent cart with the manifolds in place in cabinet #3 is shown in Figure 2.3.2.6.1. Two effluent carts were kept on hand so one could be used in processing while the other was emptied, to expedite the irradiation schedule.

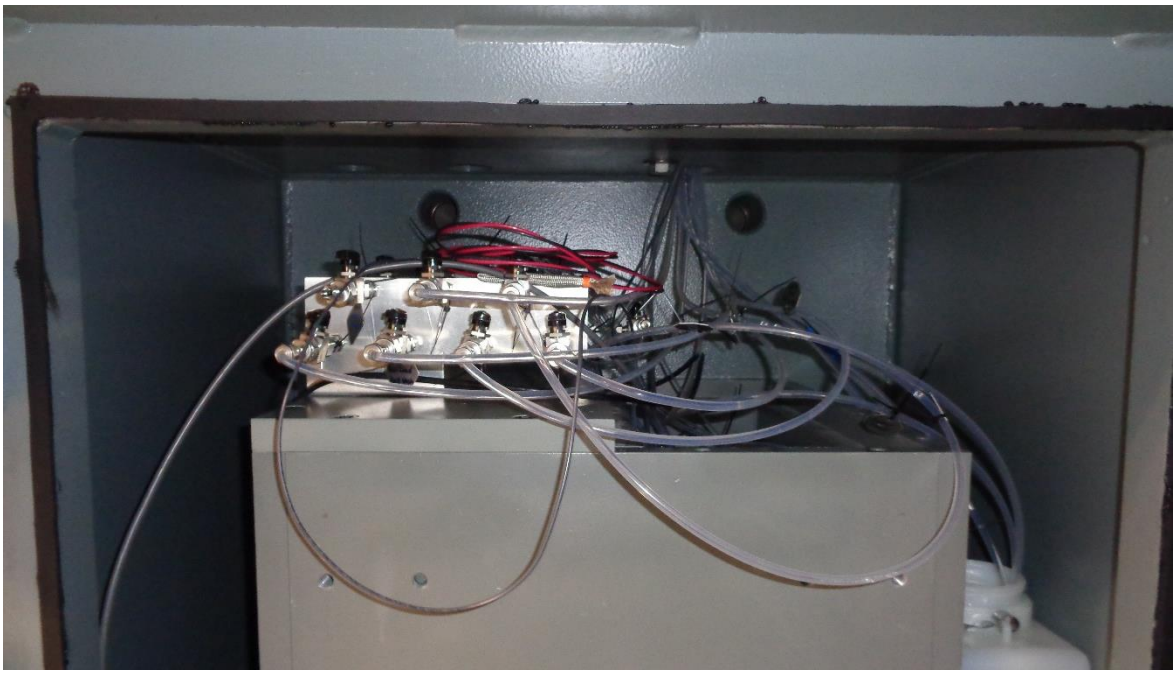

FIGURE 2.3.2.6.1 Effluent cart with lid and valve manifold on top, in place inside cabinet # 3

2.3.2.7 Verification Tank

The verification tank was used to determine the total mass of uranyl sulfate target solution and to take samples of the target solution to determine density, U concentration, and pH. The determination of these parameters was critical, as the ASE specified the maximums of these parameters. This procedure is discussed further in Section 2.3.4. Verification also was one of the few processes that utilized clear FEP lines, through which the target solution could be directly viewed and inspected to ensure that it was free of precipitate. The verification tank itself was a 35 L stainless-steel vessel (Eagle Stainless, KTT-CTH-36-316L-J, slant bottom, 1.5 in. Tri-Clamp with EPDM gasket bottom connection; the clamped lid seal was a platinum-cured silicone gasket, 10 Ra finish) that resided on a 50-kg balance (Ohaus, model D50QLUS, 0.005 kg resolution) in a shielded cart with 1-in.-thick steel sides, a diagram of which is shown in Figure 2.3.2.7.1. As in the effluent cart design, a rotating cam mechanism was installed in the bottom of the steel box to lift the verification-tank secondary tray off the balance during transport, when re-connecting the balance cables and establishing a connection between the balance and its indicator, and when taring the balance once the cart was positioned in its cabinet. Two scissor jacks were used to level the verification tank cart once it was positioned in its cabinet to ensure accurate balance readings. When connected to the glovebox system, valves V-2034, V-2035, and V-2036 on the verification tank were connected to pipes P-136 (G8), P-506 (E2), and P-126 (G6) on the glovebox system, respectively. Valve V-2037 was not connected to the glovebox system, as it is the “sparge line.”

FIGURE 2.3.2.7.1 Drawing of the verification tank resting on its balance inside the shielded verification cart

2.3.2.8 In-line Sampling Ladders

In addition to the samples taken from the verification tank prior to irradiation, it was often desirable to take samples at different points during irradiation, circulation, and processing. Owing to the high dose rates in the irradiation cell, this sampling had to be done remotely, and was accomplished using sample ladders located in the glovebox. These sample ladders were designed such that the target solution would pass through the sampling ladder "rung" until a sample was to be collected. Figure 2.3.2.8.1 illustrates the structure of an individual sample ladder rung with arrows indicating the flow path of solution. In the figure, valves V-0100 and V-0102 would be closed when a sample needed to be taken and solution would begin flowing to the next higher rung in the ladder. The other valves in the diagram, V-0101 and V-0103, were used when retrieving samples after the irradiation.

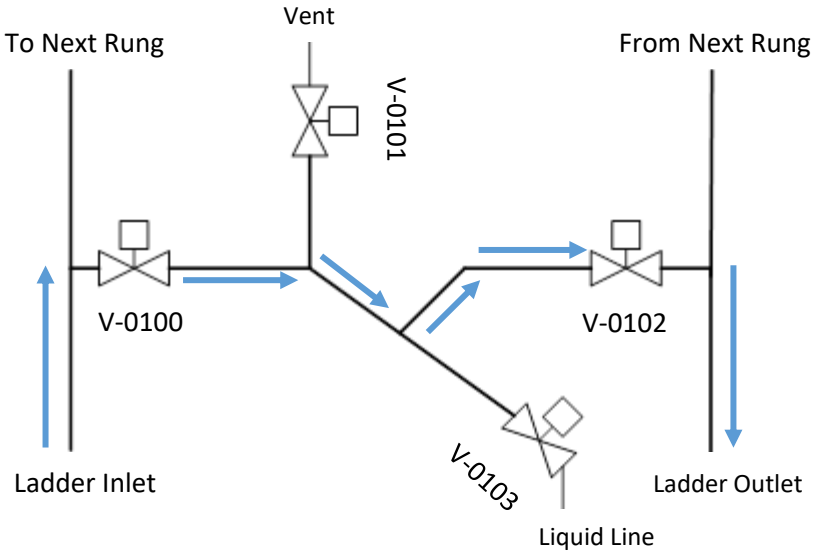

FIGURE 2.3.2.8.1 Schematic diagram of a sample ladder rung with arrows showing the flow of solution during normal operation

For the sample ladder depicted in Figure 2.3.2.8.1, V-0101 would be connected to the glovebox atmosphere through a one-way check valve (vent line), while V-0103 would be connected to a $\frac{1}{16}$ in. FEP liquid line running to the needle setup shown in Figure 2.3.2.8.2A. The concentric needles in Figure 2.3.2.8.2A penetrated through the septum of an evacuated glass vial, and the ⅛ in. vent line attached to the outer syringe needle was run to a vacuum pump through a liquid trap. When a sample needed to be retrieved, valves V-0100 and V-0102 were closed while V-0101 and V-0103 (FIGURE 2.3.2.8.1 Schematic diagram of a sample ladder rung with arrows showing the flow of solution during normal operation 2.3.2.8.1) were opened and the vacuum pump was engaged to pull the sample into the evacuated vial. Each glass vial was also held in a stainless steel or tungsten sample pot for extra shielding, as samples could be highly radioactive. An image of several of the sample retrieval vials in sample pots with the concentric needles inserted can be seen in Figure 2.3.2.8.2B.

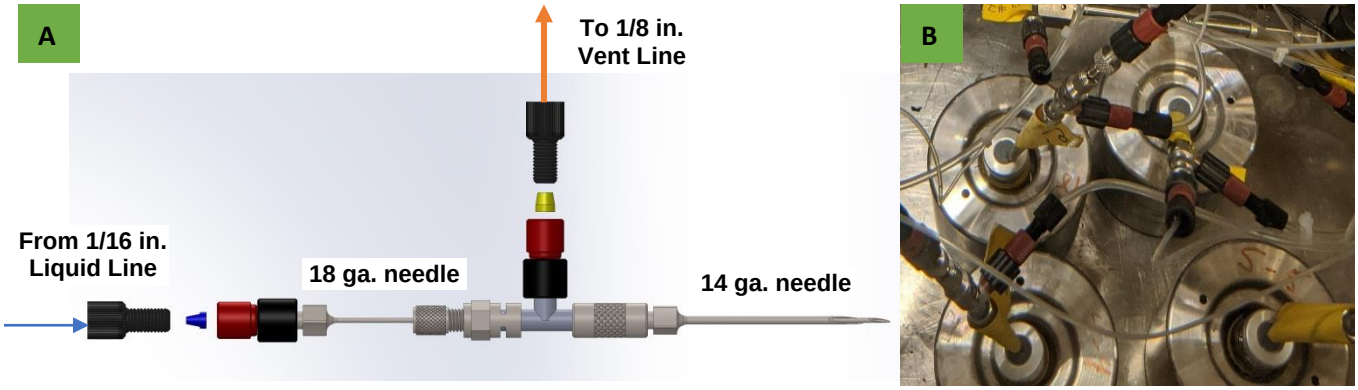

FIGURE 2.3.2.8.2 (A) Diagram of the concentric needle system used to retrieve samples from sample ladders. (B) Needle system with sample vials in sample pots in the glovebox.

2.3.2.9 Remote-operation Software

The radiation levels in the beamline cell (D035) during and directly after irradiation were much too high to operate the system manually. To give an idea of the situation, when 8 Ci of ^{99}Mo were produced, the dose rate at the face of the shielded glovebox three days after the end of bombardment (EOB) was 300 mR/hr. This radiation was generated by target solution in the sampling loops and by activation of the glovebox components during irradiation, as the vast majority of the uranyl sulfate target solution was contained in the shielded dump tank at the time. To retrieve the produced ^{99}Mo as soon after target-solution irradiation as possible required that the primary recovery system be remotely controlled. A process control program, developed in National Instruments™ LabVIEW software, was used to remotely operate all solenoid valves and record process parameters. A total of 92 control signals were used to operate the 171 solenoid valves in the system. The LabVIEW -based process control system was designed to operate in either a manual or semi-automated mode. In manual mode, the operator had full control of all the liquid solenoid valves, while in semi-automated mode the operator simply pressed the NEXT STEP button and the appropriate solenoid valves would open and close to proceed with the next step of processing. Though the main user interface display appears complicated (Figure 2.3.2.9.1), the flow path of the liquid was highlighted in accordance with the solenoid valves that were open, making it easy for the operator to check that the appropriate step was being followed.

The program captures the temperature of acid- and base-line heaters, as well as the column temperature. The pressure is also measured at six different locations throughout the system, and data are collected on the masses of the feed bottles, effluent bottles, and verification cart (when installed), as well as on the pressures of the gas collection reservoirs and temperatures in the linac. Data are collected by the LabVIEW system every two seconds, and each data point is associated with a time stamp so data from irradiations could be reviewed later.

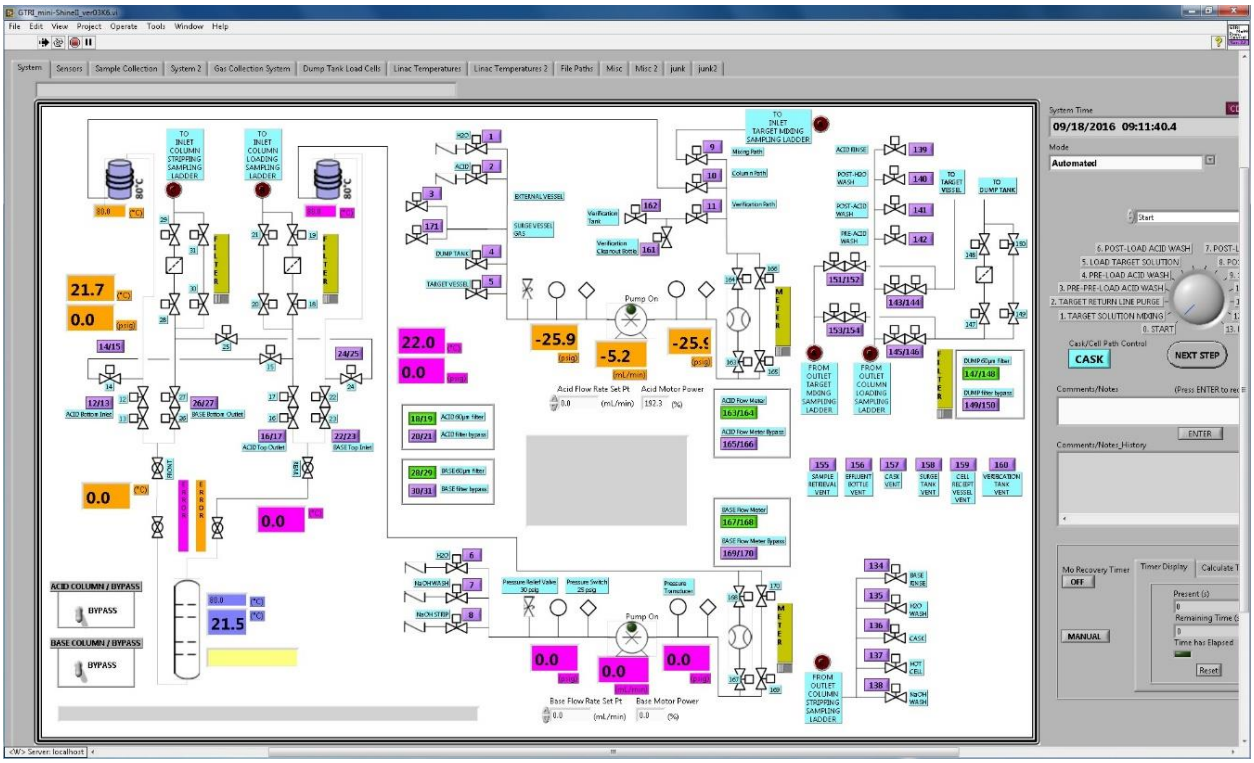

FIGURE 2.3.2.9.1 Main LabVIEW user interface for remote operation of the recovery glovebox

2.3.2.10 Leak Sensors

In addition to needing to pump solution around the system remotely, it was advantageous to be able to tell if the system was leaking at any point during processing. If a leak were occurring, processing would halt and the system put in a safe condition so that all the solution would not be pumped out into the glovebox cavity or wherever the leak was occurring. Leak sensors were distributed throughout the glovebox system. Locations included the dump tank, effluent-bottle secondary tray, feed-bottle secondary tray, surge-vessel secondary tray, and two on the floor of the glovebox. In addition, a sensor was placed in the column pot to detect possible leaks from the column. If moisture were detected by a leak sensor, a light on the alarm panel would illuminate and an audible alarm would sound until the leak sensor was dry.

The leak sensors themselves operated by having moisture complete a circuit. They were composed of two wires with approximately ½ in. of bare wire exposed, separated by a moisture-absorbing material. The moisture-absorbing material also surrounded the wires to ensure that they did not short out, and everything was zip-tied in place. Figure 2.3.2.10.1 shows an image of one of these sensors. The other ends of the two wires were then connected to the alarm system. If moisture were present, it would be absorbed by the wire-surrounding material and complete the circuit, thus sounding the alarm.

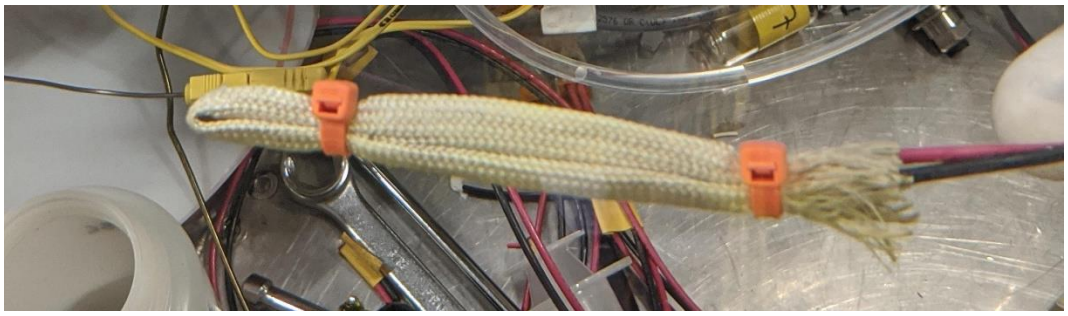

FIGURE 2.3.2.10.1 One of the leak sensors used in the glovebox system, composed of an adsorbent tube folded in half with one wire inserted in each side. The wires and adsorbent are held in place by zip-ties.

2.3.3 Sampling

All sampling was based on mass. In addition to recording the mass of all samples taken, the mass of the target solution was verified prior to each irradiation and the masses of the reagents used during processing were tracked at both the feed bottles and effluent bottles. The mass of solution used to strip the column was also verified by measuring the mass of solution transferred to the hot cell at the end of processing. This was done because it was possible to achieve lower relative uncertainty via mass than via volume. This lower uncertainty, in turn, minimized the uncertainty associated with parameters determined for the target solution and the numbers reported for production and recovery of ^{99}Mo. It also allowed for the direct calculation of total product and contaminants in the various solutions from the samples.

2.3.3.1 Target Solution

Samples were taken to assess target solution parameters directly from the verification tank via the ⅛ in. O.D. FEP sparge line, which had a manual valve attached to the end. To obtain these samples, a 60 mL syringe with a 3-way Luer valve was connected to the manual sparge-line valve (V-2037), as shown in Figure 2.3.3.1.1. A small amount of solution was drawn into the syringe, then the 3-way valve was switched and the sample was dispensed into a sample bottle. Following dispensing, the syringe was used to inject air into the sparge line and force the remaining target solution back into the verification tank.

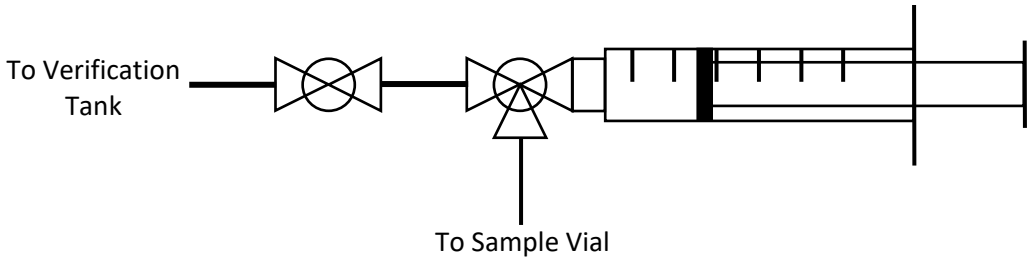

FIGURE 2.3.3.1.1 Diagram of sparge line connection used for sampling the target solution

If a sample of the target solution had to be taken when the verification tank was not installed for some reason, this could be done through valve V-2001 (G6 of Figure 2.3.2.2.4). To utilize this port, a small length of ¼ in. O.D. FEP tubing was attached to the valve using Swagelok fittings and fed through an HDPE Nalgene bottle lid. To execute the sampling, a bottle was screwed on to the lid attached to the port and the FMI pump was turned on to circulate the target solution through the system. When solution reached the open port, it would push solution into the waiting bottle. Once an adequate sample was retrieved, the FMI pump was reversed to pull any solution remaining in the FEP tubing back into the system; then the port was closed, and circulation could continue or the target solution could be returned to the TSV. After these steps, the bottle was unscrewed from the port attachment and capped before being removed from the glovebox.

2.3.3.2 In-line Samples

Following an irradiation and processing, samples taken using the in-line sampling ladders had to be retrieved. As mentioned in Section 2.3.2.9, high radiation levels prevented entry to the beamline cell for several days after the EOB. Once sufficient time had elapsed for the radiation levels to fall to reasonable levels (usually 3-4 days), entry was made and it was possible to retrieve samples from the sample ladders for gamma counting. A separate LabVIEW program was used for sample retrieval (Figure 2.3.3.2.1), and the vacuum pump used to ensure that samples moved to the evacuated vials in shielded pots had to be turned on manually at the glovebox for each retrieval. Each sample ladder was also connected to a manifold of manual valves that had to be manipulated to direct the vacuum to, and open to atmosphere, the appropriate sets of solenoid valves. An example of one of these manifolds is shown in Figure 2.3.3.2.2; the valves controlling which sample ladder rung is exposed to the glovebox atmosphere (through a one-way check valve) are along the top, and the corresponding valves controlling which sample vial and ladder rung are exposed to vacuum are along the bottom.

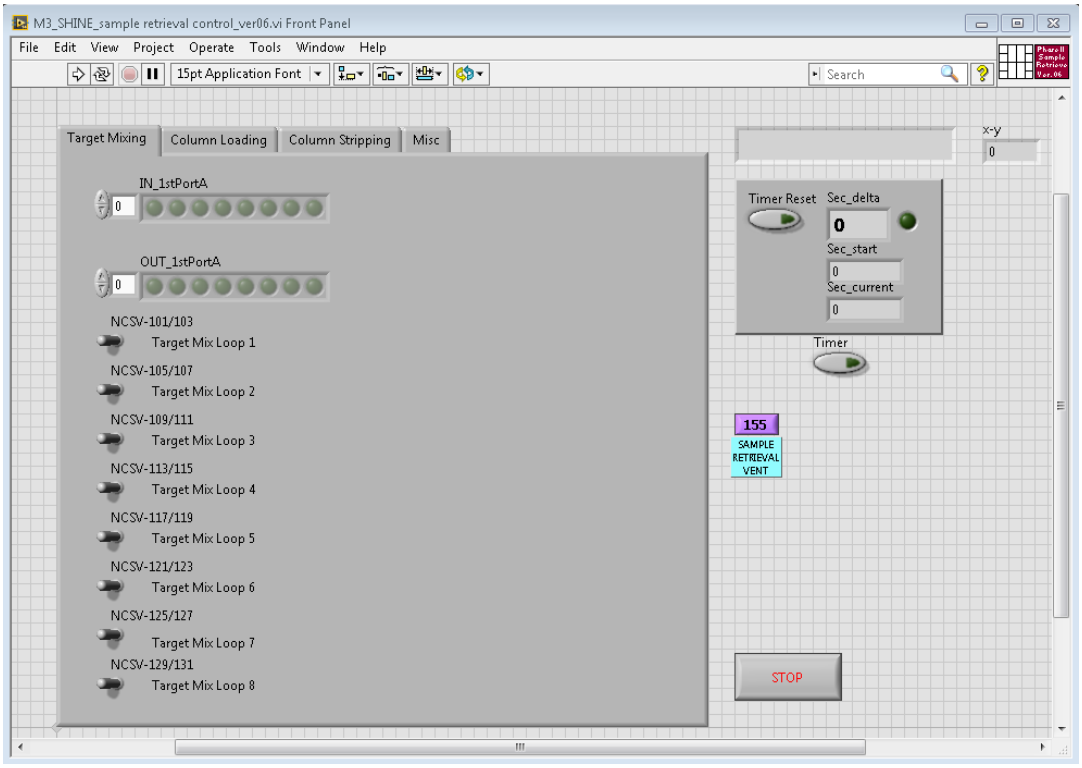

FIGURE 2.3.3.2.1 LabVIEW operator interface for sample recovery after irradiation and processing

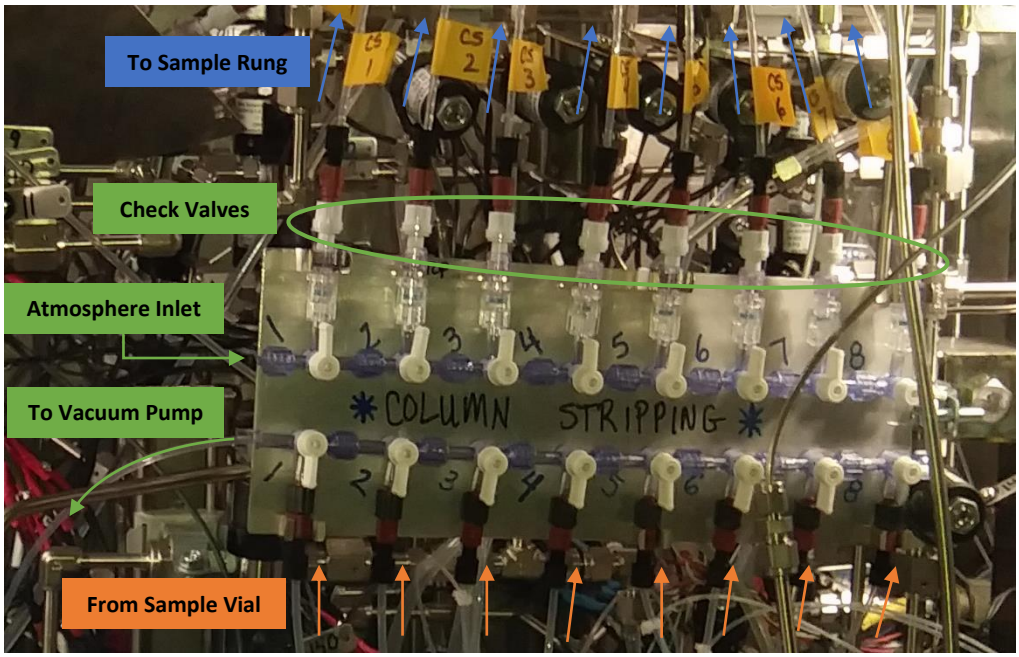

FIGURE 2.3.3.2.2 Manifold used to direct atmosphere and vacuum to the appropriate sample ladder rung. White one-way check valves preventing backflow of gases into the glovebox are circled in green.

Over time, the sampling ladders began only working intermittently. Initially, it was thought that this was because the vacuum pump used to pull the samples from the sample loops was not working. The power supply of the pump had been damaged by several rounds of radiation exposure, and replacing it fixed the pump. Unfortunately, fixing the pump did not allow retrieval of most of the samples, so it was thought that the plastic pieces used in the manifolds were leaking. This seemed plausible, as they had not been replaced in several months and had also been exposed several times to high radiation levels, which can degrade the plastic. Upon replacement, however, it was found that many of the samples still could not be retrieved from the ladders. Finally, it was determined that one or both solenoid valves used for retrieving the samples from the ladders were not opening. It is still unclear whether this was happening because of radiation damage, buildup of precipitate, uranyl sulfate residue, or some other factor (see detailed discussion in Section 3.3).

While most of the sampling loops resulted in data that were informational but not necessarily consequential to the experiment, it was not urgent to find a solution until none of the samples from the target mixing sample ladder could be recovered. This is because the target mixing sample ladder was the only place in the system where a sample could be taken that was representative of the target solution at the EOB and would allow for the calculation of ^{99}Mo production and process yields. As more of the target mixing sample ladders stopped functioning, it was decided to install an alternate sample loop that used only manual valves so a sample of the target solution at the end of irradiation could be retrieved reliably. While the manual valves slightly increased the time when workers were required to be in the high radiation field for a single sample, only one sample was captured instead of up to 24 samples that would have been captured using the sample ladders, and therefore the total time a worker spent in the radiation field was decreased. The alternate sample loop was placed in the acid-side flow meter bypass loop between solenoid valves V-0165 and V-0166 (G6 of Figure 2.3.2.2.4). In Figure 2.3.3.2.3, the alternate sample loop is shown with the vent manual valve pointing left and the retrieval manual valve pointing towards the camera. The retrieval valve was connected to ⅛ in. FEP liquid line that led to the same concentric needle setup used with the sample ladders, replacing the 1⁄16 in. liquid line. This alternate sample loop was operated in much the same way as the loops in the sample ladder, in that the solenoid valves on either side were closed while the vent and sample-line valves were opened and vacuum was pulled on the associated sample vial. When this sample loop was implemented, no other samples were collected in the normal sample ladders to minimize the dose to workers when they went to retrieve this sample. This sample loop could also be used as an alternate way to collect samples of the target solution if the verification tank was not installed.

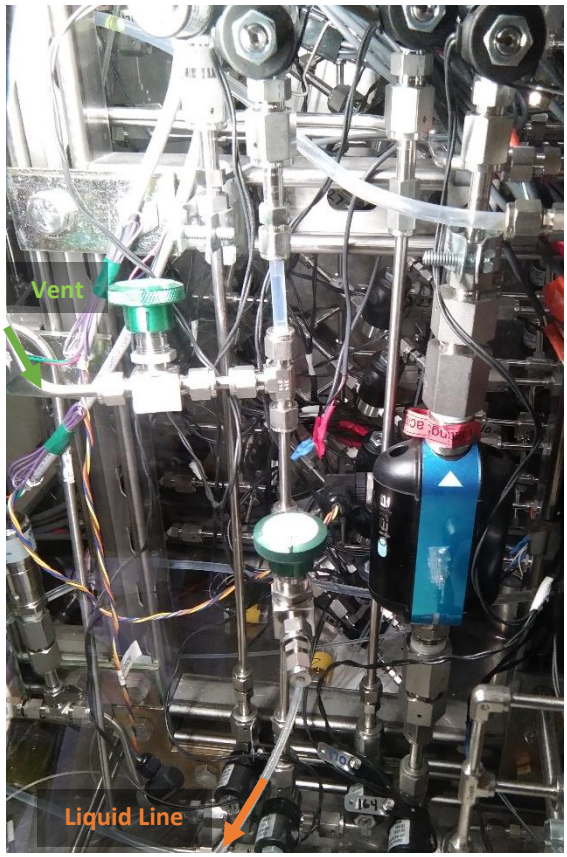

FIGURE 2.3.3.2.3 Alternate sample loop with manual valves used in later irradiations

After being retrieved from the glovebox, samples were taken to a laboratory and diluted by mass to prepare them for gamma spectroscopy. See Section 2.5 for details on gamma instrumentation and analysis methods.

2.3.3.3 Effluent Bottles

In certain instances later in the experimental sequence, it was also desired to take samples from the various effluent bottles. To do this, the ¼ in. O.D. FEP liquid lines feeding the effluent bottles were disconnected at the double-valve joint in the effluent-cart manifold and 1⁄16 in. O.D. polyether ether ketone (PEEK) tubing was threaded through the line into the bottles. A syringe was then attached to the PEEK tubing using a Luer valve fitting, and the solution was pulled into the syringe. Once the desired volume had been obtained, the Luer valve was closed and the PEEK line was cut off and allowed to drop back into the eluent bottle to minimize the chance of radiological contamination and release of fission gasses into the glovebox environment. Diagrams of the effluent bottle, tubes, valves, and syringe during sampling and after cutting off the 1⁄16 in. PEEK line are shown in Figure 2.3.3.3.1A and B, respectively.

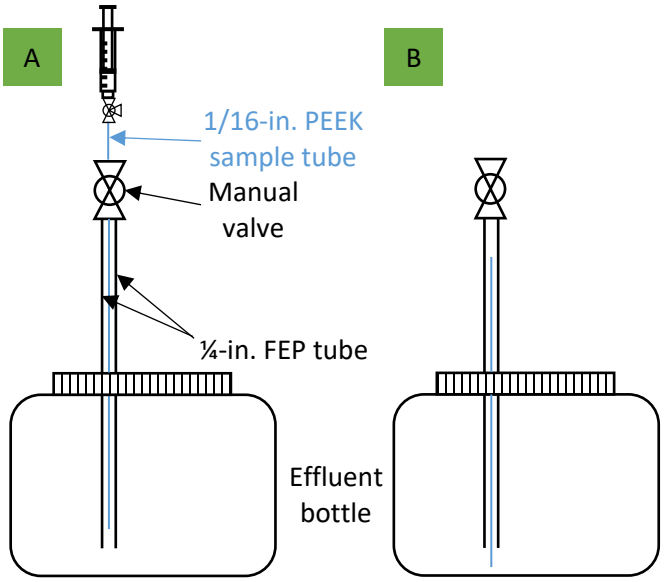

FIGURE 2.3.3.3.1 Diagram of sampling setup for effluent bottles (A) while sampling with a syringe and (B) after cutting the PEEK tubing and allowing it to drop back into the bottle

2.3.4 Pre-start Checks

Prior to irradiation, it was necessary to determine if the target solution was within acceptable specifications set by the ASE. The ASE specified the concentration of LEU at 120–140 g U/L, and total volume of the target solution between 5 and 20 L at the beginning of the irradiation, to ensure safe operation of the target vessel. The pH of the target solution was also specified to be between 0.95 and 1.05 to ensure proper loading of Mo on the column. Often, after an irradiation and processing, it was found that the uranium concentration in the target solution was low. This was a result of small amounts of acid and water being held up in the glovebox system even after purging it with N_2 after the previous irradiation and diluting the target solution as it passed through the system. This holdup also caused minor fluctuations in the pH of the target solution. In addition to the slight dilution from residual processing solutions, it was found that the irradiated target solution would precipitate uranyl peroxide upon standing unless Fe was added to the solution (this phenomenon is discussed in Reference [5]).

Samples of the target solution were retrieved directly from the verification tank when determining if ASE specifications were being met. This retrieval was carried out using the sparge line, as described in Section 2.3.3.1. If any of the ASE parameters were found to be out of specification, calculations were performed and makeup solution was added. As mentioned above, the most common issue was that the U concentration was found to be low. To correct this problem, the makeup solution was prepared by dissolving LEU from the same batch used to make the target solution in a small amount of H_2SO_4, resulting in a concentration of 636 g U/L at pH 1. It was also possible to add water or concentrated H_2SO_4 to adjust the pH, although this adjustment was not required during these experiments. In addition to adjusting the uranium

concentration and pH, a small amount of stable Mo carrier was added prior to each irradiation to facilitate chemical processing post-irradiation and when Fe was added to prevent uranyl peroxide precipitation.

All of these solutions were added to the target solution directly through the same sparge line setup described for taking samples from the verification tank. To adjust the target solution, the make-up (and carrier) solutions were mixed in a bottle, then drawn into a 60 mL syringe through the 3-way valve connected to the verification-tank sparge-line valve. Once the syringe was full, the 3-way valve was switched, the manual sparge valve was opened, and the solution was injected into the verification tank. Once all the make-up solution was injected, air was injected using the same process to ensure that all the solution was forced into the verification tank from the sparge line before closing the manual sparge-line valve and disconnecting the syringe and 3-way valve.

To mix the target solution after any adjustments, the solution was pumped to the dump tank and returned to the verification tank. Upon returning the solution to the verification tank, sampling and analysis were repeated to ensure the solution was within the ASE boundary conditions.

2.3.5 References

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[2] Stepinski, D., and Vandegrift, G.F., *SHINE and Mini-SHINE Column Designs for Recovery of Mo from 140 g-U/L Uranyl Sulfate*, ANL/NE-16/11, Argonne National Laboratory, 2016.

[3] Stepinski, D.C., Abdul, M., Youker, A.J., Rotsch, D.A., Tkac, P. Chemerisov, S., and Vandegrift, G.F., *Optimization of SHINE Process: Design and Verification of Plant-Scale AG 1 Anion-Exchange Concentration Column and Titania Sorbent Pretreatment*, ANL/NE-16/7, Argonne National Laboratory, 2016.

[4] Youker, A.J., Stepinski, D.C., Ling, L., Chung, P.-L., and Vandegrift, G.F., *Plant-Scale Column Designs for SHINE Target Solutions*, ANL/CSE-14/24, Argonne National Laboratory, 2014.

[5] Kalensky, M., Chemerisov, S., Youker, A.J., Tkac, P., Krebs, J.F., Quigley, K., Lowers, R., Bakel, A., and Vandegridt, G.F., *Means to Eliminate Uranyl Peroxide Precipitation in SHINE Target Solution*, ANL/CSE-13/21, Argonne National Laboratory, 2013.

2.4 HOT CELL PROCESSES

2.4.1 Concentration Column

2.4.1.1 Experimental Design Features of the D-024 Hot Cell

The ^{99}Mo produced by subcritical fission of an aqueous solution of uranyl sulfate was recovered from the irradiated solution on a titania column, washed with acid and then water, and eluted with base. The alkaline strip, containing the desired ^{99}Mo, was transferred directly to the D-024 Hot Cell (Figure 2.4.1.1.1) for final purification.

A solution transfer line for Phase II, which consists of 1/4-in.-diameter 304 SS tubing in 6-in. lengths, welded end-to-end to form an ~90-ft length, was installed during the installation of the Phase I line. Both lines are held inside the secondary containment. Prior to sealing the secondary containment, the lines were vacuum- and liquid-tested. Minimal solution holdup was observed while testing liquid transfers in the lines. Several liquid monitors were installed in the secondary containment for leak detection. Both lines were connected via swage lock fittings to valves within the D-024 Hot Cell.

FIGURE 2.4.1.1.1 The D-024 Hot Cell, where final purification of ^{99}Mo is performed

The interior of the D-024 Hot Cell was designed to accommodate various aspects of the project and allow for adaptation to the research being performed. An image of the interior of the D-024 Hot Cell is shown in Figure 2.4.1.1.2; various features are pointed out in the image. A quick-connect system was installed that enables three vacuum connections for simultaneous use, water in and out, and the option for compressed gas (Figure 2.4.1.1.3). These features rest on scaffolding that was welded to the inner back wall of the hot cell to increase floor space and, thus, workspace. The scaffold supports the following:

- the quick-connect system
- vacuum line
- vacuum traps
- gas scrubbers
- pumps
- pH probes
- solution vessels
- columns
- the concentration-column valve board
- valves for the ^{99}Mo transfer lines
- electrical wiring

It also serves as a barrier to protect and manage various lengths of tubing.

Electric controls for heaters, stirrers, balances, pH probe readout, lights, etc., were placed on the exterior of the hot cell. Consideration was given to the placement (interior or exterior of the hot cell) of systems that require routine replacement or recharging.

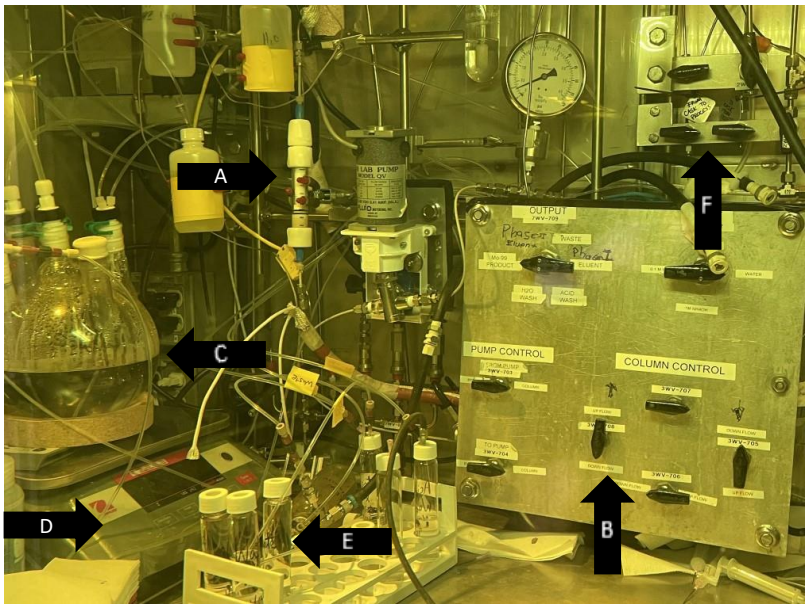

FIGURE 2.4.1.1.2 Image of interior of the D-024 Hot Cell: concentration column (A); column control board (B); acidification vessel (C); balance (D); reagent vials (E); and valves for exterior solution line (F)

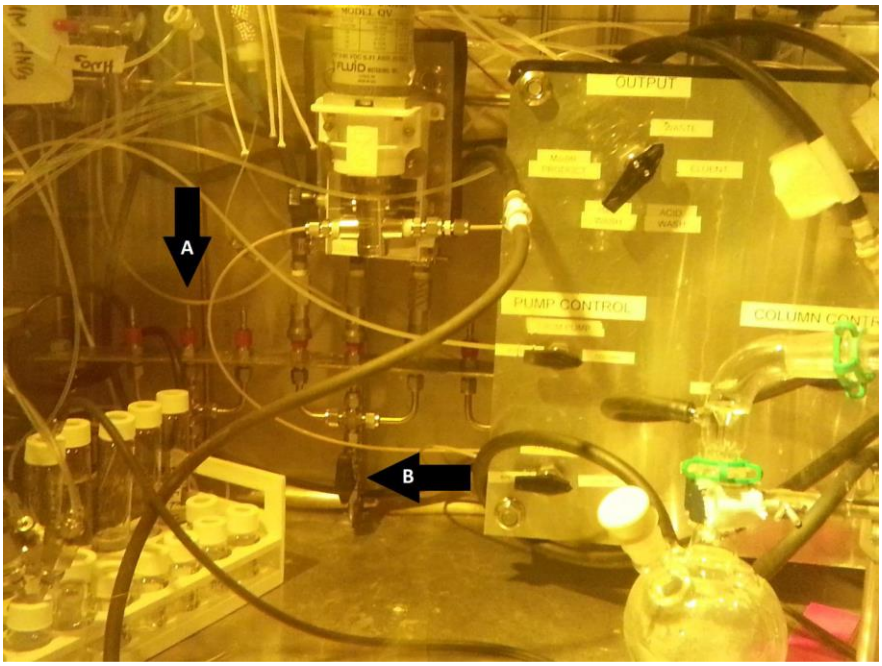

FIGURE 2.4.1.1.3 Image of quick-connect system: compressed-gas connection (A); exhaust valve connecting to gas collection system (B); and water in/out (hidden behind the board)

The new items installed for the Phase II experiments include a larger vessel—a 3-L, 5-neck, plastic-coated flask (Figure 2.4.1.1.4)—to receive the solution from the primary recovery glovebox. An ultra-thin magnetic stirrer was placed between the top-loading balance and the flask. The pre-weighed flask was held in place by a lightweight cork ring. The concentration column, which used 0.8 mL of titania sorbent in Phase I, was updated to use 1.5 mL of sorbent for the Phase II experiments. A liquid trap was installed between the GCS and the connections inside the D-024 Hot Cell as an added safety feature. Also, a series of six 5-L vessels were installed beneath the hot cell inside a plastic secondary container. These vessels had their headspace connected to the GCS to vent any residual fission gases when the irradiated solutions were sent to the vessels for storage until their ultimate disposal. These vessels were all leak-checked before installation. Finally, before the last experiment, all the plastic lines and tubing were replaced. During one of the practice runs, a leak was observed coming from behind the valve board. When troubleshooting the leak, it was found that the valve had become extremely brittle and had developed a spiral crack when the needle on the line was being placed into the receiving container. Some of the other plastic lines were found to be in a similar condition, so the decision was made to replace all lines. A protocol was established for replacing all lines within 2 years of installation, to prevent future equipment failures of this type.

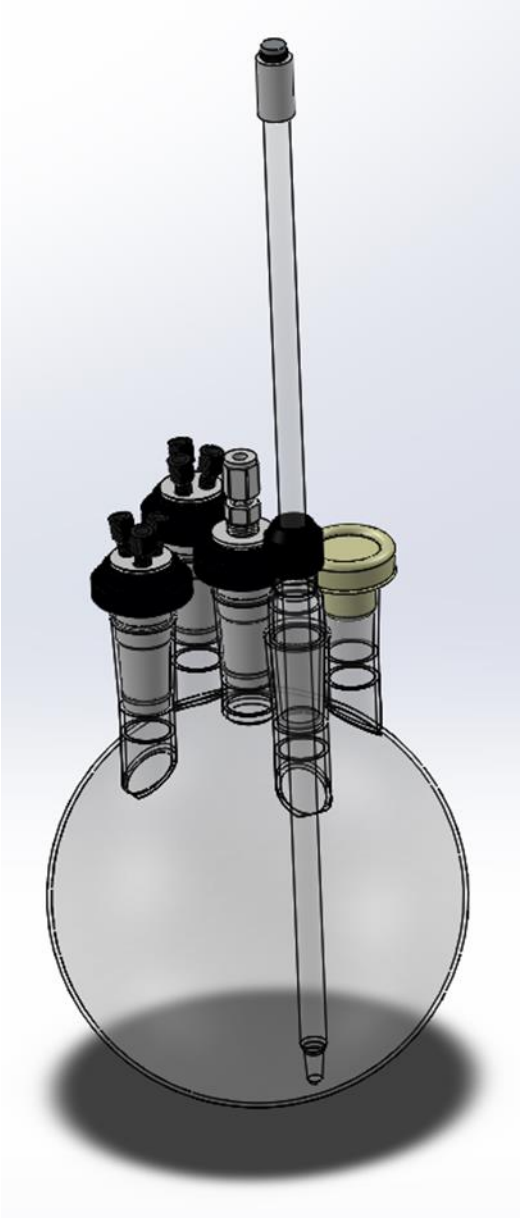

FIGURE 2.4.1.1.4 Diagram of the 3-L, 5-neck vessel used for receiving and acidifying the ^{99}Mo product prior to loading on the concentration column

Before each experiment, a checklist was followed to ensure that the hot cell was prepared for the coming run. This checklist included collecting all sampling syringes, 60-mL collection vessels, and 20-mL scintillation vials with septum tops; refilling the solution reservoirs inside the hot cell with water, 1.0 M NaOH, and 0.01 M HNO_3; placing 8 M HNO_3 inside the 3-L flask; and installing the concentration column. Before installation, the concentration column was packed with 1.5 mL of titania sorbent (S40, 40-micron particle size, 60-angstrom pore size). The sorbent was prepared in advance by putting the sorbent into purified water and shaking. Once the sorbent settled, the supernatant was removed using a pipette to remove the fine particles. This procedure was repeated multiple times, until the solution above the settled resin was clear and colorless. The sorbent was then packed into the column using the slurry method. The packed column was then attached to an FMI pump, and purified water was run through the column in the upflow and downflow directions for at least one hour in each direction. The effluents were then checked to make sure all fines had been removed. The column was then inspected for any channeling, and the column caps were tightened in case the resin had compressed during the flow tests. The pH probe was calibrated with pH standard solutions as close to the date of the experiment as possible, and the probe was then placed inside the 3-L flask. All ports were checked and confirmed to be sealed to the vessel as a final check before receiving any solution from the glovebox team.

Immediately before receiving the solution from the glovebox, a system interface step was performed, with a member of each team present to verify the conditions inside the D-024 Hot Cell. A member of each team signed off on the checklist in both sets of work instructions. The steps included ensuring that all the ports on the 5-neck vessel were properly sealed, the pH probe was in place, the balance was zeroed and the weight was recorded, and finally the proper valves were opened and connected to the GCS.

2.4.1.2 Concentration-Column Design and Operation

The ^{99}Mo product from the recovery column, in 1.0 M NaOH solution, was received into the 5-neck, 3-L vessel within the D-024 Hot Cell. The ultra-thin magnetic stirrer was activated to mix and sample the solution prior to acidification and concentration. Sampling the solution provides an accurate account of the product strip from the recovery column, which allows for determinations of experiment and equipment performance. Approximately 1700 g of solution was transferred from the recovery glovebox to the flask in the D-024 Hot Cell. This volume is too large to be handled with the LMC process (maximum volume, 100 mL). Therefore, the solution was concentrated using a second column. A schematic diagram depicts the system used during concentration-column operation (Figure 2.4.1.2.1).

The column separation system consists of an FMI positive-displacement pump, which allows for forward and reverse flow; Swagelok 3-, 4-, and 5-way switching valves; and Hamilton 3-port flow valves with a T-plug to direct the process streams. All the valves were mounted on a board, and this valve board was mounted on the previously mentioned scaffold. Reagent bottles were mounted on the scaffold and connected to the valve board using 1/8-in. HDPE tubing. Stainless steel tubing (1/8 in.) was used to connect the valves to each other and the preheater, and HDPE or PEEK tubing was used to connect the column, reagent, and collection bottles. The concentration-column system is equipped with a solution preheater, and the solution lines feeding into the column are wrapped with heating tape. During the experiment, any vessels or sample vials receiving solution were connected to the GCS described in Section 2.2.2. A liquid trap was installed between the D-024 Hot Cell and the GCS to guarantee that no liquid could be transferred into the GCS or the gas-collection lines. This trap was inspected and verified before and during the experiments and is located directly above the valve board inside the hot cell. The eluent from the feed and wash steps of the concentration column was collected in a 5-L storage vessel inside the hot cell; the headspace of this vessel was also plumbed to the GCS. The effluent was to be held in this vessel until the majority of the fission nuclides decayed. Once the radiation field had dropped sufficiently, the effluent solution was transferred to the effluent storage flasks underneath the hot cell. The effluent storage area was lined with multiple layers of lead brick and connected to the GCS. All storage vessels were leak-checked before use with actual column-effluent solution. The effluent and rinse solutions were transferred into these vessels for storage until the solutions were ready for disposal.

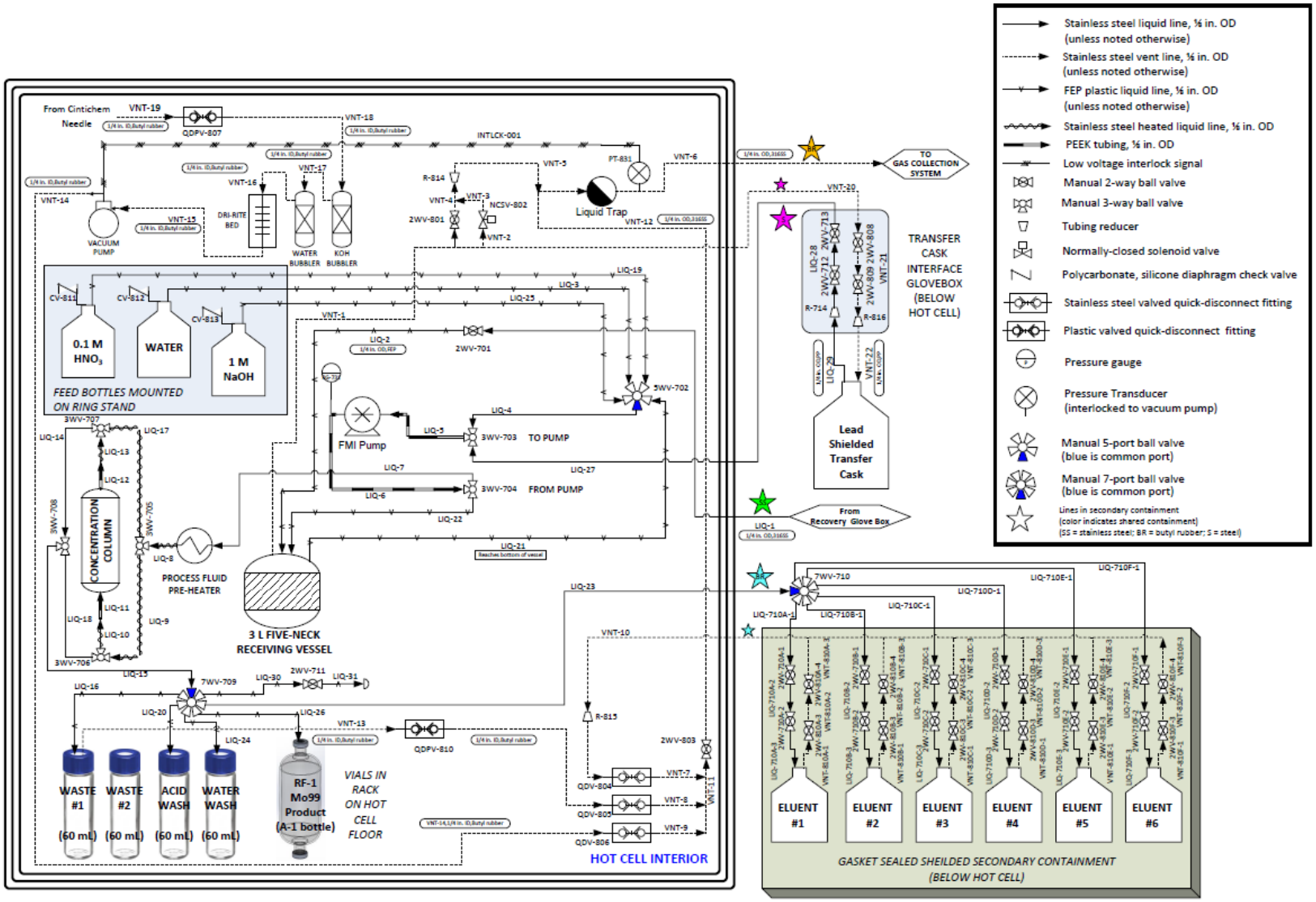

FIGURE 2.4.1.2.1 Schematic diagram of ^{99}Mo concentration column

After the ^{99}Mo solution was sampled, the pH of the 1.0 M NaOH solution in the 3-L receiving vessel was adjusted with 8 M HNO_3 to pH = 2 (monitored with a calibrated pH probe). The acidification was performed dropwise with a 60-mL syringe fitted with a 1-in. needle. The needle was inserted into the port fitted with the rubber septum. To simplify the acidification process, approximately 75 percent of the calculated value of 8 M HNO_3 necessary to reach pH 2 was placed in the 3-L flask when preparing the hot cell for the experiment. The stir plate underneath the flask was activated before final acidification to ensure proper mixing and uniformity of the solution. The system was then purged of air by passing water through the lines. The dead volume of the system (15 mL) was significant; hence, column pre-equilibration, wash, and strip volumes were increased from the volumes of previous experiments to ensure that an appropriate volume reaches the column and is collected. All equilibration, washing, and elution steps were timed steps, and therefore, a stopwatch was used by the operator working the pump. The operator working the manipulators was responsible for verifying that the solution was being transferred into the appropriate container at the desired flow rate. The pre-equilibration, loading, and washing steps were carried out at 80°C. The FMI pump was kept at a power of 67.2% for these steps, which corresponded to a flow rate of 50 mL/min. The flow rate was verified during the installation and preparation of the hot cell. An Omnifit BenchMark column packed with S40 Sachtopore sorbent (1.5 × 1 cm, 1.5-mL bed volume) from ZirChrom Separations, Inc., Anoka, MN, was pre-equilibrated with 22 mL of 0.01 M HNO_3 and subsequently loaded with 1.7 L of acidified ^{99}Mo solution flowing at 50 mL/min in the upflow direction. Subsequently, the column was washed with 44 mL of 0.01 M HNO_3 and 44 mL of water solution at 50 mL/min in the upflow direction at 80°C. During the product-stripping step, the initial 10 mL of strip solution was loaded, and eluent was collected in the water-wash collection flask (to remove the water present in the system dead volume). Subsequently, ^{99}Mo was stripped with 66 mL of 1 M NaOH in the downflow direction at 11 mL/min at 70°C. For the stripping step, the FMI pump was set to a power of 14.8%, which was also tested and verified during the pump installation. As the last step, lines were rinsed with 50 mL of water, which was collected as waste. Each process stream was collected in pre-marked and pre-weighed bottles. Samples of the eluent, acid wash, water wash, waste, and final product were collected for analysis.

Approximately one week after the experiment was completed, output lines and lines that had contained the ^{99}Mo effluent or product were flushed with water. These rinse solutions were directed into the effluent storage container inside the D-024 hot cell. The line between the primary recovery glovebox and the hot cell was also rinsed and blown dry with compressed helium. The helium was regulated at 4 psi during the process so as not to overwhelm the GCS, which was operational for all solution transfers. The rinse solutions were all held inside the hot cell for a period of time before being transferred to the 5-L flasks in the shielded solution-storage vessels below the hot cell for long-term storage while awaiting disposal.

When the final AMORE run was performed, the concentration-column operations proceeded as planned; however, there were some issues. The 3-L, 5-neck flask in the D-024 Hot Cell received 1699 g of solution from the primary recovery glovebox. The solution was adjusted to a pH of 2 with approximately 175 g of 8 M HNO_3 with a counter-correction with 7.7 g of 10 M NaOH, resulting in a total volume of 1881.7 g. This solution was loaded onto the column with a set point of 50 mL/min, but it took approximately 1 hour to completely load the column. Following the wash steps, the column was then eluted into the appropriate vessel for the LMC

process to proceed. The initial feed sample indicated that approximately 9 Ci of ^{99}Mo was delivered to the D-024 Hot Cell. However, the sample taken from the RF-1 bottle before the LMC process had only 2 Ci of activity in it. These numbers account for only a 22% recovery rate from the concentration-column process. Analysis of a sample of the concentration-column effluent demonstrated that there was breakthrough from the column, but only of 500 mCi. There are a few possibilities that could account for the percent recovery rate. First, there may have been a sampling error, i.e., the sample was not adequately mixed and/or a representative sample of the solution was not taken. It is possible that the remaining ^{99}Mo was fixed on the column and unable to be recovered. In an attempt to determine the root cause, the column was eluted a second time with 50 mL of 1 M NaOH. This solution was collected, sampled, and analyzed via gamma spectroscopy. Unfortunately, because of issues with the GCS, this operation was performed on March 8, which was over 17 half-lives after the irradiation, so no ^{99}Mo or ^{99m}Tc was detected in the sample. We concluded that there could have been a few reasons for the activity to remain on the column. The reasons include channeling in the packing of the column, which would account for some of the breakthrough and inefficient elution; incorrect pH of the concentration-column loading solution; or the need for a larger elution volume to fully elute the column to recover all the activity. The other samples taken did not show any significant amount of ^{99}Mo, so we cannot definitively say what happened to the remaining activity.

Although the concentration column did not perform as intended during this experiment, the technology has been proven to be effective. The multiple experiments during Phase I and preliminary experiments of Phase II demonstrated a recovery of at least 90 % for the ^{99}Mo. Also, previous benchtop experiments performed with cold materials [1] found that the loaded ^{99}Mo from a 2-L solution should need approximately 16 bed volumes of 1.0 M NaOH to be sufficiently recovered. For these reasons, we believe there was another underlying issue during this particular experiment.

2.4.1.3 References

[1] Stepinski, D., and Vandegrift, G., *SHINE and Mini-SHINE Column Designs for Recovery of Mo from 140 g-U/L Uranyl Sulfate*, ANL/NE-16/11, Argonne National Laboratory, 2016.

2.4.2 LEU-Modified Cintichem Process

The LMC purification process is a small-scale process that accommodates selective precipitation of Mo from acidic conditions, where Mo is predominantly present as MoO_2^{2+}, using alpha-benzoin oxime (ABO). The complexation reaction between Mo and ABO can be expressed as follows:

$$MoO_2^{2+} + 2C_6H_5CH(OH)C(:NOH)C_6H_5 \rightarrow MoO_2[C_6H_5CH(O)C(:NOH)C_6H_5]_2 + 2H^+ \quad \text{(Eq. 1)}$$

It is important for Mo to be present in relevant chemical concentrations to form a precipitate with ABO, so for trace levels of Mo (as ^{99}Mo), Mo carrier needs to be added. The process was originally developed in 1974 for purification of ^{99}Mo produced by irradiation of

highly enriched uranium as UO_2 at the Cintichem facility in New York [1]. The Cintichem process was later modified by Argonne to allow processing of irradiated LEU targets dissolved in nitric acid [2–6].

Although the LMC process was developed for purification of fission-made ^{99}Mo from irradiated U metal or uranium oxide targets, it could also be utilized, with some minor modifications, for accelerator-driven production of ^{99}Mo from uranyl sulfate solution. This process has been demonstrated and discussed previously [7]. In summary, after the strip solution is received from the concentration column in 1 M NaOH, the solution is acidified by adding 10 M HNO_3 to make ~1.2 M HNO_3. Then, radioactive iodide is co-precipitated with carrier-added NaI after mixing with $AgNO_3$ solution. Because of the low solubility of AgI (8.3×10^{-17} $mol.L^{-1}$ [8]) and fast isotopic exchange between iodine and iodide, the removal of radioactive iodine and iodide is considered near-quantitative. However, because of the low rate of iodate-iodide isotopic exchange, this procedure does not completely remove radioiodine in the form of iodate. Next, Mo carrier is added, followed by Ru and Rh hold-back carriers to prevent their coprecipitation with the Mo-ABO complex. $KMnO_4$ is added to keep Mo as Mo(VI) before complexation with ABO. After addition of ABO, the Mo-ABO precipitate is filtered and washed with 0.1 M HNO_3. After repeated washing, the precipitate is redissolved in dilute $NaOH/H_2O_2$ solution by applying heat. Precipitation of trace iodine is repeated by adding NaI and $AgNO_3$, and the solution is then loaded onto a combination column that contains three sorbents: activated charcoal (AC), hydrous zirconia (HZO, which acts as a cation exchanger), and silver-coated charcoal (Ag/C). The AgI precipitate is collected on the combination column. The I_2 does not co-precipitate with the silver ions, but can be removed by reaction with silver metal (Ag/C resin) to form the insoluble AgI. The solubility of silver iodate is also limited, but is significantly larger (3.1×10^{-8} $mol.L^{-1}$) [8] than that of AgI. It is likely that $AgIO_4$ is even more soluble than $AgIO_3$. Mo passes through the combination column, while organic residuals are retained on the AC. The purified ^{99}Mo effluent is recovered in ~0.2 M NaOH solution.

2.4.2.1 Reagents Used in LMC Process

All chemical reagents used in this work were of analytical-reagent-grade purity and were used without further purification. All aqueous solutions were prepared with deionized water with a resistivity ≥18 MΩ•cm.

Silver-coated Charcoal (Ag/C)

Twenty grams of AC (50–100 mesh) was mixed with water to form a slurry-like mixture, and 15 mL of 10% $AgNO_3$ in 0.1 M HNO_3 was added. After 5 min of mixing. 4 mL of freshly prepared 5% Na_2SO_3 was added and mixed in. Next, 40 mL of 0.1 M NaOH was added while mixing, and the mixture was heated at 80–90°C for 30 min. After cooling, the material was washed several times with water until the wash solution was clear.

Hydrous Zirconia (HZO)

HZO was prepared by dissolving $ZrOCl_2$ in water and precipitated as zirconia by adding enough NH_4OH to make a solution with pH≥8. The slurry was covered and mixed for 20 min, and the pH was checked again and adjusted with NH_4OH to pH≥8 if needed. The precipitate was allowed to settle overnight, then centrifuged and washed with water until the pH of the supernatant reached pH=5–7. After the final water wash, the precipitate was filtered under vacuum and slowly dried at ~40°C for ~3 days. After drying, the precipitate was added to water, and very fine particles were decanted using water. After decanting, the material was allowed to air-dry overnight and then sieved to a size range of 100–200 mesh. The HZO resin was washed again with water and 0.2 M NaOH and stored under 0.2 M NaOH.

Alpha Benzoin Oxime (2% ABO in 0.4 M NaOH)

ABO (0.4 g) was combined with 20 mL of 0.4 M NaOH and heated while stirring until fully dissolved. The solution was allowed to cool and was used the same day.

Mo Carrier Solution (10 mg Mo/mL)

375 mg of MoO_3 was dissolved in 15 mL of 1 M NaOH, neutralized with 5 mL of 4 M HNO_3, and diluted to 25 mL with 0.1 M NaOH. The solution was prepared within seven days of actual processing.

Rh Carrier Solution (8 mg Rh/mL)

500 mg of $RhCl_3•3H_2O$ was transferred into a volumetric flask containing 15 mL of water, and then 1 mL of 70% HNO_3 was added and the solution was diluted to 25 mL with water. The solution was mixed until the $RhCl_3$ was completely dissolved.

Ru Carrier Solution (5 mg Ru/mL)

250 mg of K_3RuCl_6 was transferred into a volumetric flask containing 11 mL of 70% HNO_3 and mixed. The solution was diluted with water to 50 mL.

2.4.2.2 Reagents Used for Product-purity Analyses

Fission Product Carrier Solution

10 mg of $RhCl_3•3H_2O$ was transferred into a flask containing 50 mL of water, acidified with 1 mL of 70% HNO_3, and mixed until fully dissolved.

11 mL of 70% HNO_3 was added to a beaker containing 5 mL of water. 10 mg of K_3RuCl_6 was added and mixed in until fully dissolved.

500 mg of MoO_3 was dissolved in 20 mL of 1 M NaOH and acidified with 2 mL of 4 M HNO_3. Rh, Ru and Mo solutions were combined in a volumetric flask and diluted to 200 mL with water.

Ethyl Acetate, Pre-equilibrated

0.33 mL of 0.1 M NaOH was combined with 3.33 mL of 10% H_2SO_4 and mixed. 167 uL of $Fe_2(SO_4)_3$ (10 mg Fe/mL) in 1% H_2SO_4 was added and mixed in, followed by 0.33 mL of 50% NH_4SCN in water and further mixing. Then 0.83 mL of 10% $SnCl_2$ in 10% HCl was added and mixed in. The solution was combined with 20 mL of ethyl acetate and mixed for 1 min using a vortex mixer. The phases were separated by centrifugation and the ethyl acetate was used the same day.

2.4.2.3 Mo-Product Analyses

Thiocyanate Extraction of Mo

Extraction was performed in a 50-mL plastic vial. 0.1 mL of fission-product carrier solution was added into 10 mL of 10% H_2SO_4 and the solution was swirled. Then 0.5 mL of $Fe_2(SO_4)_3$ (10 mg Fe/mL) in 1% H_2SO_4 was added, followed by addition of 0.1 mL of ^{99}Mo product solution obtained after the LMC process, and the solution was swirled. Then 1 mL of 50% NH_4SCN in water was added and the solution was mixed. Next, 2.5 mL of 10% $SnCl_2$ in 10% HCl was added and the solution was mixed. The aqueous solution was then combined with 12 mL of ethyl acetate (pre-equilibrated) and mixed for 2 min. The aqueous phase was separated and combined with 5 mL of fresh ethyl acetate (pre-equilibrated) and mixed for another 2 min. The aqueous phase was separated and used to determine the activity of the fission product via gamma counting.

Iodine extraction using chloroform

Extraction was performed in a 20-mL glass scintillation vial. One drop of KI in water (10 mg I/mL) was added to 10 mL of water, followed by addition of 0.1 mL of ^{99}Mo product solution obtained after the LMC process. Then one drop of 8 M HNO_3 was added and the solution was combined with 5 mL of $CHCl_3$. Then two drops of 35% HCl and 4 drops of 20% $NaNO_2$ in water were added and mixed in for 1 min. The phases were allowed to separate, and the organic phase (bottom phase) was transferred into a new glass vial. Then two drops of 20% $NaNO_2$ in water were added to the aqueous phase, and the solution was combined with 5 mL of $CHCl_3$ and mixed for 1 min. The phases were allowed to separate, and the organic phase (bottom) was transferred and combined with $CHCl_3$ from the first extraction step. The combined organic phase was used to determine the activity of ^{131}I using gamma counting.

2.4.2.4 Preparation for the LEU-Modified Cintichem Process

Prior to performing each LMC process with irradiated solution, reagent solutions were prepared according to the descriptions above. The combination column (Figure 2.4.2.4.1) was prepared by placing glass wool on the bottom of the column, then loading slurries of Ag/C, HZO, and AC in that order. Then glass wool was placed on top of the sorbents, and the column was sealed. The column was repeatedly washed with 0.2 M NaOH solution until the pH of the eluent was alkaline. The column was stored under 0.2 M NaOH solution and was washed again just before placing into the hot cell right before LMC processing. LMC glassware used for the LMC process is shown in Figure 2.4.2.4.1. Double-sided needles were prepared by combining two needles with male-to-male Luer-lock connectors. The 40-mm 0.3-µm PP filter (Zenpure) for filtration of AgI precipitate (Figure 2.4.2.4.2) was pre-wetted. Reagents used for the LMC process were loaded into syringes and transferred into the hot cell prior to processing.

One-way check valves with needles were used during vacuum transfer of solution between bottles, and only allowed air to enter the system. Normal needles were not used for venting, to minimize release of fission gasses. The needles and filter assembly are shown in Figure 2.4.2.4.2.

Needle guards used to interconnect bottles and needles are shown in Figures 2.4.2.4.3 and 2.4.2.4.4.

FIGURE 2.4.2.4.1 LMC glassware, plastic coated except for the fritted-glass column. From left to right: flat-bottom bottle, double-sided bottle, 51-mm fritted-glass column containing ~20 mL of glass beads, AgC/ZrO/AC column, charcoal filter column (shown empty). All glassware uses crimps to hold septa in place.

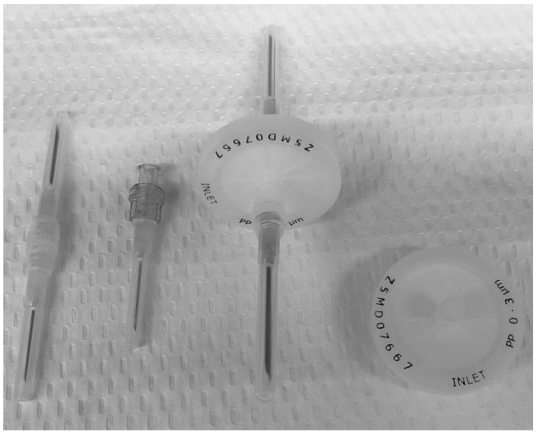

FIGURE 2.4.2.4.2 From left to right: double-sided needle with male-to-male Luer connector in the middle, one-way Luer check-valve needle, 40-mm 0.3-µm filter with needles, 40-mm 0.3-µm filter

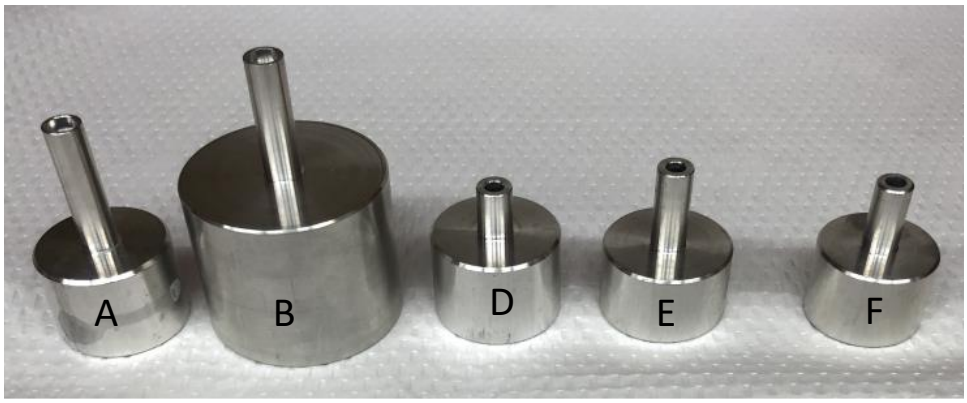

FIGURE 2.4.2.4.3 Various models of aluminum needle adapters

2.4.2.5 Acidification of Strip Solution from Concentration Column

The alkaline strip solution from the concentration column was received in the RF-1 bottle (RF denotes raw fission). The mass of solution in the RF-1 bottle was verified by weighing. An addition of 0.25 mL of 10 M HNO_3 solution was used per 1 mL of column strip solution. Before addition of an appropriate amount of 10 M HNO_3 solution, the RF-1 bottle was connected to the GCS using a needle on top of the bottle. After acidification, the RF-1 bottle contents were mixed by shaking.

FIGURE 2.4.2.4.4 D-type aluminum needle adapter with side port for vacuum line

2.4.2.6 Silver Iodide Precipitation

After acidification, 4 mg of iodide as NaI carrier was added, and the solution was mixed. Then 0.5 mL of 10% $AgNO_3$ was added, and a white precipitate formed. The bottle was mixed again. To remove excess silver, 1 mL of 1 M HCl was added, and additional precipitate formed. The solution was mixed and allowed to sit for 2 min. The solution and precipitate from the RF-1 bottle were then transferred through the 40-mm 0.3-μm PP (Zenpure) filter by applying vacuum to the bottom bottle (RF-2). A typical setup for filtration of AgI precipitation is shown in Figure 2.4.2.6.1. The RF-1 bottle was then rinsed with 11 mL of 4 M HNO_3, and the solution was passed through the filter.

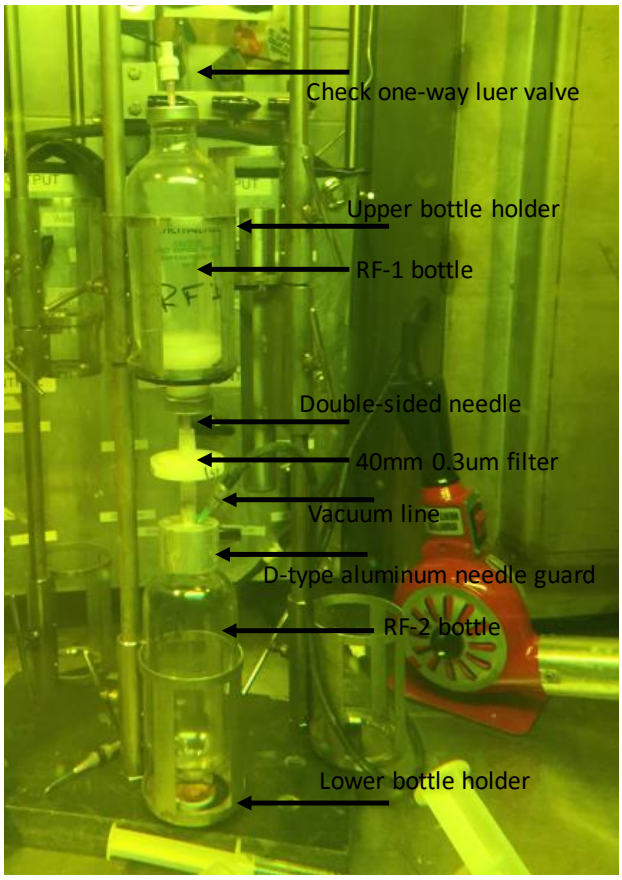

FIGURE 2.4.2.6.1 Example of AgI filtration setup

2.4.2.7 Mo-ABO Precipitation

Following the transfer of the RF-1 solution and wash into the RF-2 bottle, the bottle was evacuated, and 0.5 mL of Mo carrier solution was added (10 mg Mo/mL). Then 2.5% $KMnO_4$ solution was added dropwise until a deep pink color persisted for ~30 sec. This was followed by the addition of 1.5 mL of Rh (8 mg Rh/mL) and 2 mL of Ru (5 mg Ru/mL) carriers. The solution in the RF-2 bottle was mixed and allowed to sit for 1 min. Then 20 mL of 2% ABO in 0.4 M NaOH was added, and the resulting slurry was mixed and allowed to sit for 1 min. Upon addition of ABO, a beige precipitate formed. The slurry containing the Mo-ABO precipitate was than filtered through a fritted bottle containing glass beads by applying vacuum to the RFW (raw fission waste) bottle. The experimental setup for filtration of Mo-ABO is shown in Figure 2.4.2.7.1. The RF-2 bottle on top contains Mo-ABO precipitate after addition of ABO, and Mo-ABO precipitate is transferred into the fritted-filter bottle, while the solution containing the majority of other radionuclides passes through the filter into the RFW bottle. The fritted-filter bottle contains glass beads that allow for better mixing during the washing steps for the Mo-ABO precipitate and during the dissolution of the Mo-ABO precipitate. It should be noted that for these experiments, the fritted-filter bottle was modified from the original design (Figure 2.4.1.4.1) by the Argonne glass blower, who extended the height of the bottle to allow a better grip with remote manipulators.

FIGURE 2.4.2.7.1 Experimental setup for filtration of Mo-ABO precipitate

The Mo-ABO precipitate was washed three times with 20 mL of 0.1 M HNO_3, and five times with 10 mL of 0.1 M HNO_3. The first three washes were performed by adding the 0.1 M HNO_3 solution to the RF-2 bottle, briefly mixing it, and then passing it through the frit containing the Mo-ABO precipitate. If no significant amount of precipitate was present in the RF-2 bottle, the next 2–3 washes were applied directly to the fritted-filter bottle. The last 2–3 washes with 10 mL of 0.1 M HNO_3 were performed by loading the wash solution under the frit and pulling it through the frit by applying vacuum in the chamber above, which contains Mo-ABO precipitate. Wash solution was then transferred by vacuum to the RFW bottle. The RFW bottle was weighed, and an aliquot was taken for gamma counting. The solution in the RFW bottle first appeared clear, but over time, formation of a precipitate was observed. This formation is due to an excess of ABO that did not precipitate with Mo but, owing to its limited solubility under acidic conditions, precipitated after the filtration step. It was confirmed experimentally that the amount of the Mo-ABO complex in the RFW bottle was very small and the presence of precipitate was caused by the limited solubility of excess ABO.

2.4.2.8 Dissolution of Mo-ABO Precipitate

After complete washing of the Mo-ABO with 0.1 M HNO_3, 10 mL of 0.4 M NaOH/1% H_2O_2 was added to the Mo-ABO precipitate and heat was applied by positioning a heat gun at the fritted-filter bottle. The bottle was vented through a charcoal filter (the small glass column shown in Figure 2.4.2.8.1, filled with AC) into the GCS. The experimental setup for dissolution of Mo-ABO precipitate is shown in Figure 2.4.2.8.1. Heat was applied until bubbles started to evolve; then the solution was allowed to cool for five minutes. The resulting solution was removed from the fritted-filter bottle by connecting into the 1-A bottle. The remaining undissolved precipitate was then dissolved in the same manner by adding 10 mL of 0.2 M NaOH/1% H_2O_2 and heating. The fritted-filter bottle was then rinsed with 10 mL of 0.2 M NaOH and its contents collected into the 1-A bottle.

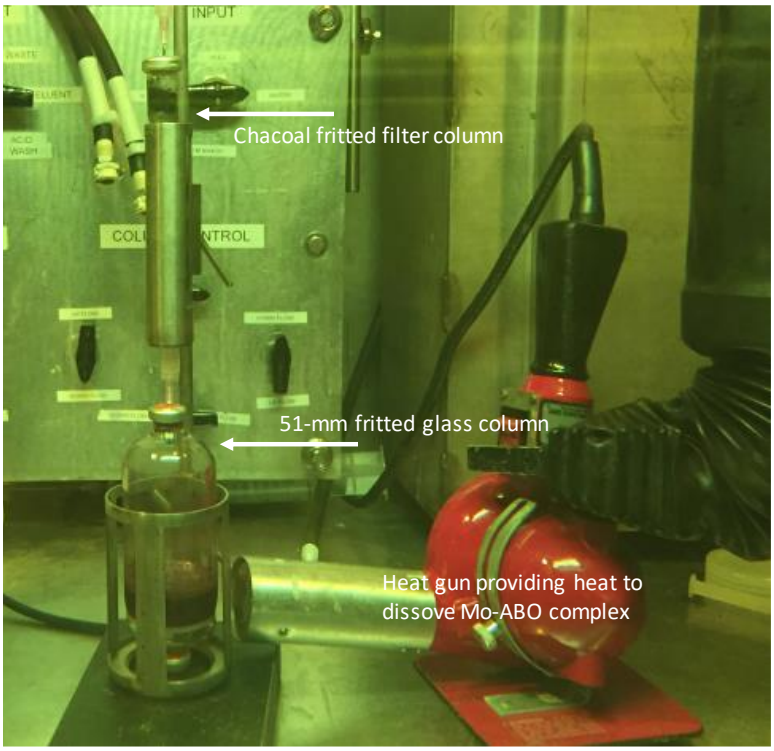

FIGURE 2.4.2.8.1 Experimental setup for dissolution of Mo-ABO precipitate

2.4.2.9 Combination Column

After dissolution of the Mo-ABO precipitate, a final purification was performed by using a combination column (AC-HZO-Ag/C). Before loading the solution from the 1-A bottle onto the column, another iodine precipitation step was performed. After adding 4 mL of NaI solution (1 mg-I/mL) to the 1-A bottle and mixing, 0.5 mL of 10% $AgNO_3$ in 0.1 M HNO_3 was added and mixed. A gray-black precipitate formed, and the solution was allowed to sit for five minutes. Then the slurry was loaded onto the column, and the effluent was collected in the 1-B bottle at the bottom. Figure 2.4.2.9.1 shows the 1-A bottle with black precipitate on top, the combination column in the middle, and the 1-B collection bottle for ^{99}Mo product at the bottom. Elution was initiated by turning on a vacuum pump connected to the 1-B bottle until a steady flow of drops was observed exiting the column. Then the vacuum pump was turned off, and the column was eluted by gravity at 1–3 mL per minute. After the initial solution passed through the column, the 1-A bottle was rinsed with 10 mL of 0.2 M NaOH, which was then run through the column. Once the additional 10 mL of solution passed through the column, a vacuum was applied to the 1-B bottle to recover all solution from the column. The resulting purified ^{99}Mo product was recovered as a ~0.2 M NaOH solution.

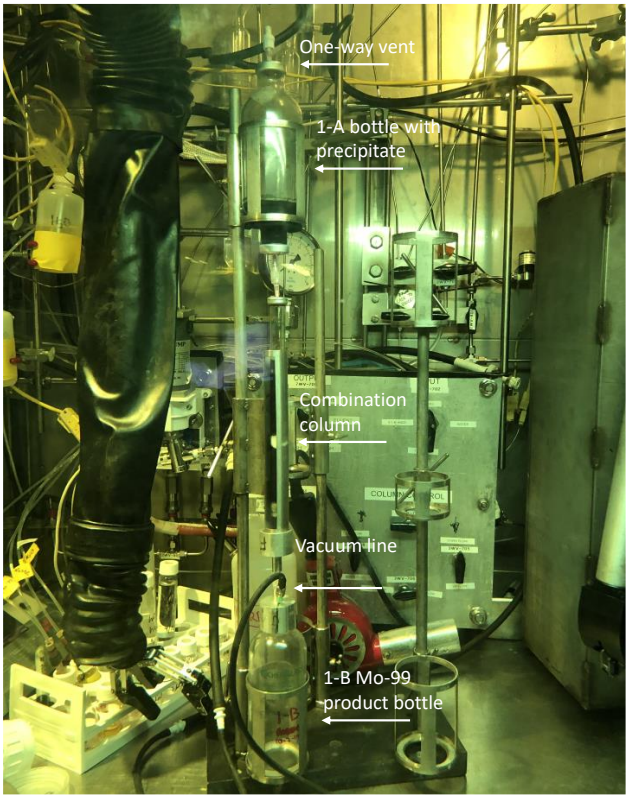

FIGURE 2.4.2.9.1 Experimental setup for combination column

2.4.2.10 References

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2.5 GAMMA COUNTING

2.5.1 Introduction

Gamma spectroscopy was performed on aqueous samples to determine the initial and produced activities of fission products at various points in solution processing. This information was then used as a way to quantify the recovery achieved by the various steps as well as the overall procedure, and to determine the points where several important isotopes/contaminants were removed from the final product.

2.5.2 Instrumentation

Gamma spectroscopy was performed using one of two high-purity germanium (HPGe) detectors, both of which were calibrated with an Eckert & Ziegler mixed-isotope standard. Both HPGe detectors used were coaxial geometry connected to ORTEC DSPEC 50 digital analyzers and were calibrated at various distances to accommodate samples of different strengths. One of the instruments was cooled using LN_2 with an ORTEC Möbius Recycler to extend the refilling interval, and was set up with simple shielding around the detector and a movable sample holder with set distances (seen in Figure 2.5.2.1). The other HPGe instrument was mechanically cooled using an ORTEC X-COOLER III and was coupled to an autosampling system with a shielded canyon where the sample resided for counting (see Figure 2.5.2.2).

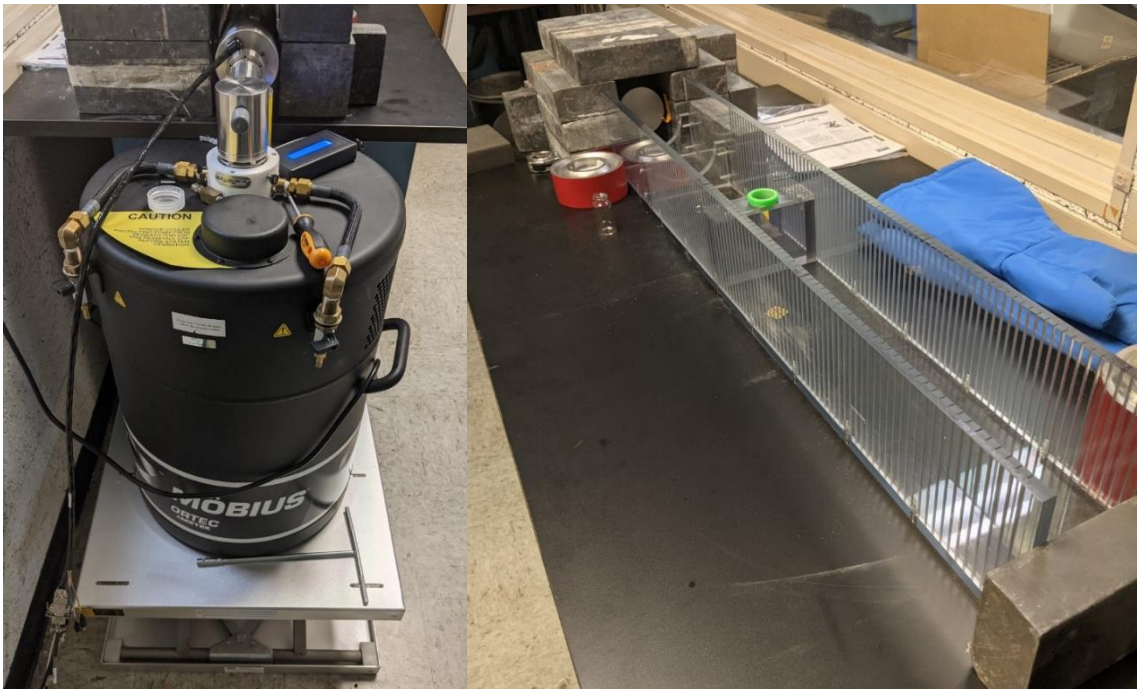

FIGURE 2.5.2.1 Coaxial HPGe cooled with LN_2 and recycler (left) and the same detector with associated shielding and movable sample holder (right)

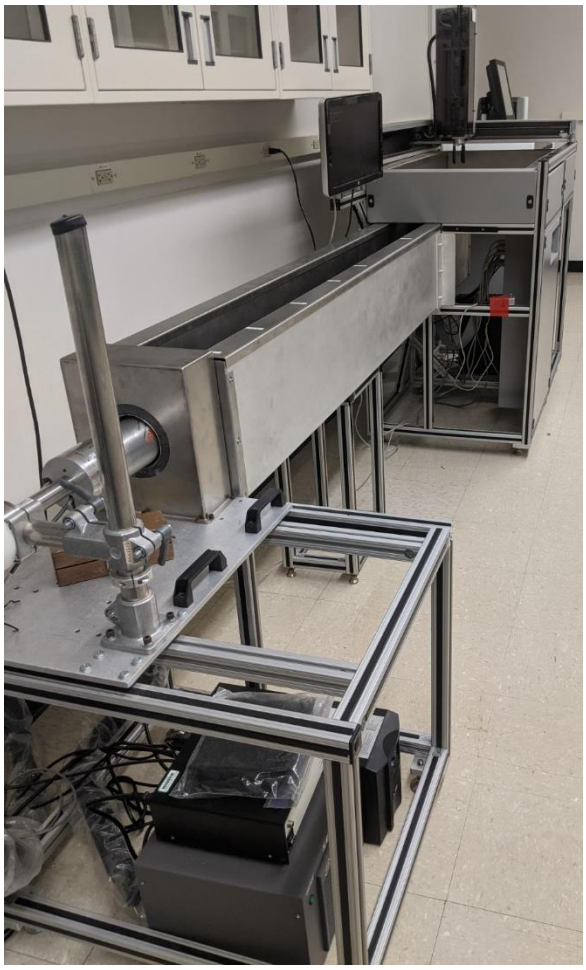

FIGURE 2.5.2.2 Mechanically cooled coaxial detector coupled to an autosampling unit with a shielded canyon for counting

Regardless of which instrument was used to collect the gamma spectra, resulting data for the different solutions were analyzed and decay-corrected with GammaVision software.

For each sample, multiple analyses were executed over time to ensure that the reported activities were not skewed by overlapping peaks. In most cases, only the 33 nuclides and associated peaks listed in Table 2.5.2.1 were quantified. In several cases, however, large increases in reported activities were observed when the same samples were counted a second or third time. This increase indicated the presence of overlapping peaks from unidentified constituents and was found to occur primarily in the target solution samples after irradiation. In these instances, a more complex library was used to subtract the minor peaks of the nuclides of interest from the spectrum, resulting in much better agreement. The only nuclides for which this approach did not work were ^{105}Rh and ^{131m}Xe. The interference on these peaks was caused by ^{147}Nd and ^{140}Ba, respectively, which were only present in the target solution and therefore did not cause discrepancies in any samples taken after glovebox processing.

It should be noted that high activity concentrations of ^{131}I, ^{133}I, ^{135}I, and ^{99}Mo in most of the samples following the initial glovebox processing meant that aliquots of even a few milligrams often resulted in significant detector dead time, which made the signal of some radionuclides with lower gamma branching ratios (such as ^{125}Sn) difficult to identify. The iodine isotopes (^{131}I, ^{132}I, ^{133}I, and ^{135}I) could all be easily identified, since their parents were either short-lived (half-life < 1 hr) or were removed by the recovery column in the glovebox (^{131m}Te, ^{132}Te). The recovery column also removed all ^{237}U and ^{239}Np from the process stream.

TABLE 2.5.2.1 Isotopes and associated peaks analyzed during gamma spectroscopy

Isotope	Peak Energy Used (keV)	Isotope	Peak Energy Used (keV)
^{95}Zr	756.7	^{97}Zr	743.4
^{95}Nb	765.8	^{92}Sr	1384.1
^{237}U	208.0	^{99m}Tc	140.5
^{156}Eu	1230.7	^{131m}Xe	163.9
^{137}Cs	661.7	^{135}Xe	249.8
^{239}Np	277.6	^{133m}Xe	233.2
^{99}Mo	181.1	^{132}Te	49.8
^{103}Ru	497.1	^{97}Nb	657.9
^{132}I	522.7	^{105}Rh	318.9
^{131}Te	793.8	^{125}Sn	1088.9
^{131}I	637.0	^{127}Sb	685.5
^{133}I	529.9	^{91}Sr	555.56
			1024.3
^{136}Cs	818.5	^{147}Nd	91.1
	1048.1		
^{140}Ba	423.7	^{151}Pm	340.06
^{156}Sm	165.7	^{93}Y	266.9
^{140}La	1596.2	^{135}I	1260.4
^{143}Ce	293.3		

2.5.3 Sample Preparation

All solutions were prepared for gamma spectroscopy by mass-based serial dilution. Because the original solution volumes were also tracked by mass, this method reduced uncertainty in dilution factors and allowed very small samples to be correlated to large initial solutions with higher confidence. Small samples sizes were often necessary because of the highly radioactive nature of the parent solutions.

3 RESULTS AND DISCUSSION

The first Phase II post-irradiation processing was performed on March 9, 2018. After successful recovery-column processing in the recovery glovebox, the solution containing ^{99}Mo and other fission products was transferred to the hot cell for further purification. At the beginning of concentration-column processing inside the hot cell, an elevated background reading occurred in the linac facility. Fission gases from the hot-cell stack were being recirculated into the building, and this caused elevated readings on the radiation detectors within the linac facility.

During the subsequent fact-finding process, a strong correlation was established between the increase in readings and the installation of a pH probe, which caused a 3-liter processing vessel to be opened to the processing-hot-cell atmosphere. This incident was investigated by internal and external committees. Both committees arrived at very similar conclusions and recommendations. On the basis of the results of the investigations, multiple corrective actions were proposed and executed. The main parts of the corrective actions were as follows:

- Develop a process for screening of the experimental work against facility safety bases.
- Develop a configuration management program for the facility.
- Conduct a design review of the building exhaust and extend the exhaust stack for the hot cell to prevent the possibility of recirculation.
- Conduct design reviews for all systems connected to the GCS.
- Setup dedicated equipment for monitoring radiation levels in LEAF during processing.
- Modify the facility Safety Assessment Document (SAD) and ASE to include additional descriptions of the experiment and additional credited controls.
- After implementation of the corrective actions, conduct an Accelerator Readiness Review for the restart of experimental activities.

In accordance with the corrective actions identified, several modifications were implemented, and a Commissioning Plan was developed. Modified operational procedures were subsequently developed and are attached as multiple appendices to this report. The purpose of the Commissioning Plan was to check that all systems were operating as intended. The Commissioning Plan consisted of testing all the systems by performing the associated operations with a solution spiked with a small portion of ^{99}Mo. The results of the test provided a baseline for separation process readiness, allowed improvements to the procedures and training, and provided integrated verification of the chemical separation, purification, and gas collection systems within the controls described in the SAD and ASE. Those two documents encompass all the safety aspects of Accelerator Facility operations: the SAD provides the bases for safe

operations of the facility and the ASE lists the limits for the operation parameters and credited controls. Because the type of accident (gas release and recirculation) was not discussed in the SAD and controls were not established in the ASE, both documents had to go through significant rewriting and a vetting process with DOE ASO.

The Commissioning Plan consisted of the following sections:

a. Prerequisite Conditions (including GCS pre-checks);
b. GCS Commissioning;
c. Recovery Glove Box Processing Commissioning;
d. D-024 Hot Cell Commissioning; and
e. Radiation Monitoring Commissioning.

A commissioning run was carried out from August 13 to 15, 2019, with a ^{99}Mo spike (no irradiation). Experimental conditions were verified prior to commencing the commissioning run, in accordance with updated SAD and ASE requirements:

- U concentration was 132g/L and was below the SAD limit of <145 g-U/L.

- No makeup solution was added, and ^{235}U enrichment remained at or below the original value of 19.86% ^{235}U, which is below the SAD limit of <20% ^{235}U.

- The LEU solution volume was ~17.6 L, which is below the SAD limit of <20 L.

During mixing of the ^{99}Mo spike with the LEU solution, the presence of a precipitate was observed in the LEU solution. A sample of the precipitate was analyzed using powder X-ray diffraction analysis (XRD) and confirmed the presence of uranyl peroxide. From the results of the XRD analysis, we concluded that this precipitate had formed during the first Phase II irradiation in March 2018. Formation of uranyl peroxide under irradiation conditions is not unexpected and was confirmed during our small-scale irradiations of a similar uranyl sulfate solution at the Van de Graaff (VDG) generator. The formation of uranium precipitate is due to complexation of the uranyl ion with peroxide that forms during the radiolysis of water. This behavior is well understood and can be prevented by addition of a Fe^{2+}/Fe^{3+} catalyst, as was confirmed by subsequent VDG irradiations [1]. The presence of precipitate did not cause any safety concerns; however, it is experimentally undesirable, as it may complicate solution transfer and sample collection. Prior to irradiation of the LEU solution, an attempt was made to remove the precipitate from the system by repeated pumping of the solution from the TSV to the verification tank through a high-capacity filter. In addition, an iron catalyst was added to the LEU solution to minimize the likelihood of future precipitation.

References

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3.1 LINAC IRRADIATIONS

Six irradiations were performed as part of the Phase II studies. Table 1.1 briefly describes these experiments. Each is discussed further in the text below.

3.1.1 Irradiation #0 with Accidental Rad Gas Release, 3/8/18

The first irradiation of the uranyl sulfate solution in the study was conducted on March 8, 2018. For the first test with the beam, the time for irradiation was limited to a maximum of 4 hours at high power. This run duration was chosen for two reasons:

- One-fifth of the planned full-production scale allowed a good estimation of the activity produced at a full 20-hour irradiation, and
- Four hours would be enough to establish thermal equilibrium in the uranyl sulfate solution, as well as steady-state gas generation rates.

This irradiation provided us with fission-product production rates, which were compared with the expectation from our Monte Carlo (MCNPX) calculations.

Preparation for the irradiation started on the day prior to irradiation. Performance of the cooling system was verified using procedures LEAF-PROC-001, -002, -003, -004, -006, and -007 (see Appendices 19–24). Initial beam tuning was performed following procedure LEAF-PROC-027 (Appendix 25).

On the day of irradiation, the beam parameters were verified. Readiness of all AMORE systems for irradiation was verified according to procedure LEAF-PROC-012 (Appendix 26). Owing to limitations for heat dissipation in the spectrometer magnet, the energy spectrum was measured at 38 MeV. The resulting energy spectrum is presented in Figure 3.1.1.1. After spectrum acquisition, the peak beam current was reduced to shift the beam energy peak to 40 MeV. After energy verification, the beam was placed on the target window at low power (500 W), and the beam shape was adjusted to produce a 16x14.1-mm FWHM[2] beam spot on the target face (Figure 3.1.1.2).

Irradiation started at 9:15 am with ~500 W of beam power on the target. Over a period of 45 min, the power was increased to 6 kW, where it stayed for 1 h. The power was then increased to 10 kW, then to 12 kW, and finally to 13.8 kW. A beam power of 13.8 kW was the highest at which we could continuously perform irradiation while maintaining the hydrogen concentration below 1% in the vessel off-gas.

[2] Full Width at Half Maximum. The beam profile has a Gaussian shape; measurement of the FWHM values for the beam intensity distribution is a well-established practice.

To test the target performance at full beam power, we decreased the beam power to 10 kW, waited until the hydrogen concentration dropped and stabilized, and then increased the power to 18 kW for a short period of time. During these short power increases, we monitored the temperature of the target cooling water. Figure 3.1.1.3 depicts the beam history for this irradiation. The irradiation was ended at 4 pm.

At the end of irradiation, the gas-analysis and -collection system was purged to reduce the amount of radioactive gas in the gas-analysis manifold. During irradiation, a significant amount of gaseous fission products is accumulated in the gas lines connected to the experiment. Those fission products generated a high radiation field in the rooms used by personnel for processing of the solution. Therefore, prior to the start of the solution processing, the gas manifolds have to be purged to reduce dose rates in the occupied areas. Processing of the irradiated solution started at ~4 pm.

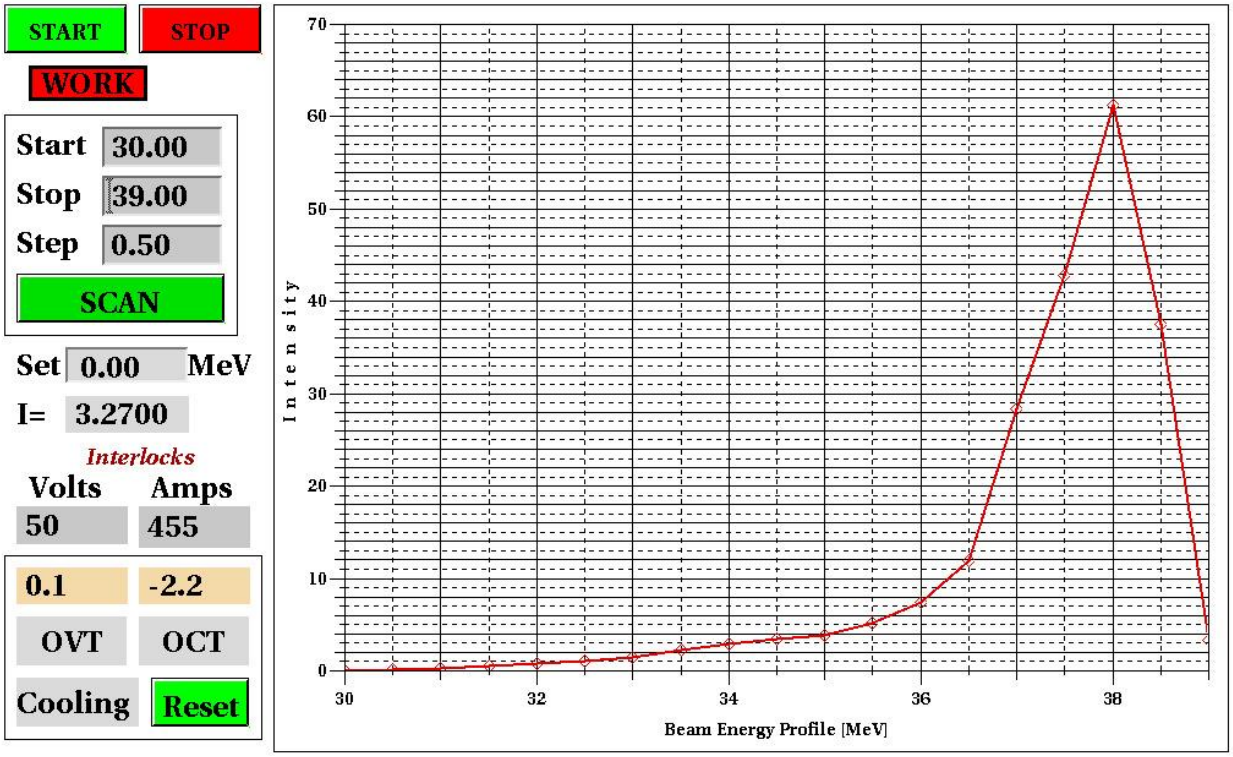


FIGURE 3.1.1.1 Beam energy spectrum at ~38 MeV for Irradiation #0 on 3/8/18. After tune-up, the injector current was lowered to shift the beam energy to ~40 MeV.

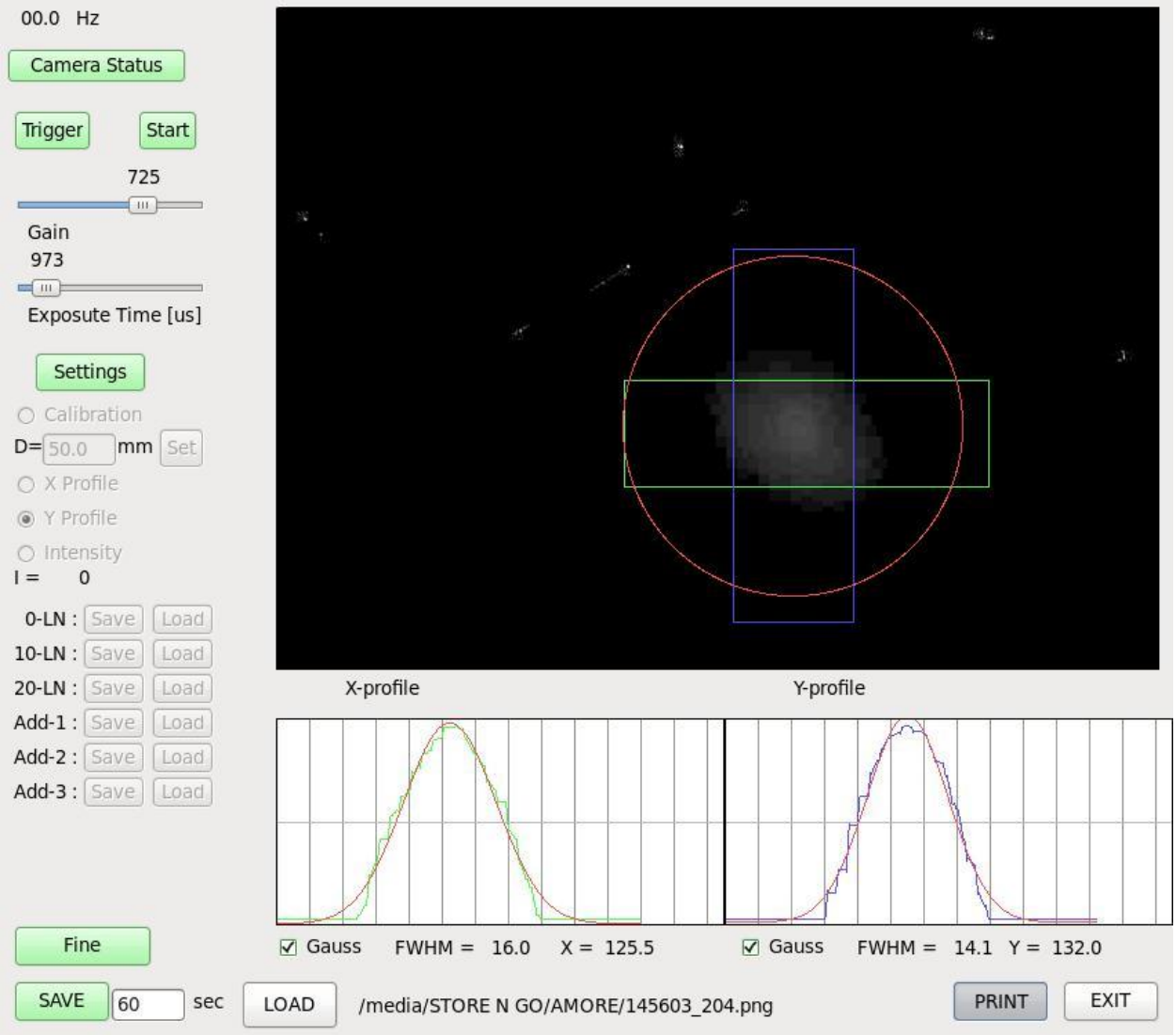


FIGURE 3.1.1.2 Beam profile on the target window for Irradiation #0. Red circle outlines the target beam window boundary.

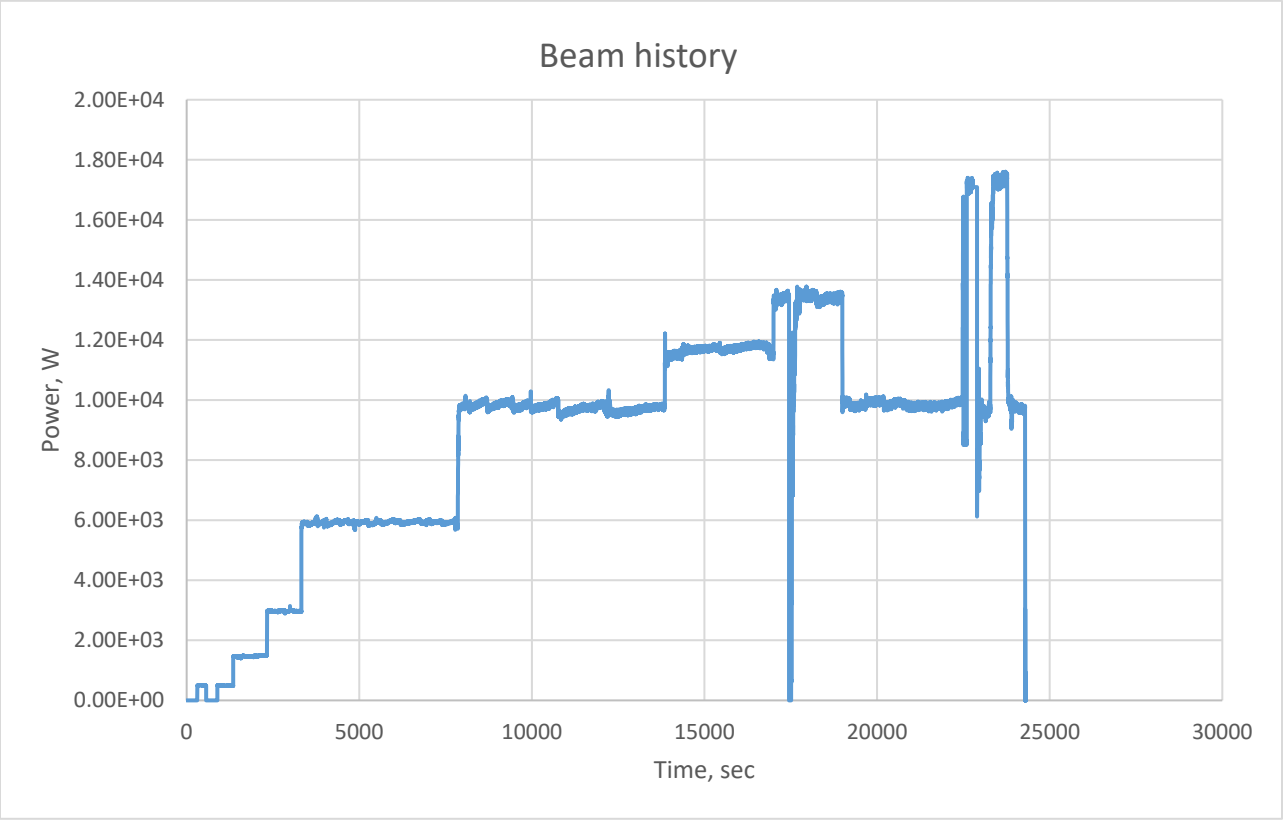


FIGURE 3.1.1.3 Beam history for Irradiation #0

A commissioning run was carried out from August 13 to 15, 2019, with a ^{99}Mo spike (no irradiation). Prior to performing the experimental work, all workers were trained and qualified (qualification cards were filled out and signed). A pre-job briefing was held on August 12 to confirm everyone's readiness to perform the work. All training documentation is stored in an online database. Experimental conditions were verified prior to commencing the commissioning run in accordance with updated SAD and ASE requirements:

- The U concentration was 132 g/L, which is below the SAD limit of <145 g-U/L;
- No makeup solution was added, and ^{235}U enrichment remained at or below the original value of 19.86% ^{235}U, which is below the SAD limit of <20% ^{235}U; and
- The LEU solution volume was ~17.6 L, which is below the SAD limit of <20L.

During mixing of the ^{99}Mo spike with the LEU solution, the presence of a precipitate was observed in the LEU solution. A sample of precipitate was analyzed using XRD, and confirmed the presence of uranyl peroxide. From the XRD analysis results, we conclude that this precipitate formed during the first Phase II irradiation in March 2018. The formation of uranyl peroxide under irradiation conditions is not unexpected, and was confirmed during our small-scale irradiations of a similar uranyl sulfate solution at the VDG generator. The formation of uranium precipitate is due to the complexation of the uranyl ion with peroxide that forms through radiolysis of water. This behavior is well understood and can be prevented by the addition of a Fe^{2+}/Fe^{3+} catalyst, as was confirmed by subsequent VDG irradiations. The presence of precipitate does not cause any safety concerns; however, it is undesirable, as it may complicate solution transfer and sample collection. Prior to irradiation of the LEU solution, we removed the precipitate from the system by repeated pumping of the solution from the TSV to the verification tank through a high-capacity filter. In addition, an iron catalyst was added to the LEU solution to minimize the likelihood of future precipitation.

The commissioning activities were completed in the manner described previously and all associated work procedures were followed. Despite some technical difficulties that involved failure of electronic components (such as power supply, heater, solenoid valve, and pressure transducer) due to radiation damage caused by irradiations performed in the D-035 glovebox for other programs, all systems performed well, and the solution was moved from the D-035 glovebox into the D-024 hot cell, where the concentration-column operation and LMC process were performed. All necessary samples were collected and analyzed.

Performance of the Gas Handling system pre-checks and interlock verification, linac pre-checks and interlock verification, and Supplemental Radiation Monitoring System was successfully completed. Gas Handling procedures were followed as written. Some lessons learned were incorporated into the procedures to improve clarity. The relevant parameters for the experimental systems were confirmed to be as described in the facility SAD and ASE. In short, all acceptance criteria were met. All follow-up items required to be completed prior to the next irradiation were tracked to completion by the ^{99}Mo Program Manager.

3.1.2 Irradiation #1, 10/1/19

The first irradiation after the restart was conducted on 10/1/19. For the restart, the time for irradiation was limited to a maximum of 4 hours at high power. Essentially, we were trying to return to the same starting point as Irradiation #0. Because the plan for the experiment included the processing of the solution immediately after irradiation without significant interruptions, the end of irradiation #1 was scheduled for 10 pm, leaving enough time to prepare for the experiment during the day.

On the morning of Irradiation #1, the beam was tuned and prepared for transport to the target. The beam energy spectrum is presented in Figure 3.1.2.1. This spectrum corresponds to a peak energy of 37 MeV at 0.53 A peak current. After spectrum acquisition, the peak beam current was reduced to 0.42 A, which shifted the beam energy peak to 40 MeV. After that, the energy verification beam was placed on the target window at low power, and the beam shape was adjusted to produce a 16.2x15.7-mm FWHM beam spot on the target face (Figure 3.1.2.2).

The irradiation began at 4 pm with ~150 W of beam power on the target. Over 45 min, the power was increased to 14 kW, where it stayed for the duration of the experiment. Around 7 pm, instabilities in the injector current caused beam position changes that led to beam losses in the beamline and a worsening vacuum. This caused ~15 minutes of interruption in irradiation. At 7:15 pm, irradiation at ~14 kW was resumed. The maximum beam power used in this experiment was 14.4 kW; the maximum power was limited by hydrogen production and the requirement to maintain hydrogen concentration under 1 % in the irradiation-vessel off-gas. Figure 3.1.2.3 depicts the beam history for the irradiation. Irradiation #1 was completed at 10 pm. After the end of irradiation, the gas-analysis and -collection system was purged to reduce the amount of radioactive gases in the gas-analysis manifold. Processing of the irradiated solution started at 11 pm.

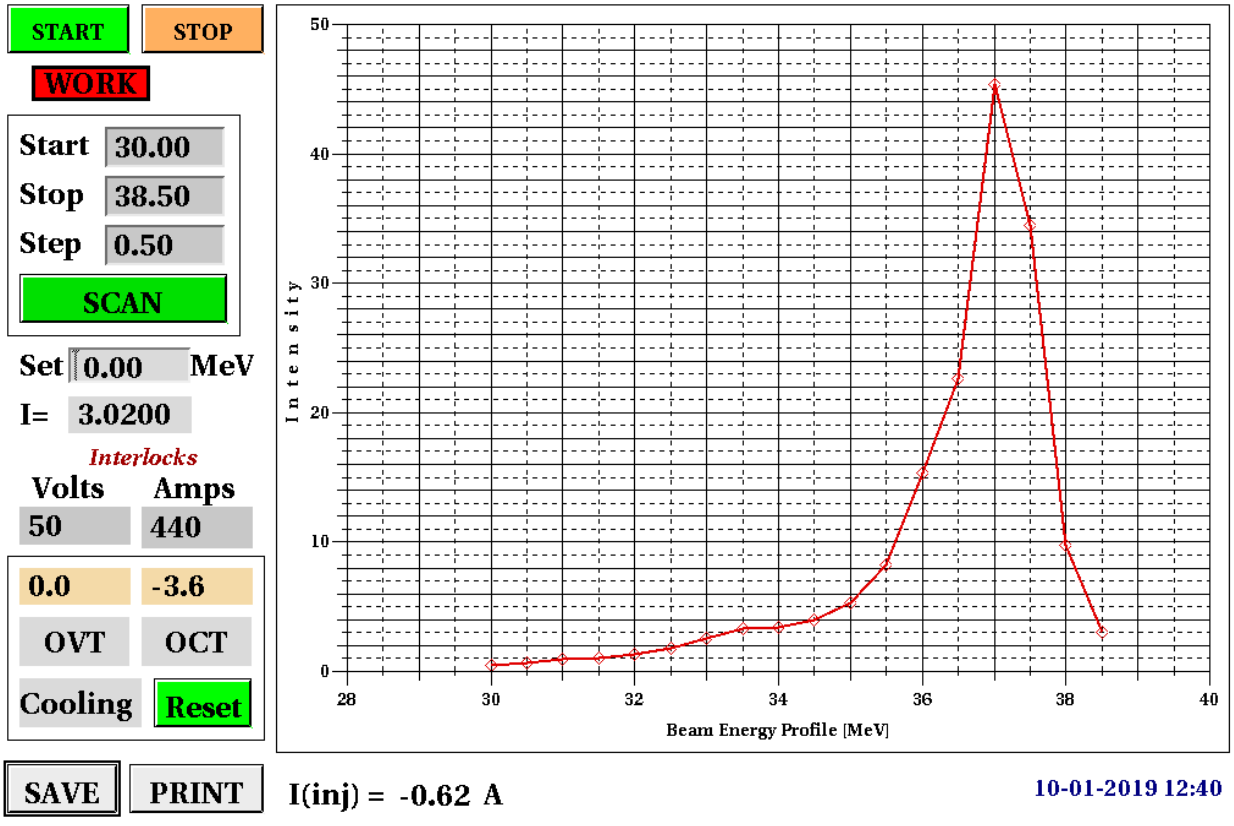


FIGURE 3.1.2.1 Beam-energy spectrum for Irradiation #1 on 10/1/19. The energy spectrum was recorded at a lower energy than 40 MeV because of spectrometer limitations. After initial tune-up, the beam peak current was reduced to adjust the peak energy to 40 MeV.

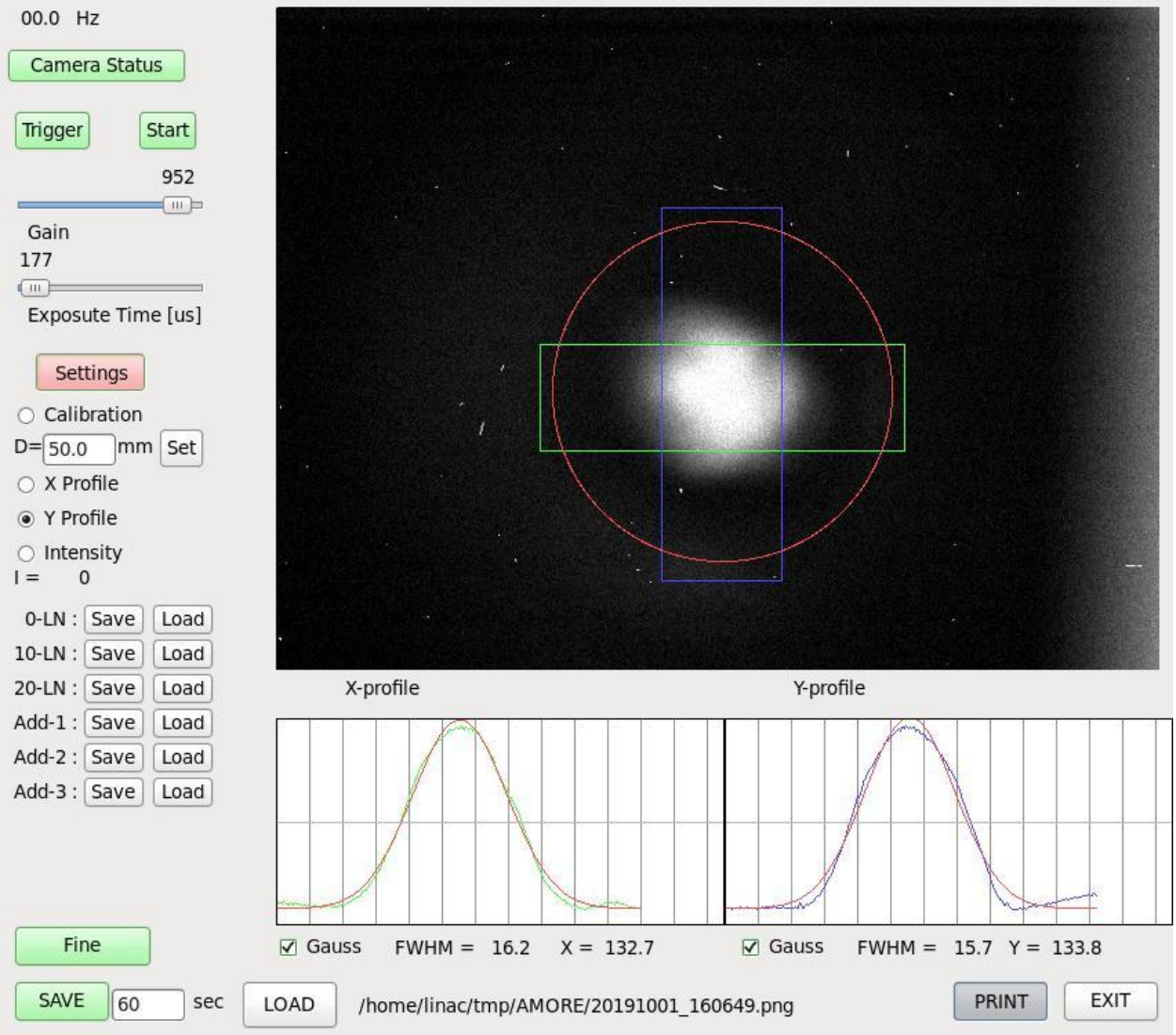


FIGURE 3.1.2.2 Beam profile on the target window for Irradiation #1. Red circle outlines the target beam window boundary.

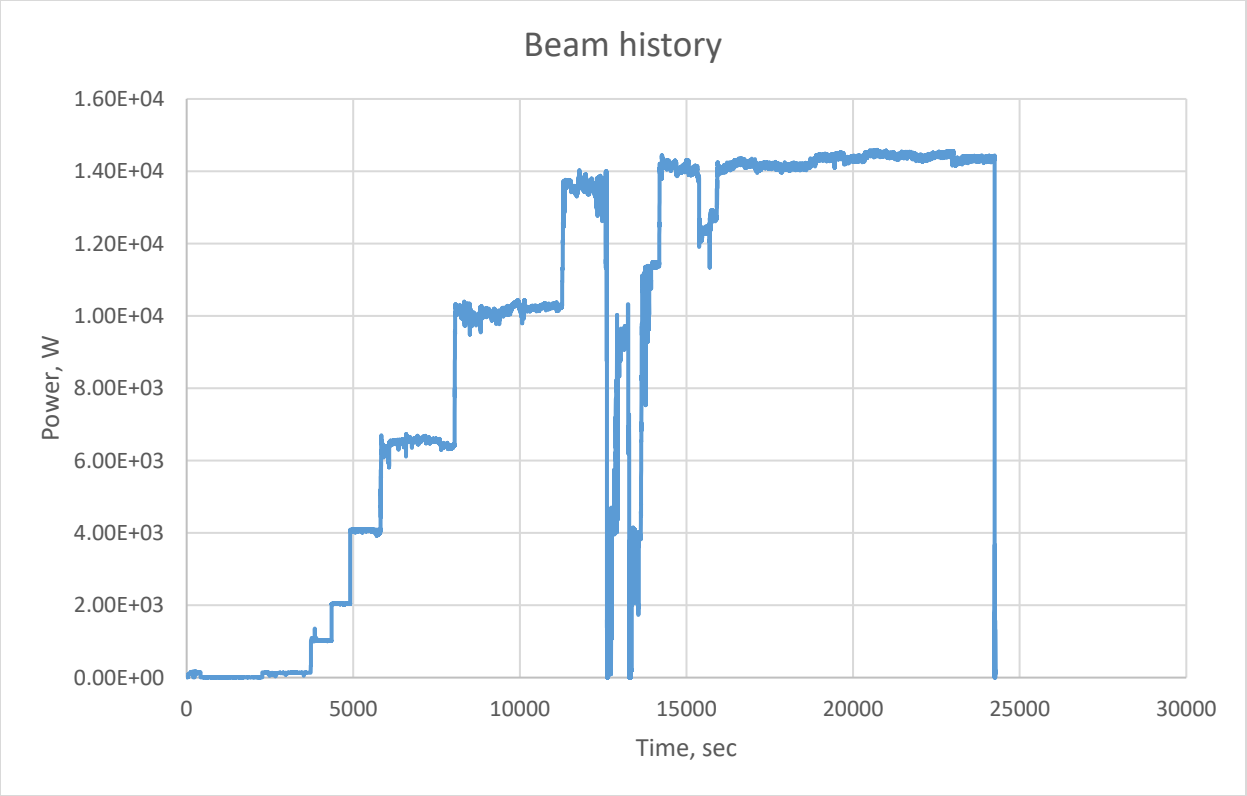


FIGURE 3.1.2.3 Beam history for Irradiation #1

3.1.3 Irradiation #2, 11/11/19

The second experiment after restart was conducted on 11/11/19. The plan was to irradiate the uranyl sulfate solution for 16 hours at a maximum beam power limited by hydrogen production/recombination. Because the plan for the experiment included the processing of the solution immediately after irradiation without significant interruptions, the end of irradiation was scheduled for 2 am. Initial beam tune-up was conducted on 11/8/19.

On the morning of the irradiation day, the beam was tuned and prepared for transport to the target. The beam energy spectrum is presented in Figure 3.1.3.1. This spectrum corresponds to a peak energy of 37 MeV at 0.57 A peak current. After spectrum acquisition, the peak beam current was reduced to 0.53 A, which shifted the beam energy peak to 40 MeV. After energy verification, the beam was placed on the target window at low power (175 W) and the beam shape was adjusted to produce a 17.1x13.5-mm FWHM beam spot on the target face (Figure 3.1.3.2).

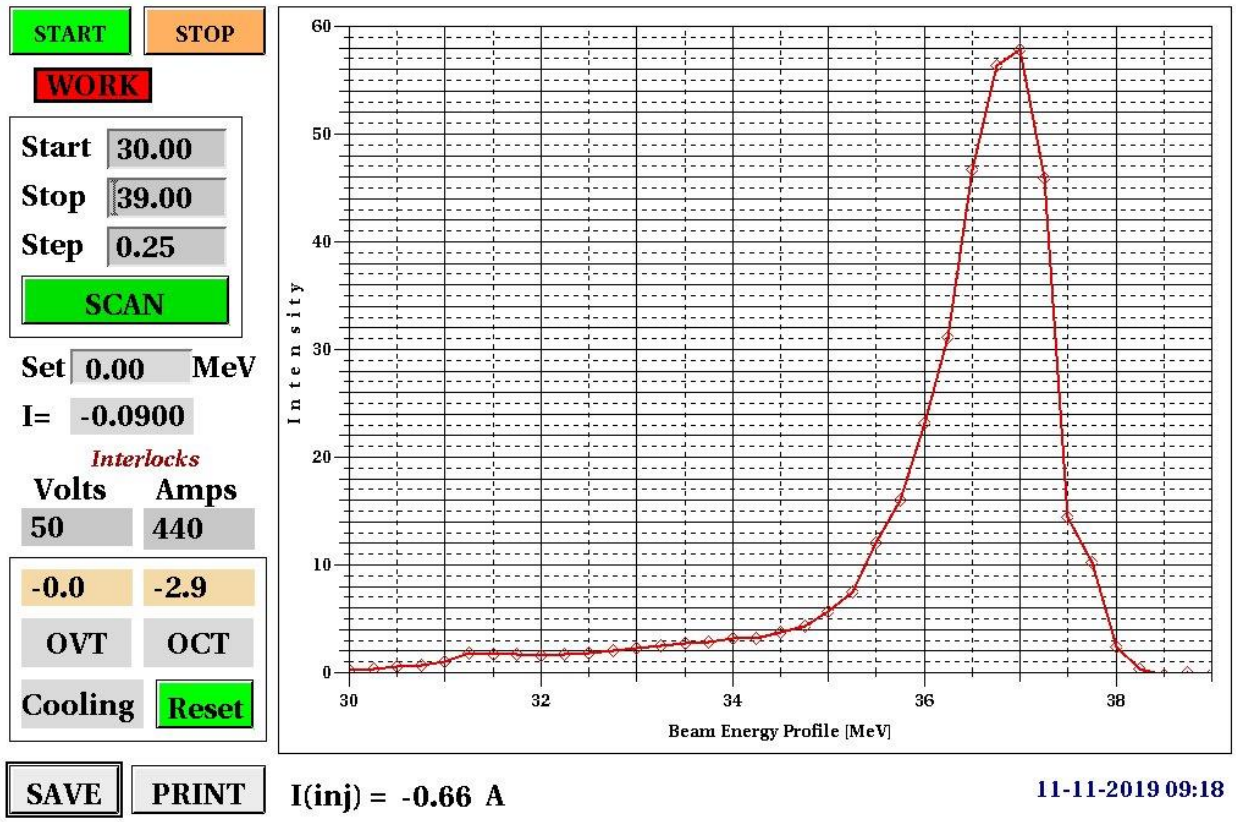


FIGURE 3.1.3.1 Beam energy spectrum for Irradiation #2 on 11/11/19. The energy spectrum was recorded at a lower energy than 40 MeV because of the thermal limitation of the spectrometer. After the initial tune-up, the beam peak current was reduced to adjust the peak energy to 40 MeV.

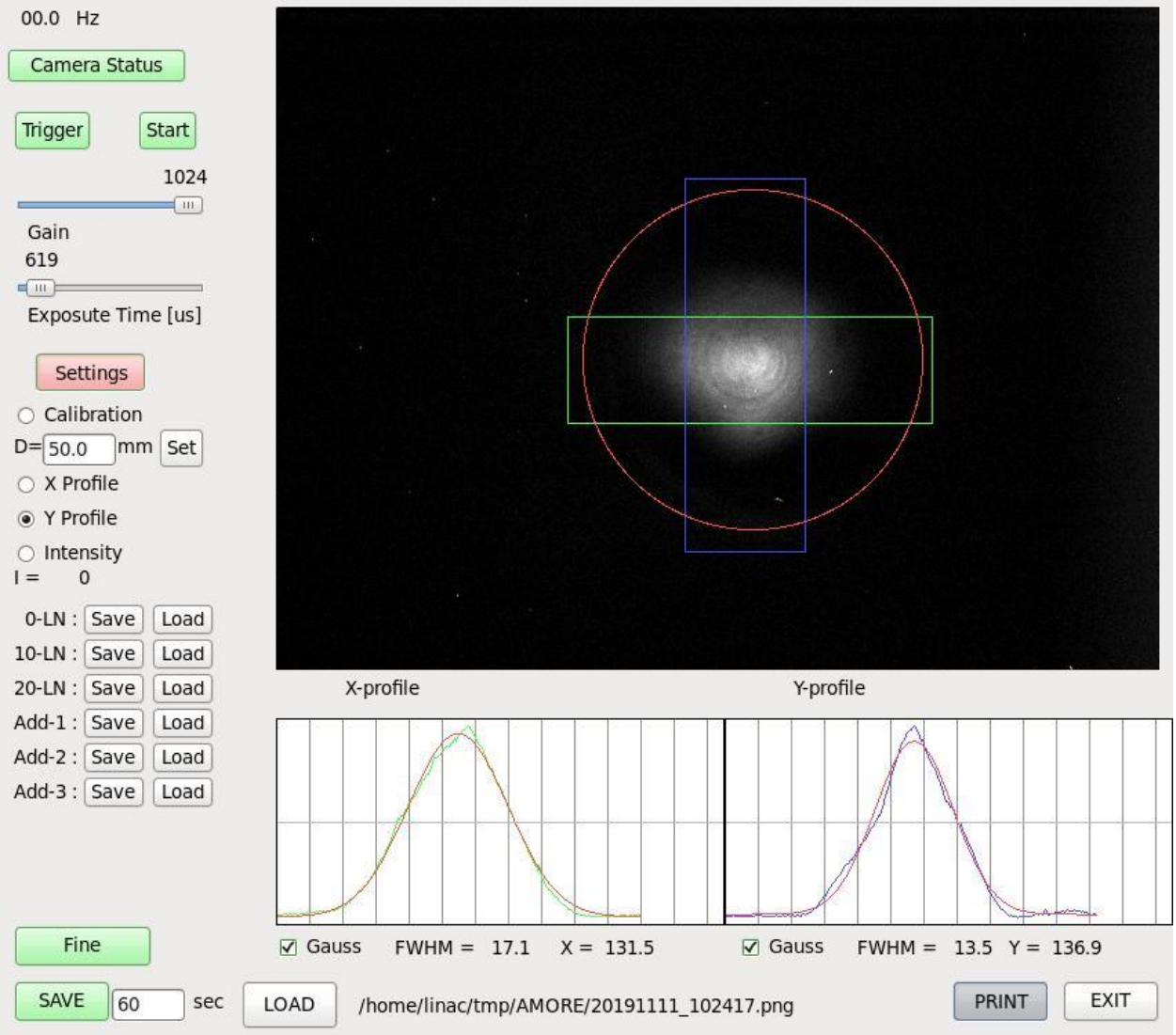


FIGURE 3.1.3.2 Beam profile on the target window for Irradiation #2. Red circle outlines the target beam window boundary.

Irradiation started at 10 am at ~175 W of beam power on the target. Over 1.5 hours, the power was increased to 15 kW. The beam was stopped at 12:11 pm and restarted at 12:15 pm. At 12:42, 16.8 kW of beam power on the target was achieved. Beam power was maintained at 17.2 kW for the duration of the irradiation. The maximum beam power used in this experiment was 17.6 kW, limited by hydrogen production and the requirement to maintain hydrogen concentration under 1%. Figure 3.1.3.3 shows the beam history for the irradiation. Irradiation was finished at 2 am the next day. At 1:55 am, the beam power was shut off, and the gas-analysis and -collection system was purged to reduce the amount of radioactive gas in the gas-analysis manifold. Processing of the irradiated solution started at 3 am.

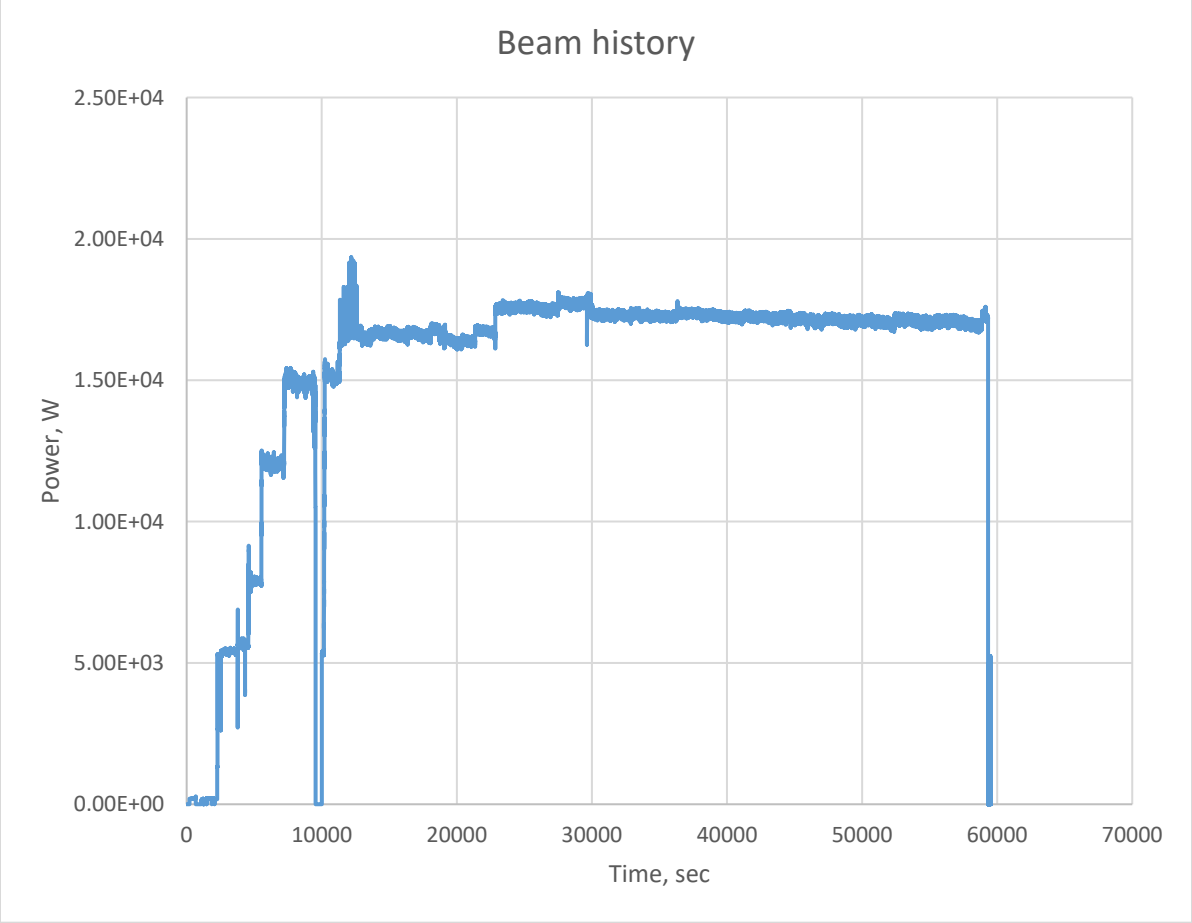


FIGURE 3.1.3.3 Beam history for Irradiation #2

3.1.4 Irradiation #3, 3/2/20

The third experiment after restart was completed on 3/2/20. The plan was to irradiate the uranyl sulfate solution for 24 hours at a maximum beam power limited by hydrogen production/recombination, to produce up to 20 Ci of ^{99}Mo. Initial beam tune-up was conducted on February 26 and 28, 2020.

At 10 am on the irradiation day, the beam was tuned and prepared for transport to the target. The beam energy spectrum is presented in Figure 3.1.4.1. This spectrum corresponds to a peak energy of 38 MeV at 0.56 A peak current. After spectrum acquisition, the peak beam current was reduced to 0.50 A, which shifted the beam energy peak to 40 MeV. After energy verification, the beam was placed on the target window at low power (175 W), and the beam shape was adjusted to produce a 20x20.5-mm FWHM beam spot on the target face (Figure 3.1.4.2).

The irradiation started at 12 pm with 175 W of beam power on the target. Over 2 hours, power was increased to 15 kW. Irradiation was stopped at 1:56 pm because of a worsening vacuum in the beamline. Irradiation was restarted at 3:43 pm. Power on the target was gradually increased to 17.5 kW. The maximum beam power used in this experiment was 18 kW, limited by hydrogen production and the requirement to maintaining hydrogen concentration under 1%. Figure 3.1.4.3 shows the beam history for the irradiation. Irradiation was interrupted at 5:28 am the next day because of a trip in the modulator and a worsening vacuum in the beamline. At that point, irradiation could not be continued and had to be canceled. Investigation of the vacuum issues revealed significant scraping of the beam in the beamline that led to the leak in the beamline. No processing of the irradiated solution was attempted because estimated production of ^{99}Mo was too low, and we would not be able to meet minimum activity requirements for the shipment.

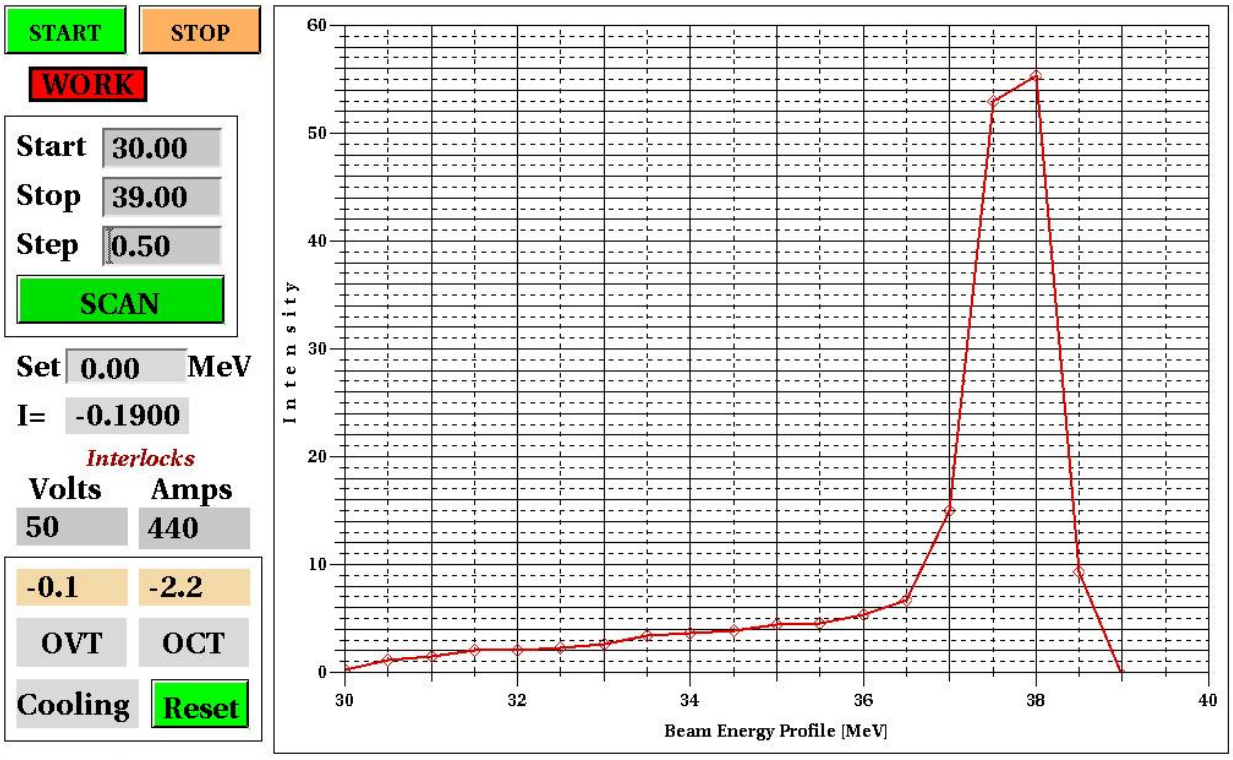


FIGURE 3.1.4.1 Beam energy spectrum for Irradiation #3 on 3/1/20. The energy spectrum was recorded at a lower energy than 40 MeV because of the thermal limitation of the spectrometer. After initial tune-up, beam peak current was reduced to adjust the peak energy to 40 MeV

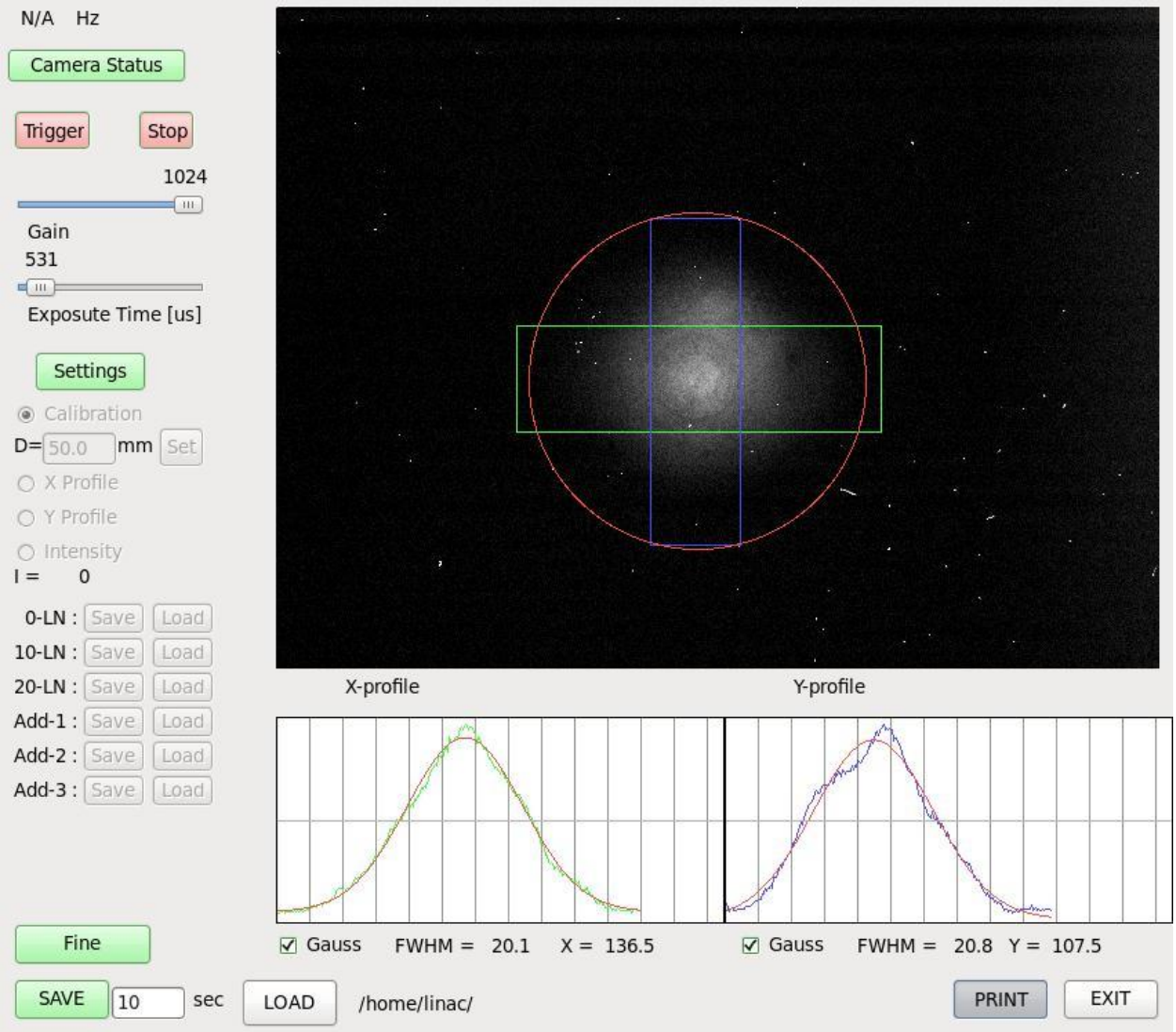


FIGURE 3.1.4.2 Beam profile on the target window for Irradiation #3. Red circle outlines the target beam window boundary.

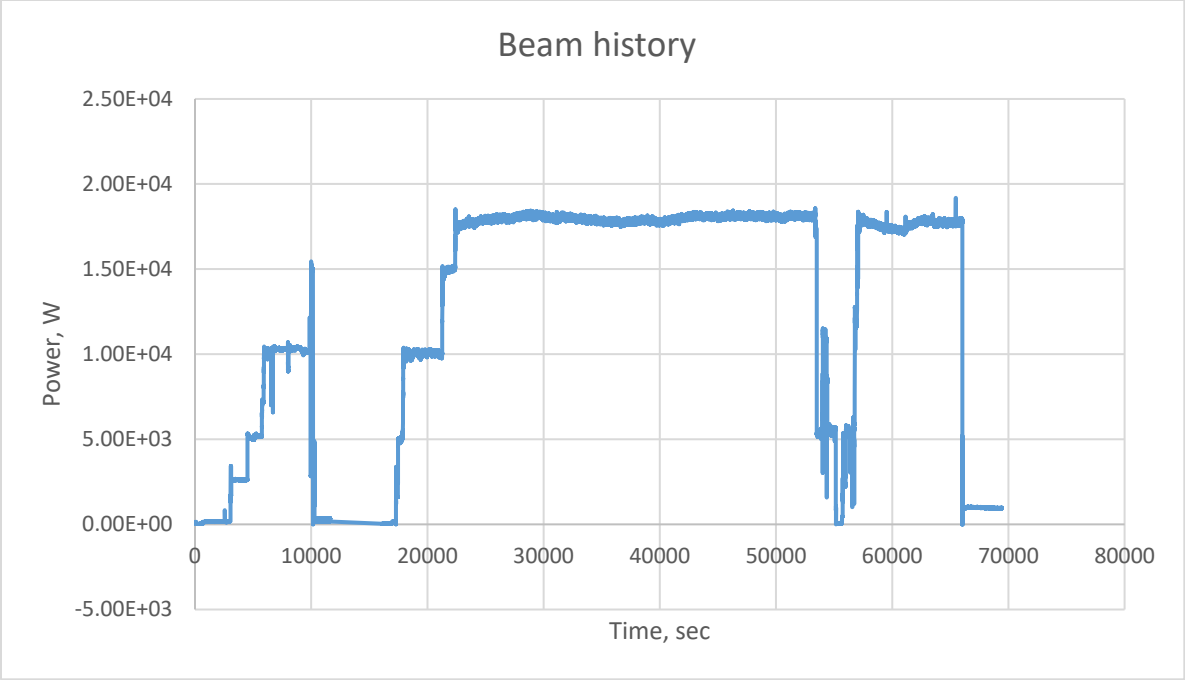


FIGURE 3.1.4.3 Beam history for Irradiation #3

3.1.5 Irradiation #4, 8/30/20

The fifth experiment after restart was conducted on 8/30/20. The plan was to irradiate uranyl sulfate solution for 10 hours at a maximum beam power limited by hydrogen production/recombination. Initial beam tune-up was conducted on 8/26/20. The final tune-up was conducted on 8/28/20. The beam energy spectrum is presented in Figure 3.1.5.1. This spectrum corresponds to a peak energy of 36 MeV at 0.64 A peak current. After spectrum acquisition, the peak beam current was reduced to 0.53 A, shifting the beam energy peak to 40 MeV.

Irradiation started at 7 am on 8/30/20. After energy verification, the beam was placed on the target window at low power (~180 W), and the beam shape was adjusted to produce a 16.1x19.4-mm FWHM beam spot on the target face (Figure 3.1.5.2). Over 1.5 hours, the power was increased to 12.5 kW. Beam power was maintained at ~12.5 kW for the duration of the irradiation. The maximum beam power used in this experiment was 12.5 kW, limited by hydrogen production and the requirement to maintain hydrogen concentration under 1%. Figure 3.1.5.3 shows the beam history for the irradiation. The irradiation was interrupted at 7 pm because of loss of vacuum in the beamline. The loss of vacuum was caused by a sudden change of the injector current, causing the beam to deviate from the proper trajectory. This deviation caused the beam to strike the wall of the vacuum chamber, leading to a loss of vacuum. At the end of irradiation, the gas-analysis and -collection system was purged to reduce the amount of radioactive gas in the gas-analysis manifold. Processing of the irradiated solution started shortly thereafter.

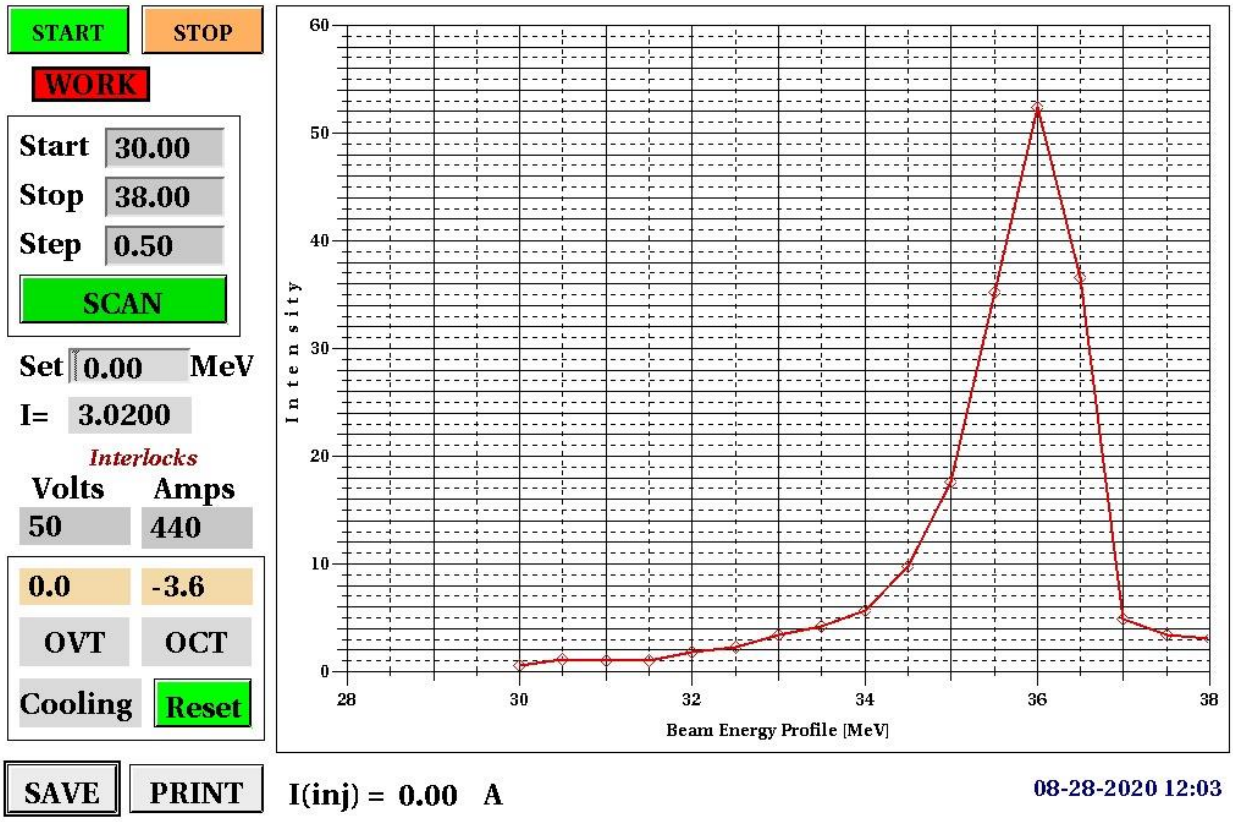


FIGURE 3.1.5.1 Beam energy spectrum for Irradiation #4 on 8/30/20. The energy spectrum was recorded on August 28 at a lower energy than 40 MeV because of the thermal limitation of the spectrometer. After initial tune-up, the beam peak current was reduced to adjust the peak energy to 40 MeV.

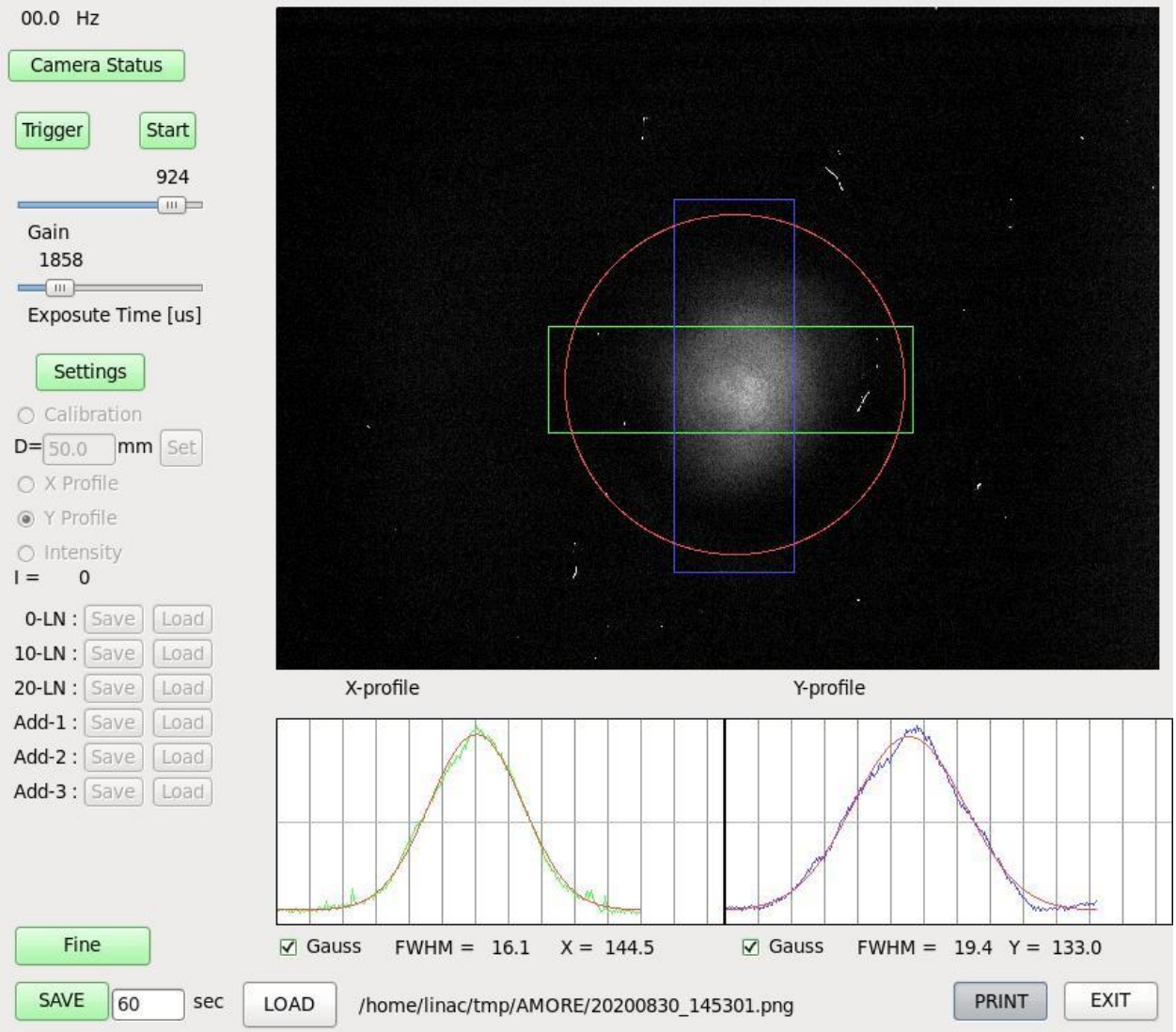


FIGURE 3.1.5.2 Beam profile on the target window for Irradiation #4. Red circle outlines the target beam window boundary.

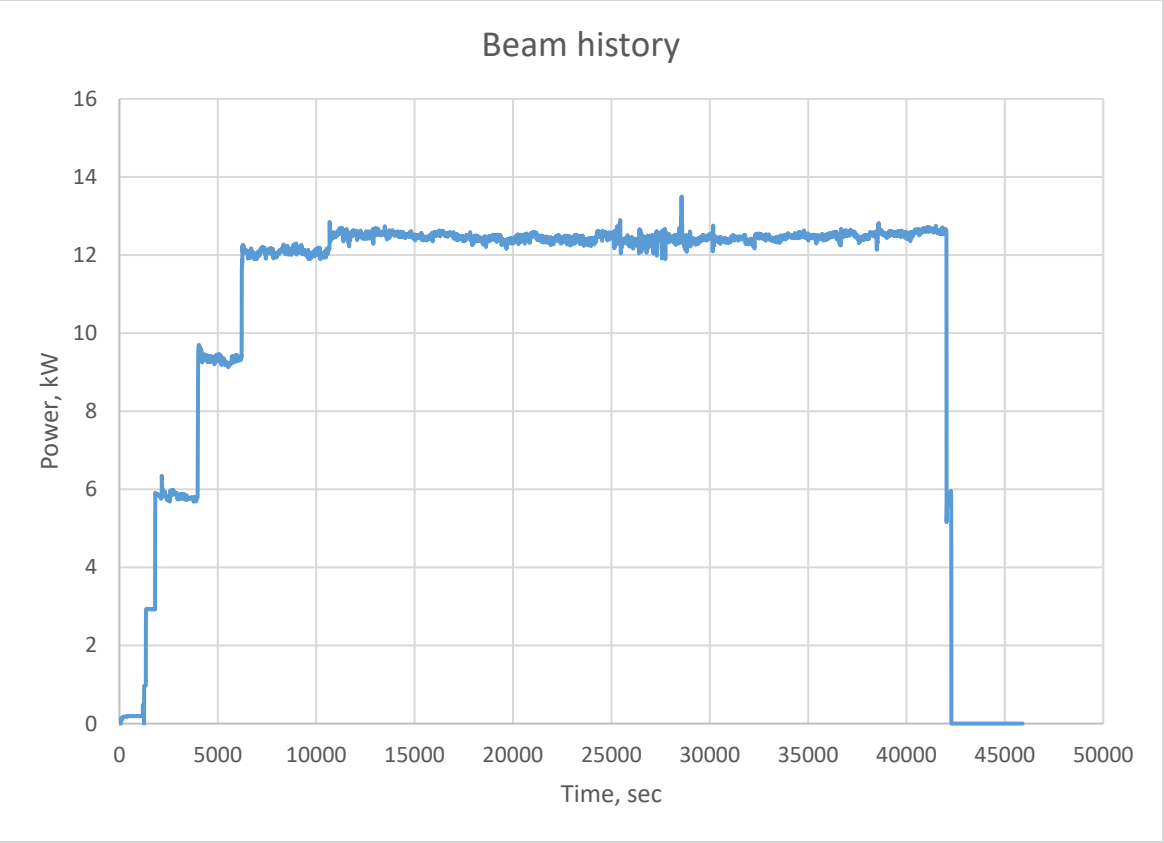


FIGURE 3.1.5.3 Beam history for Irradiation #4

3.1.6 Irradiation #5, 1/18/21

The last experiment was completed on 1/18/21. The plan was to irradiate the uranyl sulfate solution for 24 hours at a maximum beam power limited by hydrogen production/recombination. Initial beam tune-up was conducted on 1/15/21. The beam energy spectrum is presented in Figure 3.1.6.1. This spectrum corresponds to a peak energy of 37 MeV at 0.56 A peak current. After spectrum acquisition, the peak beam current was reduced to 0.46 A, which shifted the beam energy peak to 40 MeV.

Irradiation started at 7 am on 1/17/21. The beam was placed on the target window at low power and the beam shape was adjusted to produce an 18.6x21.1-mm FWHM beam spot on the target face (Figure 3.1.6.2). The beam power was gradually increased to 13.5 kW and maintained at ~12.5 kW for most of the duration of the irradiation. The maximum beam power used in this experiment was 12.5 kW, limited by hydrogen production and the requirement to maintain hydrogen concentration under 1%. The irradiation was interrupted three times: first, for two hours because of sudden changes in RF parameters of the modulator; and twice more for several minutes each because of interlock protection trips on the RF modulators. The irradiation was finished at 8 am the next day. Figure 3.1.6.3 shows the beam history for the irradiation. At the end of irradiation, the gas-analysis and -collection system was purged to reduce the amount of radioactive gas in the gas-analysis manifold. Processing of the irradiated solution started at ~9 am.

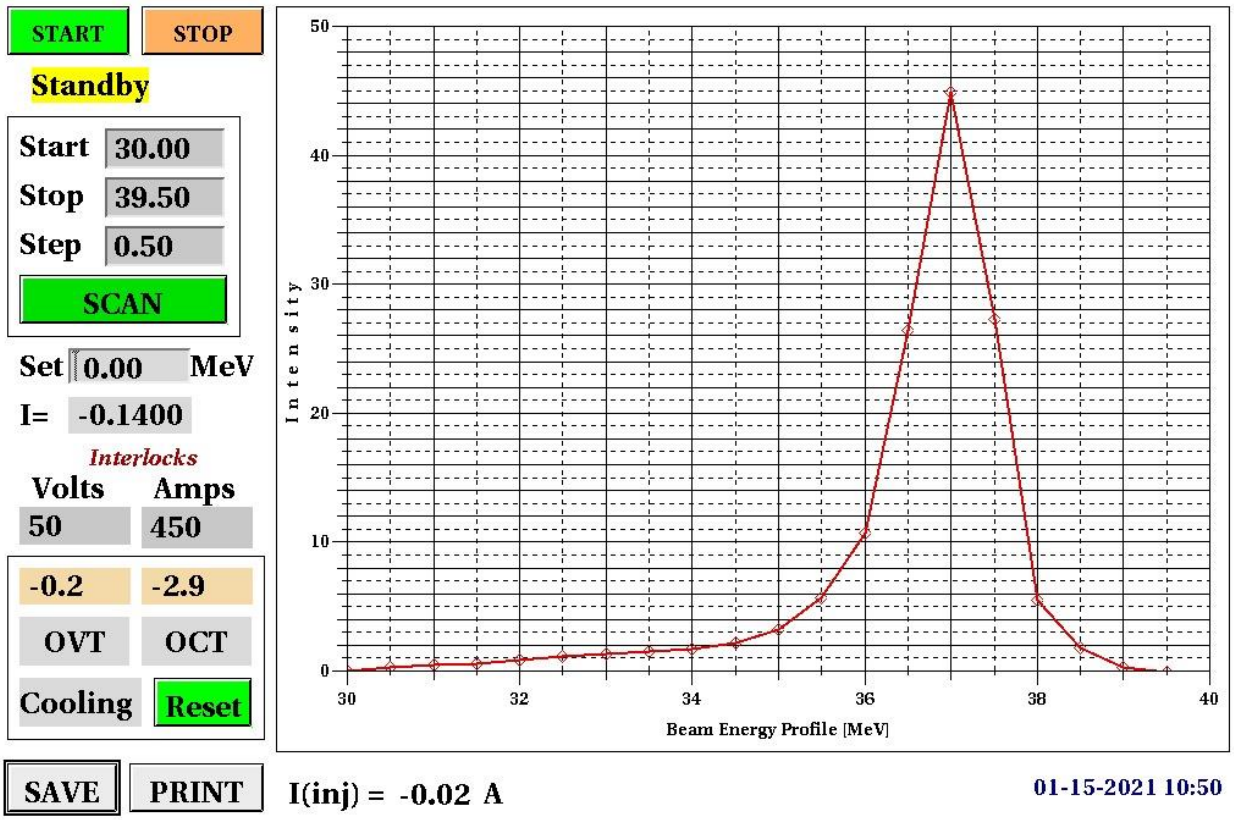


FIGURE 3.1.6.1 Beam energy spectrum for Irradiation #5 on 1/17/21. The energy spectrum was recorded on January 15 at a lower energy than 40 MeV because of the thermal limitation of the spectrometer. After initial tune-up, the beam peak current was reduced to adjust the peak energy to 40 MeV.

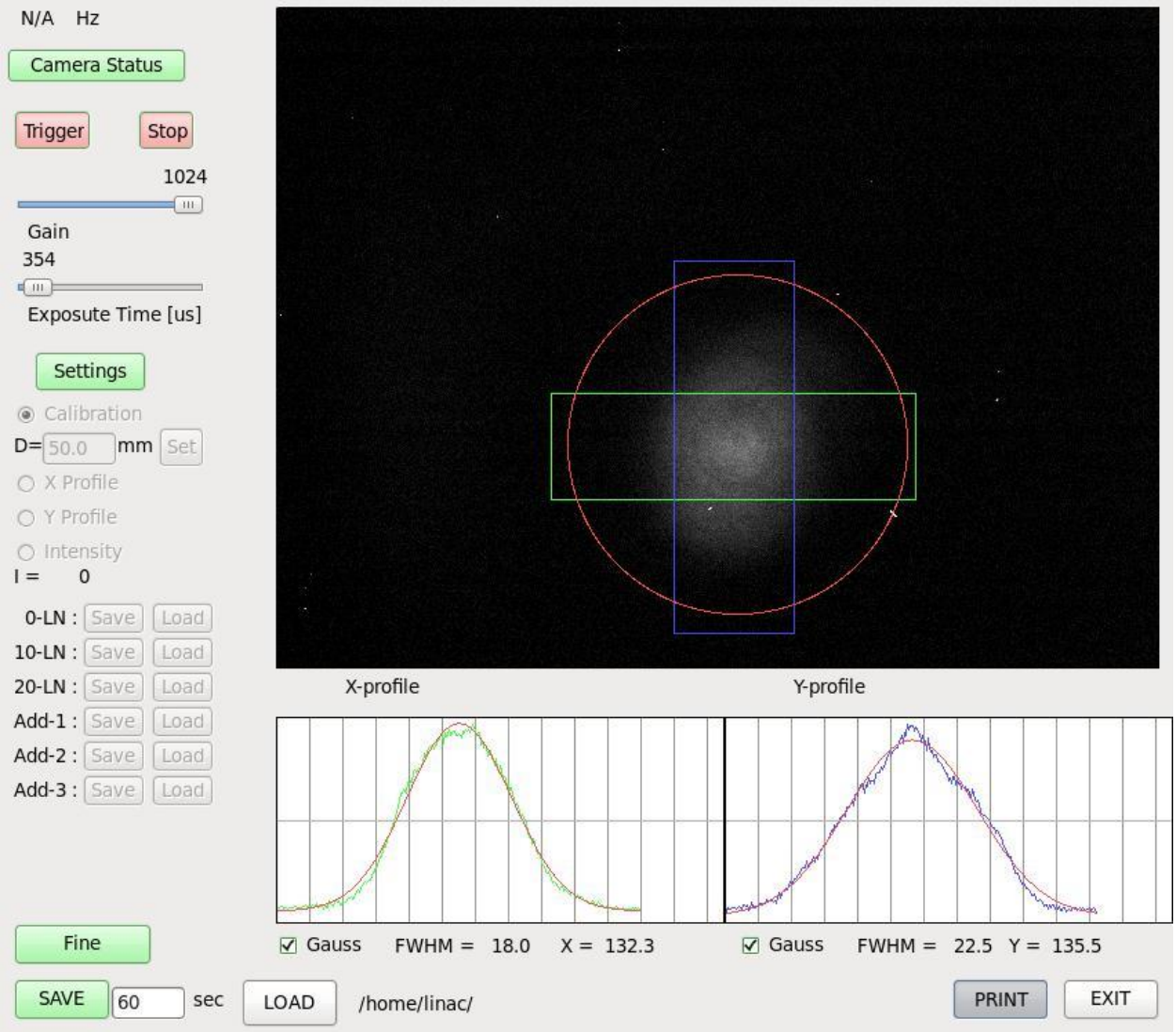


FIGURE 3.1.6.2 Beam profile on the target window for Irradiation #5. Red circle outlines the target beam window boundary.

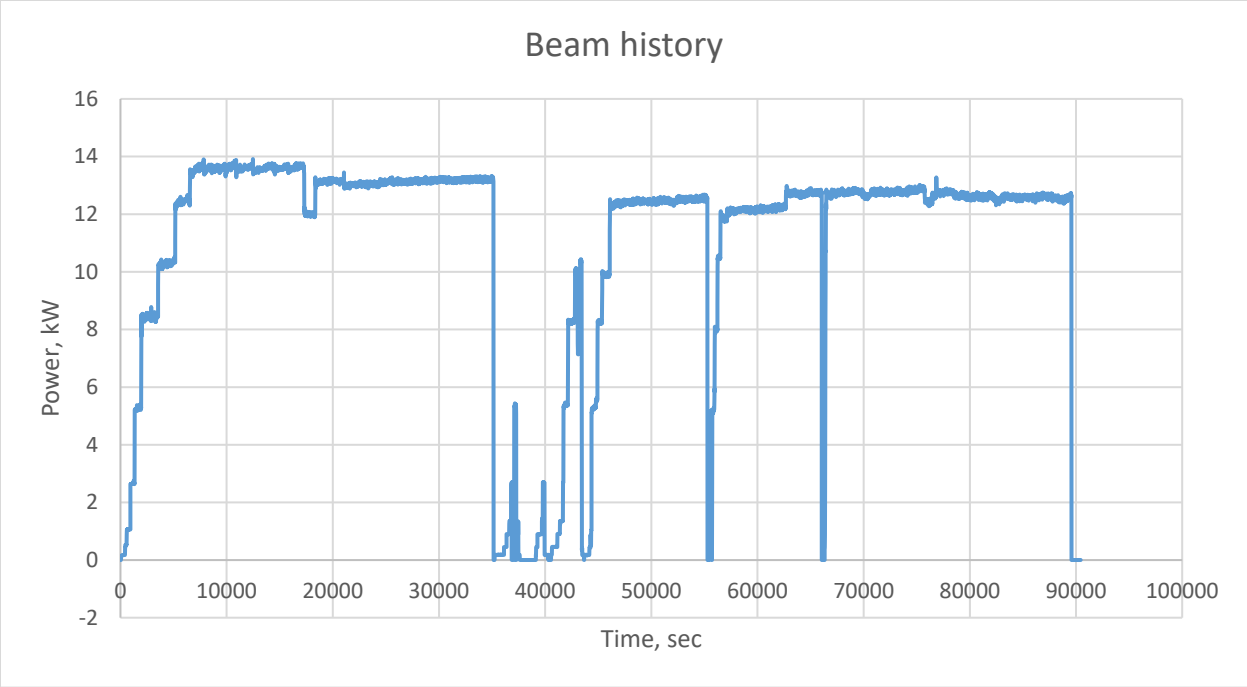


FIGURE 3.1.6.3 Beam history for Irradiation #5

3.1.7 Summary and Conclusions

A total of five irradiations of uranyl sulfate solution were conducted. On two occasions, the irradiations were interrupted because of loss of vacuum in the beamline, due to beam misplacement. Because of the chromatic nature of the beamline (beams with different energies will emerge after the bend at different points and traveling in different directions), the trajectory of the beam was very sensitive to the beam energy and stability of the accelerator parameters. An achromatic transport line (a line where beams will emerge with the same position and direction after the bend regardless of beam energy) would improve the reliability of the irradiations.

All irradiations were limited in maximum beam power delivered to the target because of hydrogen production. This was the only limitation; the target design allowed placement of the full beam power, 20 kW at 40 MeV, on the target.

3.2 GAS ANALYSIS

Each experimental section shows data generated by the RGA. The RGA was calibrated by applying a standard of known concentration. Equation (1) was used to generate a Relative Response Factor (RRF) for each analyte. The RRF is based on the ion current of the analyte ($IC_{analyte}$), the concentration of the analyte ([%Analyte]), the ion current of the internal standard (IC_{is}), and the concentration of the internal standard ([IS]). During the experiments, the analyte concentration was determined using Equation (2). Helium used to purge the solution vessel

contained 1.0 % xenon as the internal standard. Since the solution vessel was not purged before experiments, the accuracy of the hydrogen and oxygen values generated by the RGA was subject to error because of the low concentration of internal standard in the vessel headspace. The RGA data were compared to the HYOptima Hydrogen Sensor during each experiment. The hydrogen concentration values generated by the RGA were consistently lower than the sensor data. Since the hydrogen sensor does not suffer from the same error as the RGA, the percent error was calculated for the RGA data when compared to the sensor data, which are assumed to be the true values. The analytical data presented in graphical form for each experiment were generated by the RGA. The percent error for hydrogen is shown in the discussion section for each experiment, where the true value is assumed to be that of the sensor and the error is in the graphical data. Before each experiment, the RGA and hydrogen sensor were calibrated using standards prepared from a primary standard with a certified accuracy of ±2%. The certified standards were procured from a vendor. The instruments were calibrated at the same pressure that occurs during an irradiation. The calibrations were verified with check standards, and the acceptance ranges for percent recovery were ±10% for the RGA and ±5% for the Hydrogen Sensor.

Since the analytical instruments were located in an adjacent room outside the irradiation cell, there is a time delay of about 4 minutes between the actual solution vessel concentration and the analytical data.

$$RRF = IC_{analyte} \times [IS]/IC_{IS} \times [\%Analyte] \quad (1)$$

$$[\%Analyte] = IC_{analyte} \times [IS]/IC_{IS} \times RRF \quad (2)$$

3.2.1 Commissioning Run, No Irradiation, 8/15/19

The Gas Handling System was commissioned on 8/15/19. System checks were performed and gas composition was monitored in the same way that they would be during actual experiments. The GCS (see Figure 2.2.2.7) functioned as designed. This functioning was tested by adding helium to the system so that the pressure rose at about 60 mbar per minute. The pump in Chamber #1 started at 961 mbar and evacuated the system to 940 mbar, at which time the pump shut off. This protocol kept the pressure in the experiment between 940 and 961 mbar. Chamber #2 was filled with the excess gas. As the pressure in Chamber #2 rose to 1141 mbar, the compressor inside that chamber started operating and evacuated the chamber to 1020 mbar. The excess gas was pumped into the collection cylinders, causing a pressure rise of about 2 psig per compressor cycle.

The GCS interlocks were tested for the collection cylinders, Chamber #2, and Chamber #1. The function of the interlocks is to disable power to the linac during an experiment. The interlock for the collection cylinders was tested and tripped by setting the trip pressure lower than the pressure that was actually in the cylinders at the time of the test. The Chamber #2 interlock was tested and tripped by setting the trip pressure lower than the pressure in the chamber at the time of the test. The alarm state for Chamber #2 also actuates and closes a solenoid valve located between the collection cylinders and Chamber #2. The pumps in each

chamber are also disabled. The purpose is twofold: In the event the Chamber #2 compressor fails, (1) no more gas can be added to the GCS and (2) any leaks through the check valve between the cylinders and Chamber #2 are stopped, preventing the over-pressurization of Chamber #2 and release of gas through the rupture disc. The Chamber #1 interlock was tested and tripped by setting the trip pressure lower than the pressure in the chamber at the time of the test. Alarms were activated in the monitoring control room during each test.

Hydrogen alarms and interlocks were tested by introducing standards into the system. Hydrogen alarms activated with the introduction of a 1% standard. The hydrogen interlock tripped the linac interlock chain, and an alarm sounded in the monitoring control room, upon introduction of a 2% standard.

The verification of the alarm for the gas-sampling-pump flow was successfully completed. The verification of alarm and interlock responses for the catalyst pump was successful. The alarm test for solution-vessel pressure was successful. The alarm was tested by setting the alarm value lower than the actual pressure in the vessel. Oxygen introduction and a post-irradiation system purge were tested successfully. A 40% oxygen-in-helium mix was added at a rate of 50 mL/minute to the vessel. The rise in oxygen concentration was observed in the RGA monitoring data over an hour, and the increase was 3.5%.

The checklist tasks for pre-irradiation valve configuration were performed, and minor modifications were made for clarity.

Performance of the Gas Handling System pre-checks and interlocks, linac pre-checks and interlocks, and post-irradiation purge was successfully verified. Gas Handling procedures were followed as written. Some lessons learned were incorporated into a later revision of the procedures to improve clarity. The relevant parameters for the experimental systems were confirmed to be as described in the facility SAD and ASE, and all acceptance criteria were met. The checks described above were performed before each irradiation.

3.2.2 Irradiation #1, 10/1/19

Figure 3.2.2.1 shows gas concentration and linac beam power for Irradiation #1 on 10/1/19. The RGA data presented has a percent error of 22% and is lower when compared to the sensor data for hydrogen. In this experiment, hydrogen concentration began to rise when the beam energy reached 1.37 kWh, whereas the residual concentration of oxygen began to decrease through recombination with hydrogen at the catalyst at 0.34 kWh. The dip in hydrogen concentration was due to beam-power fluctuations. What is striking, and was not expected, is that we needed to add oxygen to the solution vessel. The oxygen/helium mixture was added to the system at 120 min, at a rate of 50 mL/min, to control hydrogen concentration in the vessel. The system was not purged with helium before the irradiation, and oxygen was at about 13%. It was assumed that the residual oxygen would be sufficient to compensate for the initial oxygen deficit that occurred during the initial stage of an irradiation.

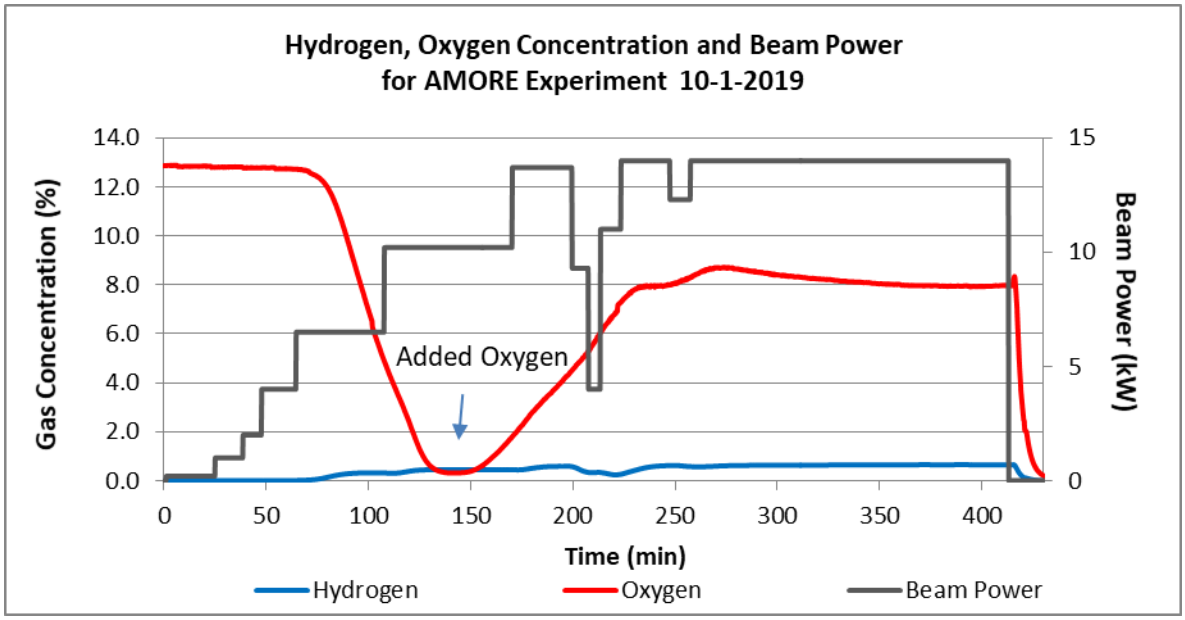


FIGURE 3.2.2.1 Gas concentration and linac beam power for Irradiation #1 on 10/1/19

Prior to this irradiation, while transferring the uranyl sulfate solution from the vessel during sample collection, uranyl peroxide precipitate was observed in the solution. An attempt was made to remove as much of the precipitate as possible. On 9/9/20, a solution of iron sulfate was added to the solution vessel at 164 ppm in an attempt to mitigate the precipitation[1]. The iron concentration was determined by Inductively Coupled Plasma Mass Spectrometry (ICPMS) analysis on 9/23/19. We observed additional precipitate every time the solution was moved, including on 11/6/19. On 2/13/20, the solution was moved for the first time with no visible precipitate, and none was observed after that.

The reaction of hydrogen peroxide and uranyl sulfate forms a precipitate, uranyl peroxide. In the initial stage of water radiolysis, H_2 and H_2O_2 are generated. Subsequently, H_2O_2 will radiolytically decompose to O_2 and H_2O. If H_2O_2 reacts with uranyl sulfate to form uranyl peroxide, this will sequester the oxygen that would normally be released into the vessel as a gas. This sequestration can cause an oxygen deficit in the vessel headspace, which was seen here. Even though iron was added before Irradiation #1, crystals already present in solution could possibly have facilitated further precipitation.

3.2.3 Irradiation #2, 11/11/19

Figure 3.2.3.1 shows gas concentration and linac beam power for Irradiation #2 on 11/11/19. The RGA data presented has a percent error of 19% and is lower when compared to the sensor data for hydrogen. In this experiment, hydrogen concentration began to rise when beam energy reached 1.40 kWh, whereas the residual concentration of oxygen began to decrease through recombination with hydrogen at the catalyst at 0.03 kWh. Hydrogen was maintained at a steady concentration throughout the experiment, with a dip when the beam dropped out. Oxygen

showed a slow, steady decline. No additional oxygen was added during this experiment. When compared to the previous experiment, the decline in oxygen could be indicating that a precipitate is forming but not as rapidly. One possible explanation is that the precipitate that formed from the previous experiment had mostly decomposed as the solution sat in the vessel between experiments. Uranyl peroxide has been shown to decompose radiolytically. Prior to this experiment, uranyl peroxide precipitate was observed in the solution. An attempt was made to remove it. Here, it is possible that the rate of precipitate formation was slower than in Irradiation #1 because there were fewer crystals in solution to facilitate precipitation.

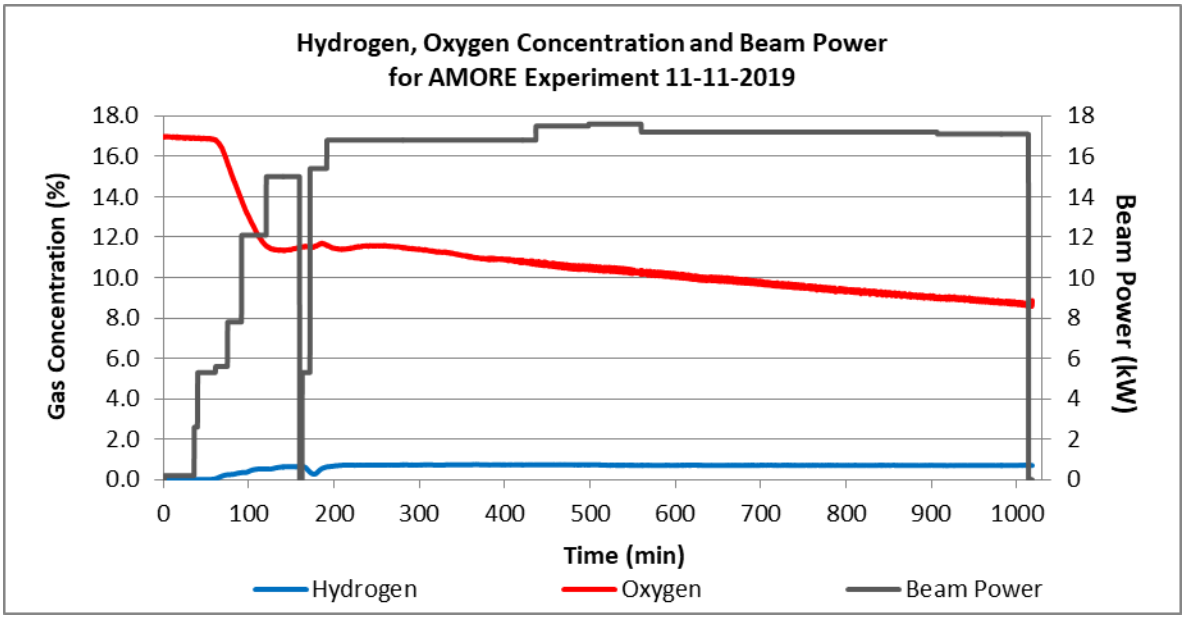


FIGURE 3.2.3.1 Gas concentration and linac beam power for Irradiation #2 on 11/11/19

3.2.4 Irradiation #3, 3/2/20

Figure 3.2.4.1 shows gas concentration and linac beam power for Irradiation #3, completed on 3/2/20. The RGA data presented have a percent error of 18% and are lower when compared to the sensor data for hydrogen. In this experiment, hydrogen concentration began to rise when beam energy reached 1.77 kWh, whereas the residual concentration of oxygen began to decrease through recombination with hydrogen at the catalyst at 0.81 kWh. In this experiment, it appears that oxygen concentration continued to show a downward trend similar to that seen in Irradiation #2, though not as dramatic. The oxygen decrease for Irradiation #2 was approximately 0.0037% per minute, whereas that for Irradiation #3 was 0.001% per minute. Each subsequent experiment following the precipitation event became more stable with respect to gas generation. Prior to Irradiation #3, no visible sign of precipitate was observed. The concentration of iron in the solution was 150 ppm, as determined by ICPMS analysis on 2/28/20. Iron concentration was lower than previously determined because there was a slight dilution of the uranium solution in the vessel due to processing.

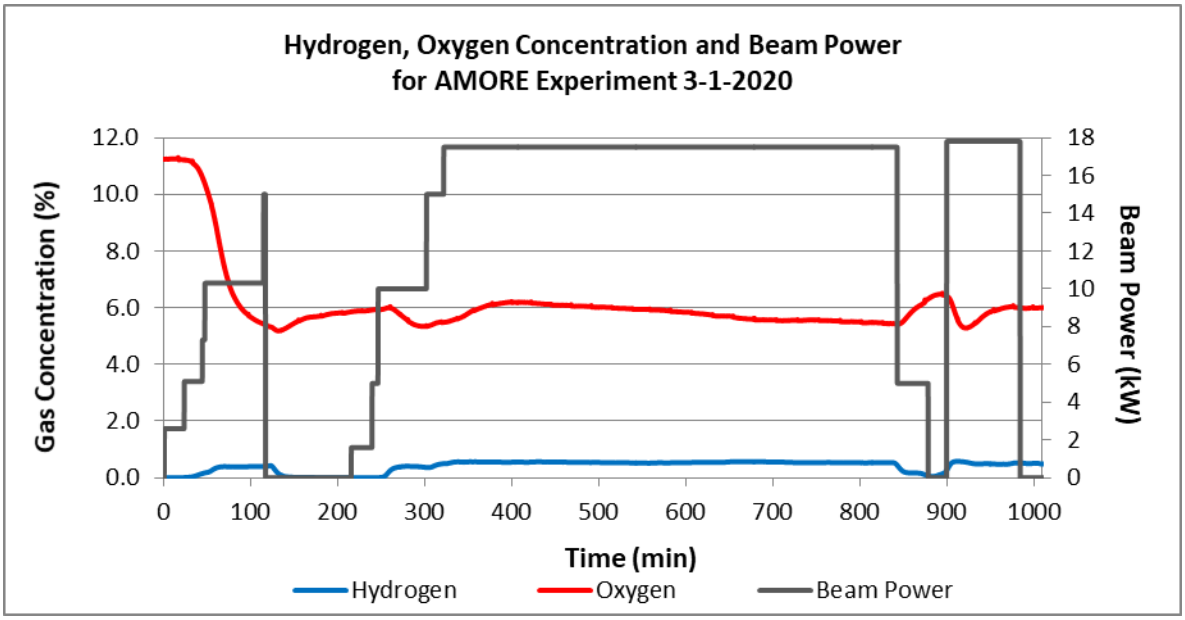


FIGURE 3.2.4.1 Gas concentration and linac beam power for Irradiation #3 on 3/1/20

3.2.5 Irradiation #4, 8/30/20

Figure 3.2.5.1 shows gas concentration and linac beam power for Irradiation #4 on 8/30/20. The RGA data presented have a percent error of 13% and are lower when compared to the sensor data for hydrogen. Before this experiment, the KNF Neuberger N186 catalyst pump needed replacement. The new pump was a Senior Aerospace Metal Bellows MB-151. The smaller replacement pump meant a reduction in flow through the catalytic recombiner. The reduced flow required limiting the beam power to about 12.5 kW in order to keep hydrogen concentration <1%. In this experiment, the hydrogen concentration began to rise when the beam energy reached 1.22 kWh, whereas the residual concentration of oxygen began to decrease through recombination with hydrogen at the catalyst at 1.70 kWh. The initial drop in oxygen occurred at a higher beam energy than in the previous experiments. After the initial drop, oxygen showed no deficit throughout the irradiation. A steady-state hydrogen and oxygen concentration was maintained throughout the experiment. This observation indicates a balanced gas generation and catalytic recombination. No precipitate was observed in the solution before or after this experiment.

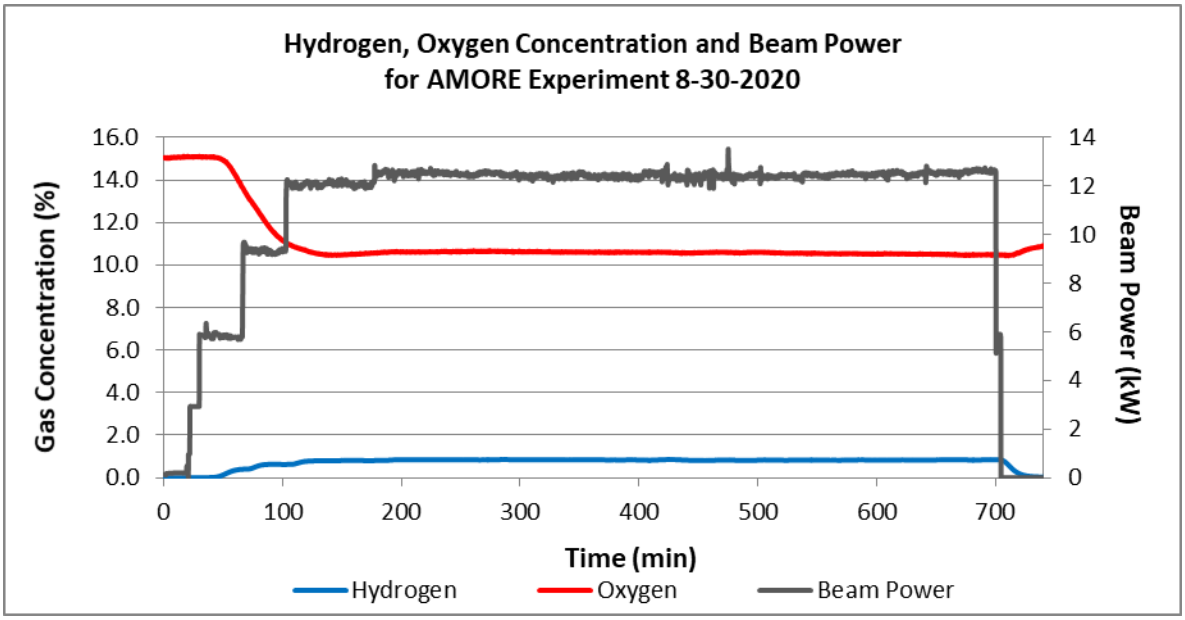


FIGURE 3.2.5.1 Gas concentration and linac beam power for Irradiation #4 on 8/30/20

3.2.6 Irradiation #5, 1/18/21

Figure 3.2.6.1 shows gas concentration and linac beam power for Irradiation #5, completed on 1/18/21. The RGA data presented have a percent error of 13% and are lower when compared to the sensor data for hydrogen. In this experiment, hydrogen concentration began to rise when beam energy reached 1.78 kWh, whereas the residual concentration of oxygen began to decrease through recombination with hydrogen at the catalyst at 0.14 kWh. The concentrations of hydrogen and oxygen remained steady until the beam dropped out. At those times, hydrogen concentration was reduced through recombination at the catalyst, and oxygen increased because it came out of the solution as hydrogen peroxide decomposed. This experiment showed similar characteristics to Irradiation #4 in that steady-state hydrogen and oxygen concentrations were maintained throughout the experiment. This observation indicates a balanced gas generation and catalytic recombination and no uranyl peroxide precipitation. No precipitate was observed in the solution before or after Irradiation #5.

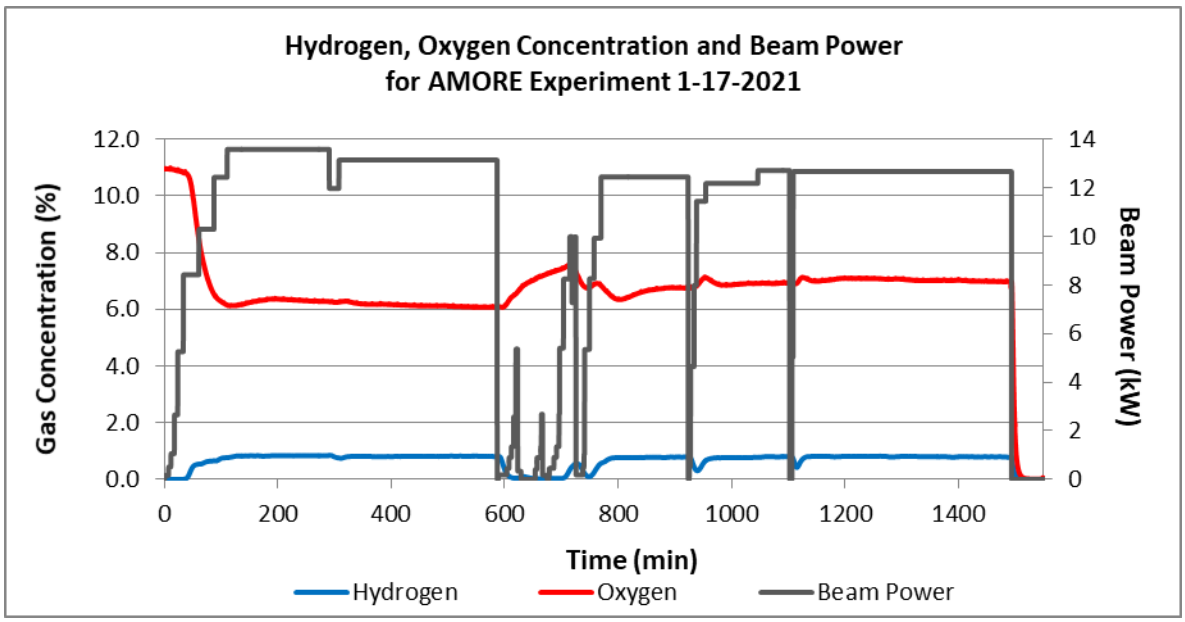


FIGURE 3.2.6.1 Gas concentration and linac beam power for Irradiation #5 on 1/17/21

3.2.7 Summary

Before Irradiation #1 on 10/1/19, a precipitate (uranyl peroxide) was observed in the uranyl sulfate solution. Iron, as ferrous sulfate, was added to mitigate further precipitation. The concentration of iron was 164 ppm in the solution vessel, as determined by ICPMS analysis on 9/23/19. Later analysis of the solution on 2/28/20 showed 150 ppm iron. We have observed a slight dilution of the uranium solution in the vessel due to processing. The oxygen deficit observed in Irradiation #1 was indicative of continued precipitation of uranium as uranyl peroxide. Uranyl peroxide crystals already present in solution could possibly have facilitated further precipitation. Hydrogen peroxide is generated by the radiolysis of water and reacts with uranyl sulfate to form uranyl peroxide instead of decomposing to oxygen and water. This reaction sequesters the oxygen that would normally be released into the vessel as a gas. Subsequent experiments showed a much smaller oxygen deficit. The final two experiments Irradiations #4 and #5 on 8/30/20 and 1/17/21, showed hydrogen and oxygen concentration stable throughout. A steady-state hydrogen and oxygen concentration was maintained throughout those experiments, indicating balanced gas generation and catalytic recombination.

In previous experiments, we observed that radiolysis can cause the uranyl sulfate solution to directly precipitate during an irradiation, form a precipitate after irradiation (delayed), and radiolytically decompose the precipitate during an irradiation [1,2]. It is possible that after the initial precipitation events that occurred before and during Irradiation #1 on 10/1/19, uranyl peroxide began to decompose as the solution sat between experiments. During subsequent irradiations, the hydrogen peroxide generated was decomposed rapidly enough by the added iron to prevent further precipitation. No precipitate was observed on 2/13/20 during solution processing and transfer.

Before Irradiation #4 on 8/30/20, the catalyst pump was replaced with a new but smaller pump. The smaller pump meant a reduction in flow through the catalytic recombiner. The reduced flow required limiting beam power to about 12.5 kW in order to keep hydrogen concentration <1%.

3.2.8 References

[1] Youker, A., Kalensky, M., Quigley, K., Brossard, T., Chemerisov, S.D., and Vandegrift, G.F. *Uranyl Sulfate Irradiations at the Van de Graaff: A Means to Combat Uranyl Peroxide Precipitation*, ANL/NE-17/17, Argonne National Laboratory, 2017.

[2] Silverman, M.D., Watson, G.M., and McDuffie, H.F., Peroxide Decomposition in Aqueous Homogeneous Reactor Fuels, *Industrial and Engineering Chemistry 1956*; 8:1238–1241.

3.3 RECOVERY COLUMN

3.3.1 Commissioning Run, No Irradiation, 8/14/19

The commissioning run was carried out by adding a known amount of ^{99}Mo to the target solution and circulating it to simulate an irradiation and exercise LEAF-PROC-024 (see Appendix 17). At the same time, a small (50 mL) solution of ferrous sulfate dissolved in 0.1 M H_2SO_4 was added to the target solution as a catalyst to ensure that any further precipitation of uranyl peroxide would be prevented. The target solution had a mass of 20.795 kg, a concentration of 131.93 g U/L, and a density of 1.18357 g/mL, resulting in a total of 2318 g U in a volume of 17.57 L for this simulated irradiation. During the pre-irradiation checks of the system, it was noted that the balances used for measuring the masses of the verification tank, feed bottles, and effluent bottles were out of calibration. Because this was a simulated irradiation, the commissioning run continued as planned with the understanding that the balances needed to be calibrated before the first real irradiation.

After loading molybdenum into the system, the solution was transferred between the verification tank and the TSV several times to allow for good mixing of the ^{99}Mo spike. During mixing of the ^{99}Mo spike with the LEU solution, the presence of uranyl peroxide precipitate (later confirmed by powder XRD analysis) was observed in the target solution. This precipitate likely formed during the first Phase II irradiation in March 2018 (prior to the addition of the iron catalyst). Formation of uranyl peroxide under irradiation conditions is not unexpected, and was confirmed during small-scale irradiations of a similar uranyl sulfate solution at the 3 MeV VDG electron accelerator. The formation of uranium precipitate is due to complexation of the uranyl ion with peroxide that forms because of the radiolysis of water. This precipitate did not impede processing during the commissioning run, and the bulk of it was removed prior to the first irradiation (shown in Figure 3.3.1.1).

FIGURE 3.3.1.1 Uranyl peroxide precipitate in target solution that was extracted from the system prior to the first irradiation

After mixing, the LEU target solution containing the ^{99}Mo spike was loaded onto the column, molybdenum and uranium were eluted from the column in separate fractions, and the solution was sent to and received in the D024 Hot Cell. It was noted that several valves and sensors inside the glovebox were not working, owing to radiation damage or precipitate buildup.

Fourteen samples were collected during the various solution processing steps, divided as shown in Table 3.3.1.1. On the basis of observations from the previous irradiation, there were several sample loops that had to be skipped during sample collection because they were not operable. These included target mixing loop 1, column loading loop 3, and column loading loop 5.

Several issues arose while trying to retrieve the various samples from sample loops. The vacuum pump used to pull solution from the sample loops to the evacuated sample vials became inoperable and had to be replaced. Once replaced, it was found that solution could not be recovered from target mixing loop 3, column loading loops 1, 6, and 7, and column stripping loops 1 and 3. It was still possible to retrieve at least one sample from each processing step, but the number of failures was concerning since they appeared to be the result of uranyl peroxide precipitate clogging the valves used to recover the samples, which could not be easily replaced in the system.

TABLE 3.3.1.1 Processing samples taken and retrieved during the commissioning run

Processing Step	No. Samples Taken	No. Samples Retrieved
Target Solution Mixing Pre-Irradiation	1	1
Target Solution Mixing During Irradiation	2	1
Column Loading	2	1
Post-Load Acid Wash	2	1
Post-Load H_2O Wash	2	1
Column Stripping	3	1
Post-Strip H_2O Wash	2	2

This simulated irradiation was also used to refine LEAF-PROC-24, which was a compilation of the various glovebox processing procedures used in the first irradiation. A lengthy list of revisions was compiled and implemented prior to the next experiment.

Though the target solution was not irradiated, the collected samples were analyzed via gamma spectroscopy to track the efficacy of the ^{99}Mo processing steps. The samples recovered from the target mixing ladder showed only 85.9% of the known ^{99}Mo, indicating that a small amount of solution was trapped in the sample retrieval system even after it was cleaned out at the end of the previous processing. The single recovered column stripping sample showed 84.1% of the added ^{99}Mo, indicating that the majority of the ^{99}Mo would be removed from the column in the first 500 mL of stripping. Following column stripping, roughly 1.3% of the ^{99}Mo was still present in the system for the Post-Strip H_2O Wash samples. Table 3.3.1.2 shows the average results.

TABLE 3.3.1.2 Results from gamma analysis of samples recovered during the commissioning run

Sample	Average Total ^{99}Mo (mCi)	Std Dev (%)	Yield (%)
^{99}Mo Added	49.1	1.12%	N/A
Target Mixing	42.2	5.70%	85.9%
Column Stripping	41.3	2.47%	84.1%
Post-Strip H_2O Wash	0.6	1.24%	1.3%

3.3.2 Irradiation #1, 10/1/19

Referred to as the "Short Irradiation," this run was used to ensure that all the systems would operate as planned during a real irradiation. Prior to this irradiation, the uranyl peroxide precipitate was removed by pumping solution from the TSV into an external bottle until no more precipitate was observed in the stream. After removing as much precipitate as possible, the U concentration was adjusted with makeup solution. After adjustment, the target solution had a mass of 20.11 kg, concentration of 135.4 g U/L, and density of 1.18274 g/mL, resulting in a total of 2298 g U in a volume of 16.97 L for this irradiation. This was the first irradiation in which LEAF-PROC-024 Rev. 2 was used for preparing the system and processing the solution. This revision removed 32 pages of redundant instructions from the first revision and moved all replacement-part descriptions to the end. Because of the issues with retrieving samples during the commissioning run, samples for this run were re-prioritized. As a result, fewer processing steps were sampled, but more samples were taken from the process steps that were deemed more important. The distribution of samples taken during this irradiation is shown in Table 3.3.2.1. Further issues with sample retrieval occurred, resulting in recovery of only seven of the 18 collected samples. The recovered samples were representative of the target mixing, column loading, and column stripping processing steps.

TABLE 3.3.2.1 Samples taken and retrieved during processing for the first irradiation

Processing Step	No. Samples Taken	No. Samples Retrieved
Target Solution Mixing Pre-Irradiation	0	0
Target Solution Mixing After Irradiation	3	2
Column Loading	7	3
Post-Load Acid Wash	0	0
Post-Load H_2O Wash	0	0
Column Stripping	6	2
Post-Strip H_2O Wash	2	0

During processing, the pressure readings measured by the system were observed to increase slowly during column stripping. The filter on the base side of the system was bypassed to remedy this issue. Because bypassing the filter did not relieve the pressure, the sampling loop was also changed in case there had been some buildup of solids during processing. This change also had no effect, but processing was able to be completed before the pressure increased to the point of tripping the system pressure switch.

Gamma spectroscopy analysis was carried out on the two samples recovered from target mixing, the three recovered from column loading, and the two recovered from column stripping. The average masses of sample counted for target mixing, column loading, and column stripping were 224 mg, 268 mg, and 122 mg, respectively. Each sample was counted for one hour on 10/4/19, then counted for a second hour roughly three days later (10/7-8) to ensure the accuracy of the results.

Because mixing was not carried out during the irradiation, all target mixing samples reflect the contents of the target solution after irradiation. Both samples were taken in the last 30 min of mixing to ensure that they were representative of the homogenized solution. Unfortunately, the first target mixing sample was diluted by some solution that remained in the sample recovery line after washing the system at the end of the sample recovery run. For this reason, only the results from the second target mixing sample (designated TM-2) are reported in Table 3.3.2.2. The results indicate the presence of ^{97}Nb/^{97}Zr, ^{239}Np, ^{99}Mo, ^{103}Ru, ^{131}I, ^{132}I, ^{133}I, ^{140}Ba/^{140}La, and ^{143}Ce in the irradiated target with high confidence. Several other isotopes, including ^{105}Rh, ^{91}Sr, ^{135}I, ^{127}Sb, ^{125}Sn, ^{135}Xe, and ^{133}Xe may have been present, but had poor peak shape or high 1σ values. The remaining radionuclides were below the minimum detectable activity (MDA) for the solution analyzed.

The recovered column loading samples were taken at 5, 10, and 40 min into the loading phase of this processing run. Relatively little Zr/Nb was seen in these samples (designated CL in Table 3.3.2.2), reinforcing the conclusion drawn during the Mini-SHINE experiments that Zr has a high affinity for the titania column. All samples showed the presence of the same set of radionuclides with high confidence (low 1σ uncertainty), including ^{237}U, ^{239}Np, ^{103}Ru, ^{133}I, ^{140}Ba/^{140}La, ^{143}Ce, ^{132}Te, ^{105}Rh, and ^{91}Sr. The results indicate that ^{239}Np, ^{143}Ce, and ^{91}Sr are not retained at all, as their activities are approximately the same across the target mixing and all column loading samples. Though ^{140}Ba/^{140}La, ^{127}Sb, ^{105}Rh, ^{132}Te, ^{133}I, ^{131}I, ^{133}Xe, and ^{103}Ru are seen in all the loading samples collected, their activities vary enough that it is likely they are retained by the titania column to some extent.

The recovered column stripping samples were taken at 3.5 and 14 min into the stripping phase of this processing run. It is unclear whether the first recovered sample (CS-2, at 3.5 min) was still composed of the post-load acid wash solution or if it was mostly solution trapped in the sample recovery line from the previous washing, but it effectively contained no activity. The other column stripping sample (CS-6, at 14 min) clearly contained ^{99}Mo, ^{103}Ru, ^{133}Xe, ^{131}I, ^{133}I, ^{97}Zr, ^{135}Xe, ^{127}Sb, and ^{135}I. All remaining isotopes had high 1σ uncertainty or were below the MDA.

TABLE 3.3.2.2 Activities of isotopes in the various samples collected, with 1σ uncertainty, for Irradiation #1. Activities are listed as total mCi in the system

Radionuclide	TM-2	1σ	CL-1	1σ	CL-2	1σ	CL-4	1σ	CS-2	1σ	CS-6	1σ
^{95}Zr	104	2.9%	0.85	58.1%	0.60	84.1%	0.93	81.9%	0.0027	37.5%	0.25	33.0%
^{95}Nb	12	10.0%	0.46	60.3%	0.56	56.2%	0.90	43.6%	0.0019	30.6%	0.043	66.1%
^{237}U	31	12.8%	32	9.3%	29	10.8%	26	14.7%	0.0064	67.8%	1.1	57.8%
^{137}Cs	3.1	14.2%	2.4	28.7%	3.0	23.1%	3.8	23.5%	0.0014	24.3%	0.065	48.9%
^{239}Np	4054	4.6%	4393	4.4%	3909	4.5%	4116	4.6%	0.027	61.3%	2.0	80.8%
^{99}Mo	2391	5.1%	76	40.5%	105	53.5%	85	62.1%	0.062	76.1%	131	5.9%
^{103}Ru	79	3.2%	13.1	5.0%	13	7.1%	43	4.1%	0.0028	31.5%	7.5	3.1%
^{131m}Xe	< 80	MDA	< 41	MDA	40	60.0%	34	90.3%	0.11	83.9%	4.3	61.5%
^{133}Xe	195	12.9%	5.9	100.6%	15	46.4%	16	36.8%	0.0060	102.5%	88	5.8%
^{131}I	276	8.1%	17	40.7%	27	29.7%	44	32.9%	0.014	74.4%	141	2.8%
^{133}I	7317	3.0%	76	7.8%	195	3.6%	227	4.0%	0.058	60.7%	3114	2.6%
^{136}Cs	< 1.1	MDA	< 0.67	MDA	3.2	23.5%	< 0.89	MDA	0.00073	53.2%	0.023	34.3%
^{140}Ba	493	3.1%	306	2.8%	383	2.7%	489	2.7%	0.0041	89.4%	0.33	60.5%
^{140}La	4285	3.2%	1766	3.2%	2230	3.2%	2769	3.2%	0.00055	68.5%	0.097	32.0%
^{143}Ce	4431	2.8%	4591	2.7%	4052	2.7%	4355	2.7%	0.057	41.1%	1.3	83.3%
^{97}Zr	7085	4.7%	80	57.7%	20	25.0%	128	59.3%	< 0.0017	MDA	11	74.8%
^{135}Xe	1619	13.9%	366	42.5%	134	93.3%	230	62.5%	0.18	75.7%	1118	3.7%
^{88}Kr	>12 half-lives		>12 half-lives		>12 half-lives		>12 half-lives		>12 half-lives		>12 half-lives	
^{132}Te	1716	10.3%	537	10.7%	750	10.5%	1379	10.3%	0.015	110.6%	1.5	89.6%
^{97}Nb	>12 half-lives		>12 half-lives		>12 half-lives		>12 half-lives		>12 half-lives		>12 half-lives	
^{105}Rh	944	6.3%	841	5.1%	746	5.5%	854	5.7%	0.058	72.0%	12	42.2%
^{125}Sn	< 5.8	MDA	5.8	75.9%	20	38.8%	8.2	91.6%	0.022	61.9%	2.7	33.3%
^{127}Sb	37.9	17.7%	13	27.1%	11	29.8%	11	28.3%	0.0047	47.2%	4.4	6.7%
^{91}Sr	16682	3.2%	13244	2.4%	11430	2.8%	13037	3.4%	< 0.031	MDA	65	34.3%
^{135}I	16042	16.2%	4521	44.8%	908	92.8%	< 666	MDA	1.1	73.5%	8320	4.1%

3.3.3 Irradiation #2, 11/11/19

This irradiation was the first full-scale irradiation following the gas release that occurred in early 2018. The target solution was not adjusted prior to this irradiation, but it was sampled to capture any dilution/loss that might have occurred during the previous irradiation and processing. Measurements determined that the target solution had a mass of 19.625 kg, concentration of 133.7 g-U/L, and density of 1.17983 g/mL, resulting in a total of 2224 g U in a volume of 16.63 L for this irradiation. To ensure the best odds of retrieving a sample that was representative of the solution after irradiation, all eight target mixing samples were collected regardless of past failures. All eight column loading samples were also collected during processing, but no other processing steps were sampled.

Several issues arose during solution processing for this irradiation. Neither the acid pre-heater nor the column heater worked when activated, meaning that the temperature of the solution being loaded onto the column was much lower than 80°C. The lowest temperature observed at the pre-heater during loading was 19°C, and on the column was 21°C. Further, the pressure switch in the system tripped three times during column loading. The issue was mitigated by pumping for a longer time at lower solution velocity to load the column. This issue was likely due to a combination of residual precipitate in the TSV that was dislodged during the previous irradiation processing and an overly packed column.

Despite the number of samples collected, none could be retrieved at the end of processing (see Table 3.3.3.1). Multiple novel strategies were attempted to retrieve the samples after allowing several days for the radiation field to decrease sufficiently. These included replacing the plastic manifold parts, reversing the direction of the pump to relieve pressure on the valves, and manually forcing the solenoid valves open using positive pressure. Though forcing the valves open worked, it required far too much time in close proximity to the sample ladder right after an irradiation, when the dose rates would be much higher. To ensure that at least one high-quality sample could be retrieved from the target mixing step in the future, an alternative sample loop with manual valves was installed in the acid flow meter bypass path.

TABLE 3.3.3.1 Samples taken and retrieved during processing for the second irradiation

Processing Step	No. Samples Taken	No. Samples Retrieved
Target Solution Mixing Pre-Irradiation	0	0
Target Solution Mixing After Irradiation	8	0
Column Loading	7	0
Post-Load Acid Wash	0	0
Post-Load H_2O Wash	0	0
Column Stripping	0	0
Post-Strip H_2O Wash	0	0

One additional sample was collected from the target solution after it had been transferred back to the TSV and left sitting for several days, to see which fission products remained in the target solution after processing. While pumping solution from the TSV, a large amount of precipitate was seen in the FEP portion of the acid line. As a result, as much precipitate as possible was removed via the same method that was used previously. It is likely that this precipitate is what caused the overpressure during column loading. After removing the precipitate, the target solution was pumped to the dump tank and sampled via the port at valve V-2001 before being returned to the TSV for storage.

Though no samples were recovered from the sample ladders, one was collected from the target solution after all processing had been completed. This sample was referred to as the processed target solution and was analyzed twice by gamma spectroscopy. The mass of target solution analyzed was 98.2 mg. The first analysis counted the sample for 10 min and was used to determine the detector dead time and ensure that no further dilution needed to occur. The second analysis occurred 2.75 hours later, counted the sample for 3.5 hours, and positively identified ^{95}Zr/^{95}Nb, ^{239}Np, ^{103}Ru, ^{132}I, ^{133}I, ^{140}Ba/^{140}La, ^{143}Ce, ^{131m}Xe, ^{132}Te, and ^{147}Nd. The radionuclides ^{237}U, ^{137}Cs, ^{131}I, ^{99m}Tc and ^{105}Rh were also detected with higher uncertainties. The detection of ^{132}Te indicates that ^{132}I was present at one time, and ^{99m}Tc indicates that small amounts of ^{99}Mo were likely present, but at quantities undetectable when using the lower-branching-ratio emission. The remaining radionuclide activities were below their respective MDAs, as indicated in Table 3.3.3.2.

TABLE 3.3.3.2 Activities of isotopes in the processed target solution sample, with 1σ uncertainty, for Irradiation #2. Activities are listed as total mCi in the system.

Radionuclide	Processed Target Solution	1σ	Radionuclide	Processed Target Solution	1σ
^{95}Zr	74	3.4%	^{97}Zr	>12 half-lives	
^{95}Nb	21	5.7%	^{92}Sr	>12 half-lives	
^{237}U7	71	27.0%	^{99m}Tc	180	11.1%
^{156}Eu	< 34	MDA	^{131m}Xe	4246	6.0%
^{137}Cs	5.9	21.7%	^{135}Xe	>12 half-lives	
^{239}Np	9658	4.0%	^{133m}Xe	< 499	MDA
^{99}Mo	< 543	MDA	^{132}Te	3284	7.4%
^{103}Ru	218	2.5%	^{97}Nb	>12 half-lives	
^{132}I	3010	4.2%	^{105}Rh	3192	13.3%
^{131m}Te	< 1382	MDA	^{125}Sn	< 80	MDA
^{131}I	222	18.5%	^{127}Sb	< 27	MDA
^{133}I	40478	4.9%	^{91}Sr	>12 half-lives	
^{136}Cs	< 3.0	MDA	^{147}Nd	490	5.1%
^{140}Ba	1115	7.5%	^{151}Pm	< 1944	MDA
^{156}Sm	>12 half-lives		^{93}Y	>12 half-lives	
^{140}La	1174	2.2%	^{135}I	>12 half-lives	
^{143}Ce	9990	4.3%			

3.3.4 Irradiation #3, 3/2/20

Prior to this irradiation, LEAF-PROC-024 was modified to reflect the addition of the alternate sample loop. In addition, the relays associated with all the system heaters were replaced along with the pressure transducers to ensure they would work, since most had malfunctioned during the previous irradiation processing due to radiation damage. To ensure that the dose rate would not be excessively high while retrieving the sample from the alternate loop, none of the original sample loops in the sample ladders were used. After adjustment and the addition of stable Mo carrier, the target solution had a mass of 20.31 kg, concentration of 140.3 g-U/L, and density of 1.19800 g/mL, resulting in a total of 2378 g U in a volume of 16.95 L for this irradiation.

This irradiation was cut short because of a malfunction in the accelerator. The target solution was mixed, but it was determined that the amount of ^{99}Mo produced was not sufficient to ship, so the solution was returned to the TSV to monitor hydrogen accumulation while the solution was stored until the next irradiation. Because the processing was aborted during mixing, no samples were taken as part of this irradiation.

3.3.5 Irradiation #4, 8/30/20

The target solution was not adjusted prior to this irradiation, but it was sampled to capture any dilution/loss that might have occurred during the aborted irradiation and mixing from irradiation #3. Measurements determined that the target solution had a mass of 20.37 kg, concentration of 140.18 g-U/L, and density of 1.19802 g/mL, resulting in a total of 2383 g U in a volume of 17.00 L for this irradiation. As was intended for the previous irradiation, the only sample taken utilized the alternate sample loop. This was the first irradiation where samples were taken from the various effluent bottles to determine which fractions contained which fission products.

The glovebox processing run for this irradiation was carried out without incident or malfunction. After irradiation, the target solution was mixed and loaded onto the column; then molybdenum and uranium were eluted from the column in separate fractions. The ^{99}Mo fraction was sent to and received in the D024 Hot Cell.

For this irradiation, one sample, which was representative of the irradiated and mixed target solution, was retrieved from the alternate sample loop for gamma spectroscopy. This sample was serially diluted until the detector dead-time was approximately 5% when counting at the furthest available distance (100 cm), which resulted in counting a net mass of 112 mg of the original solution. The target mixing sample was counted three times, for 15.5, 86, and 63.5 hours, with these analyses occurring 4, 5, and 12 days after irradiation, respectively. Table 3.3.5.1 shows the average activities of the various nuclides and their 1σ uncertainties calculated in the entire target solution volume from the three replicate analyses. Owing to the delay in counting, ^{92}Sr, ^{97}Nb, and ^{135}I had undergone > 12 half-lives of decay prior to the first analysis and could not be quantified. The radionuclides ^{156}Eu, ^{136}Cs, ^{156}Sm, ^{135}Xe, ^{133m}Xe, and

^{125}Sn were all below the MDA for all analyses as well. The analysis indicates that roughly 3.5 Ci of ^{99}Mo was produced during the irradiation.

TABLE 3.3.5.1 Activities of radionuclides in the target mixing sample, with 1σ uncertainty, for Irradiation #4. Activities are listed as total mCi in the irradiated target solution.

Radionuclide	Target Mixing	1σ	Radionuclide	Target Mixing	1σ
^{95}Zr	181	3.4%	^{97}Zr	7940	4.6%
^{95}Nb	108	3.4%	^{92}Sr	>12 Half-lives	
^{237}U	28	13.5%	^{99m}Tc	3310	6.1%
^{156}Eu	8.4	MDA	^{131m}Xe	715	10.2%
^{137}Cs	11	8.8%	^{135}Xe	< 3000	MDA
^{239}Np	5760	5.8%	^{133m}Xe	< 160	MDA
^{99}Mo	3500	5.1%	^{132}Te	2758	3.6%
^{103}Ru	172	3.7%	^{97}Nb	>12 Half-lives	
^{132}I	2130	6.0%	^{105}Rh	18400	9.1%
^{131m}Te	3530	20.6%	^{125}Sn	< 18	MDA
^{131}I	481	5.1%	^{127}Sb	61.0	14.8%
^{133}I	11650	3.0%	^{91}Sr	51000	19.1%
^{136}Cs	< 1.0	MDA	^{147}Nd	221	7.1%
^{140}Ba	797	3.7%	^{151}Pm	2230	13.7%
^{156}Sm	< 5300	MDA	^{93}Y	315000	15.4%
^{140}La	916	4.1%	^{135}I	>12 Half-lives	
^{143}Ce	6490	7.4%			

The various effluent bottles were also sampled after irradiation to determine which radionuclides could be associated with which waste streams. Generally, each sample was counted for a very short time (~ 2 min) to determine detector dead time and whether any further dilution was required. This was followed by a longer analysis that ranged from 30 min to 16 h, dependent on the sample activity. The results from the longer analyses are found in Table 3.3.5.2. The long count for the sample from the post-load water wash bottle was performed on 9/10/20, while the base rinse was counted on 9/14/20, the acid rinse was counted on 9/16/20, and the remaining samples were counted on 9/15/20. Because of the 10+ day delay before analyses were undertaken, several radionuclides had undergone > 12 half-lives of decay and could not be quantified. These include ^{133}I, ^{156}Sm, ^{97}Zr/^{97}Nb, ^{92}Sr, ^{135}Xe, ^{91}Sr, ^{93}Y, and ^{135}I, which have been omitted from Table 3.3.5.1 for brevity. Note also that all activities in Table 3.3.5.2 are in ***μCi***, while all previous tables have been in mCi. This smaller unit was used because the effluent bottle activities were much lower than the activities of samples from the processing stream.

TABLE 3.3.5.2 Average activities of radionuclides in the effluent bottle samples, with 1σ uncertainty, for Irradiation #4. Activities are listed as total μCi in the given effluent bottle.

Radionuclide	Pre-Load Acid Wash	1σ	Post-Load Acid Wash	1σ	Post-Load Water Wash	1σ	Post-Strip Water Wash	1σ	Acid Rinse	1σ	Base Rinse	1σ
^{95}Zr	< 37	MDA	< 42	MDA	41	14.1%	118	4.4%	69	2.0%	0.31	14.4%
^{95}Nb	< 24	MDA	< 27	MDA	22	11.3%	45	6.9%	55	2.0%	0.41	6.7%
^{237}U	< 480	MDA	< 631	MDA	< 80	MDA	< 152	MDA	< 2.3	MDA	< 1.7	MDA
^{156}Eu	< 469	MDA	< 530	MDA	< 61	MDA	< 52	MDA	< 1.4	MDA	< 1.0	MDA
^{137}Cs	60	16.6%	48	25.3%	< 9	MDA	7.0	26.4%	6.2	3.4%	0.64	4.9%
^{239}Np	73000	14.3%	53000	25.0%	2418	24.8%	< 4582	MDA	< 93	MDA	< 42	MDA
^{99}Mo	< 17840	MDA	< 24012	MDA	< 1420	MDA	14593	28.7%	< 109	MDA	696	4.7%
^{103}Ru	1012	3.3%	1504	3.4%	94	6.7%	3460	2.2%	23	2.3%	38	2.0%
^{132}I	15900	12.3%	67200	6.0%	10029	4.9%	< 582	MDA	98	17.0%	24	18.8%
^{131m}Te	>12 half-lives		>12 half-lives		< 10294	MDA	>12 half-lives		>12 half-lives		4170	21.1%
^{131}I	< 906	MDA	< 1276	MDA	466	24.6%	7714	3.0%	14	21.5%	121	2.5%
^{136}Cs	< 61	MDA	< 67	MDA	< 6	MDA	< 5	MDA	< 0.16	MDA	< 0.091	MDA
^{140}Ba	5970	3.3%	4730	5.1%	184	16.3%	< 30	MDA	28	3.6%	8.5	4.3%
^{140}La	7060	2.6%	5320	2.8%	146	4.7%	< 5	MDA	36	2.5%	10	2.5%
^{143}Ce	< 132558	MDA	< 174638	MDA	< 2881	MDA	< 43953	MDA	>12 half-lives		< 350.9	MDA
^{99m}Tc	< 1299	MDA	< 1695	MDA	< 96	MDA	16065	4.2%	33	11.6%	519	3.5%
^{131m}Xe	23800	9.1%	17060	15.9%	< 521	MDA	< 1525	MDA	48	12.2%	30	22.2%
^{133m}Xe	< 43559	MDA	< 31017	MDA	< 1706	MDA	< 9063	MDA	< 146	MDA	< 117	MDA
^{132}Te	18090	4.2%	68039	2.5%	10444	2.3%	< 217	MDA	110	3.6%	27	6.1%
^{105}Rh	14110000	24.6%	3660020	18.1%	< 16995	MDA	< 212223	MDA	30289	13.5%	< 1733	MDA
^{125}Sn	< 1284	MDA	< 1582	MDA	< 158	MDA	< 169	MDA	< 3.7	MDA	< 2.4	MDA
^{127}Sb	< 1240	MDA	< 1838	MDA	< 170	MDA	2348	11.8%	< 7.1	MDA	78	4.7%
^{147}Nd	2080	6.9%	585	21.7%	< 27	MDA	< 38	MDA	6.1	7.8%	< 0.63	MDA
^{151}Pm	>12 half-lives		>12 half-lives		< 13319	MDA	>12 half-lives		>12 half-lives		>12 half-lives	

Generally, it was found that ^{95}Zr/^{95}Nb were only present in samples from effluent bottles following the rinsing of the column with water, in line with the conclusion that Zr is well retained by the titania column under acidic conditions. Also found only in the water rinse samples was ^{127}Sb, indicating that it too was retained under acidic conditions. The radionuclides ^{103}Ru, ^{132}Te/^{132}I, ^{137}Cs, and ^{140}Ba/^{140}La were either found in all effluent bottles or the MDA reported was higher than the activities in the various other samples. It was observed that ^{147}Nd and ^{105}Rh were only found in acid process effluent bottles, indicating that they are not retained by the column at all. The post-strip water wash is the only effluent to contain significant amounts of ^{99}Mo/^{99m}Tc, though some is also present in the base rinse sample. This is unsurprising, since a small portion of the strip solution is directed towards the post-strip water wash during processing, and this step is followed immediately by one in which the lines are flushed out to the base rinse bottle.

3.3.6 Irradiation #5, 1/18/21

After adjustment and the addition of stable Mo carrier, the target solution had a mass of 21.18 kg, concentration of 144.8 g-U/L, and density of 1.20863 g/mL, resulting in a total of 2537 g U in a volume of 17.52 L for this irradiation. As with the previous irradiation, the only sample taken during processing utilized the alternate sample loop. One sample was also retrieved from each of the effluent bottles. An additional sample of the target solution was retrieved using valve V-2001 so that a comparison could be made between the freshly irradiated target solution and the processed target solution. The glovebox processing run for this irradiation was carried out without incident or malfunction. After irradiation, the target solution was mixed and loaded onto the column; then molybdenum and uranium were eluted from the column in separate fractions. The ^{99}Mo fraction was sent to and received in the D024 Hot Cell.

The target mixing sample was counted for 2, 4, and 64 h, while the post-processing sample was counted for 1 and 18 h. After a short count to determine dead time, effluent bottle samples were counted at least once for 1 h, with low-activity samples recounted for longer times to improve counting statistics.

Unfortunately, delays in sample preparation meant that none of the samples could be analyzed until 10 days after the irradiation. As a result, 10 of the nuclides of interest (^{133}I, ^{131m}Te, ^{156}Sm, ^{97}Zr/^{97}Nb, ^{92}Sr, ^{135}Xe, ^{91}Sr, ^{93}Y, and ^{135}I) had decayed for > 12 half-lives and did not provide usable data for any of the collected samples. For brevity, these have been removed from Table 3.3.6.1 and Table 3.3.6.2, below. Analysis indicates that roughly 5.8 Ci of ^{99}Mo was created during the irradiation, though nearly 1 Ci of that was still in the target solution after processing. Regardless of how much was left in solution, 5.8 Ci is far below the intended 10-Ci production run, and it is unclear why this occurred.

TABLE 3.3.6.1 Average activities of the various radionuclides in the irradiated target and processed target solutions, with 1σ uncertainty, for Irradiation #5. Activities are mCi present in the total target solution, which had roughly the same mass before and after irradiation.

Radionuclide	Irradiated Target Average	1σ	Post-Processing Average	1σ
^{95}Zr	293	3.8%	53	3.0%
^{95}Nb	99	4.8%	20	3.4%
^{237}U	104	17.5%	49	8.9%
^{156}Eu	< 29	MDA	< 5	MDA
^{137}Cs	9.0	12.8%	2.6	8.0%
^{239}Np	9342	7.6%	4727	5.4%
^{99}Mo	5787	11.7%	1037	20.1%
^{103}Ru	203	3.8%	51	2.8%
^{132}I	3253	6.0%	1277	5.5%
^{131}I	957	3.4%	138	3.6%
^{136}Cs	1.1	11.6%	0.7	11.8%
^{140}Ba	1308	4.4%	447	2.9%
^{140}La	84	2.5%	27	7.9%
^{143}Ce	9697	22.3%	> 12 half-lives	
^{99m}Tc	6106	6.3%	1138	5.3%
^{131m}Xe	82	9.9%	34	14.4%
^{133m}Xe	< 480	MDA	< 380	MDA
^{132}Te	7993	11.8%	3081	13.7%
^{105}Rh	11549	33.5%	17910	17.0%
^{125}Sn	< 104	MDA	< 19	MDA
^{127}Sb	203	16.4%	61	17.6%
^{147}Nd	507	7.2%	255	5.5%

When retrieving samples from the effluent bottles, it was elected to not collect samples from the acid rinse, pre-load acid wash, and base rinse bottles, since these effectively contain the same nuclides as the post-processing, post-load acid wash, and post-strip water wash bottles, respectively. Unfortunately, the post-strip water wash bottle did not have enough solution to sample, so no representative sample of the base side of the system was recovered. The radionuclide activities of the remaining post-load acid wash and post-load water wash effluent bottles are shown in Table 3.3.6.2. Though the activity was lower, the results obtained for the post-load acid and post-load water washes are similar to those obtained after the previous irradiation, reinforcing the conclusions stated above.

TABLE 3.3.6.2 Activities of radionuclides found in the effluent bottle samples, with 1σ uncertainty, for Irradiation #5. Activities are listed as total μCi in the given effluent bottle.

Radionuclide	Post-Load Acid Wash	1σ	Post-Load Water Wash	1σ
^{95}Zr	< 14	MDA	< 5.7	MDA
^{95}Nb	< 10	MDA	< 3.0	MDA
^{237}U	< 250	MDA	< 80	MDA
^{156}Eu	< 209	MDA	< 51	MDA
^{137}Cs	19	15.0%	< 3.7	MDA
^{239}Np	32475	25.7%	< 2960	MDA
^{99}Mo	< 14876	MDA	< 3615	MDA
^{103}Ru	630	28.1%	124	3.5%
^{132}I	78114	3.5%	34833	3.6%
^{131}I	2320	9.1%	1663	6.7%
^{136}Cs	< 22	MDA	< 6	MDA
^{140}Ba	5474	10.5%	< 300	MDA
^{140}La	179	25.7%	< 25	MDA
^{99m}Tc	< 1115	MDA	< 264	MDA
^{131m}Xe	< 1526	MDA	< 472	MDA
^{133m}Xe	< 29315	MDA	< 6580	MDA
^{132}Te	360480	12.5%	101714	9.4%
^{105}Rh	235389	25.2%	< 55817	MDA
^{125}Sn	< 529	MDA	< 91	MDA
^{127}Sb	< 715	MDA	< 170	MDA
^{147}Nd	900	9.1%	51	25.2%

3.4 HOT CELL PURIFICATION PROCESS

3.4.1 Concentration Column

3.4.1.1 Commissioning run (8/4/19)

The solution from the recovery glovebox was transferred via a transfer line to the D-024 hot cell for volume reduction and further purification using the D-024 hot-cell operations procedure. After the concentration-column operation, the LMC process was performed to produce the final ^{99}Mo product. ^{99}Mo samples were collected at a variety of process steps and analyzed.

The concentration-column procedure was performed by following LEAF-PROC-011 (Appendix 27). Minor adjustments were made to the procedure following this experiment to streamline the process and to allow for the researchers to ensure that the operation was completed within the scope of the procedure. During the commissioning run, a thermocouple failed during pre-checks, and, therefore, that particular heating element was not used during this process. The thermocouple was investigated, and a loose wire was found at the connection inside the D-024 hot cell. This wire was repaired before the next experiment.

The solution-transfer and system-interface steps were performed as expected. The concentration column performed as expected. ^{99}Mo was successfully loaded on the column; the column was washed; and ^{99}Mo was eluted for final processing by the LMC process. Tables 3.4.1.1.1 and 3.4.1.1.2 summarize the results for samples from the concentration-column process.

TABLE 3.4.1.1.1 Activities detected in the concentration-column fractions, decay corrected to the addition of the spike solution during the commissioning run

8/14/19 Commissioning Run

Radionuclide	Feed, mCi (1s, %)	HNO_3 Wash, mCi (1s, %)	H_2O Wash, mCi (1s, %)	Waste, mCi (1s, %)	^{99}Mo Product, mCi (1s, %)
^{99}Mo	43.8 (2)	0.05 (1.2)	0.074 (7.4)	0.33 (0.1)	16.4 (4.6)

TABLE 3.4.1.1.2 Relative distribution of ^{99}Mo in various fractions of the concentration column during the commissioning run

8/14/19 Commissioning Run

Radionuclide	HNO_3 Wash	H_2O Wash	Waste	Product	Total Recovery
^{99}Mo	0.1%	0.2%	0.8%	37.4%	38.5%

Because of the low recovery of ^{99}Mo product from the concentration column during the commissioning run, the elution volume and time were increased in the procedure for subsequent experiments.

3.4.1.2 Irradiation #1 (10/01/19)

During the processing after the first irradiation, the concentration column ran smoothly, with no issues observed. The increase of the elution volume and time led to ^{99}Mo recovery greater than 100% from the concentration column. This result was also observed during the Phase I experiments and is attributed to an inaccurate reading from the balance determining the final weight of the feed solution delivered from the glovebox team. The balance was inaccurate for several reasons, including the following: the floor of the D-024 hot cell is not completely level, and the 3-L flask has multiple liquid transfer lines, gas collection lines, and pH probes connected to it. Owing to these connections, slight shifts of equipment or lines can impact the mass reading of the received solution. However, the concentration column still performed as expected and delivered a high recovery yield during this experiment.

Using gamma spectroscopy, we tracked the ^{99}Mo product as well as fission-produced radionuclides through the process. These data showed that major fission radionuclides travel with the product through the concentration-column process. Ruthenium-103, ^{131}I, and ^{133}I had approximately 25% of their initial activity carried with the product, while ^{95}Zr, ^{105}Rh and ^{127}Sb all had values above 50%. This indicates that there was retention of these radionuclides on the column. We believe that the remainder of the activity was carried through in the solution effluent, since it was not detected in any of the other samples. It was not possible to sample the effluent during this experiment to verify this conclusion.

The calculations of the relative distribution of isotopes in Tables 3.4.1.2.1 and 3.4.1.2.2 used the initial feed sample as the means to standardize the samples.

TABLE 3.4.1.2.1 Activities detected in the concentration column fractions, decay corrected to EOB during the first irradiation

10/1/19 Irradiation #1

Radionuclide	Feed, mCi (1s, %)	HNO_3 Wash, mCi (1s, %)	H_2O Wash, mCi (1s, %)	Waste, mCi (1s, %)	^{99}Mo Product, mCi (1s, %)
^{95}Zr	2.41E-01 (20.1)	1.76E-04 (44.2)	2.46E-04 (54.9)	2.35E-03 (29.6)	1.72E-01 (24.1)
^{99}Mo	2.08E+03 (2.2)	6.16E-01 (4.6)	2.01E-02 (71.5)	1.04E+01 (2.3)	2.24E+03 (2.1)
^{103}Ru	6.85E+00 (2.8)	7.54E-02 (2.1)	2.58E-02 (2.2)	8.09E-02 (2.4)	1.71E+00 (3.9)
^{131}I	1.02E+02 (2.3)	2.12E+00 (1.8)	1.24E+00 (1.9)	2.19E+00 (1.9)	2.82E+01 (2.7)
^{133}I	2.40E+03 (2.4)	4.77E+01 (1.9)	2.82E+01 (1.9)	4.93E+01 (1.9)	6.21E+02 (2.1)
^{105}Rh	2.18E+01 (35.9)	3.06E-01 (15.4)	1.53E-01 (26.9)	1.19E-01 (40.7)	1.19E+01 (26.8)
^{127}Sb	1.41E+00 (18.9)	1.06E-03 (54.9)	8.54E-04 (48.4)	8.51E-02 (4.0)	2.21E+00 (10.0)

TABLE 3.4.1.2.2 Distribution of activity of various isotopes in the fractions collected from the concentration column during the first irradiation

10/01/19 Irradiation #1

Radionuclide	HNO_3 Wash	H_2O Wash	Waste	^{99}Mo Product	Total Recovery
^{95}Zr	0.1%	0.1%	1.0%	71.3%	72.4%
^{99}Mo	0.0%	0.0%	0.5%	107.8%	108.3%
^{103}Ru	1.1%	0.4%	1.2%	25.0%	27.7%
^{131}I	2.1%	1.2%	2.1%	27.5%	33.0%
^{133}I	2.0%	1.2%	2.1%	25.9%	31.1%
^{105}Rh	1.4%	0.7%	0.5%	54.8%	57.4%
^{127}Sb	0.1%	0.1%	6.0%	156.7%	162.9%

3.4.1.3 Irradiation #2 (11/11/19)

The processing after the second irradiation was performed, and no issues were observed during the concentration-column operation. While processing the solution, it was observed that the solution received from the primary recovery glovebox was cloudy. During Phase I experiments, a cloudy solution typically resulted in sub-optimal recovery from the concentration column. During acidification, the precipitate dissolved, but the concentration-column recovery of the ^{99}Mo product suffered, with only 65% recovery. In an attempt to avoid poor recovery, the collection time for the product was increased to collect more volume during this step. Analysis of the collected fractions showed a lower recovery of the ^{99}Mo product than in the previous experiment, and the iodine isotopes showed less retention on the column and a lower overall recovery as well. Conversely, nearly all ^{95}Zr, ^{103}Ru, ^{105}Rh and ^{127}Sb were recovered in the ^{99}Mo product during this experiment. Many of the percent recoveries were similar to the ^{99}Mo recovery, but when the recovery was above 100%, we attribute this result to either an inaccurate weight of the initial feed solution or incomplete mixing of the sample.

Once the concentration column was loaded and eluted, dose rates were taken from the 5-L storage vessels underneath the D-024 hot cell. The shielding in place for this experiment was shown to be adequate, but additional shielding was needed before the full irradiation experiment. To achieve ALARA and to maintain a safe working environment, it was decided to install an effluent holding bottle inside the D-024 hot cell. This bottle was designed to hold the solution during its initial decay before being transferred underneath the hot cell for long-term storage. Installation of the effluent bottle inside the hot cell also allowed for sampling of the effluent solution during subsequent experiments. This sampling was made possible by use of a three-way valve that was connected to a line that went to the bottom of the holding bottle. An empty syringe was attached to the sampling line on the three-way valve and the valve was turned from receiving to sampling. The syringe was then filled and the sample solution was transferred to a scintillation vial. Tables 3.4.1.3.1 and 3.4.1.3.2 summarize the results for samples from the concentration-column process.

TABLE 3.4.1.3.1 Activities detected in the concentration-column fractions, decay corrected to EOB during the second irradiation

11/11/19 Irradiation #2

Radionuclide	Feed, mCi (1s, %)	HNO_3 Wash, mCi (1s, %)	H_2O Wash, mCi (1s, %)	Waste, mCi (1s, %)	^{99}Mo Product, mCi (1s, %)
^{95}Zr	2.79E+00 (21.5)	2.48E-03	7.18E-04	7.86E-03 (18.0)	1.81E+00 (6.1)
^{99}Mo	9.74E+03 (2.1)	2.95E-01	1.02E-01	4.90E+00 (5.5)	6.16E+03 (2.2)
^{103}Ru	4.04E+00 (18.7)	1.80E-02 (6.7)	4.08E-03 (8.8)	3.88E-03	1.66E+00 (6.5)
^{131}I	7.17E+02 (3.0)	2.70E+00 (2.6)	1.62E+00 (2.2)	5.11E+00 (2.4)	1.59E+01 (12.2)
^{133}I	1.39E+04 (2.1)	6.67E+01 (2.1)	4.20E+01 (1.9)	1.24E+02 (2.1)	4.85E+02 (11.1)
^{105}Rh	8.97E+01	3.88E-01	2.62E-01 (25.3)	6.24E-01	2.88E+01
^{127}Sb	6.45E+00 (21.7)	1.03E-02	3.23E-03	5.73E-02	7.83E+00 (9.6)

TABLE 3.4.1.3.2 Distribution of activity of various isotopes in the fractions collected from the concentration column during the second irradiation

11/11/19 Irradiation #2

Radionuclide	HNO_3 Wash	H_2O Wash	Waste	^{99}Mo Product	Total Recovery
^{95}Zr	0.1%	0.0%	0.3%	65.1%	65.5%
^{99}Mo	0.0%	0.0%	0.1%	63.2%	63.3%
^{103}Ru	0.4%	0.1%	0.1%	41.2%	41.8%
^{131}I	0.4%	0.2%	0.7%	2.2%	3.5%
^{133}I	0.5%	0.3%	0.9%	3.5%	5.2%
^{105}Rh	0.4%	0.3%	0.7%	32.1%	33.5%
^{127}Sb	0.2%	0.1%	0.9%	121.5%	122.6%

3.4.1.4 Irradiation #3 (03/02/20)

The third irradiation did not have any chemical processing associated with it, so there are no additional concentration column data from that experiment.

3.4.1.5 Irradiation #4 (08/30/20)

The fourth irradiation performed was planned to be a complete run with full chemical processing. The steps leading up to the concentration column operated as expected, but during the water wash of the concentration column, a leak was detected, coming from behind the valve board. The experiment was paused to determine the cause of the leak. The source of the leak was not visible, since it was located behind the board. Also, an elevated dose rate was measured on the silver zeolite filter that was connected to the hot-cell exhaust. To avoid further contaminating the inside of the hot cell and increasing the size of the spill, the fourth experiment was stopped. For this reason, samples for gamma spectroscopy are incomplete in Table 3.4.1.5.1.

TABLE 3.4.1.5.1 Activities detected in the concentration column fractions, decay corrected to EOB during the fourth irradiation

8/30/20 Irradiation #4

Radionuclide	Feed, mCi (1s, %)	Eluent, mCi (1s, %)	HNO_3 Wash, mCi (1s, %)	H_2O Wash, mCi (1s, %)	Waste, mCi (1s, %)	^{99}Mo Product, mCi (1s, %)
^{95}Zr	1.06E+00 (13.0)	3.40E-03	2.76E-04			
^{99}Mo	4.06E+03 (0.7)	2.94E+02	4.51E+00			
^{103}Ru	6.62E+01 (0.2)	4.72E+01 (0.1)	1.17E+00			
^{131}I	4.43E+02 (3.7)	7.03E+01 (0.3)	5.59E+00			
^{133}I						
^{105}Rh						
^{127}Sb	2.93E+01	1.15E+00	9.08E-02			

A modified procedure for eluting the concentration column and transferring the solution to the 5-L holding vessels below the hot cell was prepared and presented to a safety review committee. The procedure was approved and followed. The concentration column was eluted and all lines were rinsed into the effluent holding container inside the hot cell. The resulting solution was then transferred to the 5-L holding containers below the D-024 hot cell. These efforts sufficiently reduced the radiation field inside the hot cell for a manned entry to begin troubleshooting and repairs. The inside of the D-024 hot cell was also decontaminated by manipulators with paper towels and water. Smears were taken and counted after the cleaning process until the contamination levels inside the hot cell were low enough for entry.

During the hot-cell troubleshooting and repairs, the water line connected to a multi-way valve was found to be damaged. The line that leaked had developed a spiral crack as a result of embrittlement from a combination of time and the heat and radioactivity applied to it since its installation years earlier. As a corrective action, all lines inside the hot cell were inspected and replaced. A provision was also put into the procedure for replacement of all lines in the system at least every two years, to avoid similar complications in the future.

The gamma results from the acid wash were typical and displayed minimal amounts of activity. It was exciting to finally have results from an effluent sample. The results showed some breakthrough of the ^{99}Mo at 7.2%, as well as low levels of ^{95}Zr and ^{127}Sb. The ^{131}I in the sample accounted for 16% of the ^{131}I activity when compared to the feed sample. This finding helped confirm our theory that the iodine isotopes are present through the entire processing. The ^{103}Ru had just over 70% of its activity accounted for in this sample, which means that the majority of this radionuclide is not retained on a titania column and passes with the effluent. The abbreviated results from this experiment can be found in Table 3.4.1.5.2. It should be noted that because of the contamination inside the hot cell and the commensurate corrective actions, these samples were not retrieved and counted in a timely manner, so much of the data was lost to decay during this period.

TABLE 3.4.1.5.2 Distribution of activity of various isotopes in the fractions collected from the concentration column during the fourth irradiation

8/30/20 Irradiation #4

Radionuclide	Eluent	HNO_3 Wash	Total Recovery
^{95}Zr	0.3%	0.0%	0.3%
^{99}Mo	7.2%	0.1%	7.3%
^{103}Ru	71.3%	1.8%	73.0%
^{131}I	15.9%	1.3%	17.1%
^{127}Sb	3.9%	0.3%	4.2%

3.4.1.6 Irradiation #5 (01/18/21)

The final experiment was performed, and the concentration-column operations ran smoothly. No issues were observed during the experiment. Gamma spectroscopy data showed that approximately 9 Ci of ^{99}Mo were received from the primary recovery glovebox in the 3-L flask. From the 9 Ci, only about 2 Ci of ^{99}Mo was recovered, which was less than 25% of the product. The samples from the rinse solutions showed little activity and the effluent sample only contained 5% of the total product. It is worth noting that nearly all the ^{103}Ru, ^{131}I, and ^{105}Rh were observed to be in the eluent sample. The percentages of these radionuclides recovered from the product sample also match closely with what was observed during Irradiation #1. The ^{95}Zr, ^{105}Rh and ^{127}Sb all had lower levels of activity detected in the product sample, and while nearly all the ^{105}Rh was accounted for with the eluent sample, the ^{95}Zr and ^{127}Sb had poor overall recoveries, with 44% and 15%, respectively. Tables 3.4.1.6.1 and 3.4.1.6.2 also show an extremely low recovery of ^{133}I. The likely reason was overlapping gamma lines, which resulted in inaccurate results for this radionuclide; it should be assumed that the ^{133}I behaves the same as the ^{131}I.

TABLE 3.4.1.6.1 Activities detected in the concentration column fractions, decay corrected to EOB during the fifth irradiation

1/18/21 Irradiation #5

Radionuclide	Feed, mCi (1s, %)	Eluent, mCi (1s, %)	HNO_3 Wash, mCi (1s, %)	H_2O Wash, mCi (1s, %)	^{99}Mo Product, mCi (1s, %)
^{95}Zr	5.00E-01 (5.1)	1.31E-01 (3.4)	9.56E-04	3.14E-04	8.57E-02 (16.2)
^{99}Mo	8.92E+03 (2.8)	4.80E+02 (3.0)	4.43E-01 (18.2)	9.83E-01	2.05E+03 (2.7)
^{103}Ru	6.43E+01 (2.8)	6.26E+01 (2.1)	7.88E-01 (2.2)	5.94E-01 (2.2)	1.64E+01 (2.1)
^{131}I	2.49E+02 (7.5)	3.15E+02 (1.9)	2.83E+00 (2.2)	4.34E+00 (2.1)	5.60E+01 (2.3)
^{133}I	9.32E+03 (1.9)		5.08E+01 (2.6)		5.36E+02 (2.5)
^{105}Rh	1.54E+02 (10.2)	2.33E+02	1.20E+00 (15.3)	1.01E+01	
^{127}Sb	1.26E+00	1.76E-01	4.58E-03	7.35E-03	

TABLE 3.4.1.6.2 Distribution of activity of various isotopes in the fractions collected from the concentration column during the fifth irradiation

1/18/21 Irradiation #5

Radionuclide	Eluent	HNO_3 Wash	H_2O Wash	^{99}Mo Product	Total Recovery
^{95}Zr	26.3%	0.2%	0.1%	17.1%	43.7%
^{99}Mo	5.4%	0.0%	0.0%	23.0%	28.4%
^{103}Ru	97.4%	1.2%	0.9%	25.5%	125.0%
^{131}I	126.5%	1.1%	1.7%	22.5%	151.8%
^{133}I	0.0%	0.5%	0.0%	5.7%	6.3%
^{105}Rh	150.7%	0.8%	6.5%	0.0%	158.0%
^{127}Sb	14.0%	0.4%	0.6%	0.0%	14.9%

A second elution was performed on the concentration column with 50 mL of 1 M NaOH, but no additional ^{99}Mo product was removed during the second elution. While we cannot definitively say what the problem was with the concentration column, we suggest one of the following possibilities: First, the column could have formed channels between the times of packing and the concentration column operations, which would lead to inefficient elution and loss of product. There could also have been a sampling error during the process, where either a more concentrated sample was taken from the initial feed solution or a more dilute sample was taken from the ^{99}Mo product sample. Incomplete mixing of the sampled solutions would cause this problem, and inhomogeneity of the solutions would greatly skew the results. There is also the possibility that the ^{99}Mo was fixed to the concentration column and unable to be eluted.

Each of the concentration-column operations during the irradiations was unique and presented its own challenges. A leak led to incomplete data during the fourth experiment. The eluent solution was unable to be sampled and analyzed until the fourth and fifth irradiation, so there were only two data points. Because of the wide spread of data and incomplete data points, it was difficult to find any overall trends from these experiments. For the first few experiments, we can speculate that the remainder of the activity was present in the eluent solution, but we do not have verified results and we have completed only one full experiment with the eluent sample present; therefore, it is not possible to draw any meaningful conclusions from these data. We are confident that the column washes do not remove significant amounts of activity. The product sample typically had at least a quarter of the other major radionuclides eluted with it, which illustrates retention of other nuclides on the titania column and underlines the fact that the modified Cintichem process is essential to purifying the ^{99}Mo product.

3.4.2 LEU Modified Cintichem Process

3.4.2.1 Commissioning Run 08/04/19

The LMC process was performed by following LEAF-PROC-011 (Appendix 27). However, the ruthenium and rhodium carriers were not used during this operation because they were not present in the ^{99}Mo spike solution. At the beginning of LMC operations, it was determined that the vacuum pump could not pull the Mo solution through a 0.3-µm filter during the iodine precipitation step. This issue was corrected by pre-wetting the filter inside the hot cell and reinstalling the filter into the filtration assembly. Gamma-counting results for LMC processing are shown in Table 3.4.2.1.1. The Mo-containing feed to the LMC process is designated RF-1; RF-2 is the solution after iodine precipitation; RFW is the filtrate from the ABO precipitation step; and 1-B is the Mo product following purification. During the filtration of Mo-ABO precipitate, delayed formation of precipitate was observed in the RFW bottle. This delay is likely due to the limited solubility of excess ABO under acidic conditions, which causes precipitation of ABO. On the basis of the results shown in Table 3.4.2.1.1, Mo losses in the RFW bottle represent only 0.12% of the total ^{99}Mo activity, confirming the fact that precipitate formed in the RFW bottle did not contain a significant amount of Mo. Recovery of ^{99}Mo from the LMC process was 93.6%, which is very good.

TABLE 3.4.2.1.1 Activities detected in LMC fractions

Sample	Average, mCi	1σ, mCi	1σ, %
RF-1	16.2	0.737	2.0%
RFW	0.0201	0.00079	2.0%
1-B	15.1	0.808	2.0%

3.4.2.2 Irradiation #1 (10/01/19)

The LMC operations after this short irradiation went as planned, without any issues. Photographs in Figure 3.4.2.2.1 show the major separation steps: a) initial iodine precipitation; b) precipitation and filtration of Mo-ABO precipitate; c) dissolution of Mo-ABO precipitate with heating using a heat gun; and d) final purification using iodine precipitation and the combination column (Ag/C, HZO, AC).

Major contaminants found entering the LMC process from the concentration column were ^{103}Ru, ^{125}Sn, ^{127}Sb, and iodine isotopes. The presence of ^{133}Xe (the daughter of ^{133}I) was also detected (Table 3.4.2.2.1).

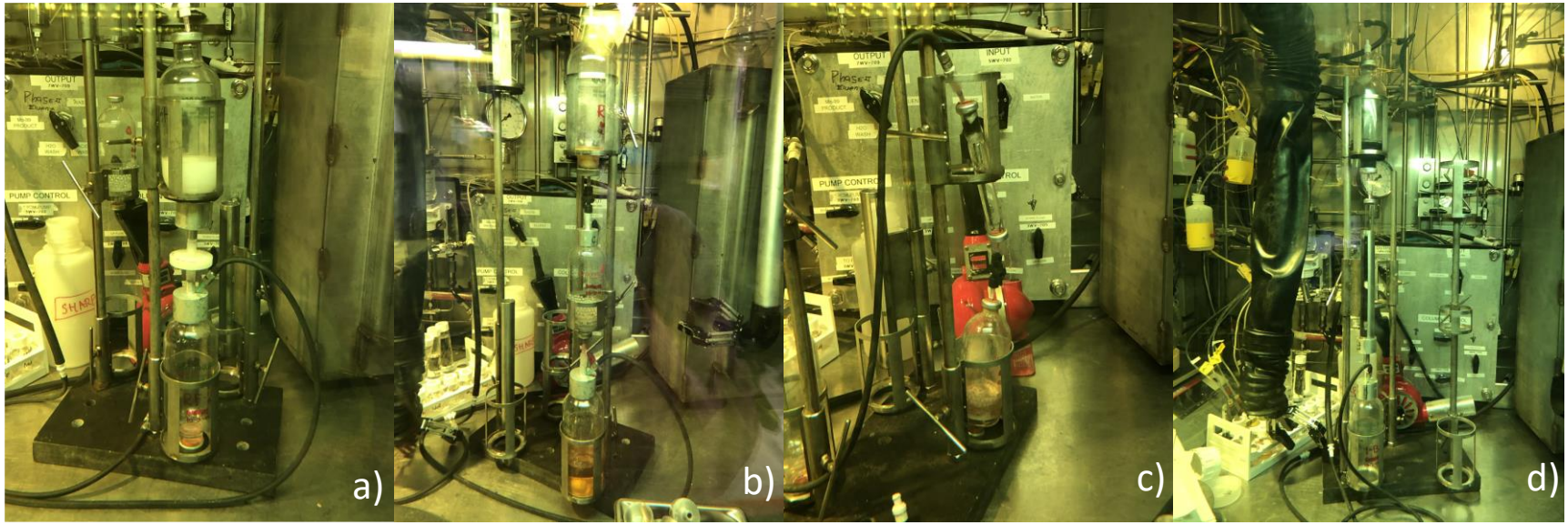


FIGURE 3.4.2.2.1 Photographs of major separation steps of LMC process: a) initial iodine precipitation; b) precipitation and filtration of Mo-ABO precipitate; c) dissolution of Mo-ABO precipitate with heating using heat gun; d) final purification using iodine precipitation and the combination column (Ag/C, HZO, CC)

TABLE 3.4.2.2.1 Activities detected in LMC fractions calculated at EOB

	A, mCi					
	RF-1	1σ	RFW	1σa	1-B product	1σ
^{95}Zr	1.72E-01	24.1%	1.22E-01	6.5%		
^{99}Mo	2.24E+03	2.10%	5.02E+00	5.50%	2.12E+03	2.10%
^{103}Ru	1.71E+00	3.90%	1.37E+00	1.90%		
^{133}Xe	2.17E+01	5.00%	8.26E+00	4.20%		
^{131}I	2.82E+01	2.70%	1.22E+01	2.10%		
^{133}I	6.21E+02	2.10%	2.76E+02	1.90%		
^{105}Rh	1.19E+01	26.8%	1.28E+00	13.6%		
^{125}Sn	1.59E+00	30.7%	8.29E-01	4.50%		
^{127}Sb	2.21E+00	10.0%	2.15E+00	3.10%		

Major radionuclides detected in the RFW fraction were ^{95}Zr, ^{103}Ru, ^{125}Sn, ^{127}Sb, iodine isotopes, and ^{133}Xe (Table 3.4.2.2.2). Results suggest that ~44% of iodine was still present after initial precipitation, which could indicate the presence of iodate, which is more difficult to remove by precipitation because of the relatively high solubility of $AgIO_3$ and the slow isotopic exchange between iodide and iodate. The majority of Ru, Zr, and Sb was found in the RFW fraction. Approximately 0.2% of the ^{99}Mo was found in the RFW, indicating that Mo is effectively removed by precipitation with ABO. No other gamma-emitting radionuclides were detected in the ^{99}Mo product. Recovery of ^{99}Mo in the LMC process was ~95%, which is very good. The total recovery of ^{99}Mo after all purification steps was ~88%.

TABLE 3.4.2.2.2 Distribution of various fission products and Mo in the RFW and ^{99}Mo product as part of the LMC process

	Fraction of Radioisotope Activity in the LMC Feed	
	RFW	1-B product
^{95}Zr	70.76%	
^{99}Mo	0.22%	94.7%
^{103}Ru	79.92%	
^{133}Xe	38.12%	
^{131}I	43.23%	
^{133}I	44.38%	
^{105}Rh	10.71%	
^{125}Sn	52.27%	
^{127}Sb	97.21%	

3.4.2.3 Irradiation #2 (11/11/19)

The LMC process followed the concentration column without any issues or spills. However, it was observed that the radiation dose rate on the silver-zeolite filter that is connected to the exhaust from the processing hot cell continuously increased during the LMC process. This increase is most likely due to the presence of radioiodine in the hot-cell atmosphere, which could be released during needle removal after solution transfers between septa bottles. No dose was detected past the silver-zeolite filter, indicating capture of iodine on the filter. Recovery yield for ^{99}Mo from the LMC process was 78.3%, which is slightly below an average of ~80% usually achieved in the LMC process. No ^{99}Mo was detected in the RFW bottle. Results can be seen in Table 3.4.2.3.1.

TABLE 3.4.2.3.1 Activities detected in LMC fractions calculated at EOB

	A, mCi					
	RF-1	1σ	RFW	1σ	1-B Product	1σ
^{95}Zr	1.81E+00	6.1%	7.38E-02	2.7%		
^{95}Nb	2.35E+00	4.4%	4.66E-02	2.7%		
^{137}Cs	2.59E+00	4.4%	2.07E-02	4.5%		
^{99}Mo	6.34E+03	2.2%			4.82E+03	2.20%
^{103}Ru	1.66E+00	6.5%	8.51E-02	2.5%		
^{131}I	1.59E+01	12.2%	5.56E+01	2.1%		
^{133}I	4.85E+02	11.1%				
^{125}Sn			3.52E+00	4.3%		
^{127}Sb	7.83E+00	9.60%	6.02E+00	3.9%		

Radionuclides present in the RFW fraction were dominated by the presence of ^{131}I. As in previous irradiations, Zr, Nb, Ru and Sb were present in the RFW fraction; however, their relative fractions compared to starting activities in the RF-1 bottle indicate that the solution in the RFW bottle might not have been mixed properly before an aliquot was taken for gamma counting (Table 3.4.2.3.2).

TABLE 3.4.2.3.2 Distribution of various fission products and Mo in the RFW and ^{99}Mo product as part of the LMC process

	Fraction of Radioisotope Activity in the LMC Feed	
	RFW	1-B product
^{95}Zr	4.08%	
^{95}Nb	1.98%	
^{137}Cs	0.80%	
^{99}Mo	0.00%	76.0%
^{103}Ru	5.13%	
^{131}I	350%	
^{127}Sb	76.9%	

No ^{99}Mo was detected in the RFW bottle after the ABO precipitation step; however, a small amount of residual Mo-ABO precipitate was visible in the RF-2 bottle. As shown previously [1], some excess of $KMnO_4$ added before precipitation of Mo with ABO might affect the recovery yield of Mo in the precipitation step. This effect is likely due to some oxidation of ABO by permanganate, which leads to formation of hard-to-work-with precipitate. The recovery of Mo in the LMC process was ~76.0%, and the total recovery of ^{99}Mo after all purification steps was ~49%. Although the recovery from the LMC process was slightly below the expected recovery (~80%), the low overall recovery yield of ^{99}Mo could be attributed mainly to the presence of cloudiness in the solution received from the primary recovery column, which negatively affected recovery yield on the concentration column.

3.4.2.4 Irradiation #3 (3/02/20)

No chemical processing was performed after the third irradiation.

3.4.2.5 Irradiation #4 (8/30/20)

A leak occurred in the multiway valve during the concentration-column processing inside the hot cell, and the experiment was paused to determine the reason for the leak. Therefore, the LMC process was not performed.

3.4.2.6 Irradiation #5 (1/18/21)

Table 3.4.2.6.1 shows the distribution of activities in the LMC fractions. About 23–26% of the iodine was found in the RFW fraction, again suggesting the presence of iodate. The composition of other fission products present in the RFW is similar to that observed in previous

irradiations. Data indicate that the majority of ^{103}Ru, ^{137}Cs and ^{125}Sn partitioned into the RFW fraction, as expected; 93.9% of the ^{99}Mo entering the LMC process was recovered. Details are presented in Tables 3.4.2.6.1 and 3.4.2.6.2.

TABLE 3.4.2.6.1 Activities detected in LMC fractions calculated at EOB

	A, mCi					
	RF-1	1σ	RFW	1σ	1-B Product	1σ
^{95}Zr			1.82E-01	2.70%		
^{95}Nb			2.71E-02	6.80%		
^{137}Cs	1.07E-01	4.80%	7.47E-02	5.30%		
^{99}Mo	2.05E+03	2.70%	4.04E+00	5.40%	1.92E+03	2.76%
^{103}Ru	1.64E+01	2.10%	2.47E+01	2.10%		
^{131}I	5.60E+01	2.30%	1.48E+01	2.20%		
^{133}I	1.07E+03	2.50%	2.48E+02	3.30%		
^{105}Rh			2.09E+01	4.00%		
^{125}Sn	1.46E+00	15.90%	1.22E+00	7.40%		

TABLE 3.4.2.6.2 Distribution of various fission products and Mo in the RFW and ^{99}Mo product as part of the LMC process

	Fraction of Radioisotope Activity in the LMC Feed	
	RFW	1-B Product
^{137}Cs	69.8%	
^{99}Mo	0.20%	93.9%
^{103}Ru	151%	
^{131}I	26.5%	
^{133}I	23.1%	
^{125}Sn	83.6%	

Aliquots of ^{99}Mo product from the LMC process were then used to detect quantities of minor fission products present in the product, using thiocyanate extraction (Figure 3.4.2.6.1) to selectively remove ^{99}Mo, and chloroform extraction to selectively separate iodine.

Radionuclides identified in the ^{99}Mo product after thiocyanate extraction and gamma counting for 57,000 s and a dead time of 0.43% are listed in Table 3.4.2.6.3, together with the activity of ^{131}I determined after selective extraction by chloroform. The iodine sample was counted for 78,000 s with a dead time of 0.79%. From the gamma analysis, it was determined that the ^{99}Mo product met radionuclide purity specifications.

FIGURE 3.4.2.6.1 Thiocyanate extraction

TABLE 3.4.2.6.3 Radionuclidic purity in ^{99}Mo product solution from the LMC process calculated at 36 hours after EOB

	Fraction of Radioisotope Activity in the LMC Feed, mCi		Ratio of Isotope Activity to ^{99}Mo Activity	
Radionuclides in 1B Product	36 h after EOB	1σ	X/^{99}Mo	Specs X/^{99}Mo
^{99}Mo	1.34E+03	2.00%		
^{137}Cs	1.42E-03	4.40%	1.06E-06	
^{103}Ru	4.65E-02	2.07%	3.47E-05	5.00E-05
^{131}I	6.09E-02	2.72%	4.54E-05	5.00E-05

3.4.2.7 References

[1] Bettinardi, D.J., and Tkac, P., *Recovery of Mo in LEU-Modified Cintichem Process at Elevated Mo Concentrations*, ANL/CFCT-19/15, Argonne National Laboratory, September 2019.

3.5 MONTE CARLO CALCULATIONS

3.5.1 Scope of the Work

This work was done to support the AMORE experiment at Argonne National Laboratory and aimed to estimate isotope accumulation in uranyl sulfate solution using Monte Carlo codes. Calculated data were compared with experimental results from the 10/1/19 irradiation to verify the model.

3.5.2 Simulation Procedure and Experimental Assembly

The experimental assembly consisted of a water-cooled target converter made of DU, a vessel with uranyl sulfate solution, and a water reflector (see the present work, Section 2, for details). MCNP and FLUKA Monte Carlo transport codes [1,2] were used for isotope yields and radiation energy deposition calculations. As a source of primary particles, a 40-MeV Gaussian electron beam with a 16-mm FWHM was used. The beam axis coincided with the converter's axis.

MCNP [1] was used to predict isotope burnup in regions of the AMORE setup containing fissionable material. The simulations involved the use of the criticality subroutine KCODE and the burnup subroutine CINDER 90. The KCODE subroutine performs iterative calculations to calculate the effective neutron multiplication factor and K_{eff}. The burnup subroutine CINDER 90 was used to simulate the production of fission-product isotopes and actinides in the solution. This subroutine tracks up to 3400 isotopes. The final data on isotope composition were calculated by considering isotope decay, burnup, and production rates in a neutron field. A 63-energy-group approximation was used in CINDER by default.

To the author's knowledge, there is no verified Monte Carlo code for burnup studies of subcritical systems driven by an external source. MCNP cannot be used directly for burnup studies of subcritical systems. But it is possible to bypass this limitation by splitting the study into two steps (Figure 3.5.2.1). First, the neutron spectrum and total number of fissions in the fissionable material are calculated. Second, the calculated neutron spectrum is used as an external source for burnup studies, and results are normalized on total number of fissions.

Stage 1: (FLUKA)

Input:
1. Primary particles: Electron beam

Output:
1. Radiation energy deposition.
2. Neutron energy spectrum generated by the irradiated target.
3. Fission events collection: number of fissions; fission fragments' A/Z range; fission energy.

Stage 2: (MCNP)

Input:
1. Primary particles: Neutron energy spectrum (from stage 1).
2. Fission power (for normalization).
3. Irradiation beam power profile (used in BURN MCNP card).

Output:
Isotope composition in uranyl sulfate solution

FIGURE 3.5.2.1 Simulation procedure

The energy deposition profile in the target region and a 3D map of deposited energy are shown in Figure 3.5.2.2. The converter absorbs almost all electromagnetic energy; only 3.0% of energy escapes the system.

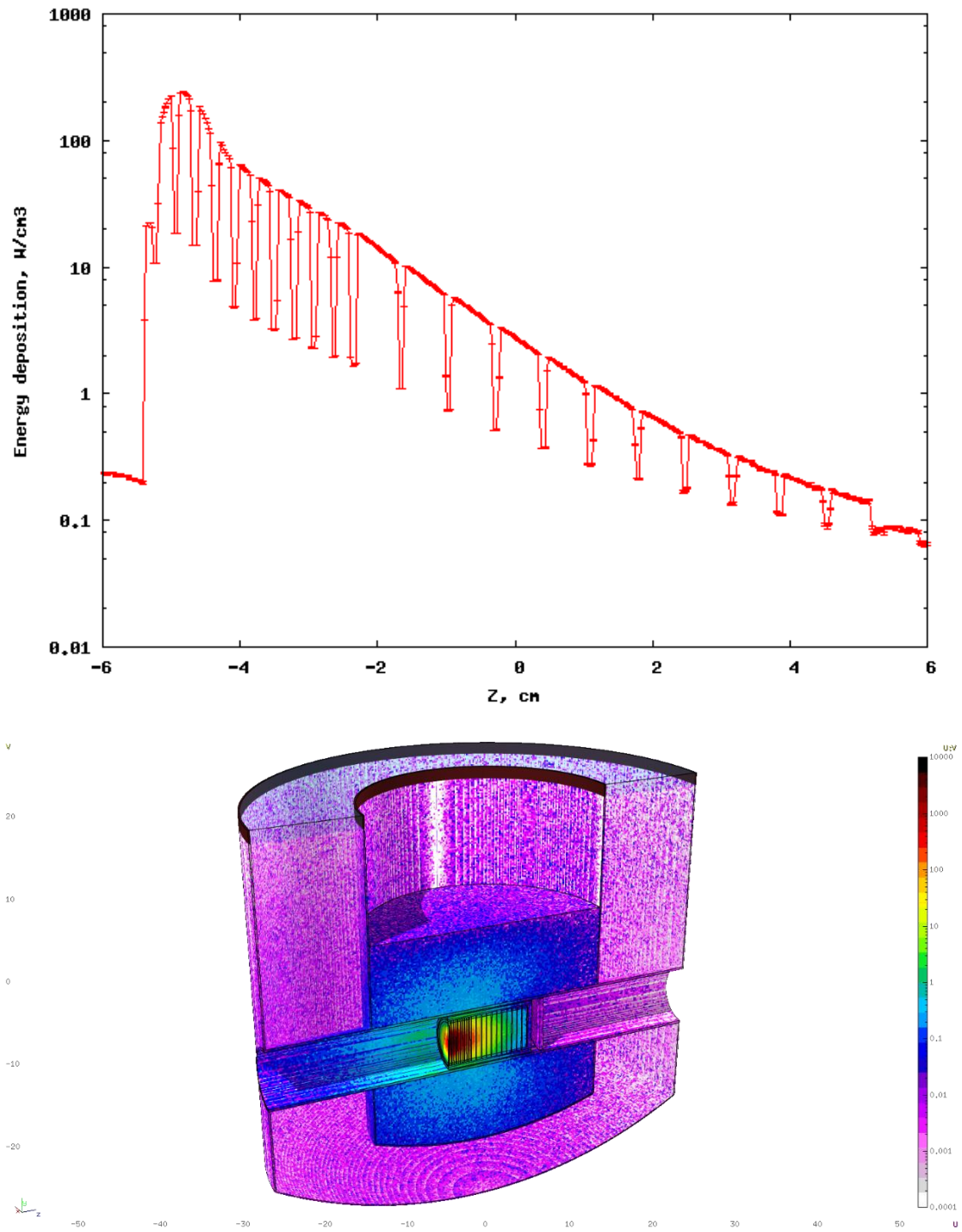


FIGURE 3.5.2.2 Radiation energy deposition: (top) energy deposition profile along the beam axis (averaged over the X,Y directions) in the target; (bottom) 3D map of energy deposition

3.5.3 Isotope Accumulation and Burnup Studies

The solution contains 135 g of uranium per liter. Uranium enrichment is 20%. The total fission rate in uranyl sulfate solution is ~ 1.99×10^{-2} fission/primary electron. The yield of ^{99}Mo was calculated as a sum of fission yields of isotopic chains, as shown in Figure 3.5.3.1. Decay mode and ^{99}Mo yield were taken from Reference [3] and are shown on the diagram. The percentage of ^{99}Mo fission was calculated as 6.14%, very close to the table value for the thermal neutron spectrum [4].

To verify the Monte Carlo model, we considered irradiation with the maximal amount of experimental data on fission fragments and fission-power gamma measurements.

The irradiation was completed on 10/1/19. The irradiation beam power profile (Figure 3.5.3.2) was provided by the linac operators and used for fission-power profile parameters in the BURN card of MCNP input. Total irradiation time was about 5.7 hours, with average power on the target 10.8 kW (14.4 max.). Total energy delivered to the target was 62 kWh. Average fission power calculated in the solution was 0.89 kW and total fission energy was 5.07 kWh. The total fission energy calculated experimentally from fission-fragment activities was 4.34 kWh.

Activities of fission fragments calculated at EOB are given in Table 3.5.3.1. The systematic difference between simulated and experimental values is in the range of 10–15% for most isotopes from MCNP Tier 3 and can be explained by a systematic error in the fission-power normalization factor and uncertainties of the simulation method.

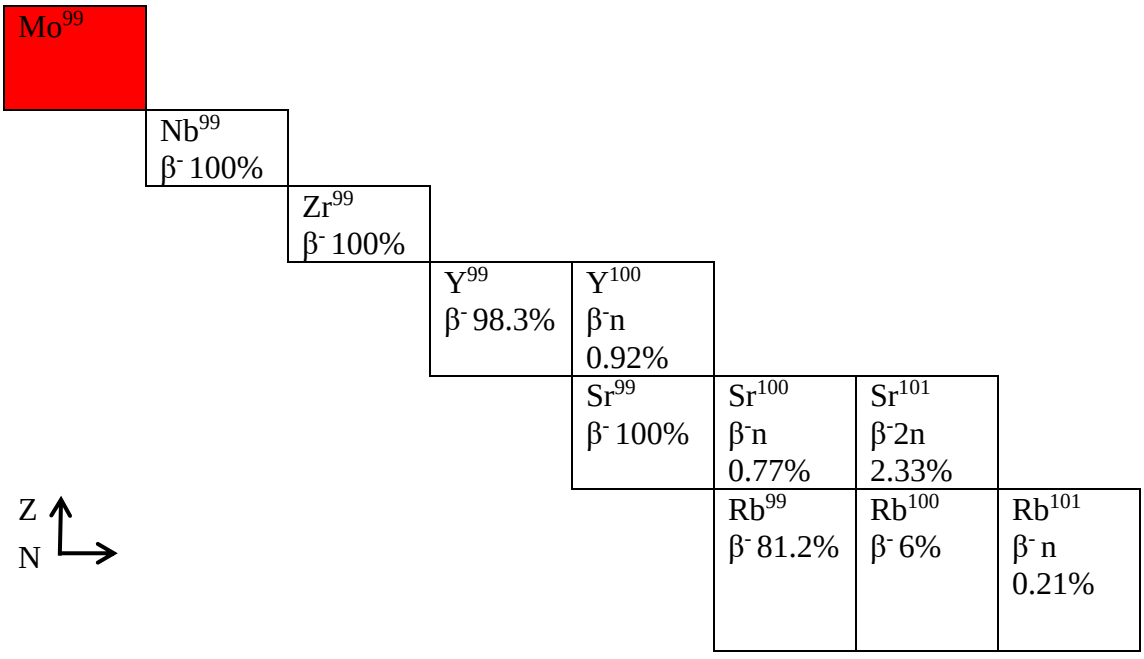


FIGURE 3.5.3.1 ^{99}Mo parent nuclides

The systematic error includes the following factors, all of which can potentially be reduced to zero:

1. Beam power measurement errors on the target caused by linac measurement-system calibrations;

2. Underestimation of neutron leaks from the system due to Monte Carlo geometry simplifications; and

3. Simplification of neutron-source parameters: energy-spectrum and spatial-distribution averaging.

Uncertainties in fission-energy determination in the solution are caused by the errors of fission-product yield cross-sections (CINDER 90) and by the calculation approach when only neutron interactions are considered. Although the simulation method we used does not take interactions with photons (photo-fissions and burnup in photo-induced reactions) into account, it gives values close to realistic ones, since most of the interactions in fissionable materials are induced by neutrons.

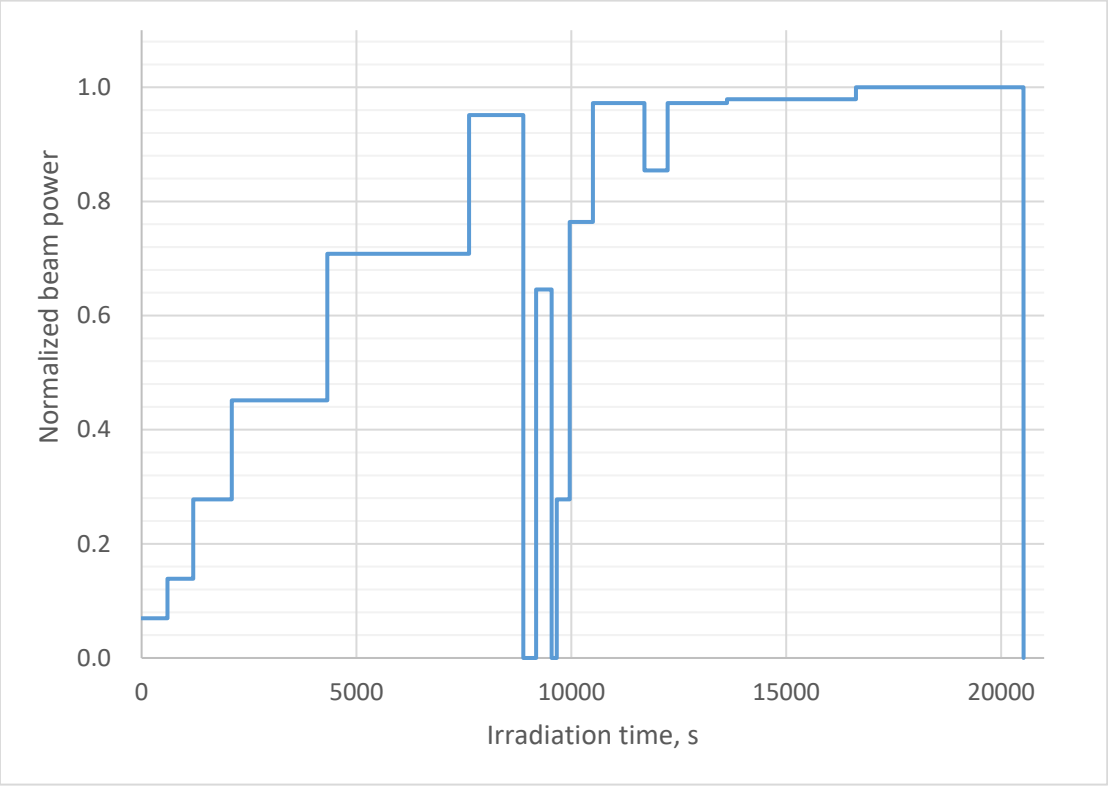


FIGURE 3.5.3.2 Irradiation beam-power profile normalized on maximum value

TABLE 3.5.3.1 Activities at EOB

	Calculation		Experiment	
Radionuclide	Activity, Ci	^{99}Mo Ratio	Activity, Ci	^{99}Mo Ratio
^{95}Zr	0.116	0.0453	0.104	0.044
^{99}Mo	2.561	1.0000	2.39	1
^{103}Ru	0.091	0.0357	0.00789	0.003
^{131}I	0.296	0.1157	0.276	0.115
^{135}Xe	4.793	1.8711	-	-
^{137}Cs	8.06E-4	0.0003	0.00314	0.0013
^{140}Ba	0.577	0.2252	0.493	0.206

3.5.4 References

[1] Los Alamos National Laboratory, A General Monte Carlo N-Particle (MCNP) Transport Code, https://mcnp.lanl.gov/.

[2] Böhlen, T.T., et al., The FLUKA Code: Developments and Challenges for High Energy and Medical Applications, *Nuclear Data Sheets* 120, 211–214 (2014).

[3] Live Chart of Nuclides, https://www-nds.iaea.org/relnsd/vcharthtml/VChartHTML.html.

[4] IAEA Physics and Nuclear Data Sections, WIMS Library Update Project, https://www-nds.iaea.org/wimsd/fpyield.htm.

4 SUMMARY

After the first Phase II irradiation of the uranyl sulfate target solution (March 2018), an elevated background reading occurred at the linac facility during concentration-column processing inside the hot cell. A small amount of fission gases from the hot-cell facility's stack was recirculated into the building, causing elevated readings on the radiation detectors within the linac facility. The experiment was put into safe conditions, and no further processing or sample collection was performed. A new process was developed for screening the experimental work against the linac facility safety bases; a configuration management program was developed; the facility exhaust stack was extended to prevent the possibility of recirculation; and the SAD and ASE were modified to include additional descriptions of the experiment and additional credited controls. In accordance with the corrective actions identified, several modifications were implemented, and modified operational procedures were developed. Phase II experiments were then restarted by performing a commissioning run using ^{99}Mo tracer without irradiation of the target solution. During preparations for the commissioning run, it was discovered that the TSV contained a precipitate that was later identified by X-ray diffraction to be uranyl peroxide. The precipitate was due to formation of uranyl peroxide during or after the first Phase II irradiation in March 2018. An attempt was made to remove the precipitate from the system, but it was not completely successful.

Gas analysis data from the first irradiation after restart (October 2019) indicated that some formation of the uranyl peroxide precipitate continued despite addition of a Fe^{2+}/Fe^{3+} catalyst prior to the irradiation. This formation was demonstrated by a high consumption of oxygen during the irradiation, which led to the necessity of actively adding oxygen into the system to ensure recombination of radiation-produced hydrogen gas. A similar trend was also observed during the second irradiation (November 2019), with a steady decrease in oxygen concentration during the irradiation. However, it appeared as if uranyl peroxide precipitate formation was slower compared to the previous irradiation, and no additional oxygen was added into the system. The third irradiation (March 2020) still showed some indication of uranyl peroxide formation, but at a much slower rate. No indication of uranyl peroxide formation was observed during the 4th and 5th irradiations.

From the irradiation perspective, on some occasions, irradiations were interrupted because of loss of the vacuum in the beamline caused by beam displacement. Because of the chromatic nature of the beamline bend (beams with different energies will emerge after the bend at different points and traveling in different directions), the beam position inside the beamline is very sensitive to beam energy and stability of accelerator parameters. An achromatic transport line (a line where beams will emerge with the same position and direction after the bend regardless of beam energy) would improve the reliability of the irradiations. All irradiations were limited in maximum beam power delivered to the target because of radiolytic hydrogen production. If the H_2 concentration reached 1%, beam power had to be decreased by 50%, and once the H_2 concentration reached 2%, the beam would turn off. For this reason, the beam power was kept as high as possible without letting the H_2 concentration reach 1%. This was the only limitation since the target design allowed placement of the full beam power (20 kW at 40 MeV) on the target.

Recovery-column data indicate that Np, Sr, and Ce were not retained by the column at all and followed U through the column during loading. Several elements, including Ba, La, Sb, Rh, and Ru, were only slightly retained by the column, evidenced by their presence at lower concentrations in samples taken during column loading. The ^{99}Mo product sent to the hot cell contained large quantities of I isotopes and their Xe daughter products, and low levels of Ru, Zr, and Sb. The contents of effluent bottles associated with the various column loading and stripping steps generally supported the trends identified by the in-line sample loops. Over the course of the 6 irradiations, there were several equipment malfunctions, the most impactful of which involved the sample retrieval solenoid valves, the column and liquid line heaters, and the pressure sensors. It is likely that the retrieval solenoid valves became clogged with precipitate over time because of their angled orientation. This idea is supported by the fact that none of the valves in the processing lines malfunctioned over the course of the experimental effort. The heaters and pressure sensors malfunctioned because of accumulated radiation damage to their control relays and the sensors themselves, respectively, even though they were shielded. These pieces will have to be replaced periodically when used in the commercial production system.

Major radionuclides identified in the feed to the concentration column besides ^{99}Mo were ^{95}Zr, ^{103}Ru, ^{105}Rh, ^{127}Sb and I isotopes. All these radionuclides were also found in the ^{99}Mo strip from the concentration column. Typically, more than 70% of iodine activity was removed during the concentration-column processing. The ^{99}Mo recovery yields from the concentration column were significantly lower than expected in four of the five runs. It appears that the main reason for incomplete ^{99}Mo recovery was an insufficient elution volume to effectively strip ^{99}Mo from the column. The content of the effluent seems to be dominated by the presence of ^{103}Ru, ^{105}Rh and iodine isotopes. Small quantities of ^{95}Zr and ^{127}Sb were also found in concentration-column effluent.

Results from the LMC process after an initial iodide precipitation step showed the presence of iodine isotopes in the bottle containing RFW (consisting of filtrate waste solution from the primary molybdenum purification step), indicating the presence of iodate species, which are more difficult to remove by precipitation as AgI because of the relatively high solubility of $AgIO_3$ and the slow isotopic exchange between iodide and iodate. It was further found that the majority of ^{103}Ru, ^{95}Zr and ^{127}Sb partitioned into the RFW. Typical ^{99}Mo recovery obtained in the LMC was ~94%, although in one instance yield was found to be 76%. Lower than expected ^{99}Mo yield could be due to a slight over-titration with $KMnO_4$, which is known to negatively impact Mo yield during Mo precipitation with ABO. Radionuclidic purity of the ^{99}Mo product was checked after the final irradiation, and only small amounts of ^{137}Cs, ^{103}Ru, ^{125}Sn and ^{131}I were detected. Activities of these radionuclides were low, and the ^{99}Mo product met radionuclide purity specifications.

APPENDIX 1

Calculation Note NE-EO-2015-05: "Thermal/Hydraulic Analysis of DU Target for mini-SHINE/MIPS"

CALCULATION COVER SHEET

Title: Thermal/Hydraulic Analysis of DU Target for mini-SHINE/MIPS
Date: 11/16/2015
Analyzed System:

PREPARER		
Philip Strons	P Philip Strons	5/16/17
Print Name	Signature	Date
REVIEWER		
Justin W. Thomas	Justin W Thomas	5/16/17
Print Name	Signature	Date
CALCULATION HAND CHECKED BY		
Justin W. Thomas	Justin W Thomas	5/16/17
Print Name	Signature	Date
FINAL APPROVER		
Jim Grudzinski		5/18/17
Print Name	Signature	Date

REVISION LOG

REVISION	CHANGES	DATE
0	Initial Release	11/18/2015
1	Updates following results of flow tests	3/8/2017

1. Objectives of the analysis

Determine via thermal/hydraulic analysis whether the DU target disks remain below safe temperature limits to prevent boiling of the water coolant and/or damage to the target disks. The data presented in this report is for guidance in operating the electron beam.

2. Background

The mini-SHINE/MIPS experiments at Argonne National Laboratory's Low Energy Accelerator Facility (LEAF) examine the possibility of Mo-99 production in a uranyl-sulfate solution. The purpose of this particular experiment, which uses the window, is to examine gas generation in the fuel solution and how it affects the reaction. This window acts both as a vacuum window and a cooling channel wall. The window design utilizes a cylindrical shape to minimize the wall thickness while maintaining sufficient stiffness.

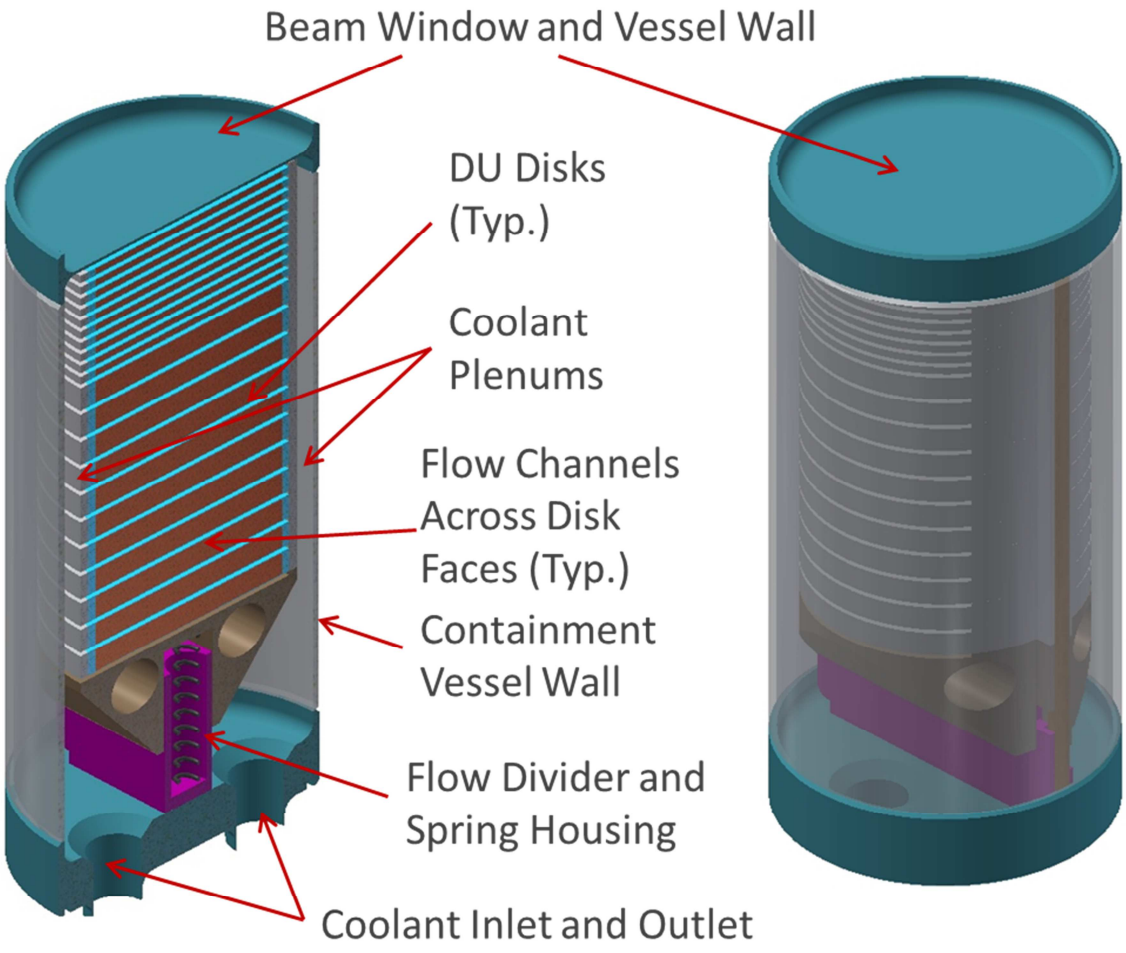


Figure 1 On the left is a cross-sectional view of the DU target assembly. The DU Disks are the subject of the analysis presented in this report. The incident electron beam is coming in from the top of the figure. On the right is a full model representation of the DU target assembly.

3. Scope of the analysis

This calculation note includes thermal/hydraulic analysis of a DU target disk only, and does not examine any of the surrounding parts of the overall target assembly. The water coolant system is merely represented by a constant volumetric flow.

4. Acceptance criteria

The temperature limits for the DU target disks are as follows:

- To prevent grain growth and clad fatigue stress, the center of the disks must not exceed 300 °C.
- To prevent boiling of the water coolant, the surface of the disks must not exceed the saturation temperatures of 126 °C at a coolant flow rate of 4.1 gpm or 134 °C at a flow rate of 5.0 gpm. (Saturation temperatures were provided based on data from the analysis in NE-CALC-2015-03 Revision 1.)

A 15% margin to boiling is assumed to be a reasonable uncertainty factor based on engineering judgment considering: a 5% error in analysis (noting that flow testing were performed on the actual target assembly and ANSYS CFX is a well validate thermal hydraulic computer code); a 5% allowance for the flow switch beam trip (considers a trip of 2gpm below the operating flow rate); 5% for beam power and width uncertainty (note that surface temperature is linear with beam power, however it is to the square of the beam width).

5. Assumptions

1. Heat is removed from the target disks through the water coolant only.
2. Flow rates: 5 gpm or 4.1 gpm per channel based on data collected during flow tests.
3. Volumetric heating of the disks is assumed to be a Gaussian distribution of the incident electron beam based calculation by CSE Division for total heat absorbed per disk.
4. A 0.25 mm layer of Zircaloy cladding is included between the uranium and the water.
5. The disk materials are assumed to be isotropic and homogeneous with the following thermal properties:

Material Thermal Properties		
	Uranium	**Zircaloy**
Thermal conductivity: k (W/m-K)	28	25

6. Method

Geometry was created in ANSYS Design Modeler [1] based on drawing number R07844. Symmetry was utilized, which can be seen in the result plots of Figures 5 and 9. The geometry of the model includes a disk of depleted uranium sandwiched between two layers of Zircaloy clad, which is cooled by two channels of water. The coolant water geometry includes an inlet plenum and an outlet plenum; each assigned a constant mass flow rate. Symmetry was utilized for all analyses except for the case of the off-center beam. Meshing of the coolant channel geometry (See Figure 3) includes inflation along the surface that interfaces with the Zircaloy clad. A k-ε turbulence model was used, and in CFX, Scalable Wall Functions are used for all turbulence models based on the ε–equation [2]. Note that both the scalable wall function and the automatic wall treatment can be run on arbitrarily fine meshes. The inlet turbulence was defined at 5% intensity and a viscosity ratio (μ_t/μ) equal to 10. The mesh was refined until the change in

results for pressure drop across the channel was less than 1%. The range of near-wall y+ values for the final mesh used was from 17 to 20.

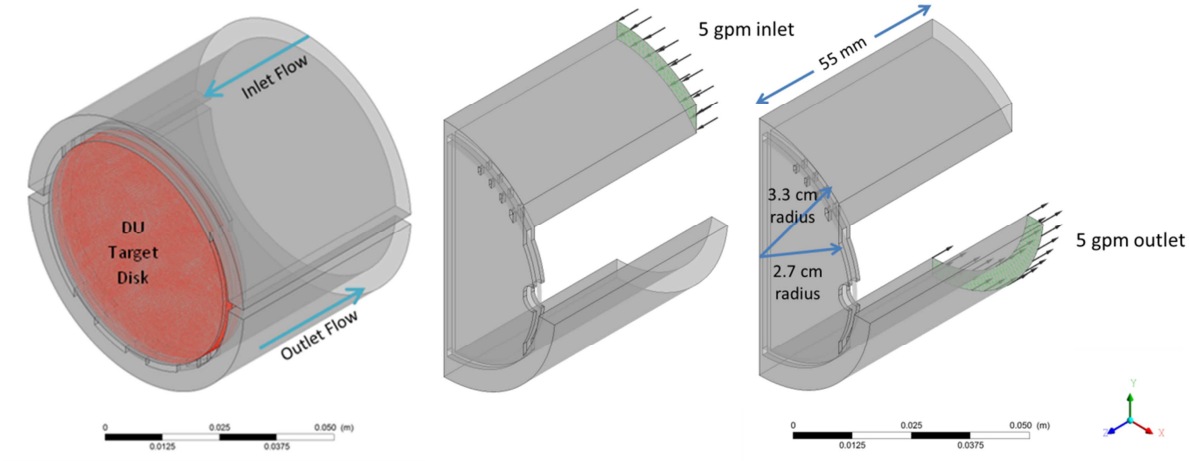


Figure 2. Simplified geometric model of a single DU target disk used in the CFD analysis. The disk is highlighted in red with the inlet plenum above and the outlet plenum below.

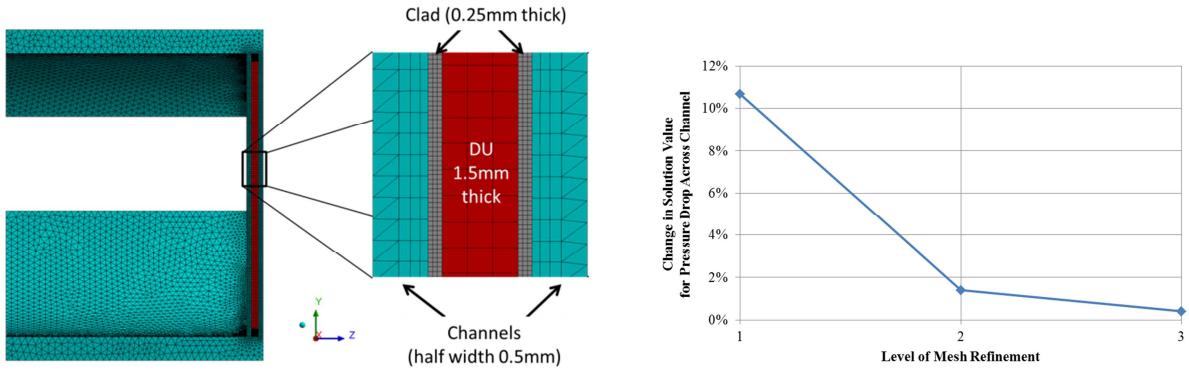


Figure 3 Meshing of the CFD model, showing the depleted uranium in red, the cladding in gray, and the water coolant in cyan. The mesh was refined until the change in results was < 1%.

The thermal/hydraulic analysis of the DU target disks is performed using ANSYS CFX [1]. A total of seven different volumetric heat generation profiles are used based on characteristics given for the incident electron beam. The profiles include three different beam widths for each of two different electron beam energies of 35 MeV and 40 MeV with two different coolant flow rates of 4.1 gpm or 5.0 gpm. Each profile was run for three different beam powers for a total of 36 cases, as well as an additional case to examine the effects of an off-center beam profile.

Table 1 Summary of Analysis Cases

Electron Beam Energy [MeV]	35						40					
Total Absorbed Power [kW]	4.06						2.30					
Coolant Flow Rate [gpm]	4.1			5.0			4.1			5.0		
Beam Width [mm]	16	18	20	16	18	20	16	18	20	14	16	18

7. Heat Absorption Profiles

The overall configuration of the DU target assembly is shown above in Figure 1, with the water coolant coming in through a plenum on one side, flowing across the face of the target disks, and exiting through the plenum on the other side. The CFD model only examines the hottest disk for each electron energy level. The values in the plot are based on a 20 kW incident electron beam, and the total heat for Disk 2 at 35 MeV is 4.06 kW and for Disk 3 at 40 MeV is 2.3 kW.

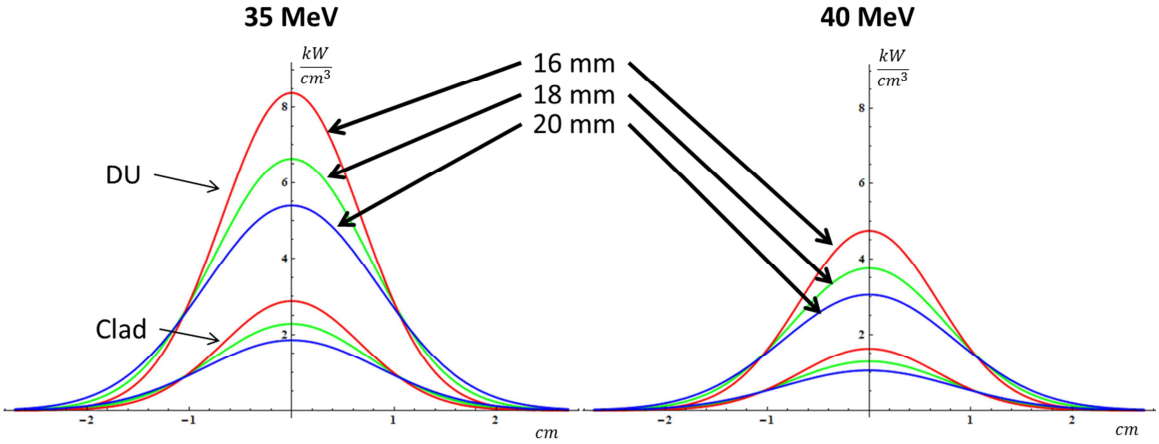


Figure 4. Power absorption profiles for the hottest disk assuming 20 kW of beam power at two different electron beam energies, 35 and 40 MeV. The power absorption is calculated separately for the depleted uranium and the Zircaloy clad, using a Gaussian distribution with FWHM beam sizes of 16, 18, and 20 mm. Total power absorbed at 35 MeV is 4.06 kW, and total power absorbed at 40 MeV is 2.30 kW.

The distributions of the volumetric heat generation were calculated in Mathematica 8 [2] to obtain a Gaussian distribution based on the FWHM size of the beam and the total power. Shown above, in Figure 4, are six separate Gaussian distributions calculated for use in the analysis. A seventh distribution, not shown in the figure, was used for a beam width of 14 mm for 40 MeV with 5.0 gpm flow rate.

8. Results

The same model setup was used throughout all steady-state analyses. Only the volumetric heat generation profile and coolant flow rate were modified for each case. For the case of the off-center beam, a full model was used with the same conditions as for the case with an 18 mm wide beam at 35 MeV and 10 kW with a flow rate of 4.1 gpm. The center of the beam was placed on a new coordinate system offset from the center by ¼ of the target disk radius.

8.1. Centered beam

Using the heat generation profiles described in Section 7, three values of beam power were selected to produce maximum surface temperature plots for each combination of electron beam energy level and coolant flow rate previously summarized in Table 1. Contour plots of the temperatures at the clad surface and the center of the DU disk are shown in Figure 5 for an 18 mm beam at 35 MeV and 10 kW with a flow rate of 4.1 gpm. In all cases, the maximum surface temperature of the Zircaloy clad was the limiting factor, and not the maximum temperature of the DU disk. Figures Figure 6 and Figure 7 summarize the effects of beam width and power for a given electron beam energy and coolant flow rate. Each plot includes a dashed horizontal line that represents the limiting saturation temperature associated with the flow rate.

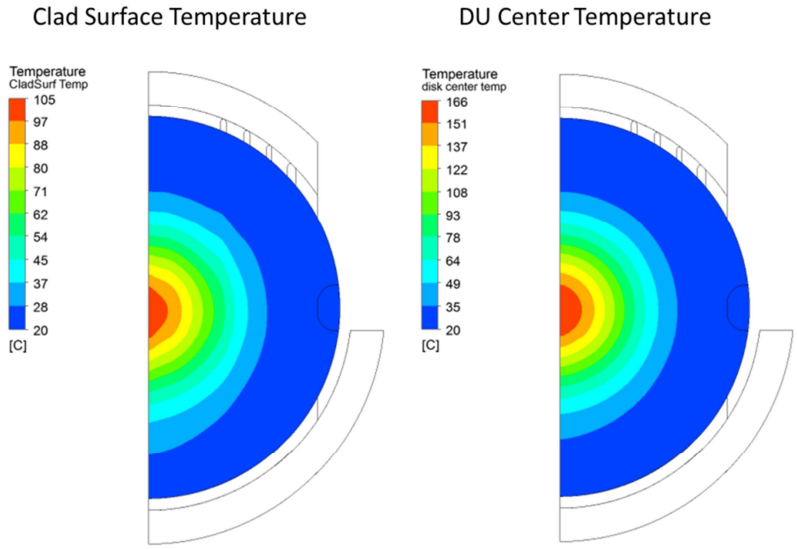


Figure 5. Steady-state temperature results, assuming a 10 kW beam at 35 MeV with a width of 18 mm FWHM and a coolant flow rate of 4.1 gpm. The plot of the left shows surface temperature distribution of the Zircaloy clad material, and the right plot shows the temperature distribution at the center of the DU target disk.

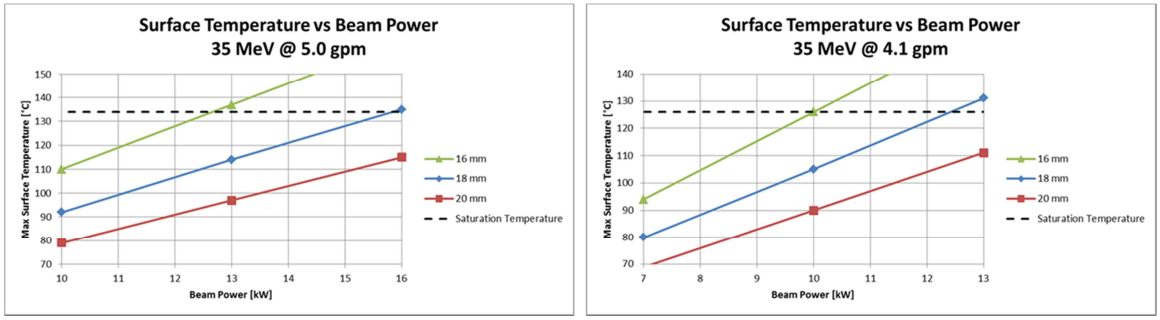


Figure 6 Maximum surface temperatures for 35 MeV for given beam size and coolant flow rate as a function of beam power. With a beam size ranging from 16 to 20 mm, the maximum beam power needs to be limited to prevent boiling at the surface of the target disk.

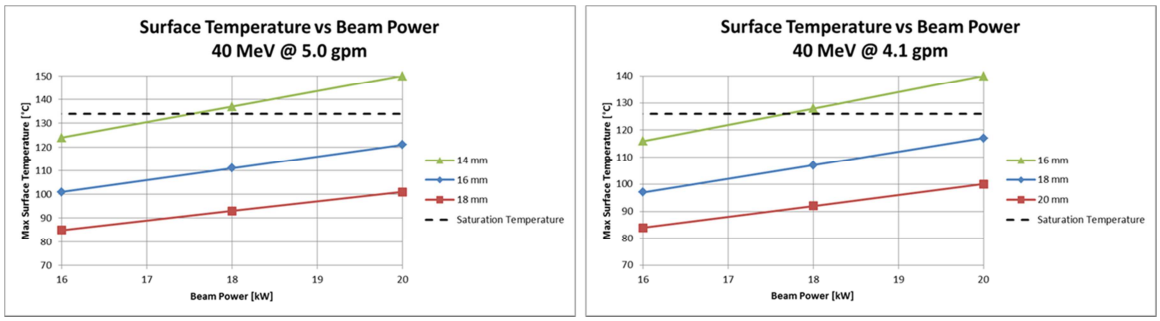


Figure 7 Maximum surface temperatures for 40 MeV for given beam size and coolant flow rate as a function of beam power. Given the right beam width, a maximum beam power of 20 kW is achievable.

8.2. Asymmetric beam

One potential accident scenario could occur in the instance of asymmetric heating of the target. Using the same Gaussian distribution of heat generation for an 18 mm beam at 35 MeV, the beam is shifted away from the center of the target disk by ¼ of the disk radius (See Figure 8). However, this deviation from off axis has no significant impact on the target disk temperature (See Figure 9).

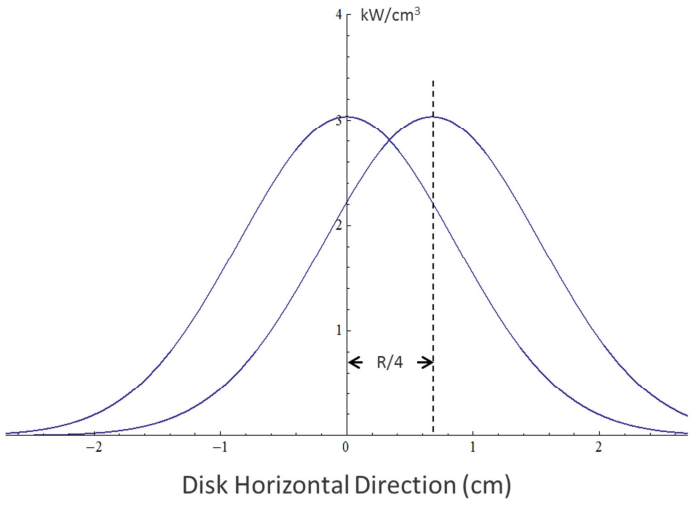


Figure 8. An illustration depicting an asymmetric beam profile based on a typical centered profile with the peak of the distribution moved away from the center by ¼ of the disk radius.

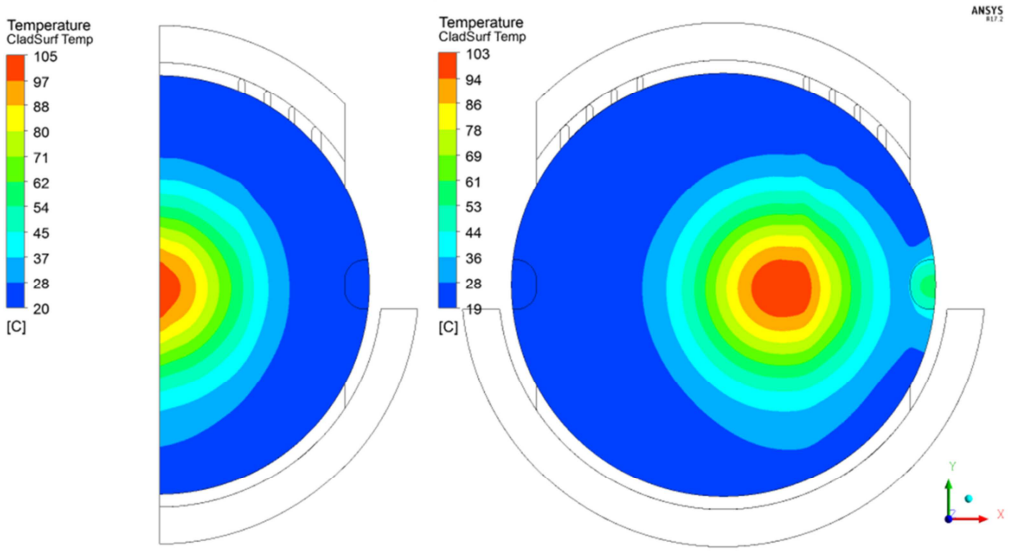


Figure 9. Temperature results from both a symmetric and asymmetric beam. All other conditions of the model remain the same. The asymmetric profile shows little difference in temperature compared to the normal operating conditions.

9. Conclusions

- Plots have been presented to show the of maximum surface temperature to be expected for a given beam size and beam power
- At 40 MeV, a 16 mm wide beam with 20 kW of power will remain below the saturation temperature provided that the water coolant is supplied at a rate of 5 gpm.
- Asymmetric beam: does not cause problematic temperature distribution, temperatures still within limits

10. References

1. ANSYS. Vers. 17.2. Canonsburg, PA: ANSYS, Inc., 2016. Computer software.
2. CFX Theory Guide, Section 2.8.1.1. Scalable Wall Functions, Canonsburg, PA: ANSYS, Inc., 2016.
3. Wolfram Mathematica. Vers. 8. Champaign, IL: Wolfram Research, Inc., 2011. Computer software.

APPENDIX 1
GENERAL CHECKING CRITERIA SHEET

CALCULATION CHECKLIST	**Yes**	**No**	**N/A**	**Comments**
1. Are analytical methods appropriate?	☒	☐	☐	
2. Are assumptions appropriate?	☒	☐	☐	
3. Is the calculation complete?	☒	☐	☐	
4. Are formulas appropriately referenced?	☒	☐	☐	
5. Are the input data appropriate?	☒	☐	☐	
6. Was utilized software appropriate for the task?	☒	☐	☐	
7. Were software input/initial conditions/properties/boundary conditions appropriate?	☒	☐	☐	Yes, but see comments below.
8. Are the results reasonable?	☒	☐	☐	

APPENDIX 1
GENERAL CHECKING CRITERIA SHEET

ADDITIONAL COMMENTS		
Number	**Comment**	**Resolution**
1.	Since this effort is focused on heat transfer (rather than flow), I would suggest using the peak clad temperature as the criterion for evaluating mesh, i.e. to replace pressure drop in Fig. 3. I would define a θ as follows: $\theta = (T_{clad} - T_{clad,\,ref})/(T_{clad} - T_{inlet})$ where T_{clad} = predicted peak cladding temperature for this (coarse) mesh $T_{clad,ref}$ = predicted peak cladding temperature for the finest mesh T_{inlet} = inlet coolant temperature And then compare θ for different meshes, and plot similarly to Fig. 3.	Although Fig 3 shows pressure drop, the thermal solution converged before the pressure drop solution.
2.	Wall functions (in STAR-CCM+ anyway) perform best for 30<y+<100, whereas near-wall turbulence models require y+~1. Wall functions have been known to produce inaccurate results for the in-between range, say 5<y+<20. I am hoping that the y+~30 range was tested during your mesh convergence study and shown to not be important for your case.	y+ values greater than 30 did not have different thermal results than the y+ values in the final solution
3.		
4.		
5.		
6.		
7.		
8.		
9.		
10.		

APPENDIX 2
ENERGY BALANCE CHECK

INPUTS		**OUTPUT**
Half Symmetric Power [kW]	**Flow Rate [kg/s]**	**ΔT [°C] from outlet average T**
4.06/2 = 2.03	0.258	1.9
	0.315	1.5
2.30/2 = 1.15	0.258	1.1
	0.315	0.9

APPENDIX 2

Calculation Note NE-CALC-2015, ver. 1: "Thermal-Hydraulic Analysis of the Stoppage of Coolant Flow"

CALCULATION COVER SHEET

Title: DU Target Assembly	Thermal-Hydraulic Analysis of the Stoppage of Coolant Flow	
Date: April 3, 2015		
Analyzed System:	DU Target Cooling	
PREPARER James L. Bailey	James L. Bailey	4/6/15
Print Name	Signature	Date
REVIEWER Philip Strons	Philip Strons	4/6/15
Print Name	Signature	Date
CALCULATION HAND CHECKED BY		
Print Name	Signature	Date
FINAL APPROVER [illegible]	Jim Grudzinski	4/8/15
Print Name	Signature	Date

REVISION LOG

REVISION	CHANGES	DATE
0	Initial Release	

1. Objectives of the analysis

The object of the analysis is to determine the maximum temperature that the coolant will reach due to decay heat in the uranium disks under the off normal occurrence of a stoppage of coolant flow through the target.

2. Background

Upon a stoppage of coolant flow through the cooling system a flow switch initiates the beam trip and shuts down the LINAC. However, the decay heat generated in the uranium continues to heat the coolant in the target. Under the worst case scenario, the total heat generated in the target immediately after shut down is 13.5W and then decays to less than a 1W after 4 hours. The target vessel is located in the tank sleeve and hence, is surrounded by a small air gap that essentially causes an insulated boundary there. Assuming these conservative conditions the maximum water coolant is calculated.

3. Scope of the analysis

The thermal hydraulic behavior of the target coolant under this off normal occurrence is to be determined.

The determination of heat generation rate is not part of this analysis.

4. Acceptance criteria

Acceptance criterion is based on avoidance of coolant boiling in the target after the stoppage of flow.

5. Methodology

Hand calculations using Excel are used calculate the required transient heat balances.

6. Assumptions

The vessel housing is insulated from its surrounding.

The residual heat in the target disks at beam shut down is negligible.

The heat capacity of the disks, vessel and inner stainless steel parts is conservatively neglected.

Only the heat capacity of the coolant water is considered.

Linear interpolation of the decay heat input curve between time steps is sufficiently accurate.

The rate of decay (time constant) for all the disks is the same as for disk 2.

Based on the thermal hydraulic analysis for the target cooling system a maximum temperature of the coolant water entering the target is 90°F.

7. Analysis Inputs

Geometry is per drawing “DU Target DU Disk Assembly + Weldment” Drg. # R07844, and associated subassemblies and parts.

Decay heat in each disk immediately after shut down is as indicated in reference 1.

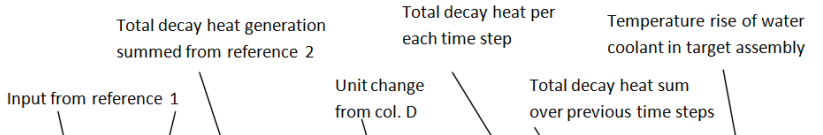
Transient decay of disk 2 is as indicated in reference 2.

Total heat generation in the target below 1W is considered negligible.

8. Calculations

This calculation is intended to determine the final maximum temperature of the coolant water and target disks after the stoppage of flow. It is assumed that all the decay heat is stored in the coolant water that remains in the target assembly after the stoppage of flow. Only the heat capacity of the water and the corresponding temperature rise is considered in the heat balance calculation.

Reference the Excel table below. Columns A and B are decay curve information for disk 2 that is taken directly from reference 1. Total heat generation in the disks immediately after shut down is stored in column E1. This value is calculated by summing the heat generation in all the disks as indicated in reference 2. Column D7 and below are the calculated total heat generation decay that includes all the disks. The values for each time step are calculated by dividing the heat generation in disk 2 at any time by the heat generation in disk 2 at time 0 and then multiplying that ratio by the total heat generation in all the disks at time 0. The implied assumption for this calculation is that the heat generation decay rate is the same for all the disks. Excel equation for this calculation is [=B7/B7*E1]. Column E7 and below simply converts the values in D from SI units to English. Column F8 and below assumes a linear interpolation over each time step from column E and then multiplies the value by the time between time steps. Excel equation for this calculation [=(E7+E8)/2*(A8-A7)]. Column G8 and below is the running sum of column F. Excel equation for this calculation [=F8+G7]. The temperature rise of the coolant water and (and disks) for each time step is calculated in Column H8 and below. This calculation conservatively assumes that all the heat calculated in column G is stored in the coolant water as sensible heat and considers only the density, heat capacity and volume of the water. Excel equation for this calculation [=G8/(E2*E3*H2)] The water volume has been estimated from the reference drawings. Water properties assume near ambient pressure and temperature. The final temperature differential of 56.6° F is indicated in column H30. If an ambient starting temperature of 90°F is assumed the final temperature of the water is [90+57=147°F]. This is assuming that the decay heat of less than 1W after 240 min., 4 hours, is negligible.



	A	B	C	D	E	F	G	H
1				q total [W]	13.54		Volume H2O [in^3]	10
2				ρ H2O [lb/ft^3]	62.3		Volume H2O [ft^3]	0.00579
3				Cp H2O [Btu/lb-F]	1		[W]--[Btu/min]	0.0569
4								
5	disk 2	Decay power		Decay power	Decay power	Decay heat	Decay heat sum over	Disk temp.
6	time	disk 2 [W]		total [W]	total [Btu/min]	Ave. [Btu]	time total [Btu]	Δ T [F]
7	0	1.599E+00		1.354E+01	7.704E-01			
8	0.5	7.43E-01		6.293E+00	3.581E-01	2.82E-01	2.82E-01	0.78
9	1	6.464E-01		5.474E+00	3.114E-01	1.67E-01	4.50E-01	1.25
10	1.5	5.92E-01		5.011E+00	2.851E-01	1.49E-01	5.99E-01	1.66
11	2	5.55E-01		4.697E+00	2.673E-01	1.38E-01	7.37E-01	2.04
12	2.5	5.27E-01		4.465E+00	2.541E-01	1.30E-01	8.67E-01	2.40
13	3	5.058E-01		4.283E+00	2.437E-01	1.24E-01	9.92E-01	2.75
14	3.5	4.88E-01		4.134E+00	2.352E-01	1.20E-01	1.11E+00	3.08
15	4	4.73E-01		4.009E+00	2.281E-01	1.16E-01	1.23E+00	3.40
16	4.5	4.61E-01		3.900E+00	2.219E-01	1.13E-01	1.34E+00	3.71
17	5	4.49E-01		3.805E+00	2.165E-01	1.10E-01	1.45E+00	4.02
18	5.5	4.392E-01		3.719E+00	2.116E-01	1.07E-01	1.56E+00	4.31
19	6	4.30E-01		3.641E+00	2.072E-01	1.05E-01	1.66E+00	4.60
20	6.5	4.22E-01		3.570E+00	2.031E-01	1.03E-01	1.76E+00	4.89
21	7	4.14E-01		3.505E+00	1.994E-01	1.01E-01	1.86E+00	5.17
22	7.5	4.07E-01		3.444E+00	1.960E-01	9.88E-02	1.96E+00	5.44
23	8	4.00E-01		3.386E+00	1.927E-01	9.72E-02	2.06E+00	5.71
24	8.5	3.94E-01		3.333E+00	1.896E-01	9.56E-02	2.16E+00	5.98
25	9	3.88E-01		3.282E+00	1.868E-01	9.41E-02	2.25E+00	6.24
26	9.5	3.82E-01		3.234E+00	1.840E-01	9.27E-02	2.34E+00	6.49
27	10	3.77E-01		3.188E+00	1.814E-01	9.14E-02	2.43E+00	6.75
28	60	1.86E-01		1.571E+00	8.938E-02	6.77E+00	9.20E+00	25.51
29	120	1.33E-01		1.124E+00	6.394E-02	4.60E+00	1.38E+01	38.26
30	240	9.57E-02		8.105E-01	4.611E-02	6.60E+00	2.04E+01	56.57

9. Discussion

The assumption that the residual heat in the target disks immediately after shut beam is negligible is based on the reasoning that the water flow has a coast down period after the beam shut down is tripped, and as a result, there is a period of time that there is still flow through the target after beam shut down, which is of the order of several seconds. Further, the transient thermal calculations indicate that the response of the disks (cool down) is less 1 second. Hence, there is sufficient time for the disks to cool to ambient temperature before complete stoppage of flow. Also, it should be noted that the heat capacity of the stainless steel internal parts and housing have conservatively not been considered.

The assumption that the decay rate for the entire disk assembly is approximately the same as for disk 2 is per verbal discussions with Brad Micklich.

10. Conclusions

Based on the above analysis it is concluded that upon an off normal occurrence of the stoppage of water coolant flow through the target, the temperature of all target components will remain below their maximum allowable design temperatures.

11. References

1. Email from Micklich, Bradley J. to Bailey, James L., Chemerisov, Sergey D. dated January 22, 2015, Subject: DU Target SAD comments.

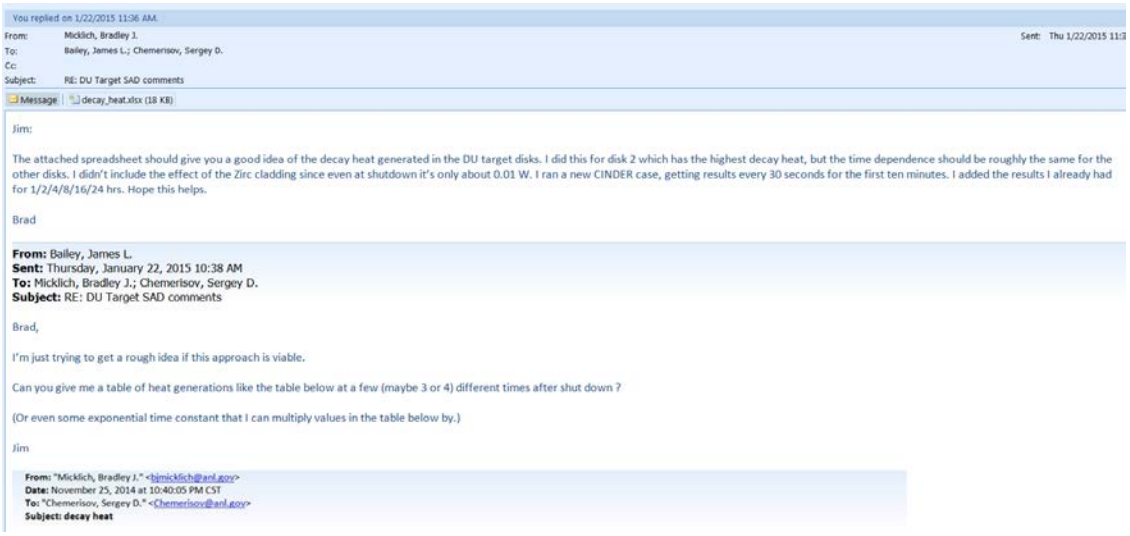
2. Email from Micklich, Bradley J. to Chemerisov, Sergey D. dated November 25, 2014, Subject: decay heat.

12. Support Documents

Reference 1 and 2 attached.

Reference Target Assembly drawing attached

Reference 1



You replied on 1/22/2015 11:36 AM.

From: Micklich, Bradley J. Sent: Thu 1/22/2015 11:3
To: Bailey, James L.; Chemerisov, Sergey D.
Cc:
Subject: RE: DU Target SAD comments

Message | decay_heat.xlsx (18 KB)

Jim:

The attached spreadsheet should give you a good idea of the decay heat generated in the DU target disks. I did this for disk 2 which has the highest decay heat, but the time dependence should be roughly the same for the other disks. I didn't include the effect of the Zirc cladding since even at shutdown it's only about 0.01 W. I ran a new CINDER case, getting results every 30 seconds for the first ten minutes. I added the results I already had for 1/2/4/8/16/24 hrs. Hope this helps.

Brad

From: Bailey, James L.
Sent: Thursday, January 22, 2015 10:38 AM
To: Micklich, Bradley J.; Chemerisov, Sergey D.
Subject: RE: DU Target SAD comments

Brad,

I'm just trying to get a rough idea if this approach is viable.

Can you give me a table of heat generations like the table below at a few (maybe 3 or 4) different times after shut down ?

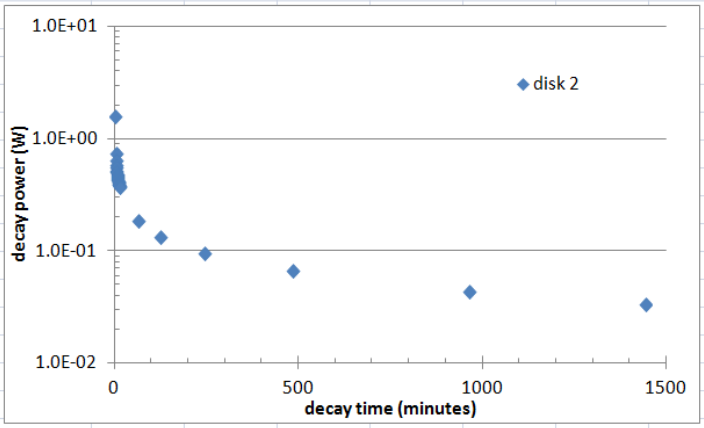
(Or even some exponential time constant that I can multiply values in the table below by.)

Jim

From: "Micklich, Bradley J." <bjmicklich@anl.gov>
Date: November 25, 2014 at 10:40:05 PM CST
To: "Chemerisov, Sergey D." <Chemerisov@anl.gov>
Subject: decay heat

Attachment for reference 1 email

	A	B	C	D	E	F	G	H	I	J	K
3											
4											
5	disk 2										
6	time	decay power (W)									
7	0	1.599E+00									
8	0.5	7.43E-01									
9	1	6.464E-01									
10	1.5	5.92E-01									
11	2	5.55E-01									
12	2.5	5.27E-01									
13	3	5.058E-01									
14	3.5	4.88E-01									
15	4	4.73E-01									
16	4.5	4.61E-01									
17	5	4.49E-01									
18	5.5	4.392E-01									
19	6	4.30E-01									
20	6.5	4.22E-01									
21	7	4.14E-01									
22	7.5	4.07E-01									
23	8	4.00E-01									
24	8.5	3.94E-01									
25	9	3.88E-01									
26	9.5	3.82E-01									
27	10	3.77E-01									
28	60	1.86E-01									
29	120	1.33E-01									
30	240	9.57E-02									
31	480	6.67E-02									
32	960	4.39E-02									
33	1440	3.39E-02									
34											



Reference 2

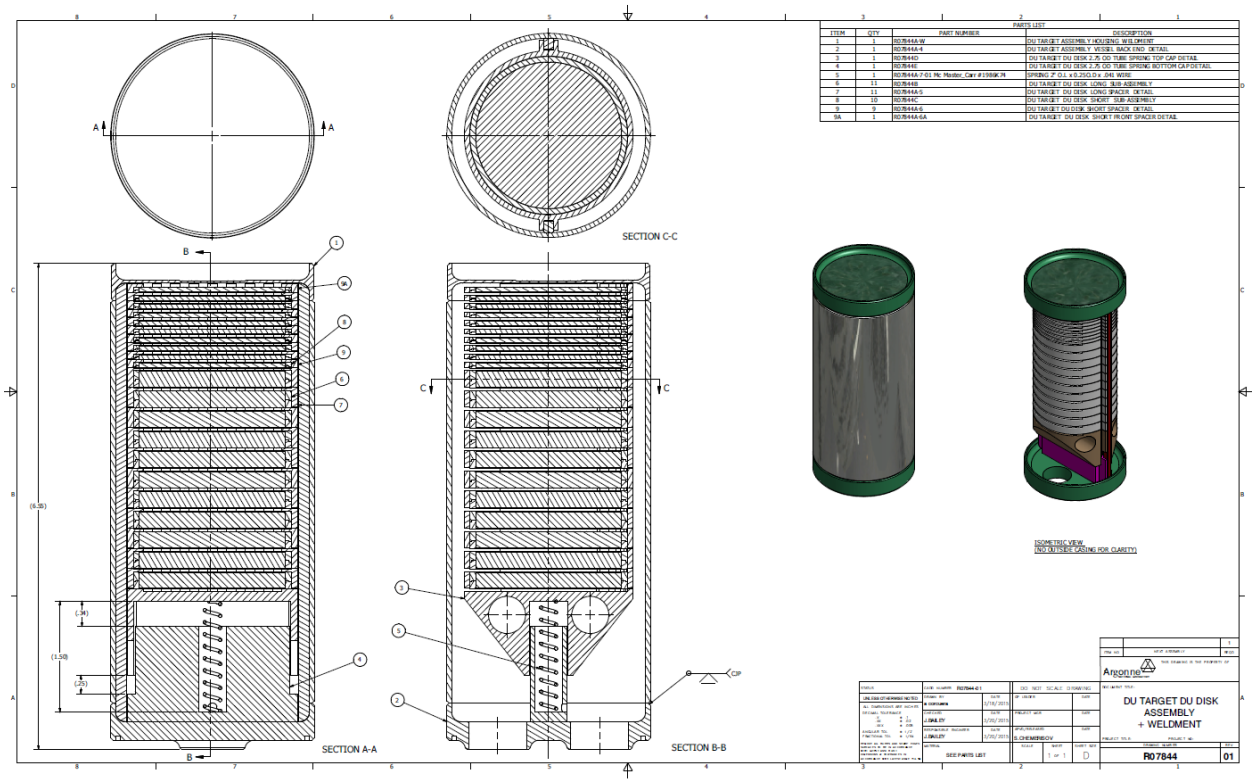
From: "Micklich, Bradley J." <bjmicklich@anl.gov>
Date: November 25, 2014 at 10:40:05 PM CST
To: "Chemerisov, Sergey D." <Chemerisov@anl.gov>
Subject: decay heat

Sergey:

The decay heat in the solution at shutdown after the 5th irradiation is about 123 W. Here are the decay heats (in watts) in the individual DU disks at shutdown after the 5th irradiation. Hope this is what you need. Brad

9.655E-001
1.599E+000
1.486E+000
1.232E+000
1.010E+000
8.416E-001
7.036E-001
5.945E-001
5.037E-001
4.221E-001
1.061E+000
7.049E-001
4.994E-001
3.805E-001
3.004E-001
2.554E-001
2.173E-001
1.892E-001
1.683E-001
1.515E-001
1.340E-001
1.201E-001

Reference Target Assembly Drawing



APPENDIX 1
GENERAL CHECKING CRITERIA SHEET

CALCULATION CHECKLIST	Yes	No	N/A	Comments
1. Are analytical methods appropriate?	☒	☐	☐	
2. Are assumptions appropriate?	☒	☐	☐	
3. Is the calculation complete?	☒	☐	☐	
4. Are formulas appropriately referenced?	☐	☐	☒	
5. Are the input data appropriate?	☒	☐	☐	
6. Was utilized software appropriate for the task?	☒	☐	☐	
7. Were software input/initial conditions/properties/boundary conditions appropriate?	☐	☐	☒	
8. Are the results reasonable?	☒	☐	☐	

APPENDIX 1
GENERAL CHECKING CRITERIA SHEET

ADDITIONAL COMMENTS		
Number	**Comment**	**Resolution**
1.		
2.		
3.		
4.		
5.		
6.		
7.		
8.		
9.		
10.		

APPENDIX 3

Calculation Note NE-CALC-2015-03: "Thermal-Hydraulic Analysis of the Overall Performance of the DU Target Cooling System"

CALCULATION COVER SHEET

Title:	Thermal-Hydraulic Analysis of the Overall Performance of the DU Target Cooling System
Date: August 3, 2015	
Analyzed Systems:	DU Target Cooling and DU Target Assembly

PREPARER		
James L. Bailey	James L Bailey	5/15/17
Print Name	Signature	Date
REVIEWER		
Victor Guarino	Victor Guarino	5/15/17
Print Name	Signature	Date
CALCULATION HAND CHECKED BY		
Print Name	Signature	Date
FINAL APPROVER		
Print Name	James Grudzinski	5/23/17
	Signature	Date

REVISION LOG

REVISION	CHANGES	DATE
0	Initial Release	
1	Revised analyses based on flow tests and analyzed system considering a larger pump	March 2, 2017

1. Objectives of the analysis

The object of the analysis is to verify that the coolant flow distribution through the target assembly is adequate to provide the cooling that is required to maintain the design temperatures for the target disks during normal operation.

2. Background

During normal operation heat is generated within the zircaloy-4 clad depleted uranium target disks due to impingement of the electron beam on the target. The target disks are cooled by DI water flowing between the disks (Refer to Figure 1). The coolant supply and return are connected to the existing DU target cooling system that provides the required heat exchanger, pump, strainer and DI unit (Refer to Figure 2).

3. Scope of the analysis

The thermal hydraulic behavior of the target assembly and associated cooling system under normal operation is to be determined.

The determination of heat generation rate is not part of this analysis and is considered input to this analysis. (Reference 1)

The local heat transfer analyses (ANSYS CFX) at the target disks are not part of this calculation note and is presented in reference 1. Only the results from these analyses are used in this note.

The hydraulic performance through the DU target assembly is not part of this calculation note and was determined by flow tests on the actual target as reported in reference 8. The flow resistances as determined by these test were input to this calculation note.

Two separate analyses are performed. The system is first analyzed considering a smaller capacity pump and then the system is reanalyzed considering a larger pump. The results for the larger pump are noted in brackets following the results for the smaller pump.

4. Acceptance criteria

Acceptance criterion is based on avoidance of coolant boiling in the target and allowable maximum material temperature of the uranium. (I.e. surface temperature of the disks to be below saturation temperature at the operating pressure and below 572°F (300°) within the disks. (Criteria is based on Reference 1 and thermal hydraulic flow stability in the channels, I.e. avoidance of channel boiling)

5. Methodology

ANSYS CFX is used to determine the thermal hydraulic performance locally at the critical disk locations in the target assembly. (Reference 1)

Flow characteristics as determined from the hydraulic tests on the DU Target Assembly (Reference 8) are used as input to the AFT FATHOM overall thermal hydraulic model.

FATHOM's library of piping components and manufactures' data are used to model the cooling system with the exception of the DU Target Assembly.

6. Analysis Assumptions and Inputs

Geometry of the target assembly is per drawing “DU Target DU Disk Assembly + Weldment” Drg. # R07844, and associated subassemblies and parts. (Figure 1)

Cooling system is per the P/I in Figure 2

Heat generation is per Reference 1

The heat generation in the vessel and inner stainless steel parts negligible.

The vessel housing and cooling system tubing and components are insulated from their surroundings.

The performance of the existing Haskris chiller (Currently located on the service floor in Building 211) is as indicated in the email from the vendor. (Reference 2)

The performance of the pumps is as indicated on the manufacturer’s pump curves (Reference 3)

The performance of the plate heat exchanger is as indicated on the vendors quote (Reference 4)

The pressure differential of the particle filter is as indicated in the email from the vendor (Reference 5)

Pressure losses through the valves, pipe and tube are calculated using data from the AFT FATHOM Version 7.0 library (Note: In general this data is in good agreement with that presented in the reference “Fluid Mechanics”, 3rd Edition, R.C.Binder.)

The expansion tank is vented to atmospheric pressure

Flow through the system will be manually balanced at start up and under normal operation the flow will remain essentially constant without feedback control.

The temperature of the system will be controlled by the constant temperature of the coolant out of the Haskris chiller (55°F). As a result, the temperature of the coolant in the primary system will be allowed to vary dependent upon the heat load up to a maximum inlet temperature of 67°F at the DU Target. (I.e. maximum heat load condition, 16kW)

Coolant fluid is DI water

7. Calculations

First, thermal hydraulic analyses were performed on the three critical disks (#2, #4, and #15) using ANSYS CFX. These disks were selected based on the total heat generation in the disk (highest) and the total coolant flow over the faces of the disks (lowest). These conditions result in the worst cases when considering the above acceptance criteria (maximum allowable temperatures). The three worst case disks are noted here. The CFX model included both the heat conduction within the disks and the convective cooling at the surface. The hydraulic performance was performed considering a 5.8psid [9.0psid] from inlet plenum to outlet plenum at the flow rates indicated below and as determined from the flow tests that were performed on the actual target (Reference 8). The flows were balanced between disks by varying the size of the flow control orifices on the spacers (Refer to Figure 1). The pressure drop through the target inlet and outlet plenums was found to be negligible and hence, the pressure differential across each disk face was the same. As a result only three different orifice sizes were required in order to balance the flows. (4.1gpm [5.0gpm] for disks #1 to #3, 1.75gpm [2.21gpm] for disks #4 to #14, and 1.00gpm [1.33gpm] for disks #15 to #22) (Refer to Reference 1 for the details of this analysis). As a result of these flow conditions at the disks the total flow through the target is 42gpm [53gpm] with the corresponding

pressure loss of 5.8psi [9.0psi]. These conditions were found to be compatible with the existing cooling system by iteration between the CFX and FATHOM models. The total internal heat generation for the critical disk thermal analyses was obtained from reference 1. The target disks are as shown on the design drawing R0744B and C and are depleted uranium with 0.010in. Zircaloy-4 clad on the faces. The CFX thermal hydraulic analyses for the target disks summarized in Figure 3 plot are discussed in detail in the report noted in Reference 1.

The commercial computer code AFT FATHOM, Version 7.0, was used to model the overall target cooling system. The AFT FATHOM computer model is shown in Figure 3. The pipe and junction numbers used in the output are referenced on the model shown in Figure 3. The DU target's internal flow configuration is modeled at the top half of the diagram. The beam direction is horizontal from right to left. The flow across the face of each disk is simulated by a parallel channel with an equivalent flow resistance as obtained from the flow tests in Reference 8. This resistance is modeled as an orifice at the channel inlet and a rectangular duct with an average length. These channels are connected to the inlet and outlet plenums shown above and below the parallel channels. The flow resistances in the plenums are also modeled. The expansion and contraction losses for the coolant supply and return at the back of the target assembly are modeled at the upper left of the diagram. Also, the total heat generation from all the disks are modeled thermally as a single 16 kW input at the back of the target. The support cooling system in the associated enclosure and expansion tank are shown in the lower half of the diagram. The throttle valve at the discharge of the pump is full for both pump analyses. The valve in the DI bypass, J51 is 85 degrees closed in order to reduce the flow to approximately 1.0gpm [1.5gpm]. All other valves are full open. The valves are standard ball valves from the FATHOM Database. Tubing and fittings are stainless steel 16BWG and are also from the FATHOM Database.

The pump performance curve was input as shown in the appendix using the manufacturer's data for the 5-3/8" [6-5/8"] impeller as indicated in reference 4.

The Particle Filter pressure resistance curve shown in reference 5 was determined from a single point for a clean filter from the vendor's data (I.e. 0.7psi at 50gpm). In order to develop the complete resistance curve the flow through the strainer was assumed turbulent, and therefore, the pressure drop was assumed proportional to the velocity squared.

The hydraulic performance curve of the heat exchanger, HX-1, was input using the manufacturer's data indicated in reference 4 (I.e. 10psi at 50gpm). In order to develop the complete resistance curve the flow through the heat exchanger was assumed turbulent, and therefore, the pressure drop was assumed proportional to the velocity squared.

The thermal performance of the heat exchanger, HX-1, was input from the manufacturer's data as indicated in reference 4.

The expansion tank is modeled by expansion and contraction losses (I.e. area changes, J44 and J45) and tubes, P105 and P106. The tubes have the same dimensions as the actual expansion tank with J47 maintaining atmospheric pressure at J46.

The input to the components in the FATHOM model is shown in reference 6.

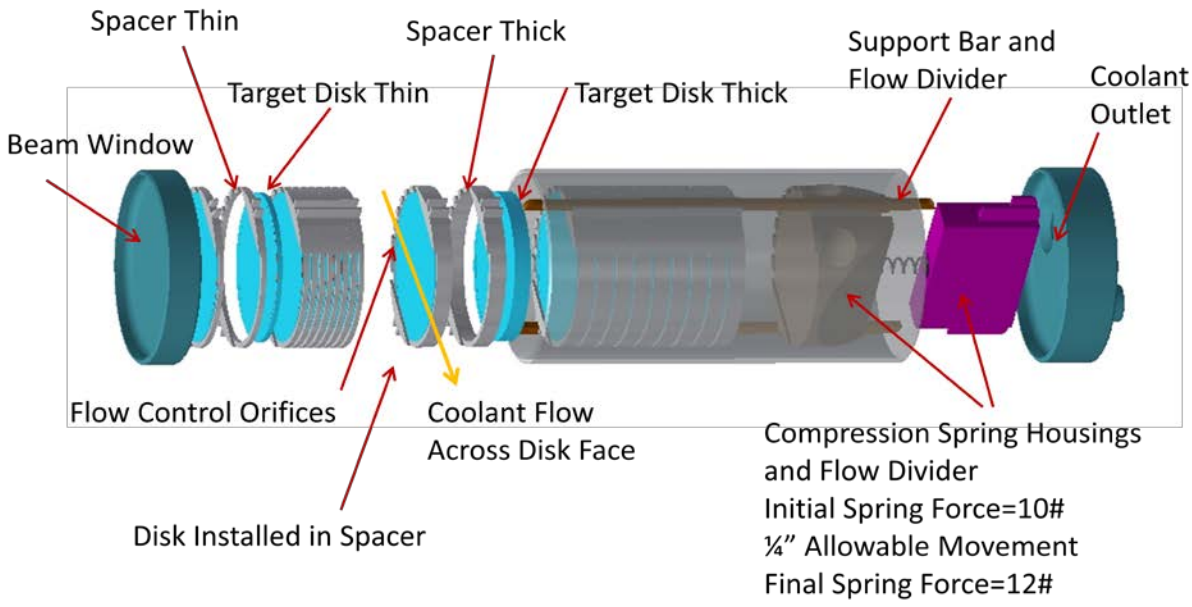
8. Conclusions

The FATHOM results are shown in reference 7. Volume flow rate through the DU Target is indicated as 42gpm [53gpm]. Noting that the throttle valve at the discharge of the pump is set at full open indicates that the system is operating at full flow required capacity. Also, the temperature of the coolant entering the target is indicated as 67F (Inlet to J87) at the target heat load of 16kW. Full heat load is only allowable for the high flow rate provided by the larger pump. Further, considering the values shown in the FATHOM results for the tubes and fitting, all flow velocities and pressure drops are within accepted design practices.

The maximum hydrostatic pressure in the system is determined by the maximum pressure at zero flow as indicated by the pump curve in the appendix. Therefore, assuming that the pressure in the expansion tank

is always at atmospheric pressure the maximum operating pressure of the system is 53psig [72psig]. Also, the maximum operating pressure on the target beam window is 26psig [33psig]. This pressure value can be used for the structural design of the beam window.

9. Figures



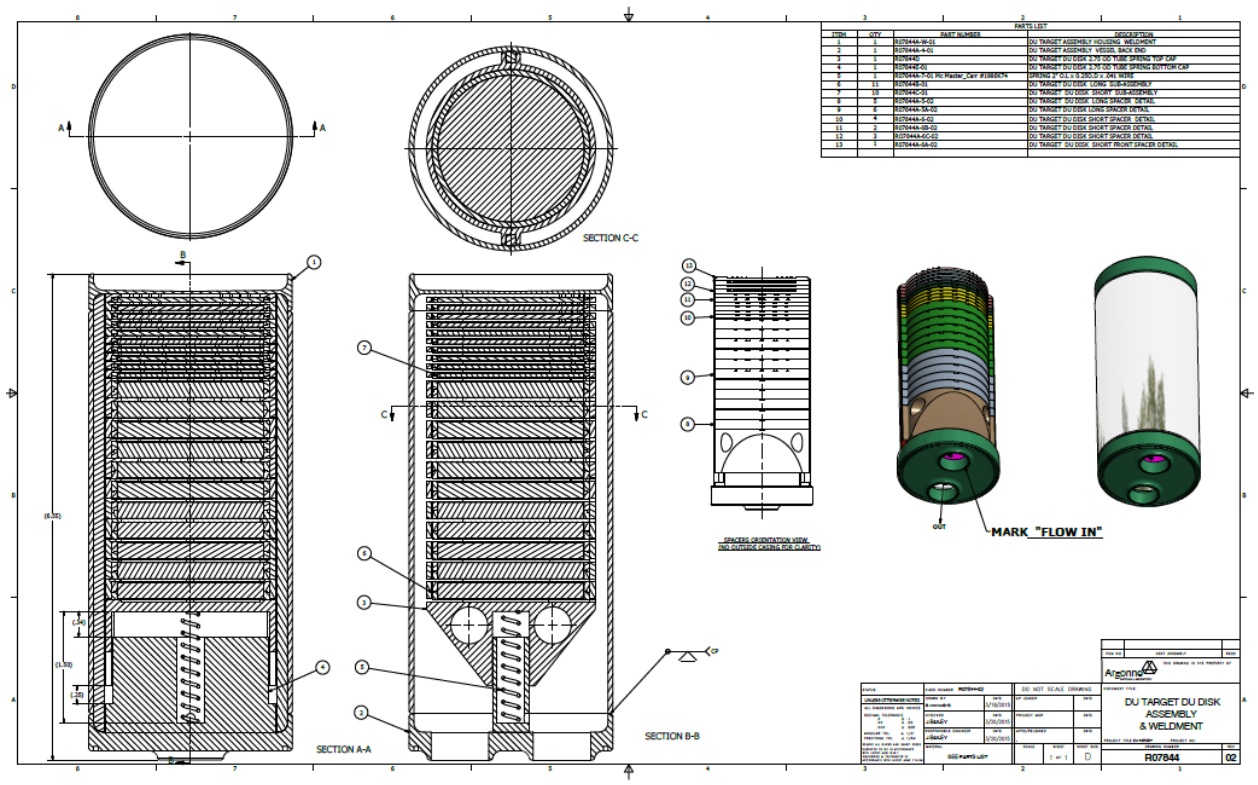


Figure 1 DU Target Assembly

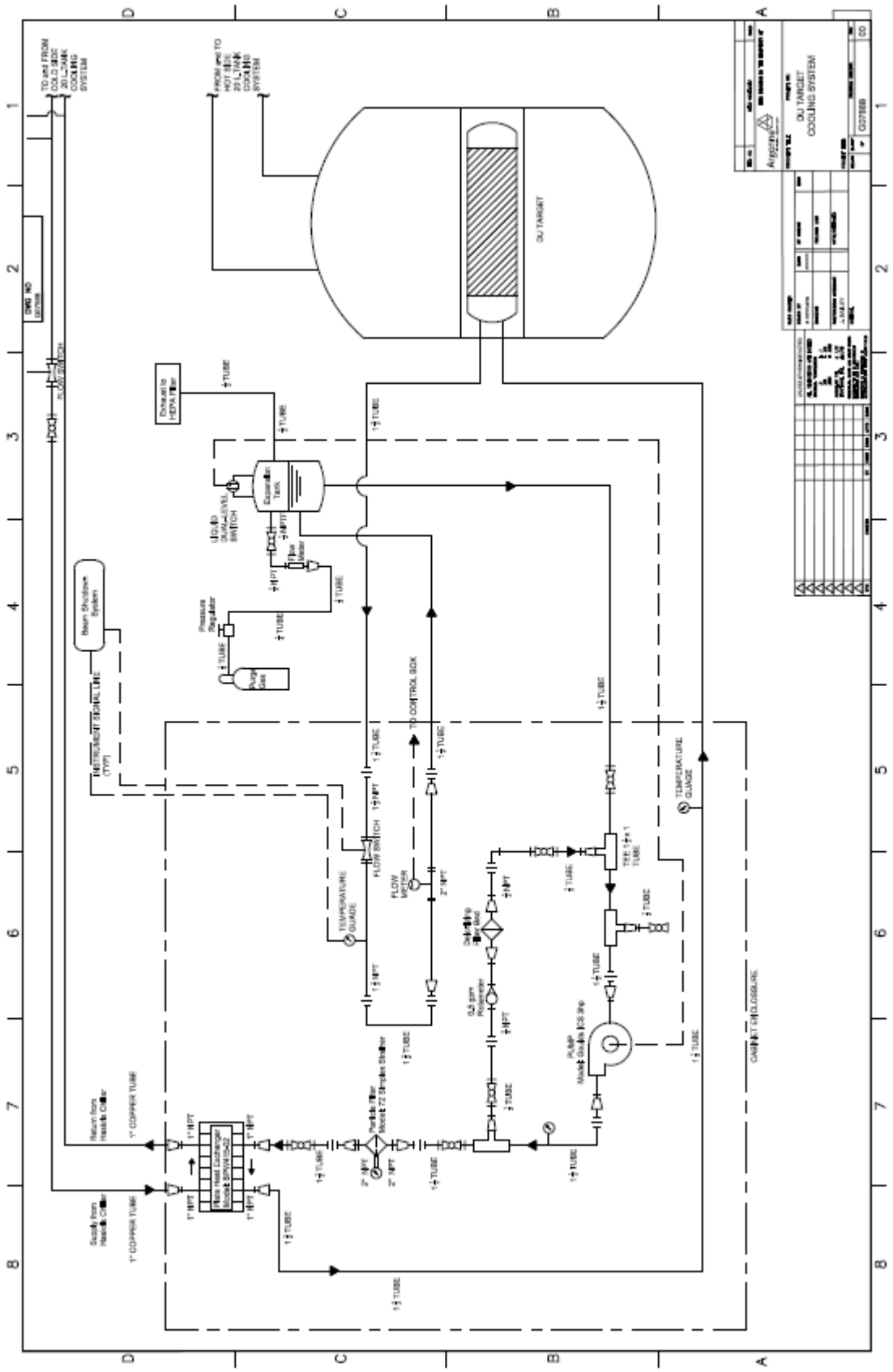


Figure 2 Cooling System P/I

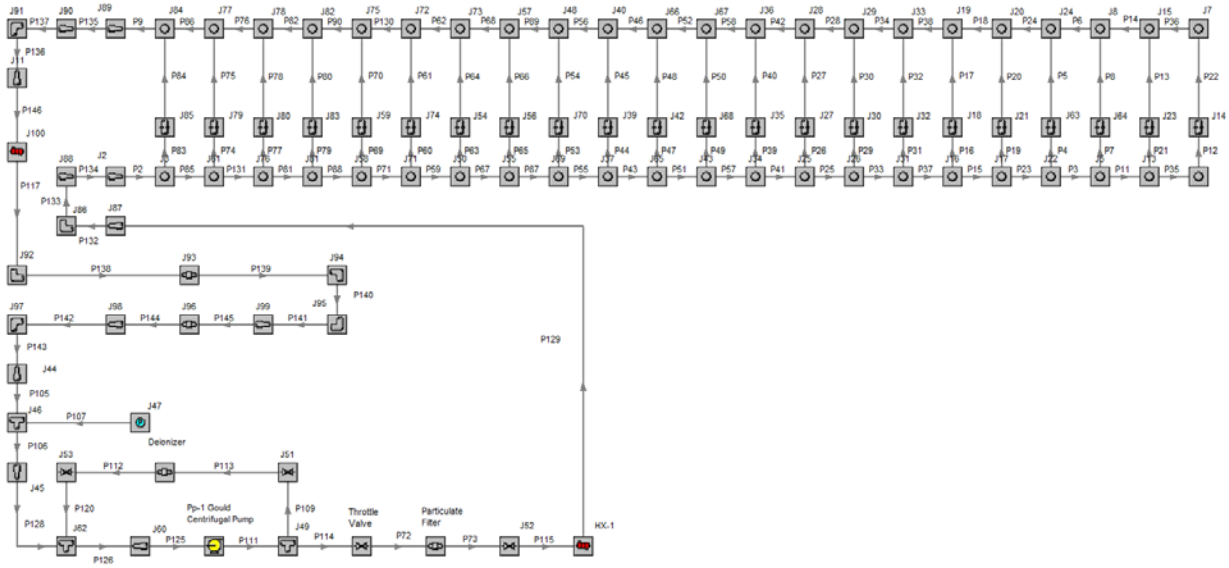


Figure 3 FATHOM Model

APPENDIX 1
GENERAL CHECKING CRITERIA SHEET

CALCULATION CHECKLIST	Yes	No	N/A	Comments
1. Are analytical methods appropriate?	☑	☐	☐	
2. Are assumptions appropriate?	☑	☐	☐	
3. Is the calculation complete?	☑	☐	☐	
4. Are formulas appropriately referenced?	☑	☐	☐	
5. Are the input data appropriate?	☑	☐	☐	
6. Was utilized software appropriate for the task?	☑	☐	☐	
7. Were software input/initial conditions/properties/boundary conditions appropriate?	☑	☐	☐	
8. Are the results reasonable?	☑	☐	☐	

From:	Bailey, James L.
To:	Guarino, Victor
Cc:	Grudzinski, James J.; Strons, Philip S.
Subject:	Response to comments NE-003
Date:	Tuesday, April 25, 2017 11:45:33 AM
Attachments:	NE-CALC-2015-03 Revised-2.docx

Vic,

See below for my response to your comments from NE003.

Jim Bailey
Engineer
Nuclear Engineering Division
Argonne National Laboratory

In reference to comments for NE-003:

Comment; "Several places it is stated that turbulent flow is assumed. The pipe and flow rates are known so why assume and why not just calculate Re?"

Reply; I have found two places in the report were "assumed turbulent" is stated:

1. "In order to develop the complete resistance curve the flow through the strainer was assumed turbulent, and therefore, the pressure drop was assumed proportional to the velocity squared."

The flow condition through a mesh or screen is uncertain, particularly near the entrance where flow is not fully developed. Here Re# is rather useless. E.g. flow through a HEPA filter goes laminar. The assumption of turbulence was used because it is conservative considering pressure drop. Note that the manufacturer has provided a test data point near the design flow that is used for development of the resistance curve. Also, the pressure drop is small compared to the overall system resistance.

2. "In order to develop the complete resistance curve the flow through the heat exchanger was assumed turbulent, and therefore, the pressure drop was assumed proportional to the velocity squared."

The heat exchanger is a standard catalog item with performance specified at only the design condition. The internal design of the exchanger is not known, and therefore, a Re# cannot be calculated. Further, it is almost a certainty that the flow in the exchanger is turbulent in order to provide a reasonable heat transfer coefficient. Here again, the single design point given by the manufacturer was used to develop the full resistance curve.

Comment; "I think it should be made clearer that the flow rates have been confirmed by tests."

Reply; There are three places in this Calc Note that state that the flow rates have been confirmed by tests:

1. Section 3; “The hydraulic performance through the DU target assembly is not part of this calculation note and was determined by flow tests on the actual target as reported in reference 8. The flow resistances as determined by these test were input to this calculation note.”

2. Section 5; “Flow characteristics as determined from the hydraulic tests on the DU Target Assembly (Reference 8) are used as input to the AFT FATHOM overall thermal hydraulic model.”

3. Section 7; “The hydraulic performance was performed considering a 5.8psid [9.0psid] from inlet plenum to outlet plenum at the flow rates indicated below and as determined from the flow tests that were performed on the actual target (Reference 8).”

Also, Reference 8 is the complete flow test report and is provided in the appendix.

I believe this to be enough clarity.

Also, I have corrected an error in the numbering of the Figures. Attached is the revised Calc Note.

Thermal-Hydraulic Analysis of the Overall Performance of the DU Target Cooling System

Appendix 2

Reference 1

Calculation Note: NE-CALC-2015-05

Reference 2

Email from the chiller manufacturer verifying performance characteristics

Hi James,

Thank you for contacting . Attached is the manual that is sent with each of our chillers; since we build each unit to order, it only includes general installation, operation, and maintenance. Below are the specifications of the unit:

Voltage: 208/230V – 3 phase – 60Hz
FLA = 30A
MOCP = 40A

Maximum cooling capacity: 23kW @ 65°F supply water set-point
Condenser: Water-cooled (heat dissipated into secondary source of water)
Refrigerant: R22 (17lbs, 6oz)
Water temperature connections: 65 – 69°F

Pump Capacity: 12.5GPM @ 45psi
Tank size: 30 gallons
Supply and return connections: 3/4" FPT Brass
Condenser water connections: 3/4" FPT Brass

If you have any questions, please let me know.

Regards,

Reference 3

Gould pump information (Small pump)

Good afternoon Jim,

Please see the quotation below per your request. If you have any questions or need any additional assistance please let me know!

Reference Quotation Number: UQ0414308

QTY(1) 1SS1H4A0, Goulds model ICS

- 1 X 1-1/4 - 5
- 316SS
- 3HP, 3500RPM
- 60Hz/1PH/TEFC
- 5-3/8" Impeller Diameter
- Carbon/Silicon Carbide/Viton Mechanical Seal
- Your net price each:
- Typically ships in about 7-10 Days

Prices quoted are net
Quotation is valid for 30 Days
Freight: Pre-Pay & Add or Collect, FOB: Shipping Point
*** standard terms & conditions apply***

(Large pump)

Attn: ACCOUNTS PAYABLE

Requested By: Jim Bailey

PO Number	*Carrier*	*Contact*
	BEST WAY	

Quantities					*Item ID* *Item Description*	*Pricing UOM* *Unit Size*	*Unit Price*	*Extended Price*
Quoted	*Allocated*	*Remaining*	*UOM* *Unit Size*	*Disp.*				
1.00	0.00	1.00	EA		10SH2K55C2	EA 1.0		

Reference 4

Plate heat exchanger information

Quotation No. SA 2014-4-29-10:04

		Quote Date:	4/29/2014
		Date:	4/29/2014 2:02:11 PM
Phone:		Terms:	
Fax:		Freight:	Freight Prepaid and Add
		Job:	ARGONNE LAB HEAT EXCHANGER
TO:			
ATTN:		Engineer:	None Selected

We are pleased to quote you on the following equipment for the above job subject to approval. Quantities listed are not guaranteed and should be verified. Prices will be adjusted accordingly. This quotation is subject to change without notice and void after 30 days unless otherwise stated below. All Contracts or Orders are subject to acceptance by the Company and are contingent upon non-occurrence of strikes or other delays beyond their control. In addition to prices named herein, you are to pay any applicable sales taxes.

Qty	Description & Tag	Wt (lbs)	Net Price Ea.	Total Net Price
	BRAZED PLATE HEAT EXCHANGERS			
1	B&G Model - BPDW415 - 92 Plate Heat Exchanger - Consisting of a Brazed Pack Unit with 92 Plates. Thermal Plates are SA240 S31603 X 0.0157 in. tk. This unit has the following connections: Port-1: 1" NPT Male Thread, Port-2: 1" NPT Male Thread, Port-3: 1" NPT Male Thread, Port-4: 1" NPT Male Thread, Working Pressure: 435 psig. Mounting Options: , ASME CODE: NO. Hot Side: 50 GPM of Water from 67 F to 65 F with 10 psi pressure drop; Cold Side: 10 GPM of Water from 55 F to 65 F.	101		
	Total BRAZED PLATE HEAT EXCHANGERS			

Reference 5

Particle filter information 2” Model 72 Simplex

Jim,

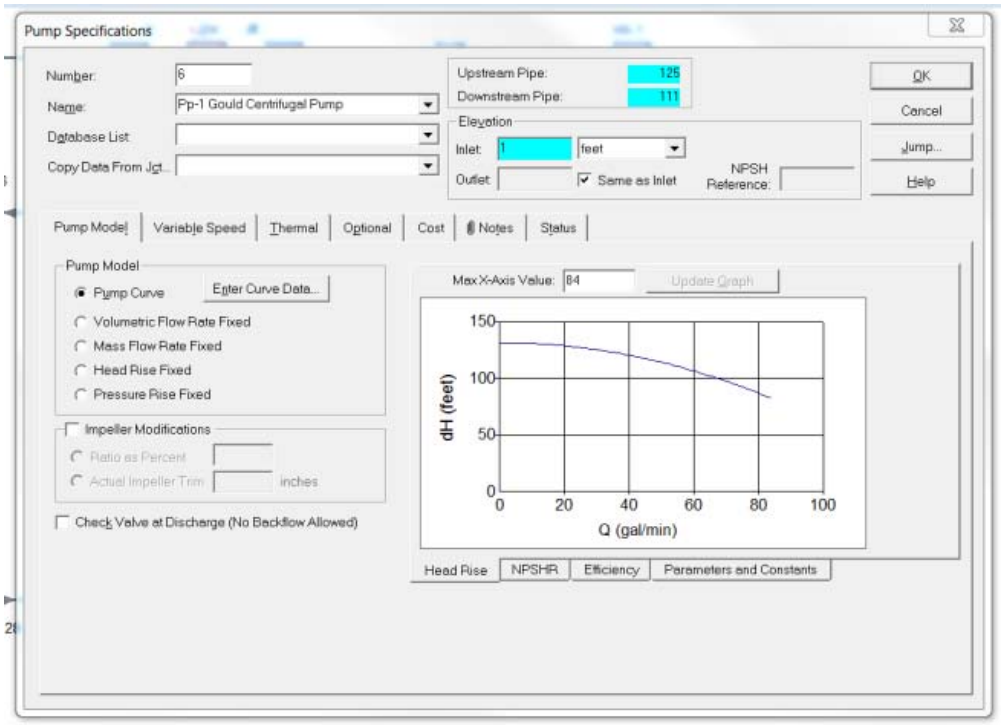
Understand your question , but I decided years ago to standardize their ½" through 2" sizes around a 600# ANSI class to cover most steam applications. There is no lower pressure version offered, and the initial request was for the Y-strainer so the model 85 was quoted, however , we can certainly go to a basket strainer , model 72 Simplex that would have a 0.7 psi initial clean drop at 50 gpm fitted with a 60 mesh screen. Reason is that a 2" model 72 Simplex basket has about 51 sq. in. of gross screen area whereas the Y-strainer has 30.4. The model 72 is also easier for maintenance. Only drawback is that it's not standardly offered with socket weld connections.
I'm assuming the pipe run is horizontal , necessary for the model 72.

The part # for the 316 SS 2" NPT #72 is ST0720200T2C……. 200 PSIG @ 100 deg. F, 2" NPT , Viton seal and includes one 316 SS 60 mesh lined basket……..$ 991.00 net each , same 3 to 5 day lead time to ship.

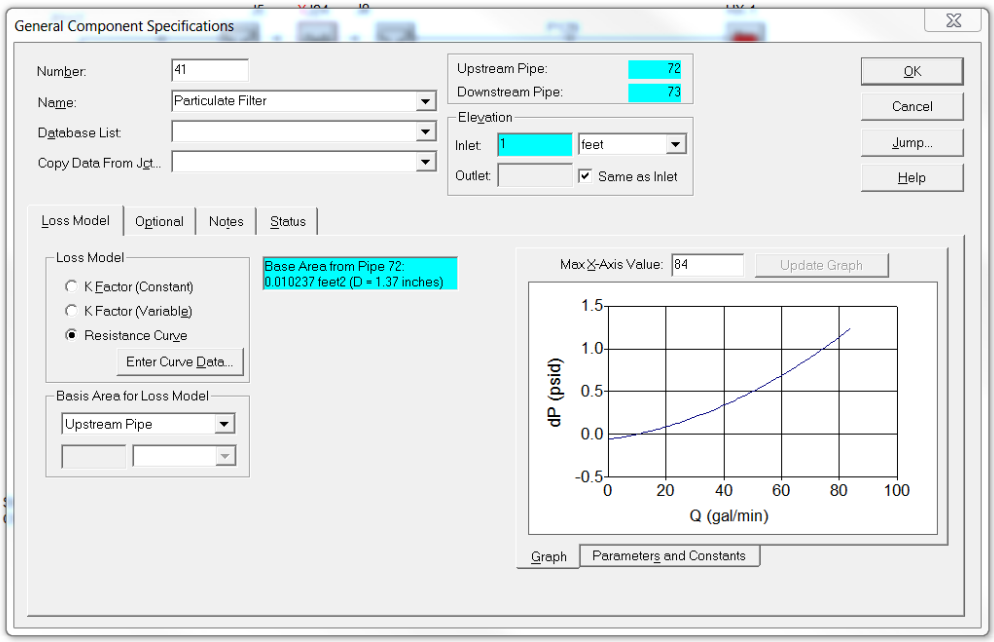
Support data attached. The appropriate curve for the 72 is at the bottom half of page one of the 'Curves' attachment.

Reference 6

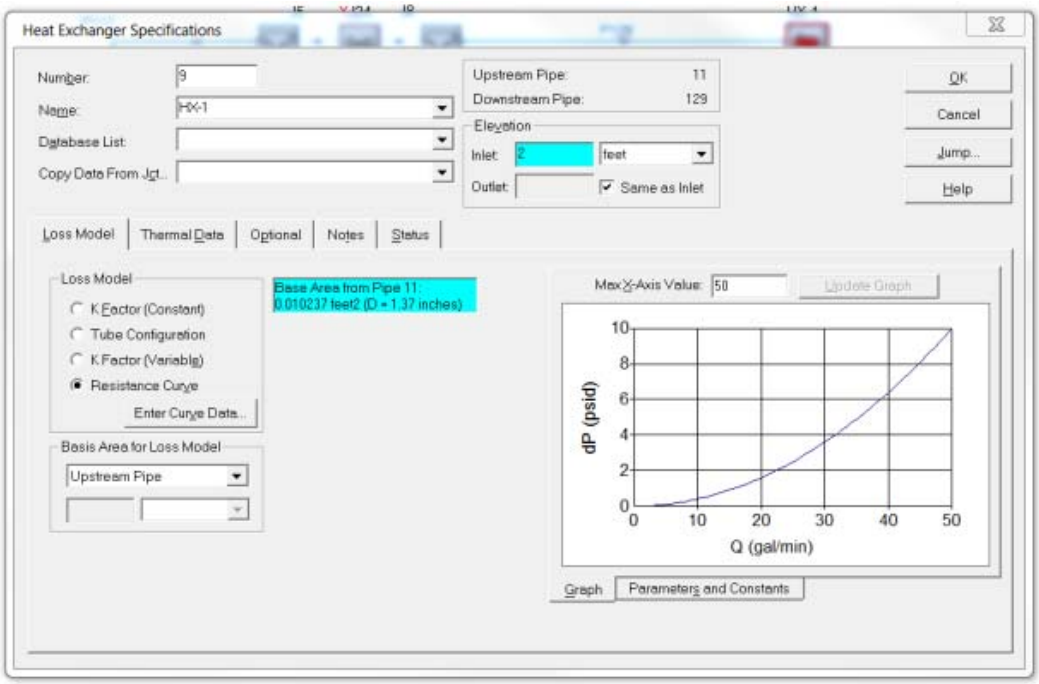
Input to the components in the FATHOM Model



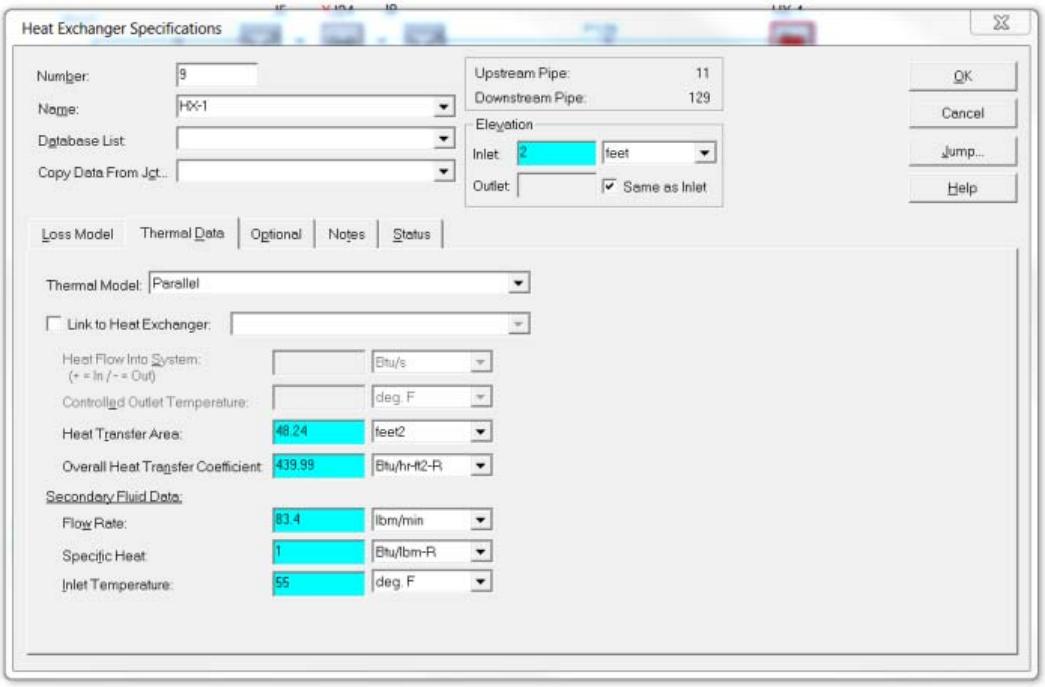
Pump curve



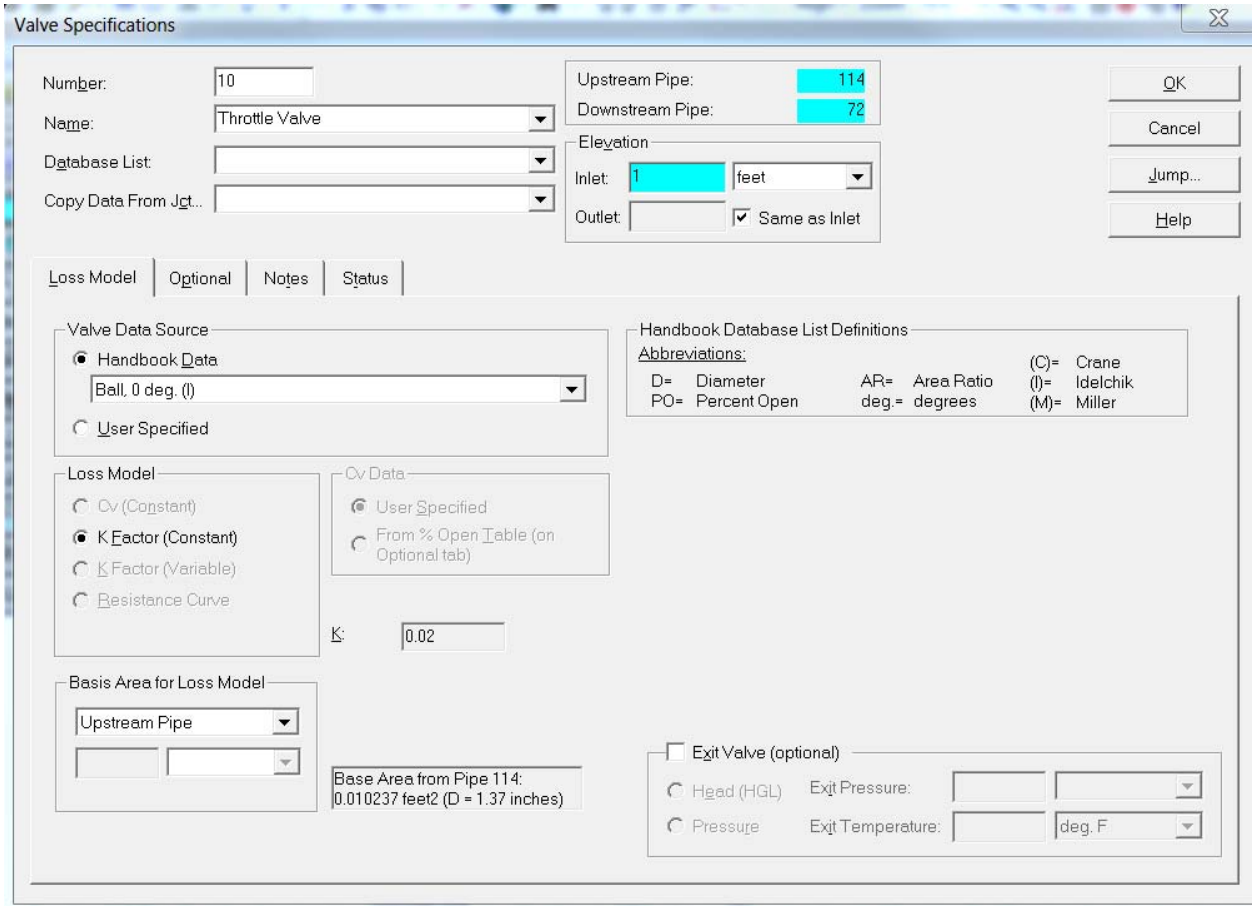
Pressure drop curve for the Particle Filter



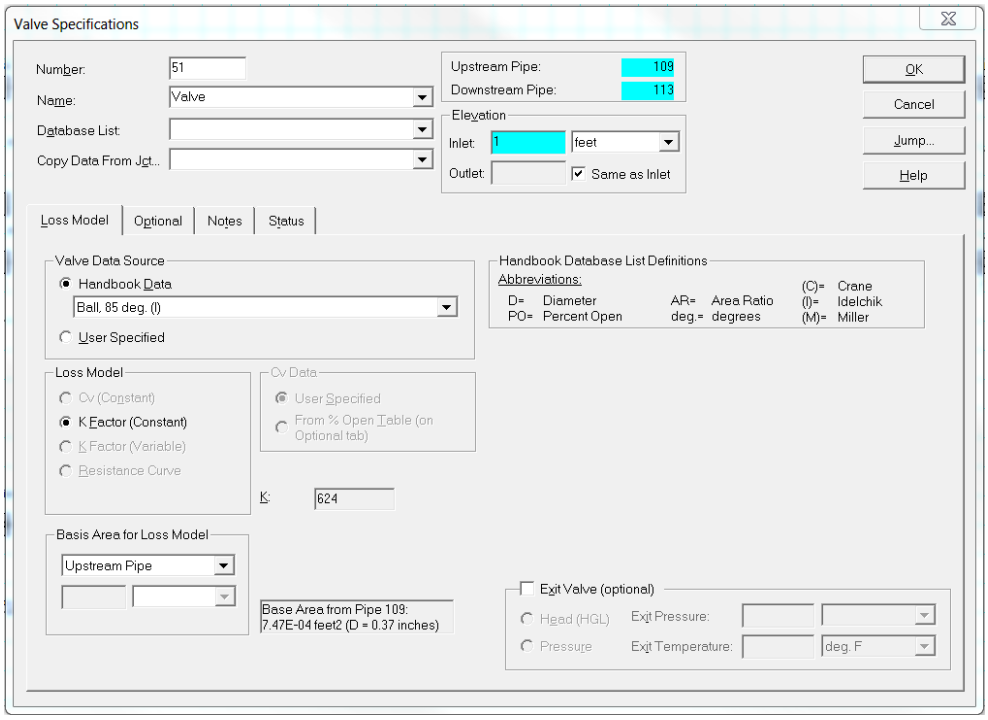
Heat Exchanger Hydraulic Performance Curve



Heat Exchanger Thermal Performance



Throttle Valve at Discharge of Pump



Valve in the DI Bypass Line for Throttling Flow through the DI Unit

Reference 7

Results from the FATHOM Model (Small pump) (Note that the input for tube size and lengths are also noted here)

General

Title: AFT Fathom Model
Analysis run on: 11/17/2015 12:34:47 PM
Application version: AFT Fathom Version 7.0 (2012.11.30)
Input File: P:\Documents\CSE Projects\Acc211\DU Target\Analysis Reports\Mo99 Isotope Production Cooling System DU Target -2.fth
Output File: P:\Documents\CSE Projects\Acc211\DU Target\Analysis Reports\Mo99 Isotope Production Cooling System DU Target -2_1.out

Execution Time= 2.12 seconds
Total Number Of Head/Pressure Iterations= 560
Total Number Of Flow Iterations= 221
Total Number Of Temperature Iterations= 563
Number Of Pipes= 112
Number Of Junctions= 90
Matrix Method= Gaussian Elimination

Pressure/Head Tolerance= 0.0001 relative change
Flow Rate Tolerance= 0.0001 relative change
Temperature Tolerance= 0.0001 relative change
Flow Relaxation= (Automatic)
Flow relaxation automatically lowered to 0.1
Pressure Relaxation= (Automatic)

Heat Transfer with Energy Balance
Fluid Database: AFT Standard
Fluid: Water at 1 atm
Max Fluid Temperature Data= 212 deg. F
Min Fluid Temperature Data= 32 deg. F
Default Temperature= 65 deg. F
Default Density= 62.34301 lbm/ft3
Default Viscosity= 2.53423 lbm/hr-ft
Default Vapor Pressure= 0.30169 psia
Viscosity Model= Newtonian

Atmospheric Pressure= 1 atm
Gravitational Acceleration= 1 g
Turbulent Flow Above Reynolds Number= 4000
Laminar Flow Below Reynolds Number= 2300

Total Inflow= 3.452E-04 lbm/sec
Total Outflow= 3.436E-04 lbm/sec
Total Energy Inflow= 15.57 Btu/s
Total Energy Outflow= 15.57 Btu/s
Maximum Static Pressure is 66.63 psia at Pipe 111 Inlet
Minimum Static Pressure is 14.28 psia at Pipe 128 Inlet
Maximum Static Temperature is 68.94 deg. F at Junction 52 Inlet
Minimum Static Temperature is 65.00 deg. F at Junction 47 Inlet

Pump Summary

Jct	Name	Vol. Flow (gal/min)	Mass Flow (lbm/sec)	dP (psid)	dH (feet)	Overall Efficiency (Percent)	Speed (Percent)	Overall Power (hp)	BEP (gal/min)	% of BEP (Percent)
6	Pp-1 Gould Centrifugal Pump	43.22	6.001	51.29	118.5	56.78	100.0	2.277	55.01	78.58

Jct	NPSHA (feet)	NPSHR (feet)
6	36.01	N/A

Valve Summary

Jct	Name	Valve Type	Vol. Flow (gal/min)	Mass Flow (lbm/sec)	dP Stag. (psid)	dH (feet)	P Inlet Static (psia)	Cv	K	Valve State
10	Throttle Valve	REGULAR	42.071	5.8411	5.187367	11.988946	66.47	18.4630	9.20000	Open
51	Valve	REGULAR	1.156	0.1605	49.919853	115.354574	66.24	0.1635	624.00000	Open

Jct	Name	Valve Type	Vol. Flow (gal/min)	Mass Flow (lbm/sec)	dP Stag. (psid)	dH (feet)	P Inlet Static (psia)	Cv	K	Valve State
52	Valve	REGULAR	42.071	5.8411	0.011277	0.026059	60.73	395.9863	0.02000	Open
53	Valve	REGULAR	1.156	0.1605	0.001600	0.003697	16.13	28.8830	0.02000	Open

Heat Exchanger Summary

Jct	Name	Vol. Flow (gal/min)	Mass Flow (lbm/sec)	dP (psid)	dH (feet)	dT Loss (deg. F)	Heat Rate In (Btu/s)	T Inlet (deg. F)	T Outlet (deg. F)	T 2nd Inlet (deg. F)	T 2nd Outlet (deg. F)
9	HX-1	42.07	5.841	7.080	16.36	2.665	-15.57	68.94	66.27	55.00	66.20
92	Heat Generation	42.06	5.841	0.000	0.00	-2.598	15.18	66.33	68.93	N/A	N/A

Pipe Output Table

Pipe	Name	Vol. Flow Rate (gal/min)	Velocity (feet/sec)	dP Stag. Total (psid)	dP Static Total (psid)	P Static In (psig)	P Static Out (psig)	P Stag. In (psig)	P Stag. Out (psig)	T Inlet (deg. F)
2	Pipe	42.058	22.04849	0.02150172	0.02150172	20.49501419	20.47351456	23.76532745	23.7438	66.27
3	Pipe	14.857	7.78876	0.00377948	0.00377948	22.80838776	22.80461121	23.21648788	23.2127	66.31
4	Pipe	1.710	0.89628	0.00004868	0.00004868	23.21108246	23.21103668	23.21648788	23.2164	66.31
5	Pipe	1.710	7.52124	0.39702490	0.39702490	17.91522598	17.51819992	18.29577255	17.8987	66.31
6	Pipe	14.857	7.78876	0.00377939	0.00377939	17.49442673	17.49064636	17.90252686	17.8987	66.32
7	Pipe	4.851	2.54283	0.00047444	0.00047444	23.16921234	23.16873550	23.21271133	23.2122	66.31
8	Pipe	4.851	21.33848	2.45297241	2.45297241	17.59873962	15.14576721	20.66180801	18.2088	66.31
9	Pipe	42.058	22.04862	0.02138883	0.02138883	14.10114479	14.07975578	17.37147522	17.3501	66.33
11	Pipe	10.007	5.24593	0.00188694	0.00188694	23.02758408	23.02569580	23.21271133	23.2108	66.31
12	Pipe	5.003	2.62282	0.00050047	0.00050047	23.16398239	23.16348267	23.21025848	23.2098	66.31
13	Pipe	5.004	22.01215	2.59189963	2.59189963	17.23678589	14.64488602	20.49631500	17.9044	66.31
14	Pipe	10.007	5.24593	0.00188692	0.00188692	17.71928787	17.71739960	17.90441513	17.9025	66.32
15	Pipe	18.272	9.57887	0.00544870	0.00544870	22.60927582	22.60382462	23.22652054	23.2211	66.31
16	Pipe	1.707	0.89476	0.00004860	0.00004860	23.22113419	23.22108459	23.22652054	23.2265	66.31
17	Pipe	1.707	7.50846	0.39587143	0.39587143	17.94326019	17.54738617	18.32251740	17.9286	66.31
18	Pipe	18.272	9.57888	0.00544846	0.00544846	17.27691841	17.27147102	17.89416504	17.8887	66.32
19	Pipe	1.705	0.89383	0.00004855	0.00004855	23.21569443	23.21564865	23.22106934	23.2210	66.31
20	Pipe	1.705	7.50071	0.39516491	0.39516491	17.94870377	17.55353928	18.32717896	17.9320	66.31
21	Pipe	5.004	2.62311	0.00050057	0.00050057	23.16453552	23.16403580	23.21082306	23.2103	66.31
22	Pipe	5.003	22.00967	2.59143424	2.59143424	17.23756790	14.64613342	20.49636078	17.9049	66.31
23	Pipe	16.567	8.68503	0.00458131	0.00458131	22.71364212	22.70906067	23.22106934	23.2165	66.31
24	Pipe	16.567	8.68504	0.00458115	0.00458115	17.39131927	17.38673782	17.89874649	17.8942	66.32
25	Pipe	23.399	12.26654	0.00845415	0.00845415	22.23652267	22.22806931	23.24874115	23.2403	66.30
26	Pipe	1.714	0.89851	0.00004881	0.00004881	23.24330902	23.24326324	23.24874115	23.2487	66.30
27	Pipe	1.714	7.53997	0.39874700	0.39874700	17.92104340	17.52229309	18.30348969	17.9047	66.30
28	Pipe	23.399	12.26657	0.00845357	0.00845357	16.86273003	16.85427666	17.87496041	17.8665	66.32
29	Pipe	1.711	0.89709	0.00004873	0.00004873	23.23487473	23.23482513	23.24028778	23.2402	66.30
30	Pipe	1.711	7.52800	0.39765397	0.39765397	17.92949295	17.53184128	18.31072617	17.9131	66.30
31	Pipe	1.709	0.89584	0.00004866	0.00004866	23.22750473	23.22745514	23.23290253	23.2329	66.30
32	Pipe	1.709	7.51752	0.39669803	0.39669803	17.93687820	17.54018021	18.31706093	17.9204	66.30
33	Pipe	21.688	11.36946	0.00738560	0.00738560	22.37070847	22.36332321	23.24028778	23.2329	66.30
34	Pipe	21.688	11.36948	0.00738515	0.00738515	17.01275444	17.00536919	17.88233566	17.8750	66.32
35	Pipe	5.003	2.62282	0.00056553	0.00056553	23.16454697	23.16398239	23.21082306	23.2103	66.31
36	Pipe	5.003	2.62282	0.00051233	0.00051233	17.85865021	17.85813904	17.90492630	17.9044	66.31
37	Pipe	19.979	10.47362	0.00638365	0.00638365	22.49495697	22.48857498	23.23290253	23.2265	66.30
38	Pipe	19.979	10.47364	0.00638331	0.00638331	17.15077209	17.14439011	17.88871765	17.8823	66.32
39	Pipe	1.717	0.90013	0.00004890	0.00004890	23.25288010	23.25283051	23.25833130	23.2583	66.30
40	Pipe	1.717	7.55353	0.39998624	0.39998624	17.91145325	17.51146698	18.29527664	17.8953	66.30
41	Pipe	25.113	13.16505	0.00958904	0.00958904	22.09239197	22.08280182	23.25833130	23.2487	66.30

Pipe	Name	Vol. Flow Rate (gal/min)	Velocity (feet/sec)	dP Stag. Total (psid)	dP Static Total (psid)	P Static In (psig)	P Static Out (psig)	P Stag. In (psig)	P Stag. Out (psig)	T Inlet (deg. F)
42	Pipe	25.113	13.16508	0.00958831	0.00958831	16.70055580	16.69096565	17.86649704	17.8569	66.32
43	Pipe	30.289	15.87860	0.03131220	0.03131220	21.64691544	21.61560440	23.34302902	23.3117	66.29
44	Pipe	1.744	0.91428	0.00004967	0.00004967	23.33740616	23.33735657	23.34302902	23.3430	66.29
45	Pipe	1.744	7.67227	0.41089070	0.41089070	17.82672119	17.41583252	18.22270584	17.8118	66.29
46	Pipe	30.289	15.87865	0.03130918	0.03130918	16.10740852	16.07609749	17.80352783	17.7722	66.32
47	Pipe	1.734	0.90907	0.00004939	0.00004939	23.30615997	23.30611038	23.31171799	23.3117	66.29
48	Pipe	1.734	7.62858	0.40686482	0.40686482	17.85805130	17.45118713	18.24953842	17.8427	66.29
49	Pipe	1.725	0.90436	0.00004913	0.00004913	23.27803802	23.27799225	23.28353882	23.2835	66.30
50	Pipe	1.725	7.58906	0.40323606	0.40323606	17.88623810	17.48300171	18.27368164	17.8704	66.30
51	Pipe	28.555	14.96953	0.02817789	0.02817789	21.80425262	21.77607346	23.31171799	23.2835	66.29
52	Pipe	28.555	14.96958	0.02817535	0.02817535	16.32423401	16.29605865	17.83170319	17.8035	66.32
53	Pipe	1.755	0.92000	0.00004998	0.00004998	23.37195206	23.37189865	23.37764740	23.3776	66.29
54	Pipe	1.755	7.72028	0.41533512	0.41533512	17.79207993	17.37674713	18.19303513	17.7777	66.29
55	Pipe	32.033	16.79287	0.03461473	0.03461473	21.48058701	21.44596863	23.37764740	23.3430	66.29
56	Pipe	32.033	16.79293	0.03461118	0.03461118	15.87515068	15.84053993	17.77221680	17.7376	66.32
57	Pipe	26.830	14.06517	0.02520887	0.02520887	21.95271301	21.92750549	23.28353882	23.2583	66.30
58	Pipe	26.830	14.06521	0.02520677	0.02520677	16.52607918	16.50087547	17.85690689	17.8317	66.32
59	Pipe	35.809	18.77235	0.04228093	0.04228093	21.12751770	21.08523941	23.49817276	23.4559	66.28
60	Pipe	1.021	0.53542	0.00002909	0.00002909	23.49624252	23.49621582	23.49817276	23.4981	66.28
61	Pipe	1.021	4.49307	0.10658273	0.10658273	17.60144806	17.49486542	17.73725510	17.6307	66.28
62	Pipe	35.809	18.77244	0.04227568	0.04227568	15.28870010	15.24642563	17.65936661	17.6171	66.33
63	Pipe	1.014	0.53158	0.00002888	0.00002888	23.45399475	23.45396423	23.45589447	23.4559	66.29
64	Pipe	1.014	4.46084	0.10456698	0.10456698	17.64345551	17.53888702	17.77732086	17.6728	66.29
65	Pipe	1.007	0.52791	0.00002868	0.00002868	23.41386414	23.41383743	23.41573715	23.4157	66.29
66	Pipe	1.007	4.43001	0.10266230	0.10266230	17.68336105	17.58069992	17.81538010	17.7127	66.29
67	Pipe	34.795	18.24077	0.04015498	0.04015498	21.21759796	21.17744084	23.45589447	23.4157	66.29
68	Pipe	34.795	18.24085	0.04015033	0.04015033	15.46121025	15.42106056	17.69951630	17.6594	66.33
69	Pipe	1.029	0.53943	0.00002931	0.00002931	23.54069138	23.54066086	23.54264832	23.5426	66.28
70	Pipe	1.029	4.52671	0.10871423	0.10871423	17.55727386	17.44855881	17.69512177	17.5864	66.28
71	Pipe	36.830	19.30777	0.04447257	0.04447257	21.03483200	20.99035645	23.54264832	23.4982	66.28
72	Pipe	42.071	9.15647	0.09042238	0.09042238	46.58879471	46.49837112	47.15263748	47.0622	68.94
73	Pipe	42.071	9.15647	0.09042238	0.09042238	46.12927628	46.03885269	46.69311905	46.6027	68.94
74	Pipe	1.054	0.55250	0.00003002	0.00003002	23.68784332	23.68781281	23.68989563	23.6899	66.28
75	Pipe	1.054	4.63634	0.11585154	0.11585154	17.41110611	17.29525185	17.55570984	17.4399	66.28
76	Pipe	39.942	20.93889	0.05145040	0.05145040	14.52742577	14.47597694	17.47684479	17.4254	66.33
77	Pipe	1.045	0.54797	0.00002978	0.00002978	23.63641739	23.63638687	23.63843918	23.6384	66.28
78	Pipe	1.045	4.59833	0.11334326	0.11334326	17.46217346	17.34882736	17.60441589	17.4911	66.28
79	Pipe	1.037	0.54361	0.00002954	0.00002954	23.58739090	23.58736038	23.58937836	23.5893	66.28
80	Pipe	1.037	4.56180	0.11096551	0.11096551	17.51087189	17.39990616	17.65086365	17.5399	66.28
81	Pipe	38.896	20.39081	0.04905951	0.04905951	20.84138870	20.79232788	23.63843918	23.5894	66.28
82	Pipe	38.896	20.39092	0.04905233	0.04905233	14.72883224	14.67977905	17.52589798	17.4768	66.33
83	Pipe	1.063	0.55720	0.00003028	0.00003028	23.74173737	23.74170685	23.74382782	23.7438	66.27
84	Pipe	1.063	4.67584	0.11849676	0.11849676	17.35760117	17.23910522	17.50468063	17.3862	66.27
85	Pipe	40.995	21.49127	0.05392968	0.05392968	20.63672256	20.58279037	23.74382782	23.6899	66.27
86	Pipe	40.996	21.49139	0.05392107	0.05392107	14.31827545	14.26435471	17.42539597	17.3715	66.33
87	Pipe	33.788	17.71287	0.03809321	0.03809321	21.30512238	21.26703262	23.41573715	23.3776	66.29
88	Pipe	37.859	19.84720	0.04673153	0.04673153	20.93947601	20.89274597	23.58937836	23.5426	66.28
89	Pipe	33.788	17.71294	0.03808910	0.03808910	15.62698364	15.58889389	17.73760605	17.6995	66.33
90	Pipe	37.859	19.84730	0.04672503	0.04672503	14.92270851	14.87598228	17.57262421	17.5259	66.33
105	Pipe	42.071	0.07040	0.00001648	0.00001648	-0.00001717	-0.00003338	0.00001621	0.0000	68.93
106	Pipe	42.071	0.07040	-0.43275058	-0.43275058	-0.00003338	0.43271732	0.00000000	0.4328	68.93
107	Pipe	0.000	0.00000	0.00000000	0.00000000	0.00000000	0.00000000	0.00000000	0.0000	65.00
109	Pipe	1.156	3.44900	0.08090679	0.08090679	51.62053299	51.53962326	51.70053482	51.6196	68.94
111	Pipe	43.224	9.40752	0.09526737	0.09526737	51.93068314	51.83541489	52.52586746	52.4306	68.94
112	Pipe	1.156	3.44900	0.08090679	0.08090679	1.51574898	1.43484116	1.59574890	1.5148	68.94
113	Pipe	1.156	3.44900	0.08090679	0.08090679	1.61977196	1.53886604	1.69977188	1.6189	68.94

Pipe	Name	Vol Flow Rate (gal/min)	Velocity (feet/sec)	dP Stag. Total (psid)	dP Static Total (psid)	P Static In (psig)	P Static Out (psig)	P Stag. In (psig)	P Stag. Out (psig)	T Inlet (deg. F)
114	Pipe	42.071	9.15647	0.09042238	0.09042238	51.86658096	51.77616501	52.43042374	52.3400	68.94
115	Pipe	42.071	9.15647	0.52317375	0.52317375	46.02757645	45.50440216	46.59141922	46.0682	68.94
117	Pipe	42.071	9.15648	5.30923080	5.30923080	5.30060959	-0.00862026	5.86445236	0.5552	68.93
120	Pipe	1.156	3.44900	0.08189765	0.08189765	1.43324089	1.35134315	1.51324081	1.4313	68.94
125	Pipe	43.224	9.27167	0.10084618	0.10084618	0.75816154	0.65731525	1.33628273	1.2354	68.94
126	Pipe	43.224	9.40752	0.09493705	0.09493705	0.83615685	0.74122047	1.43134308	1.3364	68.94
128	Pipe	42.071	9.15648	-1.27888775	-1.27888775	-0.41138744	0.86749935	0.15245628	1.4313	68.93
129	Pipe	42.058	9.15375	1.86673355	1.86673355	38.42479706	36.55806351	38.98847580	37.1217	66.27
130	Pipe	36.830	19.30786	0.04446671	0.04446671	15.10926437	15.06479645	17.61709213	17.5726	66.33
131	Pipe	39.941	20.93877	0.05145827	0.05145827	20.74049377	20.68903732	23.68989563	23.6384	66.28
132	Pipe	42.058	19.86436	1.18063545	1.18063545	34.17090607	32.99026871	36.82539368	35.6448	66.27
133	Pipe	42.058	19.86436	4.72254181	4.72254181	31.73947525	27.01693344	34.39396286	29.6714	66.27
134	Pipe	42.058	28.45761	0.11757526	0.11757526	23.88290405	23.76533127	29.33079529	29.2132	66.27
135	Pipe	42.058	28.45777	0.11755820	0.11755820	8.63183403	8.51427460	14.07975578	13.9622	66.33
136	Pipe	42.058	19.86447	1.18045437	1.18045437	5.00078773	3.82033348	7.65529060	6.4748	66.33
137	Pipe	42.058	19.86447	4.72181749	4.72181749	10.97340775	6.25159073	13.62791061	8.9061	66.33
138	Pipe	42.058	9.15381	0.09106785	0.09106785	5.39183998	5.30077171	5.95552063	5.8645	66.33

Pipe	T Outlet (deg. F)	f	Length (feet)	Hyd. Diameter (inches)
2	66.27	0.02124	0.008333	0.32467
3	66.31	0.02661	0.009417	0.32467
4	66.31	0.02925	0.008333	0.32467
5	66.31	0.03985	0.166667	0.07641
6	66.32	0.02661	0.009417	0.32467
7	66.31	0.03541	0.008333	0.32467
8	66.31	0.03059	0.166667	0.07641
9	66.33	0.02123	0.008333	0.32467
11	66.31	0.02929	0.009417	0.32467
12	66.31	0.03511	0.008333	0.32467
13	66.31	0.03038	0.166667	0.07641
14	66.32	0.02929	0.009417	0.32467
15	66.31	0.02536	0.009417	0.32467
16	66.31	0.02930	0.008333	0.32467
17	66.31	0.03987	0.166667	0.07641
18	66.32	0.02536	0.009417	0.32467
19	66.31	0.02933	0.008333	0.32467
20	66.31	0.03988	0.166667	0.07641
21	66.31	0.03511	0.008333	0.32467
22	66.31	0.03038	0.166667	0.07641
23	66.31	0.02594	0.009417	0.32467
24	66.32	0.02594	0.009417	0.32467
25	66.30	0.02400	0.009417	0.32467
26	66.30	0.02918	0.008333	0.32467
27	66.30	0.03983	0.166667	0.07641
28	66.32	0.02400	0.009417	0.32467
29	66.30	0.02922	0.008333	0.32467
30	66.30	0.03984	0.166667	0.07641
31	66.30	0.02926	0.008333	0.32467
32	66.30	0.03986	0.166667	0.07641
33	66.30	0.02440	0.009417	0.32467
34	66.32	0.02440	0.009417	0.32467
35	66.31	0.03511	0.009417	0.32467
36	66.31	0.03511	0.009417	0.32467
37	66.30	0.02485	0.009417	0.32467
38	66.32	0.02485	0.009417	0.32467

Pipe	T Outlet (deg. F)	f	Length (feet)	Hyd. Diameter (inches)
39	66.30	0.02913	0.008333	0.32467
40	66.30	0.03981	0.166667	0.07641
41	66.30	0.02363	0.009417	0.32467
42	66.32	0.02363	0.009417	0.32467
43	66.29	0.02270	0.022000	0.32467
44	66.29	0.02868	0.008333	0.32467
45	66.29	0.03964	0.166667	0.07641
46	66.32	0.02270	0.022000	0.32467
47	66.29	0.02884	0.008333	0.32467
48	66.29	0.03970	0.166667	0.07641
49	66.30	0.02899	0.008333	0.32467
50	66.30	0.03976	0.166667	0.07641
51	66.29	0.02299	0.022000	0.32467
52	66.32	0.02299	0.022000	0.32467
53	66.29	0.02850	0.008333	0.32467
54	66.29	0.03957	0.166667	0.07641
55	66.29	0.02244	0.022000	0.32467
56	66.32	0.02244	0.022000	0.32467
57	66.30	0.02330	0.022000	0.32467
58	66.32	0.02329	0.022000	0.32467
59	66.28	0.02193	0.022000	0.32467
60	66.28	0.04898	0.008333	0.32467
61	66.28	0.02996	0.166667	0.07641
62	66.33	0.02193	0.022000	0.32467
63	66.29	0.04933	0.008333	0.32467
64	66.29	0.02982	0.166667	0.07641
65	66.29	0.04967	0.008333	0.32467
66	66.29	0.02968	0.166667	0.07641
67	66.29	0.02206	0.022000	0.32467
68	66.33	0.02206	0.022000	0.32467
69	66.28	0.04861	0.008333	0.32467
70	66.28	0.03010	0.166667	0.07641
71	66.28	0.02181	0.022000	0.32467
72	68.94	0.01831	1.000000	1.37000
73	68.94	0.01831	1.000000	1.37000
74	66.28	0.04747	0.008333	0.32467
75	66.28	0.03058	0.166667	0.07641
76	66.33	0.02145	0.022000	0.32467
77	66.28	0.04786	0.008333	0.32467
78	66.28	0.03041	0.166667	0.07641
79	66.28	0.04824	0.008333	0.32467
80	66.28	0.03025	0.166667	0.07641
81	66.28	0.02157	0.022000	0.32467
82	66.33	0.02157	0.022000	0.32467
83	66.27	0.04707	0.008333	0.32467
84	66.27	0.03075	0.166667	0.07641
85	66.27	0.02135	0.022000	0.32467
86	66.33	0.02134	0.022000	0.32467
87	66.29	0.02220	0.022000	0.32467
88	66.28	0.02169	0.022000	0.32467
89	66.33	0.02219	0.022000	0.32467
90	66.33	0.02168	0.022000	0.32467
105	68.93	0.03225	0.250000	15.62400
106	68.93	0.03225	1.250000	15.62400
107	65.00	0.00000	1.000000	0.43000
109	68.94	0.03118	1.000000	0.37000

Pipe	T Outlet (deg. F)	f	Length (feet)	Hyd. Diameter (inches)
111	68.94	0.01821	1.000000	1.37000
112	68.94	0.03118	1.000000	0.37000
113	68.94	0.03118	1.000000	0.37000
114	68.94	0.01831	1.000000	1.37000
115	68.94	0.01831	1.000000	1.37000
117	68.93	0.01831	30.000000	1.37000
120	68.94	0.03118	1.000000	0.37000
125	68.94	0.02006	1.000000	1.38000
126	68.94	0.01821	1.000000	1.37000
128	68.93	0.01831	5.000000	1.37000
129	66.27	0.01845	30.000000	1.37000
130	66.33	0.02181	0.022000	0.32467
131	66.28	0.02146	0.022000	0.32467
132	66.27	0.01723	2.000000	0.93000
133	66.27	0.01723	8.000000	0.93000
134	66.27	0.01677	0.083333	0.77700
135	66.33	0.01677	0.083333	0.77700
136	66.33	0.01723	2.000000	0.93000
137	66.33	0.01723	8.000000	0.93000
138	66.33	0.01844	1.000000	1.37000

All Junction Table

Jct	Name	Vol. Flow Rate Thru Jct (gal/min)	dP Stag. Total (psid)	dP Static Total (psid)	P Static In (psia)	P Static Out (psia)	P Stag. In (psia)	P Stag. Out (psia)	T Inlet (deg. F)
2	Area Change	42.058	5.4478931	3.2703140	38.46	35.19	43.91	38.46	66.27
3	Branch	N/A	0.0000000	0.0000000	36.99	36.99	38.44	38.44	66.27
4	Branch	5.003	0.0000000	0.0000000	37.86	37.86	37.91	37.91	66.31
5	Branch	N/A	0.0000000	0.0000000	37.73	37.73	37.91	37.91	66.31
6	Pp-1 Gould Centrifugal Pump	43.224	-51.2904282	-51.2733650	15.35	66.63	15.93	67.22	68.94
7	Branch	5.003	0.0000000	-3.2125158	29.34	32.55	32.60	32.60	66.31
8	Branch	N/A	See Mult. Losses	N/A	31.71	31.71	32.60	32.60	66.32
9	HX-1	42.071	7.0797696	7.0796022	60.20	53.12	60.76	53.68	68.94
10	Throttle Valve	42.071	5.1873674	5.1873674	66.47	61.28	67.04	61.85	68.94
11	Area Change	42.058	0.5193158	-1.5715067	18.52	20.09	21.17	20.65	66.33
13	Branch	N/A	0.0000000	0.0000000	37.82	37.82	37.91	37.91	66.31
14	Orifice	5.003	2.7133958	5.9259119	37.86	31.93	37.91	35.19	66.31
15	Branch	N/A	0.0000000	0.0000000	31.93	31.93	32.60	32.60	66.31
16	Branch	N/A	0.0000000	0.0000000	37.59	37.59	37.92	37.92	66.30
17	Branch	N/A	0.0000000	0.0000000	37.64	37.64	37.92	37.92	66.31
18	Orifice	1.707	4.9039555	5.2778249	37.92	32.64	37.92	33.02	66.31
19	Branch	N/A	See Mult. Losses	N/A	32.02	32.02	32.58	32.58	66.32
20	Branch	N/A	See Mult. Losses	N/A	32.09	32.09	32.59	32.59	66.32
21	Orifice	1.705	4.8938427	5.2669415	37.91	32.64	37.92	33.02	66.31
22	Branch	N/A	0.0000000	0.0000000	37.69	37.69	37.91	37.91	66.31
23	Orifice	5.004	2.7140083	5.9272490	37.86	31.93	37.91	35.19	66.31
24	Branch	N/A	0.0000000	0.0000000	32.16	32.16	32.59	32.59	66.32
25	Branch	N/A	0.0000000	0.0000000	37.43	37.43	37.94	37.94	66.30
26	Branch	N/A	0.0000000	0.0000000	37.49	37.49	37.94	37.94	66.30
27	Orifice	1.714	4.9452057	5.3222198	37.94	32.62	37.94	33.00	66.30
28	Branch	N/A	See Mult. Losses	N/A	31.75	31.75	32.56	32.56	66.32
29	Branch	N/A	See Mult. Losses	N/A	31.84	31.84	32.57	32.57	66.32
30	Orifice	1.711	4.9295125	5.3053308	37.93	32.63	37.94	33.01	66.30
31	Branch	N/A	0.0000000	0.0000000	37.54	37.54	37.93	37.93	66.30
32	Orifice	1.709	4.9156039	5.2905769	37.92	32.63	37.93	33.01	66.30

Jct	Name	Vol. Flow Rate Thru Jct (gal/min)	dP Stag. Total (psid)	dP Static Total (psid)	P Static In (psia)	P Static Out (psia)	P Stag. In (psia)	P Stag. Out (psia)	T Inlet (deg. F)
33	Branch	N/A	See Mult. Losses	N/A	31.93	31.93	32.58	32.58	66.32
34	Branch	N/A	0.0000000	0.0000000	37.36	37.36	37.95	37.95	66.30
35	Orifice	1.717	4.9630060	5.3413773	37.95	32.61	37.95	32.99	66.30
36	Branch	N/A	See Mult. Losses	N/A	31.65	31.65	32.55	32.55	66.32
37	Branch	N/A	0.0000000	0.0000000	37.20	37.20	38.04	38.04	66.29
38	Deionizer	1.156	0.0231173	0.0231173	16.23	16.21	16.31	16.29	68.94
39	Orifice	1.744	5.1202750	5.5106363	38.03	32.52	38.04	32.92	66.29
40	Branch	N/A	See Mult. Losses	N/A	31.25	31.25	32.47	32.47	66.32
41	Particulate Filter	42.071	0.3690963	0.3690963	61.19	60.83	61.76	61.39	68.94
42	Orifice	1.734	5.0621295	5.4480581	38.00	32.55	38.01	32.95	66.29
43	Branch	N/A	0.0000000	0.0000000	37.31	37.31	37.98	37.98	66.29
44	Area Change	42.071	0.5552060	-0.0086038	14.69	14.70	15.25	14.70	68.93
45	Area Change	42.071	0.2802944	0.8441046	15.13	14.28	15.13	14.85	68.93
46	Tee or Wye	N/A	See Mult. Losses	N/A	14.70	14.70	14.70	14.70	68.93
47	Assigned Pressure	0.000	0.0000000	0.0000000	14.70	14.70	14.70	14.70	65.00
48	Branch	N/A	See Mult. Losses	N/A	31.10	31.10	32.43	32.43	66.32
49	Tee or Wye	N/A	See Mult. Losses	N/A	66.76	66.76	67.13	67.13	68.94
50	Branch	N/A	0.0000000	0.0000000	37.10	37.10	38.15	38.15	66.28
51	Valve	1.156	49.9198532	49.9198532	66.24	16.32	66.32	16.40	68.94
52	Valve	42.071	0.0112769	0.0112769	60.73	60.72	61.30	61.29	68.94
53	Valve	1.156	0.0016000	0.0016000	16.13	16.13	16.21	16.21	68.94
54	Orifice	1.014	5.6785450	5.8105083	38.15	32.34	38.15	32.47	66.29
55	Branch	N/A	0.0000000	0.0000000	37.12	37.12	38.11	38.11	66.29
56	Orifice	1.007	5.6003289	5.7304745	38.11	32.38	38.11	32.51	66.29
57	Branch	N/A	See Mult. Losses	N/A	31.18	31.18	32.40	32.40	66.32
58	Branch	N/A	0.0000000	0.0000000	37.06	37.06	38.24	38.24	66.28
59	Orifice	1.029	5.8474951	5.9833846	38.24	32.25	38.24	32.39	66.28
60	Area Change	43.224	0.0001241	-0.0169410	15.44	15.45	16.03	16.03	68.94
61	Branch	N/A	0.0000000	0.0000000	37.00	37.00	38.39	38.39	66.27
62	Tee or Wye	N/A	0.0000000	0.0000000	15.77	15.77	16.13	16.13	68.93
63	Orifice	1.710	4.9208676	5.2968117	37.91	32.61	37.91	32.99	66.31
64	Orifice	4.851	2.5504286	5.5699997	37.86	32.29	37.91	35.36	66.31
65	Branch	N/A	0.0000000	0.0000000	37.25	37.25	38.01	38.01	66.29
66	Branch	N/A	See Mult. Losses	N/A	31.39	31.39	32.50	32.50	66.32
67	Branch	N/A	See Mult. Losses	N/A	31.52	31.52	32.53	32.53	66.32
68	Orifice	1.725	5.0096100	5.3917499	37.97	32.58	37.98	32.97	66.30
69	Branch	N/A	0.0000000	0.0000000	37.14	37.14	38.07	38.07	66.29
70	Orifice	1.755	5.1845589	5.5798211	38.07	32.49	38.07	32.89	66.29
71	Branch	N/A	0.0000000	0.0000000	37.08	37.08	38.19	38.19	66.28
72	Branch	N/A	See Mult. Losses	N/A	30.96	30.96	32.31	32.31	66.33
73	Branch	N/A	See Mult. Losses	N/A	31.07	31.07	32.36	32.36	66.33
74	Orifice	1.021	5.7608914	5.8947687	38.19	32.30	38.19	32.43	66.28
75	Branch	N/A	See Mult. Losses	N/A	30.84	30.84	32.27	32.27	66.33
76	Branch	N/A	0.0000000	0.0000000	37.02	37.02	38.33	38.33	66.28
77	Branch	N/A	See Mult. Losses	N/A	30.47	30.47	32.12	32.12	66.33
78	Branch	N/A	See Mult. Losses	N/A	30.60	30.60	32.17	32.17	66.33
79	Orifice	1.054	6.1341581	6.2767096	38.38	32.11	38.39	32.25	66.28
80	Orifice	1.045	6.0339942	6.1742177	38.33	32.16	38.33	32.30	66.28
81	Branch	N/A	0.0000000	0.0000000	37.04	37.04	38.29	38.29	66.28
82	Branch	N/A	See Mult. Losses	N/A	30.72	30.72	32.22	32.22	66.33
83	Orifice	1.037	5.9384856	6.0764899	38.28	32.21	38.29	32.35	66.28
84	Branch	N/A	See Mult. Losses	N/A	30.33	30.33	32.07	32.07	66.33
85	Orifice	1.063	6.2391162	6.3841066	38.44	32.05	38.44	32.20	66.27
86	Bend	42.058	1.2507950	1.2507950	47.69	46.44	50.34	49.09	66.27
87	Area Change	42.058	0.2963510	2.3871624	51.25	48.87	51.82	51.52	66.27
88	Area Change	42.058	0.3406229	3.1340277	41.71	38.58	44.37	44.03	66.27

Jct	Name	Vol. Flow Rate Thru Jct (gal/min)	dP Stag. Total (psid)	dP Static Total (psid)	P Static In (psia)	P Static Out (psia)	P Stag. In (psia)	P Stag. Out (psia)	T Inlet (deg. F)
89	Area Change	42.058	3.2703311	5.4479218	28.78	23.33	32.05	28.78	66.33
90	Area Change	42.058	0.3342863	-2.4591331	23.21	25.67	28.66	28.32	66.33
91	Bend	42.058	1.2508016	1.2508016	20.95	19.70	23.60	22.35	66.33
92	Heat Generation	42.058	0.0000000	0.0001633	20.00	20.00	20.56	20.56	66.33

Jct	T Outlet (deg. F)	Loss Factor (K)
2	66.27	1.0000000
3	66.27	0.0000000
4	66.31	0.0000000
5	66.31	0.0000000
6	68.94	0.0000000
7	66.31	0.0000000
8	66.32	See Mult. Losses
9	66.27	12.5562496
10	68.94	9.1999998
11	66.33	0.1956359
13	66.31	0.0000000
14	66.31	58.6336441
15	66.31	0.0000000
16	66.30	0.0000000
17	66.31	0.0000000
18	66.31	910.5529785
19	66.32	See Mult. Losses
20	66.32	See Mult. Losses
21	66.31	910.5529785
22	66.31	0.0000000
23	66.31	58.6336441
24	66.32	0.0000000
25	66.30	0.0000000
26	66.30	0.0000000
27	66.30	910.5529785
28	66.32	See Mult. Losses
29	66.32	See Mult. Losses
30	66.30	910.5529785
31	66.30	0.0000000
32	66.30	910.5529785
33	66.32	See Mult. Losses
34	66.30	0.0000000
35	66.30	910.5529785
36	66.32	See Mult. Losses
37	66.29	0.0000000
38	68.94	0.2889668
39	66.29	910.5529785
40	66.32	See Mult. Losses
41	68.94	0.6546068
42	66.29	910.5529785
43	66.29	0.0000000
44	68.93	0.9846816
45	68.93	8,408.9638672
46	68.93	See Mult. Losses
47	65.00	0.0000000
48	66.32	See Mult. Losses
49	68.94	See Mult. Losses
50	66.28	0.0000000
51	68.94	624.0000000

Jct	T Outlet (deg. F)	Loss Factor (K)
52	68.94	0.0200000
53	68.94	0.0200000
54	66.29	2,987.1892090
55	66.29	0.0000000
56	66.29	2,987.1892090
57	66.32	See Mult. Losses
58	66.28	0.0000000
59	66.28	2,987.1892090
60	68.94	0.0002085
61	66.27	0.0000000
62	68.93	0.0000000
63	66.31	910.5529785
64	66.31	58.6336441
65	66.29	0.0000000
66	66.32	See Mult. Losses
67	66.32	See Mult. Losses
68	66.30	910.5529785
69	66.29	0.0000000
70	66.29	910.5529785
71	66.28	0.0000000
72	66.33	See Mult. Losses
73	66.33	See Mult. Losses
74	66.28	2,987.1892090
75	66.33	See Mult. Losses
76	66.28	0.0000000
77	66.33	See Mult. Losses
78	66.33	See Mult. Losses
79	66.28	2,987.1892090
80	66.28	2,987.1892090
81	66.28	0.0000000
82	66.33	See Mult. Losses
83	66.28	2,987.1892090
84	66.33	See Mult. Losses
85	66.27	2,987.1892090
86	66.27	0.4712000
87	66.27	0.5257463
88	66.27	0.1283196
89	66.33	1.0000000
90	66.33	0.0613603
91	66.33	0.4712000
92	68.93	0.0000000

Junction Loss Table

Jct	Pipe #	Pipe Dir.	dP Stag. Total (psid)	Loss Factor (K)
8	P6	Out	0.000	0.000
	P8	In	0.3063	0.1000
	P14	In	0.000	0.000
19	P17	In	0.03793	0.1000
	P18	In	0.000	0.000
	P38	Out	0.000	0.000
20	P18	Out	0.000	0.000
	P20	In	0.03785	0.1000
	P24	In	0.000	0.000
28	P27	In	0.03824	0.1000

Jct	Pipe #	Pipe Dir.	dP Stag. Total (psid)	Loss Factor (K)
	P28	In	0.000	0.000
	P42	Out	0.000	0.000
29	P28	Out	0.000	0.000
	P30	In	0.03812	0.1000
	P34	In	0.000	0.000
33	P32	In	0.03802	0.1000
	P34	Out	0.000	0.000
	P38	In	0.000	0.000
36	P40	In	0.03838	0.1000
	P42	In	0.000	0.000
	P58	Out	0.000	0.000
40	P45	In	0.03960	0.1000
	P46	In	0.000	0.000
	P56	Out	0.000	0.000
46	P106	Out	1.333E-11	4.000E-07
	P105	In	0.000	0.000
	P107	In	0.000	0.000
48	P54	In	0.04010	0.1000
	P56	In	0.000	0.000
	P89	Out	0.000	0.000
49	P114	Out	1.702E-04	3.019E-04
	P111	In	0.000	0.000
	P109	Out	0.7301	9.126
57	P66	In	0.01320	0.1000
	P68	Out	0.000	0.000
	P89	In	0.000	0.000
66	P46	Out	0.000	0.000
	P48	In	0.03915	0.1000
	P52	In	0.000	0.000
67	P50	In	0.03874	0.1000
	P52	Out	0.000	0.000
	P58	In	0.000	0.000
72	P61	In	0.01358	0.1000
	P62	In	0.000	0.000
	P130	Out	0.000	0.000
73	P62	Out	0.000	0.000
	P64	In	0.01339	0.1000
	P68	In	0.000	0.000
75	P70	In	0.01378	0.1000
	P90	Out	0.000	0.000
	P130	In	0.000	0.000
77	P75	In	0.01446	0.1000
	P76	In	0.000	0.000
	P86	Out	0.000	0.000
78	P76	Out	0.000	0.000
	P78	In	0.01422	0.1000
	P82	In	0.000	0.000
82	P80	In	0.01400	0.1000
	P82	Out	0.000	0.000
	P90	In	0.000	0.000
84	P9	Out	0.000	0.000
	P84	In	0.01471	0.1000
	P86	In	0.000	0.000

Large pump

General

Title: AFT Fathom Model
Analysis run on: 2/28/2017 2:14:00 PM
Application version: AFT Fathom Version 7.0 (2012.11.30)
Input File: P:\Documents\CSE Projects\Acc211\DU Target\Test and Assembly\Procedures\FullSystem switch-meter new pump.fth
Output File: P:\Documents\CSE Projects\Acc211\DU Target\Test and Assembly\Procedures\FullSystem switch-meter new pump_1.out

Execution Time= 1.29 seconds
Total Number Of Head/Pressure Iterations= 277
Total Number Of Flow Iterations= 53
Total Number Of Temperature Iterations= 364
Number Of Pipes= 120
Number Of Junctions= 98
Matrix Method= Gaussian Elimination

Pressure/Head Tolerance= 0.0001 relative change
Flow Rate Tolerance= 0.0001 relative change
Temperature Tolerance= 0.0001 relative change
Flow Relaxation= (Automatic)
Pressure Relaxation= (Automatic)

Heat Transfer with Energy Balance
Fluid Database: AFT Standard
Fluid: Water at 1 atm
Max Fluid Temperature Data= 212 deg. F
Min Fluid Temperature Data= 32 deg. F
Default Temperature= 65 deg. F
Default Density= 62.34301 lbm/ft3
Default Viscosity= 2.53423 lbm/hr-ft
Default Vapor Pressure= 0.30169 psia
Viscosity Model= Newtonian

Atmospheric Pressure= 1 atm
Gravitational Acceleration= 1 g
Turbulent Flow Above Reynolds Number= 4000
Laminar Flow Below Reynolds Number= 2300

Total Inflow= 2.030E-03 lbm/sec
Total Outflow= 2.029E-03 lbm/sec
Total Energy Inflow= 16.68 Btu/s
Total Energy Outflow= 16.68 Btu/s
Maximum Static Pressure is 98.99 psia at Pipe 111 Inlet
Minimum Static Pressure is 13.79 psia at Pipe 128 Inlet
Maximum Static Temperature is 69.36 deg. F at Junction 52 Inlet
Minimum Static Temperature is 65.00 deg. F at Junction 47 Inlet

Warnings

WARNING HGL, EGL and head loss results may not be meaningful for variable density systems.

Pump Summary

Jct	Name	Vol. Flow (gal/min)	Mass Flow (lbm/sec)	dP (psid)	dH (feet)	Overall Efficiency (Percent)	Speed (Percent)	Overall Power (hp)	BEP (gal/min)	% of BEP (Percent)
6	Pp-1 Gould Centrifugal Pump	54.43	7.556	84.50	195.3	30.89	100.0	8.685	215.6	25.25

Jct	NPSHA (feet)	NPSHR (feet)
6	34.85	N/A

Valve Summary

Jct	Name	Valve Type	Vol. Flow (gal/min)	Mass Flow (lbm/sec)	dP Stag. (psid)	dH (feet)	P Inlet Static (psia)	Cv	K	Valve State
10	Throttle Valve	REGULAR	52.933	7.3488	8.211288	18.975554	98.76	18.4630	9.20000	Open
51	Valve	REGULAR	1.485	0.2061	82.363846	190.335493	98.37	0.1635	624.00000	Open
52	Valve	REGULAR	52.933	7.3488	0.017851	0.041251	89.73	395.9863	0.02000	Open
53	Valve	REGULAR	1.485	0.2061	0.002640	0.006100	15.73	28.8830	0.02000	Open

Heat Exchanger Summary

Jct	Name	Vol. Flow (gal/min)	Mass Flow (lbm/sec)	dP (psid)	dH (feet)	dT Loss (deg. F)	Heat Rate In (Btu/s)	T Inlet (deg. F)	T Outlet (deg. F)	T 2nd Inlet (deg. F)	T 2nd Outlet (deg. F)
9	HX-1	52.93	7.349	11.21	25.90	2.270	-16.68	69.36	67.09	55.00	67.00
100	Heat Exchanger	52.92	7.349	0.00	0.00	-2.065	15.18	67.28	69.34	N/A	N/A

Pipe Output Table

Pipe	Name	Vol. Flow Rate (gal/min)	Velocity (feet/sec)	P Static Max (psig)	P Static Min (psig)	Elevation Inlet (feet)	Elevation Outlet (feet)	dP Stag. Total (psid)	dP Static Total (psid)	dP Gravity (psid)
2	Pipe	52.919	27.7421	34.9533157	34.9222832	0.000	0.000	0.03103379	0.03103379	0.0000
3	Pipe	15.420	8.0838	38.8692207	38.8652000	0.000	0.000	0.00402333	0.00402333	0.0000
4	Pipe	5.143	2.6963	39.2598783	39.2593536	0.000	0.000	0.00052308	0.00052308	0.0000
5	Pipe	5.143	22.6265	32.9972115	30.2840843	0.000	0.000	2.71312714	2.71312714	0.0000
6	Pipe	15.420	8.0838	29.8485413	29.8445168	0.000	0.000	0.00402310	0.00402310	0.0000
7	Pipe	5.141	2.6951	39.2559013	39.2553787	0.000	0.000	0.00052265	0.00052265	0.0000
8	Pipe	5.141	22.6162	32.9989700	30.2881012	0.000	0.000	2.71086621	2.71086621	0.0000
9	Pipe	52.920	27.7425	24.3169479	24.2846375	0.000	0.000	0.03230901	0.03230901	0.0000
11	Pipe	10.279	5.3887	39.1094322	39.1074600	0.000	0.000	0.00197130	0.00197130	0.0000
12	Pipe	5.139	2.6943	39.2533684	39.2528458	0.000	0.000	0.00052238	0.00052238	0.0000
13	Pipe	5.140	22.6112	32.9998093	30.2900696	0.000	0.000	2.70973730	2.70973730	0.0000
14	Pipe	10.279	5.3887	30.0947418	30.0927734	0.000	0.000	0.00197123	0.00197123	0.0000
15	Pipe	22.722	11.9115	38.3691025	38.3611031	0.000	0.000	0.00800003	0.00800003	0.0000
16	Pipe	2.160	1.1325	39.3148537	39.3147736	0.000	0.000	0.00008354	0.00008354	0.0000
17	Pipe	2.160	9.5031	30.8611908	30.2693825	0.000	0.000	0.59180957	0.59180957	0.0000
18	Pipe	22.722	11.9115	29.3230019	29.3150024	0.000	0.000	0.00799918	0.00799918	0.0000
19	Pipe	2.158	1.1314	39.3068896	39.3067856	0.000	0.000	0.00008334	0.00008334	0.0000
20	Pipe	2.158	9.4946	30.8682632	30.2773819	0.000	0.000	0.59088171	0.59088171	0.0000
21	Pipe	5.140	2.6945	39.2539520	39.2534294	0.000	0.000	0.00052244	0.00052244	0.0000
22	Pipe	5.139	22.6096	33.0000725	30.2900352	0.000	0.000	2.71003842	2.71003842	0.0000
23	Pipe	20.563	10.7801	38.5337944	38.5270958	0.000	0.000	0.00669902	0.00669902	0.0000
24	Pipe	20.563	10.7801	29.5023918	29.4956932	0.000	0.000	0.00669852	0.00669852	0.0000
25	Pipe	29.209	15.3124	37.7791596	37.7666359	0.000	0.000	0.01252464	0.01252464	0.0000
26	Pipe	2.168	1.1366	39.3476334	39.3475494	0.000	0.000	0.00008433	0.00008433	0.0000
27	Pipe	2.168	9.5378	30.8321496	30.2365456	0.000	0.000	0.59560215	0.59560215	0.0000
28	Pipe	29.209	15.3125	28.6718903	28.6593704	0.000	0.000	0.01252249	0.01252249	0.0000
29	Pipe	2.165	1.1350	39.3351326	39.3350487	0.000	0.000	0.00008403	0.00008403	0.0000
30	Pipe	2.165	9.5245	30.8432274	30.2490692	0.000	0.000	0.59415793	0.59415793	0.0000
31	Pipe	2.162	1.1336	39.3242416	39.3241577	0.000	0.000	0.00008376	0.00008376	0.0000
32	Pipe	2.162	9.5130	30.8528786	30.2599792	0.000	0.000	0.59289789	0.59289789	0.0000
33	Pipe	27.044	14.1775	37.9917717	37.9808578	0.000	0.000	0.01091360	0.01091360	0.0000
34	Pipe	27.044	14.1775	28.9079437	28.8970299	0.000	0.000	0.01091192	0.01091192	0.0000
35	Pipe	5.139	2.6943	39.2539597	39.2533684	0.000	0.000	0.00059029	0.00059029	0.0000
36	Pipe	5.139	2.6943	30.2412415	30.2412071	0.000	0.000	0.00003700	0.00003700	0.0000
37	Pipe	24.882	13.0439	38.1884232	38.1790161	0.000	0.000	0.00940517	0.00940517	0.0000
38	Pipe	24.882	13.0439	29.1249123	29.1155090	0.000	0.000	0.00940396	0.00940396	0.0000
39	Pipe	2.171	1.1384	39.3618469	39.3617592	0.000	0.000	0.00008468	0.00008468	0.0000

Pipe	Name	Vol. Flow Rate (gal/min)	Velocity (feet/sec)	P Static Max (psig)	P Static Min (psig)	Elevation Inlet (feet)	Elevation Outlet (feet)	dP Stag. Total (psid)	dP Static Total (psid)	dP Gravity (psid)
40	Pipe	2.171	9.5528	30.8195534	30.2223129	0.000	0.000	0.59724224	0.59724224	0.0000
41	Pipe	31.377	16.4489	37.5505867	37.5363464	0.000	0.000	0.01423789	0.01423789	0.0000
42	Pipe	31.377	16.4490	28.4165535	28.4023170	0.000	0.000	0.01423510	0.01423510	0.0000
43	Pipe	37.919	19.8785	36.8387833	36.7920418	0.000	0.000	0.04674156	0.04674156	0.0000
44	Pipe	2.201	1.1541	39.4878502	39.4877625	0.000	0.000	0.00008780	0.00008780	0.0000
45	Pipe	2.201	9.6848	30.7077827	30.0960922	0.000	0.000	0.61168909	0.61168909	0.0000
46	Pipe	37.919	19.8787	27.4847755	27.4380455	0.000	0.000	0.04672991	0.04672991	0.0000
47	Pipe	2.190	1.1483	39.4412003	39.4411125	0.000	0.000	0.00008664	0.00008664	0.0000
48	Pipe	2.190	9.6361	30.7491684	30.1428223	0.000	0.000	0.60634702	0.60634702	0.0000
49	Pipe	2.180	1.1431	39.3992805	39.3991966	0.000	0.000	0.00008560	0.00008560	0.0000
50	Pipe	2.180	9.5922	30.7863503	30.1848106	0.000	0.000	0.60154015	0.60154015	0.0000
51	Pipe	35.729	18.7303	37.0902481	37.0482483	0.000	0.000	0.04199915	0.04199915	0.0000
52	Pipe	35.729	18.7304	27.8249741	27.7829819	0.000	0.000	0.04198937	0.04198937	0.0000
53	Pipe	2.214	1.1605	39.5394897	39.5394020	0.000	0.000	0.00008909	0.00008909	0.0000
54	Pipe	2.214	9.7384	30.6619606	30.0443687	0.000	0.000	0.61759502	0.61759502	0.0000
55	Pipe	40.120	21.0326	36.5729408	36.5212021	0.000	0.000	0.05174000	0.05174000	0.0000
56	Pipe	40.121	21.0328	27.1204529	27.0687294	0.000	0.000	0.05172601	0.05172601	0.0000
57	Pipe	33.548	17.5873	37.3274765	37.2899704	0.000	0.000	0.03750885	0.03750885	0.0000
58	Pipe	33.548	17.5874	28.1417046	28.1042061	0.000	0.000	0.03750092	0.03750092	0.0000
59	Pipe	44.923	23.5505	35.9984589	35.9350128	0.000	0.000	0.06344625	0.06344625	0.0000
60	Pipe	1.307	0.6853	39.7260094	39.7259750	0.000	0.000	0.00003679	0.00003679	0.0000
61	Pipe	1.307	5.7505	30.0709610	29.8638039	0.000	0.000	0.20716086	0.20716086	0.0000
62	Pipe	44.924	23.5508	26.1964760	26.1330528	0.000	0.000	0.06342432	0.06342432	0.0000
63	Pipe	1.299	0.6809	39.6626053	39.6625710	0.000	0.000	0.00003655	0.00003655	0.0000
64	Pipe	1.299	5.7137	30.1308060	29.9272232	0.000	0.000	0.20358212	0.20358212	0.0000
65	Pipe	1.291	0.6767	39.6024704	39.6024361	0.000	0.000	0.00003632	0.00003632	0.0000
66	Pipe	1.291	5.6785	30.1875839	29.9873772	0.000	0.000	0.20020464	0.20020464	0.0000
67	Pipe	43.625	22.8697	36.1476021	36.0874290	0.000	0.000	0.06017315	0.06017315	0.0000
68	Pipe	43.625	22.8699	26.4692230	26.4090691	0.000	0.000	0.06015397	0.06015397	0.0000
69	Pipe	1.316	0.6899	39.7927895	39.7927513	0.000	0.000	0.00003704	0.00003704	0.0000
70	Pipe	1.316	5.7890	30.0079536	29.7970047	0.000	0.000	0.21094762	0.21094762	0.0000
71	Pipe	46.230	24.2357	35.8450241	35.7782021	0.000	0.000	0.06682043	0.06682043	0.0000
72	Pipe	52.933	11.5205	75.8554993	75.7188263	1.000	1.000	0.13667215	0.13667215	0.0000
73	Pipe	52.933	11.5205	75.1664276	75.0297546	1.000	1.000	0.13667215	0.13667215	0.0000
74	Pipe	1.345	0.7049	40.0144386	40.0144005	0.000	0.000	0.00003785	0.00003785	0.0000
75	Pipe	1.345	5.9150	29.7989655	29.5753098	0.000	0.000	0.22365433	0.22365433	0.0000
76	Pipe	50.220	26.3273	24.9906006	24.9130211	0.000	0.000	0.07757699	0.07757699	0.0000
77	Pipe	1.348	0.7068	39.9368095	39.9367714	0.000	0.000	0.00003795	0.00003795	0.0000
78	Pipe	1.348	5.9312	29.6652718	29.4399109	0.000	0.000	0.22536026	0.22536026	0.0000
79	Pipe	1.325	0.6947	39.8630409	39.8630028	0.000	0.000	0.00003730	0.00003730	0.0000
80	Pipe	1.325	5.8293	29.9416924	29.7267380	0.000	0.000	0.21495362	0.21495362	0.0000
81	Pipe	48.871	25.6201	35.5249290	35.4510460	0.000	0.000	0.07388151	0.07388151	0.0000
82	Pipe	48.872	25.6205	25.3114166	25.2375679	0.000	0.000	0.07384939	0.07384939	0.0000
83	Pipe	1.355	0.7103	40.0958023	40.0957642	0.000	0.000	0.00003815	0.00003815	0.0000
84	Pipe	1.355	5.9606	29.7223091	29.4939346	0.000	0.000	0.22837397	0.22837397	0.0000
85	Pipe	51.564	27.0317	35.1840172	35.1026001	0.000	0.000	0.08141539	0.08141539	0.0000
86	Pipe	51.565	27.0322	24.6600418	24.5786667	0.000	0.000	0.08137801	0.08137801	0.0000
87	Pipe	42.334	22.1930	36.2925301	36.2355309	0.000	0.000	0.05699896	0.05699896	0.0000
88	Pipe	47.546	24.9255	35.6872101	35.6169128	0.000	0.000	0.07029801	0.07029801	0.0000
89	Pipe	42.334	22.1932	26.7313194	26.6743355	0.000	0.000	0.05698255	0.05698255	0.0000
90	Pipe	47.547	24.9259	25.6178665	25.5475960	0.000	0.000	0.07026962	0.07026962	0.0000
105	Pipe	52.932	0.1409	-0.0001335	-0.0006933	6.000	6.000	0.00055976	0.00055976	0.0000
106	Pipe	52.945	0.1409	0.4325924	-0.0001335	6.000	5.000	-0.43272620	-0.43272620	-0.4327
107	Pipe	0.000	0.0000	0.0000000	0.0000000	6.000	6.000	0.00000000	0.00000000	0.0000
109	Pipe	1.485	4.4303	83.8016205	83.6766594	1.000	1.000	0.12505458	0.12505458	0.0000
111	Pipe	54.427	11.8458	84.2962952	84.1526184	1.000	1.000	0.14367439	0.14367439	0.0000

Pipe	Name	Vol. Flow Rate (gal/min)	Velocity (feet/sec)	P Static Max (psig)	P Static Min (psig)	Elevation Inlet (feet)	Elevation Outlet (feet)	dP Stag. Total (psid)	dP Static Total (psid)	dP Gravity (psid)
112	Pipe	1.485	4.4303	1.1579695	1.0329142	1.000	1.000	0.12505458	0.12505458	0.0000
113	Pipe	1.485	4.4303	1.3127174	1.1876631	1.000	1.000	0.12505458	0.12505458	0.0000
114	Pipe	52.933	11.5205	84.2034607	84.0667877	1.000	1.000	0.13667215	0.13667215	0.0000
115	Pipe	52.933	11.5205	75.0119019	74.4425049	1.000	2.000	0.56940198	0.56940198	0.4327
117	Pipe	52.932	11.5204	10.8014336	4.9701614	0.000	4.000	5.83127117	5.83127117	1.7309
120	Pipe	1.485	4.4303	1.0302744	0.9061832	1.000	1.000	0.12409154	0.12409154	0.0000
125	Pipe	54.427	11.6747	-0.0223379	-0.1773243	1.000	1.000	0.15498601	0.15498601	0.0000
126	Pipe	54.427	11.8458	0.0945339	-0.0491972	1.000	1.000	0.14373083	0.14373083	0.0000
128	Pipe	52.945	11.5233	0.1452131	-0.9026184	5.000	1.000	-1.04783118	-1.04783118	-1.7309
129	Pipe	52.919	11.5175	63.2353249	59.9763603	2.000	0.000	3.25896692	3.25896692	-0.8657
130	Pipe	46.231	24.2360	25.9127808	25.8459854	0.000	0.000	0.06679527	0.06679527	0.0000
131	Pipe	50.219	26.3269	35.3555794	35.2779694	0.000	0.000	0.07761186	0.07761186	0.0000
132	Pipe	52.919	24.9939	56.1974831	54.4080009	0.000	0.000	1.78948557	1.78948557	0.0000
133	Pipe	52.919	24.9939	52.4279900	45.2700424	0.000	0.000	7.15794230	7.15794230	0.0000
134	Pipe	52.919	35.8062	40.3088722	40.1302299	0.000	0.000	0.17864089	0.17864089	0.0000
135	Pipe	52.920	35.8068	15.6604919	15.4819260	0.000	0.000	0.17856525	0.17856525	0.0000
136	Pipe	52.920	24.9944	10.2400208	8.4513416	0.000	0.000	1.78867829	1.78867829	0.0000
137	Pipe	52.920	24.9944	19.3747711	12.2200584	0.000	0.000	7.15471315	7.15471315	0.0000
138	Pipe	52.932	11.5204	4.5860195	4.3126621	4.000	4.000	0.27335656	0.27335656	0.0000
139	Pipe	52.932	11.5204	3.1919346	3.0552559	4.000	4.000	0.13667828	0.13667828	0.0000
140	Pipe	52.932	11.5204	2.6711140	2.6597233	4.000	4.000	0.01138986	0.01138986	0.0000
141	Pipe	52.932	11.5204	2.2755814	2.2072430	4.000	4.000	0.06833913	0.06833913	0.0000
142	Pipe	52.932	11.5204	1.6715584	1.6373882	4.000	4.000	0.03416957	0.03416957	0.0000
143	Pipe	52.932	11.5204	1.2532454	-0.0222511	4.000	6.000	1.27549648	1.27549648	0.8655
144	Pipe	52.932	5.0609	2.4608040	2.4440937	4.000	4.000	0.01671079	0.01671079	0.0000
145	Pipe	52.932	5.0609	2.8322964	2.7988758	4.000	4.000	0.03342158	0.03342158	0.0000
146	Pipe	52.920	11.5177	10.9390602	10.8016415	0.000	0.000	0.13741986	0.13741986	0.0000

Pipe	dH (feet)	P Static In (psig)	P Static Out (psig)	P Stag. In (psig)	P Stag. Out (psig)	T Inlet (deg. F)	T Outlet (deg. F)	f
2	0.07169785	34.9533157	34.9222832	40.1302299	40.0992	67.09	67.09	0.02027
3	0.00929626	38.8692207	38.8652000	39.3087807	39.3048	67.20	67.20	0.02630
4	0.00120848	39.2598763	39.2593536	39.3087807	39.3083	67.20	67.20	0.03473
5	6.26825113	32.9972115	30.2840843	36.4409065	33.7278	67.20	67.20	0.03011
6	0.00929475	29.8485413	29.8445168	30.2881012	30.2841	67.22	67.22	0.02630
7	0.00120751	39.2559013	39.2553787	39.3047600	39.3042	67.20	67.20	0.03473
8	6.26303085	32.9969700	30.2881012	36.4396103	33.7286	67.20	67.20	0.03011
9	0.07464552	24.3169479	24.2846375	29.4939308	29.4616	67.28	67.28	0.02026
11	0.00455438	39.1094322	39.1074600	39.3047600	39.3028	67.20	67.20	0.02900
12	0.00120687	39.2533684	39.2528458	39.3021965	39.3017	67.21	67.21	0.03473
13	6.26042655	32.9998093	30.2900696	36.4388161	33.7291	67.21	67.21	0.03011
14	0.00455423	30.0947418	30.0927734	30.2900696	30.2881	67.21	67.21	0.02900
15	0.01848277	38.3691025	38.3611031	39.3234787	39.3155	67.19	67.19	0.02409
16	0.00019300	39.3148537	39.3147736	39.3234787	39.3234	67.19	67.19	0.03144
17	1.36728058	30.8611908	30.2693825	31.4686508	30.8768	67.19	67.19	0.03726
18	0.01848088	29.3230019	29.3150024	30.2773781	30.2694	67.22	67.22	0.02408
19	0.00019256	39.3068695	39.3067856	39.3154793	39.3154	67.19	67.19	0.03142
20	1.36513788	30.8682632	30.2773819	31.4746399	30.8838	67.19	67.19	0.03727
21	0.00120702	39.2539520	39.2534294	39.3027878	39.3023	67.21	67.21	0.03473
22	6.26112225	33.0000725	30.2900352	36.4386139	33.7286	67.21	67.21	0.03011
23	0.01547701	38.5337944	38.5270958	39.3154793	39.3088	67.19	67.19	0.02462
24	0.01547590	29.5023918	29.4956932	30.2840767	30.2774	67.22	67.22	0.02462
25	0.02893611	37.7791595	37.7666359	39.3563232	39.3438	67.17	67.17	0.02282
26	0.00019484	39.3476334	39.3475494	39.3563232	39.3562	67.17	67.17	0.03151
27	1.37604046	30.8321495	30.2365456	31.4440498	30.8484	67.17	67.17	0.03723
28	0.02893133	28.6718903	28.6593704	30.2490616	30.2365	67.23	67.23	0.02281

Pipe	dH (feet)	P Static In (psig)	P Static Out (psig)	P Stag. In (psig)	P Stag. Out (psig)	T Inlet (deg. F)	T Outlet (deg. F)	f
29	0.00019414	39.3351326	39.3350487	39.3437996	39.3437	67.18	67.18	0.03148
30	1.37270467	30.8432274	30.2490692	31.4534340	30.8593	67.18	67.18	0.03724
31	0.00019352	39.3242416	39.3241577	39.3328857	39.3328	67.18	67.18	0.03146
32	1.36979412	30.8528786	30.2599792	31.4616089	30.8687	67.18	67.18	0.03726
33	0.02521409	37.9917717	37.9808578	39.3437996	39.3329	67.18	67.18	0.02319
34	0.02521034	28.9079437	28.8970299	30.2599754	30.2491	67.23	67.23	0.02319
35	0.00136377	39.2539597	39.2539684	39.3027878	39.3022	67.21	67.21	0.03473
36	0.00008548	30.2412071	30.2412415	30.2900352	30.2901	67.21	67.21	0.03473
37	0.02172911	38.1884232	38.1790161	39.3328857	39.3235	67.18	67.18	0.02361
38	0.02172842	29.1249123	29.1155090	30.2693787	30.2600	67.23	67.23	0.02361
39	0.00019664	39.3618469	39.3617592	39.3705635	39.3705	67.17	67.17	0.03154
40	1.37982860	30.8195534	30.2223129	31.4333801	30.8361	67.17	67.17	0.03722
41	0.03289427	37.5505867	37.5363464	39.3705635	39.3563	67.17	67.17	0.02248
42	0.03288807	28.4165535	28.4023170	30.2365417	30.2223	67.23	67.23	0.02247
43	0.10798837	36.8387833	36.7920418	39.4968109	39.4501	67.15	67.15	0.02163
44	0.00020284	39.4878502	39.4877625	39.4968109	39.4967	67.15	67.15	0.03182
45	1.41320302	30.7077827	30.0960922	31.3386955	30.7270	67.15	67.15	0.03708
46	0.10796255	27.4847755	27.4380455	30.1428146	30.0961	67.24	67.24	0.02162
47	0.00020017	39.4412003	39.4411125	39.4500694	39.4500	67.15	67.15	0.03171
48	1.40086196	30.7491684	30.1428223	31.3737564	30.7674	67.15	67.15	0.03713
49	0.00019777	39.3992805	39.3991966	39.4080696	39.4080	67.16	67.16	0.03162
50	1.38975749	30.7863503	30.1848106	31.4052544	30.8037	67.16	67.16	0.03718
51	0.09703191	37.0902481	37.0482483	39.4500694	39.4081	67.15	67.15	0.02189
52	0.09701018	27.8249741	27.7829819	30.1848068	30.1428	67.24	67.24	0.02188
53	0.00020582	39.5394897	39.5394020	39.5485497	39.5485	67.14	67.14	0.03193
54	1.42684662	30.6619606	30.0443687	31.2998772	30.6823	67.14	67.14	0.03703
55	0.11953633	36.5729408	36.5212021	39.5485497	39.4968	67.14	67.14	0.02138
56	0.11950527	27.1204529	27.0687294	30.0960846	30.0444	67.24	67.24	0.02138
57	0.08665790	37.3274765	37.2899704	39.4080696	39.3706	67.16	67.16	0.02217
58	0.08664029	28.1417046	28.1042061	30.2223053	30.1848	67.24	67.24	0.02217
59	0.14658132	35.9864589	35.9350128	39.7291679	39.6657	67.12	67.12	0.02092
60	0.00008499	39.7260094	39.7259750	39.7291679	39.7291	67.12	67.12	0.03781
61	0.47860850	30.0709610	29.8638039	30.2933960	30.0862	67.12	67.12	0.03574
62	0.14653269	26.1964760	26.1330528	29.9272232	29.8638	67.25	67.25	0.02091
63	0.00008444	39.6626053	39.6625710	39.6657219	39.6657	67.13	67.13	0.03805
64	0.47034075	30.1308060	29.9272232	30.3504028	30.1468	67.13	67.13	0.03558
65	0.00008391	39.6024704	39.6024361	39.6055489	39.6055	67.14	67.14	0.03829
66	0.46253791	30.1875839	29.9873772	30.4044876	30.2043	67.14	67.14	0.03542
67	0.13901951	36.1476021	36.0874290	39.6657219	39.6055	67.13	67.13	0.02104
68	0.13897693	26.4692230	26.4090691	29.9873772	29.9272	67.24	67.24	0.02103
69	0.00008557	39.7927895	39.7927513	39.7959900	39.7960	67.12	67.12	0.03756
70	0.48735688	30.0079536	29.7970047	30.2333794	30.0224	67.12	67.12	0.03591
71	0.15437669	35.8450241	35.7782021	39.7959900	39.7292	67.12	67.12	0.02080
72	0.31583714	75.8554993	75.7188263	76.7480316	76.6114	69.36	69.36	0.01748
73	0.31583714	75.1664276	75.0297546	76.0589600	75.9223	69.36	69.36	0.01748
74	0.00008745	40.0144386	40.0144005	40.0177803	40.0177	67.11	67.11	0.03677
75	0.51671268	29.7969655	29.5753098	30.0343132	29.8107	67.11	67.11	0.03646
76	0.17923089	24.9906006	24.9130211	29.6528854	29.5753	67.28	67.28	0.02046
77	0.00008768	39.9368095	39.9367714	39.9401703	39.9401	67.11	67.11	0.03667
78	0.52065397	29.6652718	29.4399109	29.9019089	29.6765	67.11	67.11	0.03653
79	0.00008617	39.8630409	39.8630028	39.8662872	39.8662	67.11	67.11	0.03731
80	0.49661172	29.9416924	29.7267380	30.1702614	29.9553	67.11	67.11	0.03608
81	0.17068981	35.5249290	35.4510460	39.9401703	39.8663	67.11	67.11	0.02058
82	0.17061860	25.3114166	25.2375679	29.7267342	29.6529	67.27	67.27	0.02057
83	0.00008813	40.0958023	40.0957642	40.0991974	40.0992	67.10	67.10	0.03649
84	0.52761623	29.7223091	29.4939346	29.9612961	29.7329	67.10	67.10	0.03666

Pipe	dH (feet)	P Static In (psig)	P Static Out (psig)	P Stag. In (psig)	P Stag. Out (psig)	T Inlet (deg. F)	T Outlet (deg. F)	f
85	0.18809535	35.1840172	35.1026001	40.0991974	40.0178	67.10	67.10	0.02037
86	0.18801260	24.6600418	24.5786667	29.5753059	29.4939	67.28	67.28	0.02036
87	0.13168616	36.2925301	36.2355309	39.6055489	39.5485	67.14	67.14	0.02116
88	0.16241092	35.6872101	35.6169128	39.8662872	39.7960	67.11	67.11	0.02069
89	0.13164978	26.7313194	26.6743355	30.0443611	29.9874	67.24	67.24	0.02115
90	0.16234797	25.6178665	25.5475960	29.7970047	29.7267	67.26	67.26	0.02068
105	0.00129355	-0.0006933	-0.0001335	-0.0005598	0.0000	69.34	69.34	0.02856
106	0.00001067	-0.0001335	0.4325924	0.0000000	0.4327	69.34	69.34	0.02856
107	0.00000000	0.0000000	0.0000000	0.0000000	0.0000	65.00	65.00	0.00000
109	0.28898997	83.8016205	83.6765594	83.9336090	83.8086	69.36	69.36	0.02921
111	0.33201824	84.2962952	84.1526184	85.2399445	85.0963	69.35	69.35	0.01739
112	0.28898997	1.1579695	1.0329142	1.2899628	1.1649	69.36	69.36	0.02921
113	0.28898997	1.3127174	1.1876631	1.4447117	1.3197	69.36	69.36	0.02921
114	0.31583714	84.2034607	84.0667877	85.0959930	84.9593	69.36	69.36	0.01748
115	0.31583714	75.0119019	74.4425049	75.9044342	75.3350	69.36	69.36	0.01748
117	9.47551656	10.8014336	4.9701614	11.6939564	5.8627	69.34	69.34	0.01748
120	0.28676446	1.0302744	0.9061832	1.1622677	1.0382	69.36	69.36	0.02921
125	0.35815835	-0.0223379	-0.1773243	0.8942490	0.7393	69.35	69.35	0.01945
126	0.33214868	0.0945339	-0.0491972	1.0381765	0.8944	69.35	69.35	0.01739
128	1.57856130	-0.9026184	0.1452131	-0.0096550	1.0382	69.34	69.34	0.01748
129	9.52924120	63.2353249	59.9763603	64.1276245	60.8687	67.09	67.09	0.01759
130	0.15432084	25.9127808	25.8459854	29.8638000	29.7970	67.25	67.25	0.02079
131	0.17930809	35.3555794	35.2779694	40.0177803	39.9402	67.11	67.11	0.02047
132	4.13427603	56.1974831	54.4080009	60.3956399	58.6101	67.09	67.09	0.01650
133	16.53710411	52.4279900	45.2700424	56.6300468	49.4721	67.09	67.09	0.01650
134	0.41271679	40.3088722	40.1302299	48.9328957	48.7543	67.09	67.09	0.01610
135	0.41255043	15.6604919	15.4819260	24.2846375	24.1061	67.28	67.28	0.01609
136	4.13249495	10.2400208	8.4513416	14.4421387	12.6535	67.28	67.28	0.01649
137	16.52997982	19.3747711	12.2200584	23.5766890	16.4222	67.28	67.28	0.01649
138	0.63170116	4.5860195	4.3126621	5.4785423	5.2052	69.34	69.34	0.01748
139	0.31585058	3.1919346	3.0552559	4.0844574	3.9478	69.34	69.34	0.01748
140	0.02632088	2.6711140	2.6597233	3.5636368	3.5522	69.34	69.34	0.01748
141	0.15792527	2.2755814	2.2072430	3.1681042	3.0998	69.34	69.34	0.01748
142	0.07896264	1.6715584	1.6373882	2.5640812	2.5299	69.34	69.34	0.01748
143	0.94755167	1.2532454	-0.0222511	2.1457691	0.8703	69.34	69.34	0.01748
144	0.03861706	2.4608040	2.4440937	2.6330471	2.6163	69.34	69.34	0.02005
145	0.07723412	2.8322964	2.7988758	3.0045395	2.9711	69.34	69.34	0.02005
146	0.31748968	10.9390602	10.8016415	11.8313751	11.6940	67.28	67.28	0.01758

All Junction Table

Jct	Name	P Static In (psig)	P Static Out (psig)	Vol. Flow Rate Thru Jct (gal/min)	T Inlet (deg. F)	T Outlet (deg. F)	P Stag. In (psig)	P Stag. Out (psig)
2	Area Change	40.13022995	34.95331955	52.919	67.09	67.09	48.7543	40.1302299
3	Branch	37.79774857	37.79774857	N/A	67.09	67.09	40.0992	40.0991974
4	Branch	39.25336838	39.25336838	5.139	67.21	67.21	39.3022	39.3021965
5	Branch	39.10933685	39.10933685	N/A	67.20	67.20	39.3048	39.3047600
6	Pp-1 Gould Centrifugal Pump	-0.17732430	84.29629517	54.427	69.35	69.35	0.7393	85.2399445
7	Branch	30.29003525	30.24120712	5.139	67.21	67.21	33.7286	30.2900352
8	Branch	29.31430435	29.31430435	N/A	67.21	67.21	30.2881	30.2881012
9	HX-1	74.44250488	63.23532486	52.933	69.36	67.09	75.3350	64.1276245
10	Throttle Valve	84.06678772	75.85549927	52.933	69.36	69.36	84.9593	76.7480316
11	Area Change	8.45134163	10.93906021	52.920	67.28	67.28	12.6535	11.8313751
13	Branch	39.21594238	39.21594238	N/A	67.20	67.20	39.3028	39.3027878
14	Orifice	39.25284576	33.00007248	5.139	67.21	67.21	39.3017	36.4386139

Jct	Name	P Static In (psig)	P Static Out (psig)	Vol. Flow Rate Thru Jct (gal/min)	T Inlet (deg. F)	T Outlet (deg. F)	P Stag. In (psig)	P Stag. Out (psig)
15	Branch	29.58561707	29.58561707	N/A	67.21	67.21	30.2901	30.2900696
16	Branch	38.81470490	38.81470490	N/A	67.18	67.18	39.3235	39.3234787
17	Branch	38.89120102	38.89120102	N/A	67.19	67.19	39.3155	39.3154793
18	Orifice	39.31477356	30.86119080	2.160	67.19	67.19	39.3234	31.4686508
19	Branch	29.38167953	29.38167953	N/A	67.22	67.22	30.2694	30.2693787
20	Branch	29.50287628	29.50287628	N/A	67.22	67.22	30.2774	30.2773781
21	Orifice	39.30678558	30.86826324	2.158	67.19	67.19	39.3154	31.4746399
22	Branch	38.96128082	38.96128082	N/A	67.19	67.19	39.3088	39.3087807
23	Orifice	39.25342941	32.99980927	5.140	67.21	67.21	39.3023	36.4388161
24	Branch	28.99702454	28.99702454	N/A	67.21	67.21	30.2841	30.2840787
25	Branch	38.54724503	38.54724503	N/A	67.17	67.17	39.3563	39.3563232
26	Branch	38.64265442	38.64265442	N/A	67.17	67.17	39.3438	39.3437996
27	Orifice	39.34754944	30.83214951	2.168	67.17	67.17	39.3562	31.4440498
28	Branch	28.96141434	28.96141434	N/A	67.23	67.23	30.2365	30.2365417
29	Branch	29.11109543	29.11109543	N/A	67.22	67.22	30.2491	30.2490616
30	Orifice	39.33504868	30.84322739	2.165	67.18	67.18	39.3437	31.4534340
31	Branch	38.73182678	38.73182678	N/A	67.18	67.18	39.3329	39.3328857
32	Orifice	39.32415771	30.85287857	2.162	67.18	67.18	39.3328	31.4616089
33	Branch	29.25112915	29.25112915	N/A	67.22	67.22	30.2600	30.2599754
34	Branch	38.44562912	38.44562912	N/A	67.16	67.16	39.3706	39.3705635
35	Orifice	39.36175919	30.81955338	2.171	67.17	67.17	39.3705	31.4333801
36	Branch	28.80186081	28.80186081	N/A	67.23	67.23	30.2223	30.2223053
37	Branch	38.17398834	38.17398834	N/A	67.14	67.14	39.4968	39.4968109
38	Deionizer	1.18766308	1.15796947	1.485	69.36	69.36	1.3197	1.2899628
39	Orifice	39.48776245	30.70778275	2.201	67.15	67.15	39.4967	31.3386955
40	Branch	28.18226624	28.18226624	N/A	67.23	67.23	30.0961	30.0960846
41	Particulate Filter	75.71882629	75.16642761	52.933	69.36	69.36	76.6114	76.0589600
42	Orifice	39.44111252	30.74916840	2.190	67.15	67.15	39.4500	31.3737564
43	Branch	38.35898590	38.35898590	N/A	67.15	67.15	39.4081	39.4080696
44	Area Change	-0.02225113	-0.00069332	52.932	69.34	69.34	0.8703	-0.0005598
45	Area Change	0.43259239	-0.90261841	52.945	69.34	69.34	0.4327	-0.0096550
46	Tee or Wye	-0.00005913	-0.00005913	N/A	69.34	69.34	0.0000	0.0000000
47	Assigned Pressure	0.00000000	0.00000000	0.000	65.00	65.00	0.0000	0.0000000
48	Branch	27.94720459	27.94720459	N/A	67.23	67.23	30.0444	30.0443611
49	Tee or Wye	84.51898193	84.51898193	N/A	69.35	69.35	85.0963	85.0962677
50	Branch	38.00719833	38.00719833	N/A	67.12	67.12	39.6657	39.6657219
51	Valve	83.67655945	1.31271744	1.485	69.36	69.36	83.8086	1.4447117
52	Valve	75.02975464	75.01190186	52.933	69.36	69.36	75.9223	75.9044342
53	Valve	1.03291416	1.03027439	1.485	69.36	69.36	1.1649	1.1622677
54	Orifice	39.66257095	30.13080597	1.299	67.13	67.13	39.6657	30.3504028
55	Branch	38.04153442	38.04153442	N/A	67.13	67.13	39.6055	39.6055489
56	Orifice	39.60243607	30.18758392	1.291	67.14	67.14	39.6055	30.4044876
57	Branch	28.06256866	28.06256866	N/A	67.24	67.24	29.9874	29.9873772
58	Branch	37.93814850	37.93814850	N/A	67.11	67.11	39.7960	39.7959900
59	Orifice	39.79275131	30.00795364	1.316	67.12	67.12	39.7960	30.2333794
60	Area Change	-0.04919720	-0.02233791	54.427	69.35	69.35	0.8944	0.8942490
61	Branch	37.83267975	37.83267975	N/A	67.10	67.10	40.0178	40.0177803
62	Tee or Wye	0.46078014	0.46078014	N/A	69.34	69.34	1.0382	1.0381765
63	Orifice	39.25935364	32.99721146	5.143	67.20	67.20	39.3083	36.4409065
64	Orifice	39.25537872	32.99897003	5.141	67.20	67.20	39.3042	36.4395103
65	Branch	38.26841354	38.26841354	N/A	67.15	67.15	39.4501	39.4500694
66	Branch	28.40269852	28.40269852	N/A	67.23	67.23	30.1428	30.1428146
67	Branch	28.60906601	28.60906601	N/A	67.23	67.23	30.1848	30.1848068
68	Orifice	39.39919662	30.78635025	2.180	67.16	67.16	39.4080	31.4052544
69	Branch	38.07572937	38.07572937	N/A	67.14	67.14	39.5485	39.5485497
70	Orifice	39.53940201	30.66196060	2.214	67.14	67.14	39.5485	31.2998772

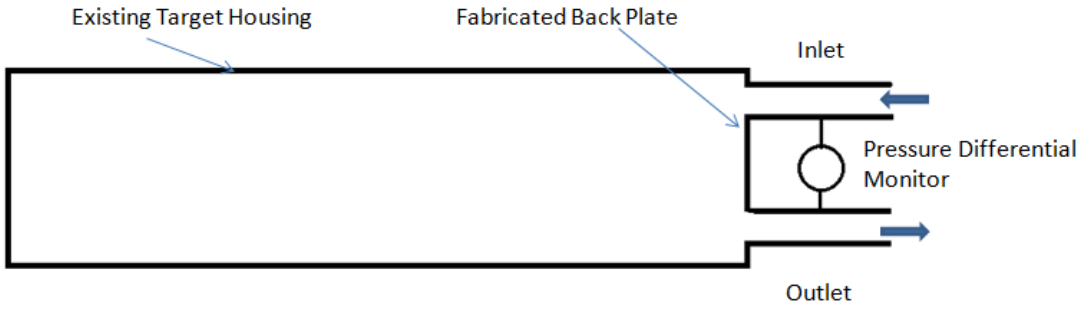
Jct	Name	P Static In (psig)	P Static Out (psig)	Vol. Flow Rate Thru Jct (gal/min)	T Inlet (deg. F)	T Outlet (deg. F)	P Stag. In (psig)	P Stag. Out (psig)
71	Branch	37.97274017	37.97274017	N/A	67.12	67.12	39.7292	39.7291679
72	Branch	27.72107315	27.72107315	N/A	67.25	67.25	29.8638	29.8638000
73	Branch	27.89530945	27.89530945	N/A	67.24	67.24	29.9272	29.9272232
74	Orifice	39.72597504	30.07096100	1.307	67.12	67.12	39.7291	30.2933960
75	Branch	27.53961563	27.53961563	N/A	67.25	67.25	29.7970	29.7970047
76	Branch	37.86754990	37.86754990	N/A	67.11	67.11	39.9402	39.9401703
77	Branch	26.94874573	26.94874573	N/A	67.27	67.27	29.5753	29.5753059
78	Branch	27.14851761	27.14851761	N/A	67.26	67.26	29.6529	29.6528854
79	Orifice	40.01440048	29.79896545	1.345	67.11	67.11	40.0177	30.0343132
80	Orifice	39.93677139	29.66527176	1.348	67.11	67.11	39.9401	29.9019089
81	Branch	37.90344238	37.90344238	N/A	67.11	67.11	39.8663	39.8662872
82	Branch	27.35077286	27.35077286	N/A	67.26	67.26	29.7267	29.7267342
83	Orifice	39.86300278	29.94169235	1.325	67.11	67.11	39.8662	30.1702614
84	Branch	26.73630142	26.73630142	N/A	67.27	67.27	29.4939	29.4939308
85	Orifice	40.09576416	29.72230911	1.355	67.10	67.10	40.0992	29.9612961
86	Bend	54.40800095	52.42798996	52.919	67.09	67.09	58.6101	56.6300468
87	Area Change	59.97636032	56.19748306	52.919	67.09	67.09	60.8687	60.3995399
88	Area Change	45.27004242	40.30887222	52.919	67.09	67.09	49.4721	48.9328957
89	Area Change	24.28463364	15.66049194	52.920	67.28	67.28	29.4616	24.2846375
90	Area Change	15.48192596	19.37477112	52.920	67.28	67.28	24.1061	23.5768890
91	Bend	12.22005844	10.24002075	52.920	67.28	67.28	16.4222	14.4421387
92	Bend	4.97016144	4.58601952	52.932	69.34	69.34	5.8627	5.4785423
93	General Component	4.31266212	3.19193459	52.932	69.34	69.34	5.2052	4.0844574
94	Bend	3.05525589	2.67111397	52.932	69.34	69.34	3.9478	3.5636368
95	Bend	2.65972328	2.27558136	52.932	69.34	69.34	3.5622	3.1681042
96	General Component	2.79887581	2.46080399	52.932	69.34	69.34	2.9711	2.6330471
97	Bend	1.63738823	1.25324535	52.932	69.34	69.34	2.5299	2.1457691
98	Area Change	2.44409370	1.67155838	52.932	69.34	69.34	2.6163	2.5640812
99	Area Change	2.20724297	2.83229637	52.932	69.34	69.34	3.0998	3.0045395
100	Heat Exchanger	10.80164337	10.80143356	52.920	67.28	69.34	11.6940	11.6939564

Summary of DU Target Flow Testing

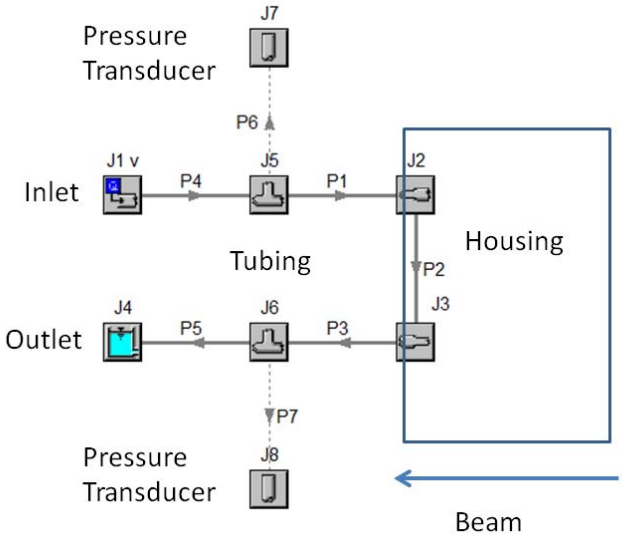
The flow tests were used to develop the complete FATHOM model by validating each component as it was added to the model. (I.e. Pre-test and then configurations 1 -4) as described below. At each step a description the test arrangement is first shown, then the corresponding FATHOM model and finally a plot showing the comparison between the test and model of pressure loss (as determined by the pressure transducers) verse flow. The final flow configuration 4 was then incorporated into the actual model of the cooling system.

Test Description Pre-test Configuration

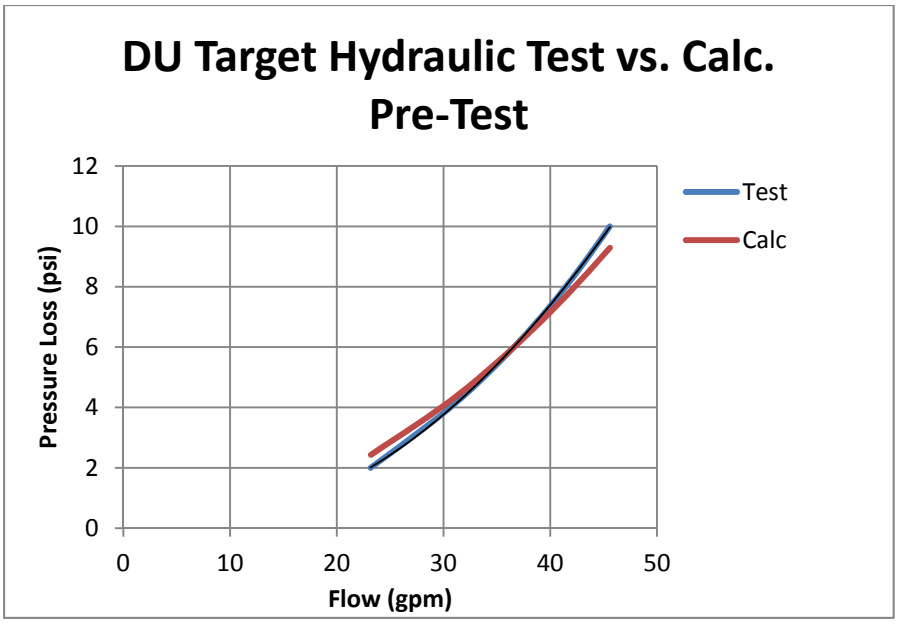
The test arrangement for the pre-test configuration is shown below. The purpose of this test is to determine the pressure losses inherent in the test system between the pressure taps for the differential pressure gages. Pressure losses in the empty target housing are assumed negligible; however, entrance and exit losses at the back of the housing are significant and will be accounted for.



Test Arrangement



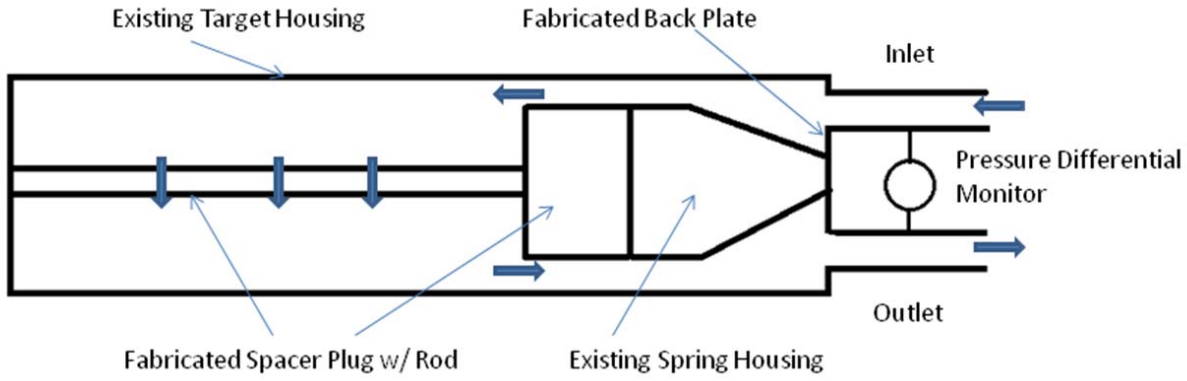
FATHOM Diagram (Note reversed image from Test Arrangement)



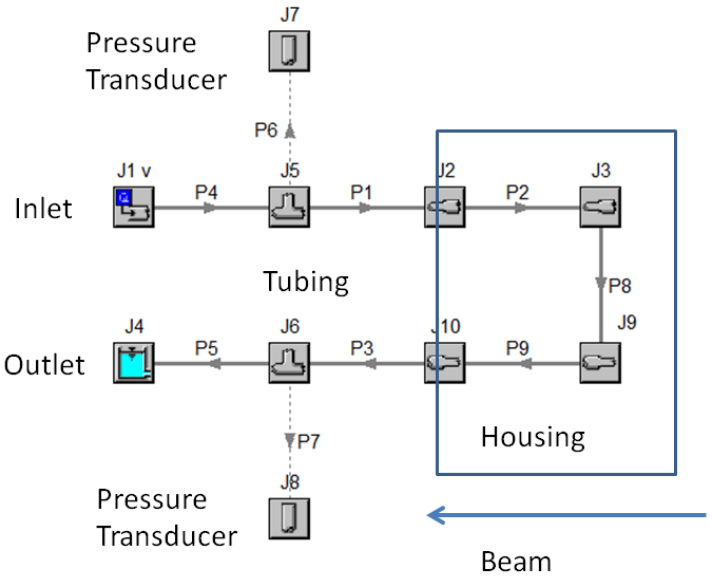
FATHOM vs Test

Test Description Configuration 1

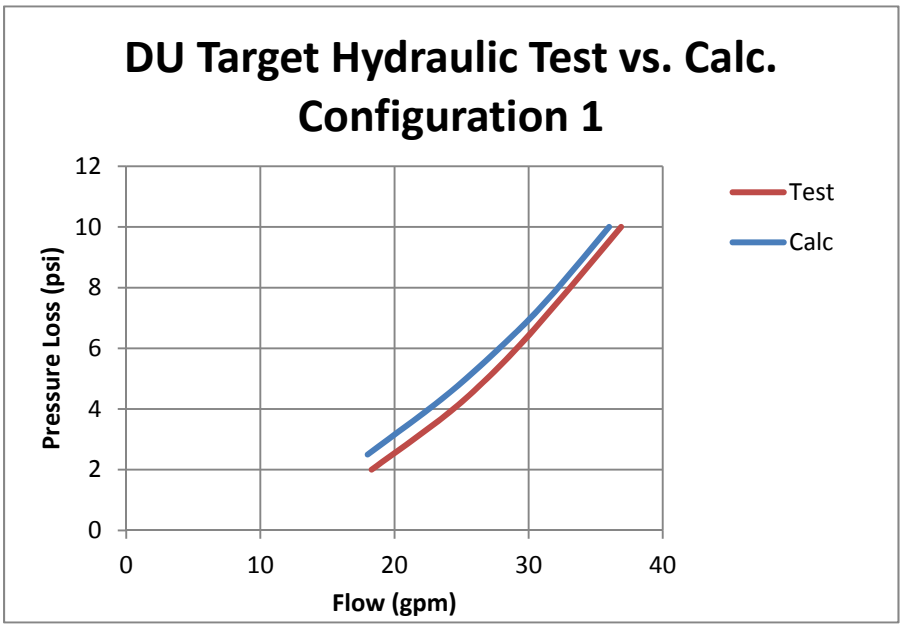
The test arrangement for configuration 1 is shown below. The purpose of this test is to validate the pressure losses associated with the flow through the inlet and outlet of the target. This configuration allows for a large flow area within the target housing and, therefore, the pressure loss within the target is assumed negligible relative to those at the inlet and outlet. This test will be used to validate the FATHOM simulation of losses at the target inlet and outlet.



Test Arrangement



FATHOM Diagram (Note reversed image from Test Arrangement)

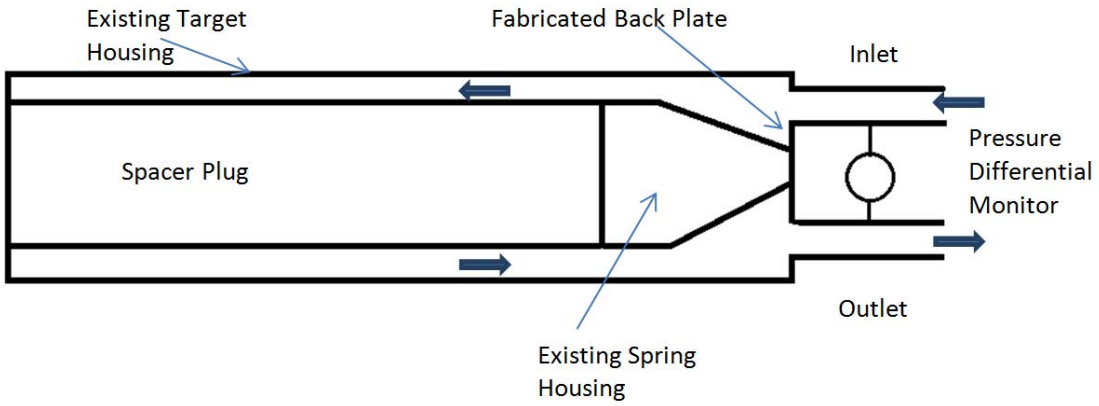


FATHOM vs Test

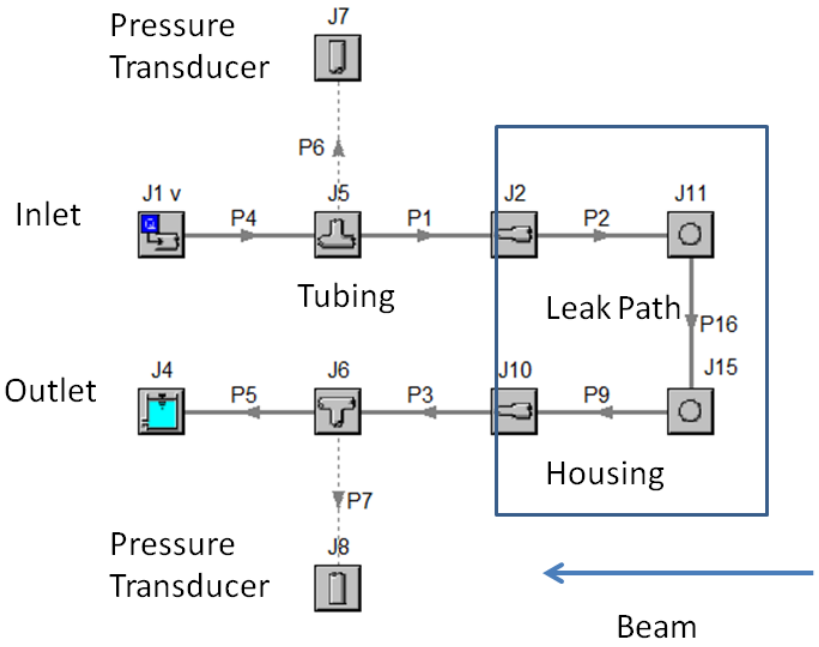
Test Description Configuration 2

The test arrangement for the full plug is shown below. The purpose of this test is to validate the losses associated with the flow around the sides of the spacers and disks, what will be referred to as 'leakage'. The back plate used for configuration 1 is also used for this test. This configuration allows for a

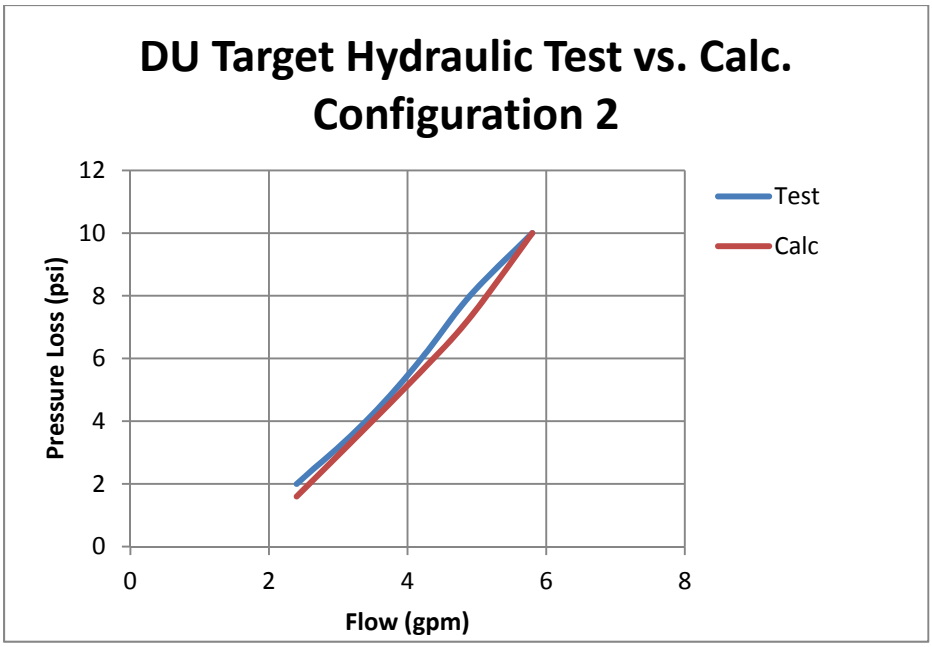
closed flow area within the target housing and, therefore, all flow through the target would be due to leakage. This test was used to validate the FATHOM simulation of losses from flow around the disks.



Test Arrangement



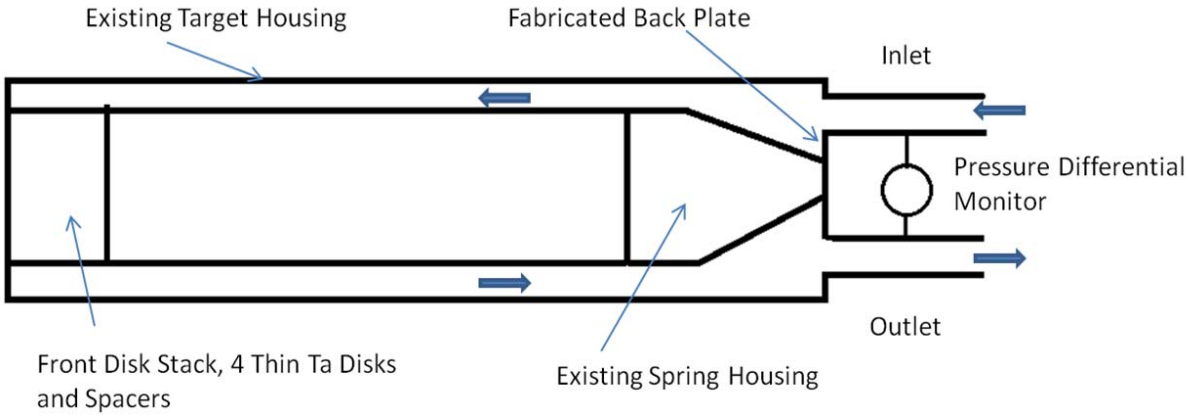
FATHOM Diagram (Note reversed image from Test Arrangement)



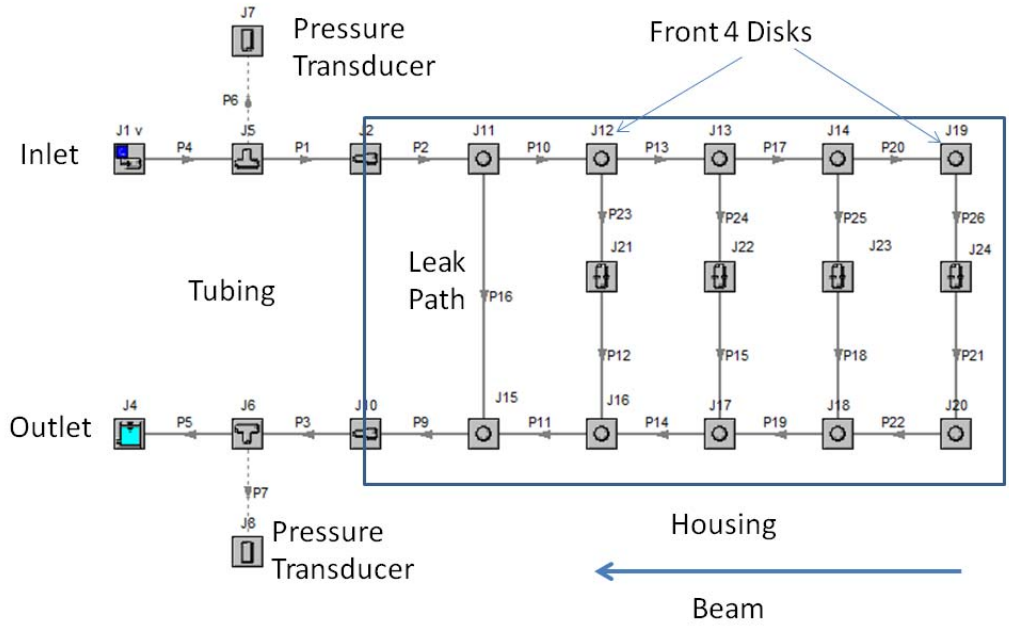
FATHOM vs Test

Test Description Configuration 3

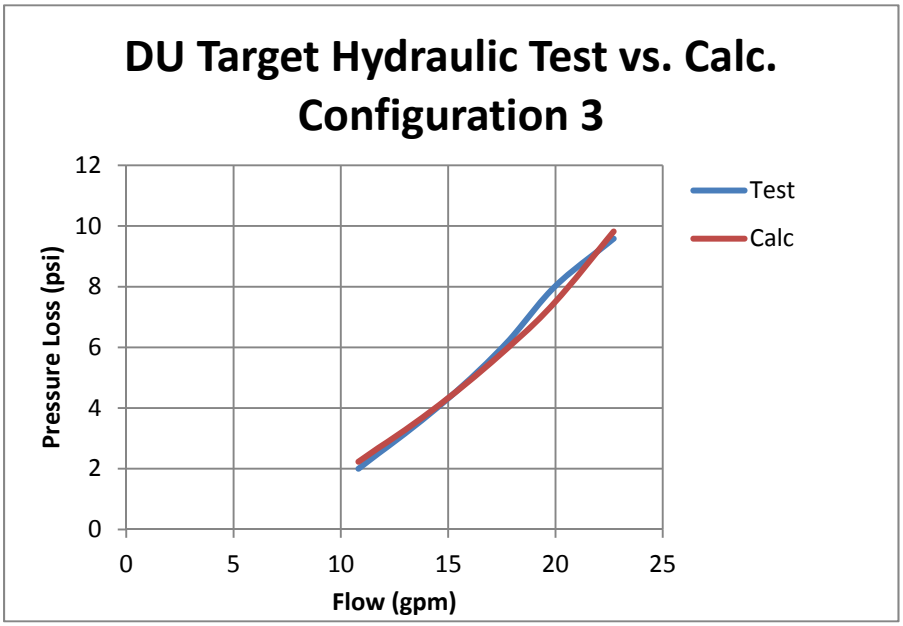
The test arrangement for configuration 3 is shown below. The purpose of this test is to validate the pressure losses associated with the flow across the front 4 disks in the target.



Test Arrangement



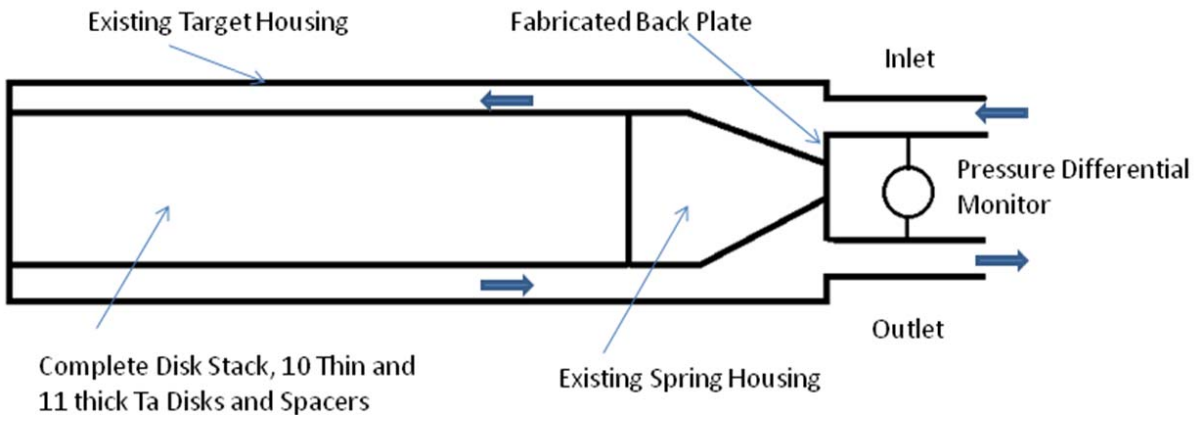
FATHOM Diagram (Note reversed image from Test Arrangement)



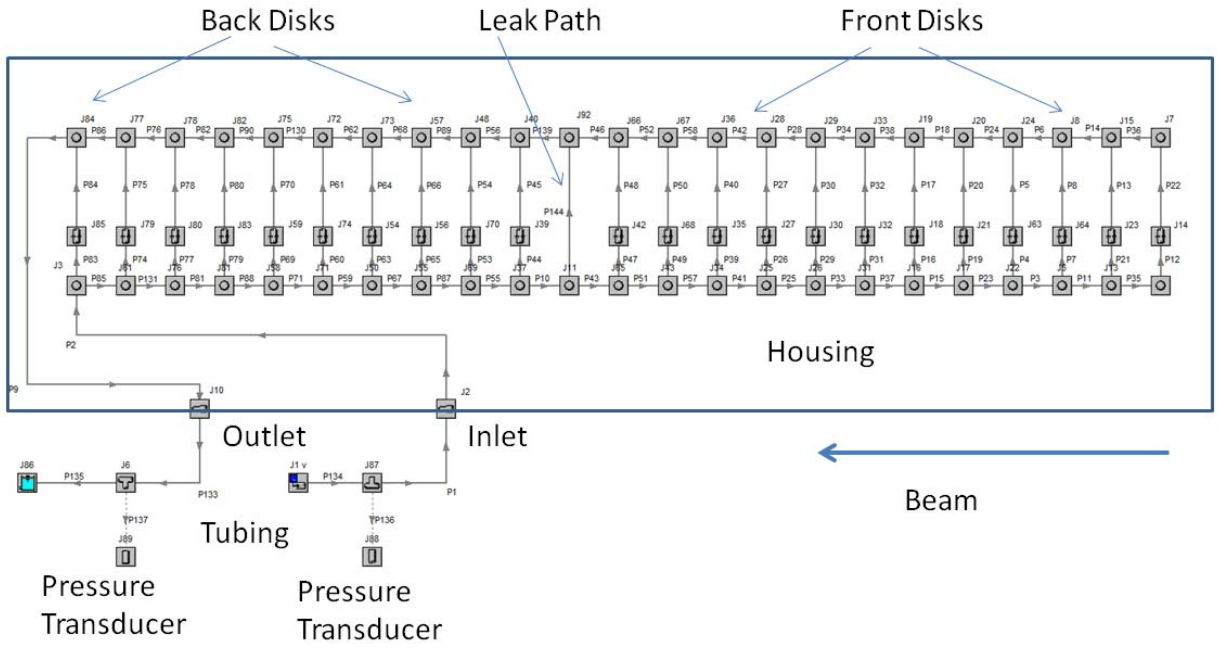
FATHOM vs Test

Test Description Configuration 4

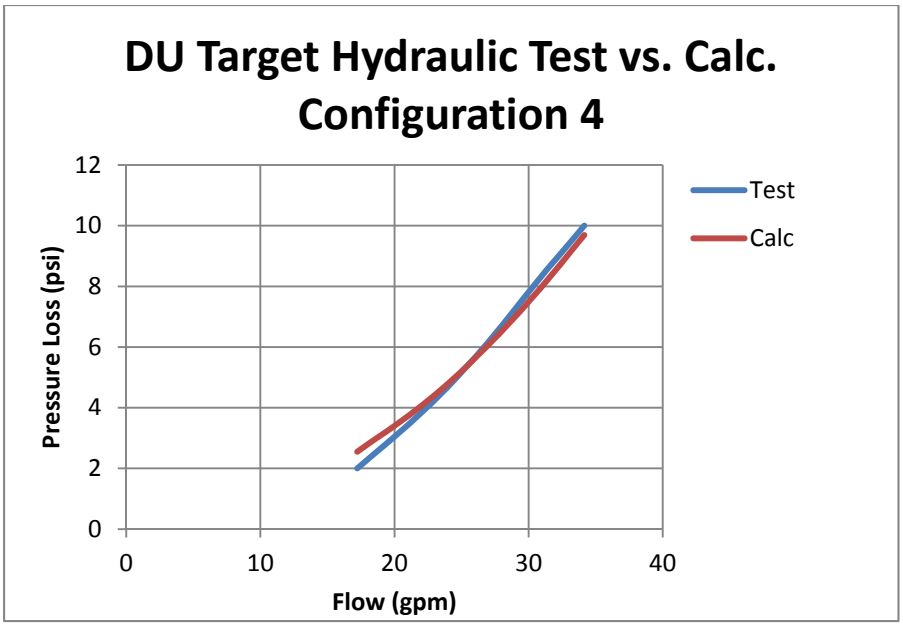
The test arrangement for configuration 4 is shown below. The purpose of this test is to validate the pressure losses associated with the overall flow through the target assembly from entrance to exit as simulated by the FATHOM code. All of the Ta disks and spacers are installed in the target housing along with the spring housing.



Test Arrangement



FATHOM Diagram (Note reversed image from Test Arrangement)



FATHOM vs Test

APPENDIX 4

Calculation Note NE-CALC-2015-04: "Evaluation of the Structural Integrity of Zircaloy-4 Clad Containment for the DU Target Disks"

CALCULATION COVER SHEET

Title: Evaluation of the Structural Integrity of the Zircaloy-4 Clad Containment for the DU Target Disks		
Date: March 14, 2017		
Analyzed System: DU Target Assembly		
PREPARER		
James L. Bailey (Print Name)	James L Bailey (Signature)	10/6/17 (Date)
REVIEWER		
James Grudzinski (Print Name)	(Signature)	10/25/17 (Date)
CALCULATION HAND CHECKED BY		
N/A (Print Name)	(Signature)	(Date)
FINAL APPROVER		
James Grudzinski (Print Name)	(Signature)	10/25/17 (Date)

REVISION LOG

REVISION	CHANGES	DATE
0	Initial Release	
1	Revised analyses based on flow tests and analyzed system considering a larger pump	March 14,2017

1. Objectives of the analysis

The object of this note is to verify that the Zircaloy-4 clad on the depleted uranium disks will provide satisfactory containment under normal and off normal operating conditions.

2. Background

During normal operation heat is generated within the Zircaloy-4 clad depleted uranium target disks due to impingement of the electron beam on the target. The target disks are cooled by DI water flowing between the disks (Refer to Figure 1). Because of differences in the thermal expansion between the Zircaloy-4 clad and the uranium inner disk, thermal stresses in the clad occur which eventually create cracks in the clad as a result of fatigue failure, that in turn, allows fission products from the uranium to enter the cooling water, thereby, limiting the usable life of the target. Thermal hydraulic analyses, thermal stress analyses, and UT testing has previously been performed for these disks. These references provide the basis for this calculation note.

3. Scope of the analysis

This note is intended to verify the structural integrity of Zircaloy-4 clad on the uranium disks during operation using the referenced information.

Essentially, all required analyses and tests have previously been performed as reported in these references. This note summarizes this work and provides a concise evaluation of their results.

4. Acceptance criteria

Acceptance criterion is based on a minimum number of allowable operating cycles of the target assembly. A cycle is defined as heat up of the disks from ambient temperature to a steady state condition with the beam at maximum power and then a complete cool down back to ambient temperature. An acceptable minimum number of cycles are 10,000. This requirement includes both normal and off normal conditions.

5. Methodology

The thermal hydraulic analysis of the disks is first address using reference 1. The worst case disk is identified and the thermal parameters are determined. Next, these parameters are used in the thermal stress calculations (reference 2) to determine the stresses and possible fatigue failure limit. Also, a parametric analysis of the effect of un-bonded areas of the clad to uranium is investigated. Lastly, UT test results (reference 3) are used to determine the size and location of possible un-bonded areas of the actual fabricated disks. By comparing the thermal stress calculation results and the UT results an allowable number of cycles are estimated.

6. Analysis Assumptions and Inputs

All assumptions are described in the references.

The references provide all inputs to this note.

Supplemental references provide additional information about the determination of the internal heat generation assumed in these reports. References 7, 8 and 9.

7. Discussion

Summary and Results for the Thermal/Hydraulic Analysis Note (Reference 1)

The object of this analysis was to determine the operating temperatures of the disks under normal and off normal conditions. The assumed acceptance criteria was to maintain the surface temperature of the worst case disk to below the saturation temperature at the operating pressure to prevent boiling in the coolant channel and to maintain the maximum temperature in the uranium to below 300°C to minimize thermal stress in the clad. The commercial computer code ANSYS CFX was used for these analyses. The internal heat generation distribution input for this analysis was a Gaussian profile with total generation obtained from a separate computer analysis. Flow conditions were obtained from the overall hydraulic analysis of the target cooling system and from flow tests performed on the actual target assembly (reference 5). The worst case disk was found to be a thin disk at location 2 and was the only disk analyzed for all cases. The normal and off normal cases were examined. The results of this analysis indicated that the disk temperatures were below the acceptable limit.

Summary and Results for the Thermal/Stress Analysis Note (Reference 2)

The object of this analysis was to determine the thermal stress in the Zircaloy-4 target clad for the worst case disk and the corresponding fatigue cycle life. The commercial code ANSYS was used for the finite element analysis. A thermal analysis was performed first using a heat generation rate and an effective convective coefficient at the surface of the disk from preliminary thermal hydraulic analysis. Note that the results of this preliminary analysis were found to be in agreement with the thermal convective coefficient and calculated disk temperatures as determined in the subsequent finalized thermal hydraulic report (reference 1), and therefore, assures that these stress results are correct and applicable to the final design.

The normal case for disk 2 was used for all the parametric study. This study evaluated un-bond areas by assuming small insulated circular areas between the clad and the uranium. The diameters of the circular areas were varied as well as their offset distance from the center. The worst case condition was found at the radial center of the disk with the largest area studied (I.e. 2mm diameter). The corresponding cladding life was 354, 000 cycles. This result is considerably above the minimum acceptable limit of 1,000cycles.

Summary and Results for the UT Tests (Reference 3)

The object of the UT tests was to determine the bonding status after final machining of the disks. These tests were performed by the fabricators of the disks (LANL). UT images for both the thin and thick disks are shown in the test reports. The thin disk UT results indicate negligible un-bonded area and essential conclude that these disks are completely bonded. The UT test resolution is noted as 0.125mm x 0.125mm and, as noted in the reference 4 email, any area larger than this size would be show up on the images, hence, it can be concluded that any small un-bonded area are smaller than this resolution. This resolution criterion is to be compared to the 2mm diameter un-bonded area evaluated in the thermal stress (reference 2). The UT results for the thicker disks do show notable un-bonded areas; however, because of the negligible heat generation in these thicker back disks their operating temperature is near ambient and thus has negligible thermal stress in the clad.

8. Conclusion

Thermal Hydraulic Analysis

The results of the thermal hydraulic analyses (reference 1) are shown in the plots below. Disk surface temperature verse beam power for several beam width are plotted. Figure 1 and 2 plots are considering a

flow rate for the smaller pump that was analyzed in reference 5. The saturation temperature of 126°C at the pressure calculated in the flow channel is also shown. Figure 1 plot indicates that for a beam width of 20mm FWHM a beam power of 13kW would provide a margin to boiling in the channel of 15%. And, for a beam width of 18mm FWHM the beam power is reduced to10.5kW in order to provide a 15% margin. Figure 2 plot indicates that for a beam width of 18mm FWHM a beam power of 18.5kW would provide a margin to boiling in the channel of 15%. Note that the electron energy of 40Mev allows for significantly higher beam power than the 35Mev energy for the same beam width and flow rate. This result is due to the axial spreading of the heat generation. Figure 3 plot indicates that for a beam width of 18mm FWHM a beam power of 18.5kW would provide a margin to boiling in the channel of 15%. Figure 3 and 4 plots are considering a flow rate for the larger pump that was analyzed in reference 5. The saturation temperature of 134°C at the pressure calculated in the flow channel is also shown. Figure 3 plot indicates that for a beam width of 20mm FWHM a beam power of 16kW would provide a margin to boiling in the channel of 15%. And, for a beam width of 18mm FWHM the beam power is reduced to13kW in order to provide a 15% margin. Figure 4 plot indicates that for a beam width of 18mm FWHM a beam power of 20kW would provide a margin to boiling in the channel of greater than 15%.

A 15% margin to boiling is assumed to be a reasonable uncertainty factor based on engineering judgment considering: a 5% error in analysis (noting that flow testing were performed on the actual target assembly and ANSYS CFX is a well validate thermal hydraulic computer code); a 5% allowance for the flow switch beam trip (considers a trip of 2gpm below the operating flow rate); 5% for beam power and width uncertainty (note that surface temperature is linear with beam power, however it is to the square of the beam width).

In conclusion, it is recommended that the target systems be operated at the 15% uncertainty factor as described above.

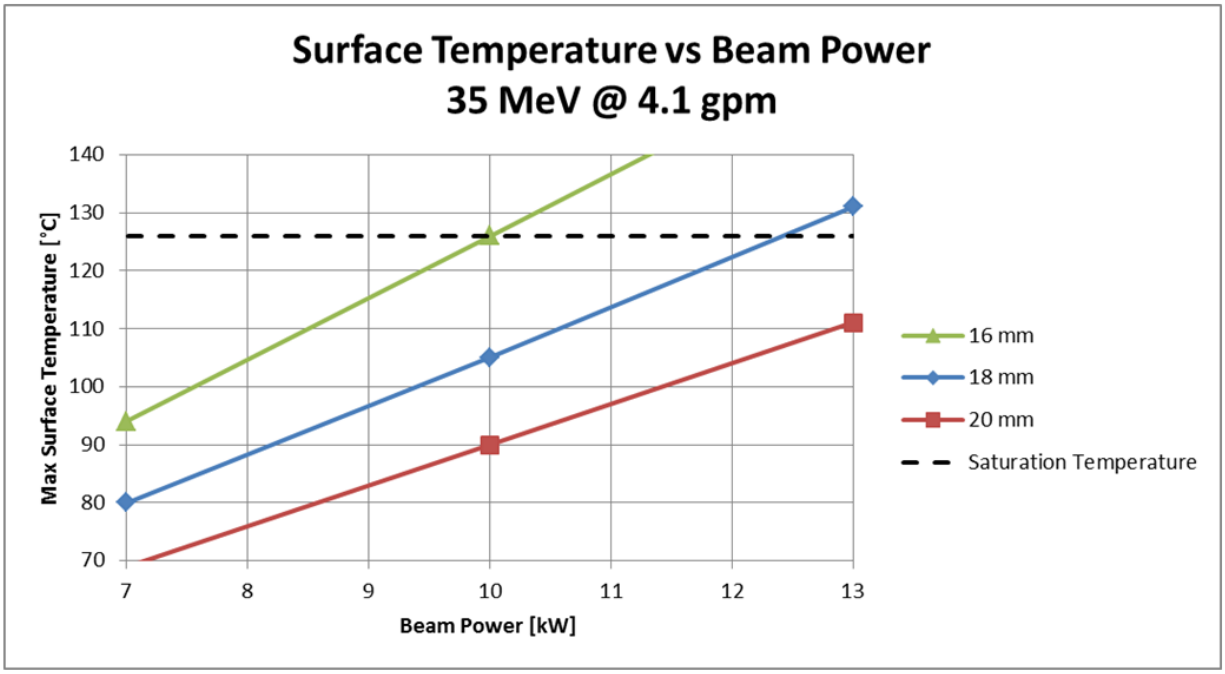


Figure 1 Plot

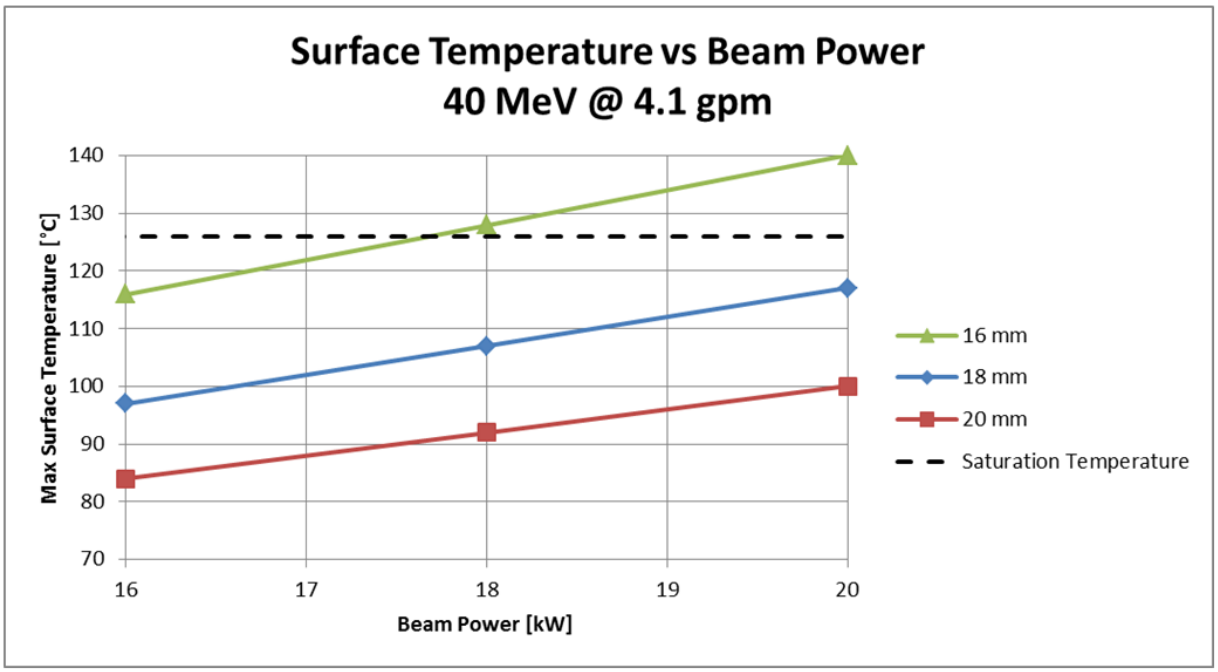


Figure 2 Plot

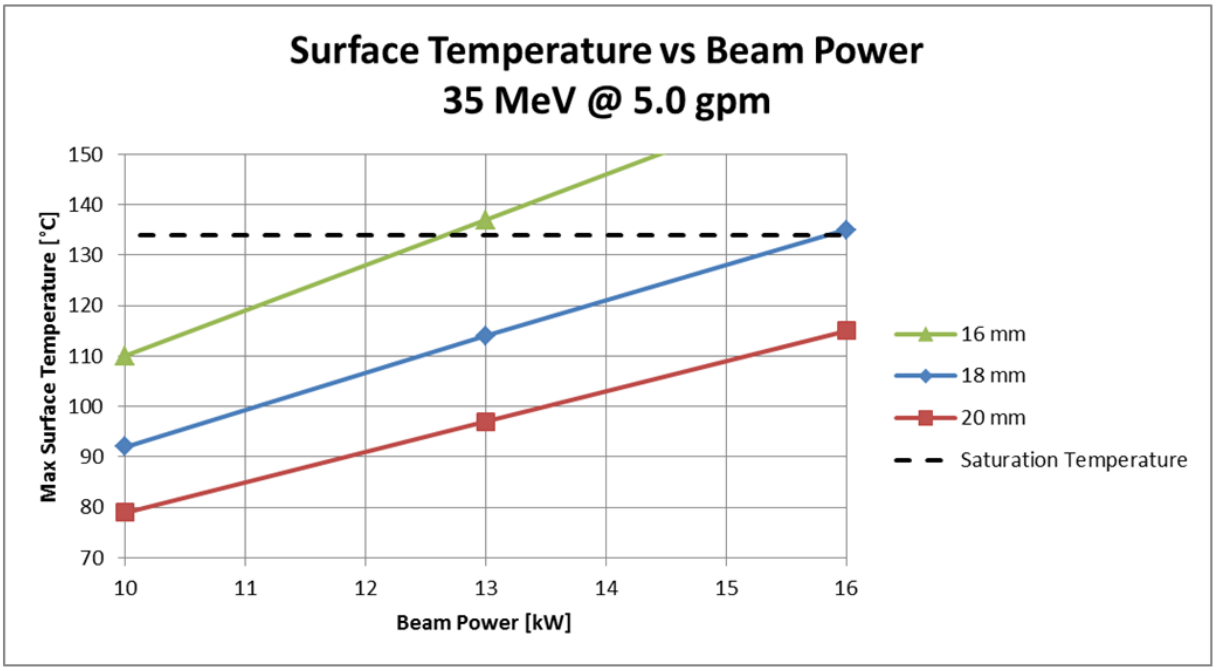


Figure 3 Plot

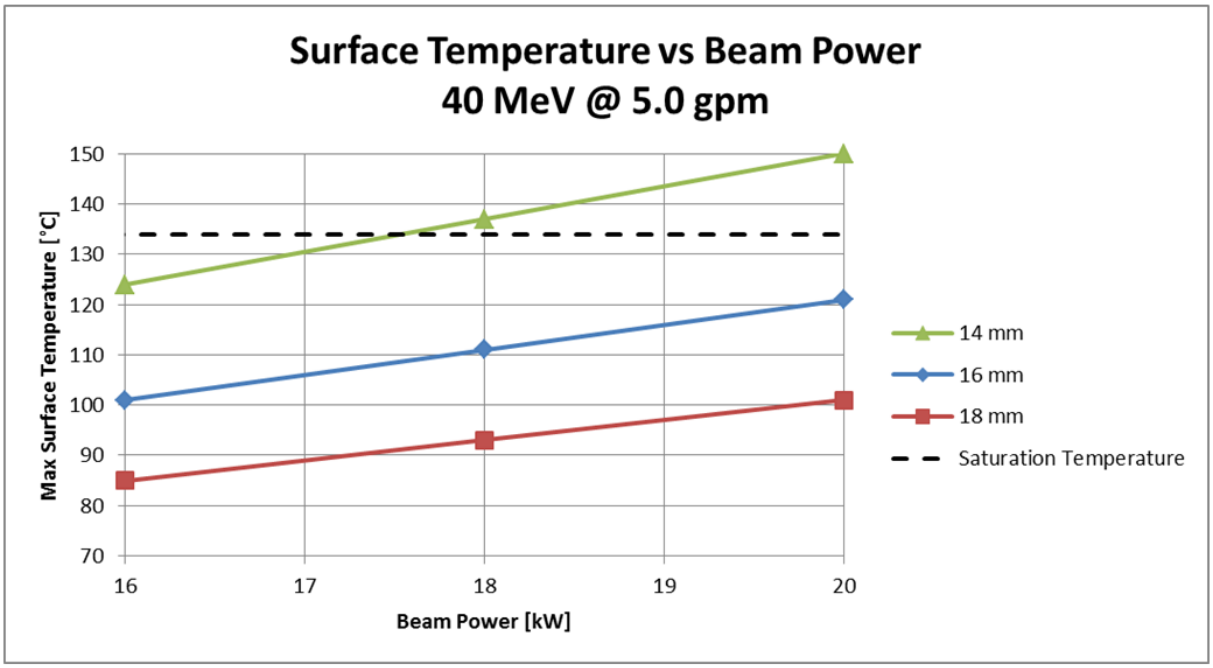


Figure 4 Plot

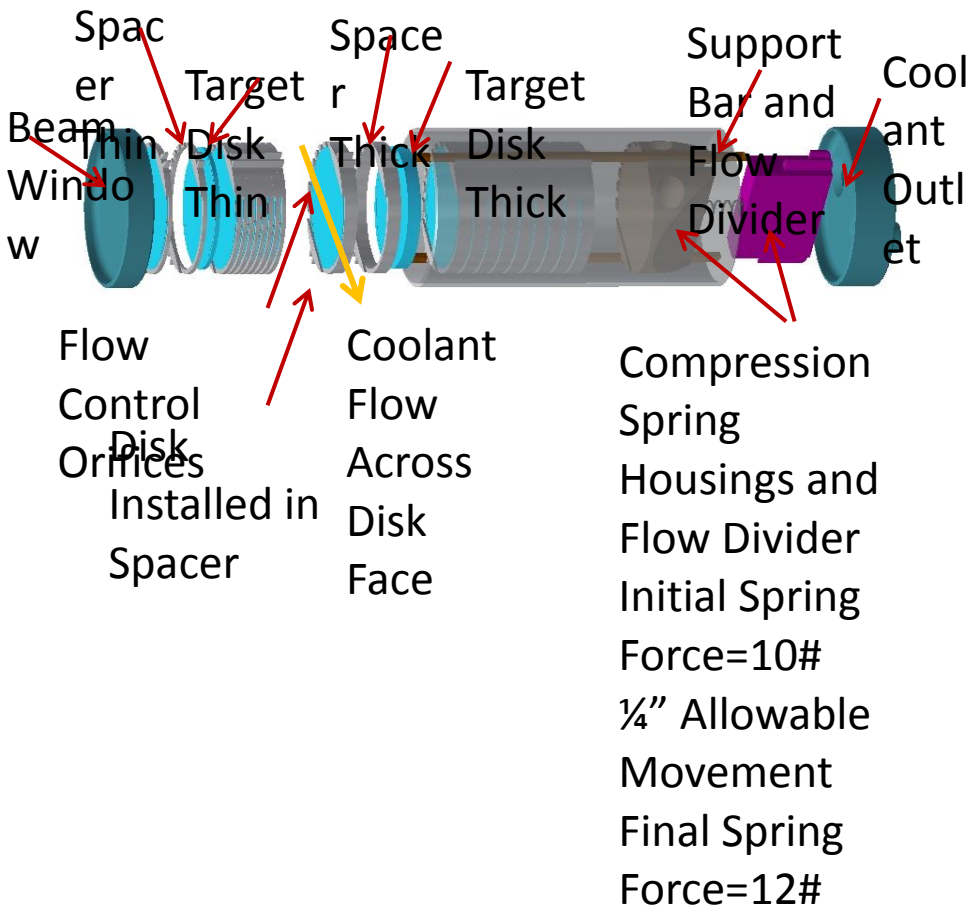
Clad Stress Analysis

The thermal stress analyses (reference 2) determined that the worst case condition was at the radial center of the disk with the largest area studied (I.e. 2mm diameter). The corresponding cladding life was 354, 000 cycles. This result is considerably above the minimum acceptable limit of 10,000cycles. Further the UT tests for the thin disks (Reference 3) indicated that there was negligible un-bonded areas (I.e. any un-bonded areas were smaller than 0.125mm x 0.125mm) This resolution is below the worst case size studied in the thermal stress analyses (2mm diameter), hence, the actual cycle life of the disks is expected to be greater than the 354,000 cycles indicated by the analyses which is far greater than the required 10,000 cycles. Because the heat generation in the thicker disks is negligible they were not analyzed.

Also, the off normal occurrence of the stoppage of coolant through the DU Target was analyzed (reference 6). And based on this study, it was concluded that upon an off normal occurrence of the stoppage of water coolant flow through the target, the temperature of all target components will remain below their maximum allowable design temperatures.

Based on the above referenced analyses and tests it is concluded that the disks will provide satisfactory containment under normal and off normal operating conditions.

9. Figure



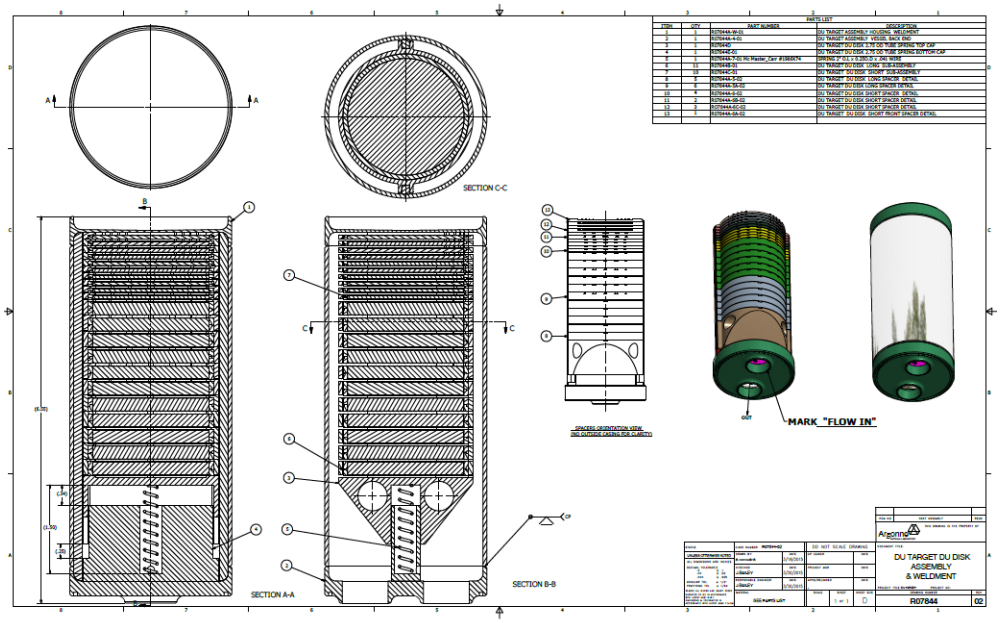


Figure 5 DU Target Assembly

APPENDIX 1
GENERAL CHECKING CRITERIA SHEET

CALCULATION CHECKLIST	Yes	No	N/A	Comments
1. Are analytical methods appropriate?	☐	☐	☐	
2. Are assumptions appropriate?	☐	☐	☐	
3. Is the calculation complete?	☐	☐	☐	
4. Are formulas appropriately referenced?	☐	☐	☐	
5. Are the input data appropriate?	☐	☐	☐	
6. Was utilized software appropriate for the task?	☐	☐	☐	
7. Were software input/initial conditions/properties/boundary conditions appropriate?	☐	☐	☐	
8. Are the results reasonable?	☐	☐	☐	

APPENDIX 1
GENERAL CHECKING CRITERIA SHEET

ADDITIONAL COMMENTS		
Number	**Comment**	**Resolution**
1.		
2.		
3.		
4.		
5.		
6.		
7.		
8.		
9.		
10.		

APPENDIX 2
REFERENCES

Reference 1

Calculation Note: NE-CALC-2015-05 Rev. 1

Reference 2

Calculation Note: NE-CALC-2015-06

Reference 3

Memorandum

Applied Engineering Technologies
AET-6 Nondestructive Testing & Evaluation
To/MS: Maria Pena, MST-6, x7-4119
From/MS: D. A. Summa, AET-6, MS P915
Phone/Fax 5-1854 / Fax 5-7176
Symbol: SHINE-Thin Post Machining UT-A Disks
Date: 9 March 2015
Ultrasonic Inspection of Thin SHINE Assemblies

Reference 4

Hi Maria—

Pixels for these inspections are .125mmx.125mm (~ 0.005" x 0.005"). For something to be unbonded and show up as bonded, it would have to be considerably smaller than the pixel. How much smaller is a good question, I'm not sure I can give you a definitive answer. (If I had to hazard a guess, I'd say maybe ¼ of the pixel size? But that's purely just a guess. This is where having that standard with known defects that Don Bucholz wanted to make would be helpful.)The transducer is essentially averaging returns over the spatial area of the pixel. A really small bad area within a single pixel would change the return signal only a little, while a larger area would make for a bigger change. Part of setting the threshold involves looking at the waveform and trying to figure out what the cutoff is. Another thing to consider is that we've looked at these several times with similar results—there is no way we are able to replace/re-align the part exactly to its previous location, meaning that a smallish defect that happened to straddle pixels and thus perhaps not show up in one scan would be unlikely to have the same thing happen in a subsequent scan.

Deb

Maria I. Peña , Ph.D.

Los Alamos National Laboratory

MST-6, MS G770

Phone: 505-667-4119

Reference 5

Calculation Note: NE-CALC-2015-03

Reference 6

Calculation Note: NE-CALC-2015-69-v1

Supplemental References

Reference 7

Power deposition for 35Mev electron beam

Thu 3/13/2014 5:45 PM

Vakho Makarashvili <makarashvili@anl.gov>

Power deposition in DU disks

To Bailey, James L.

Cc Chemerisov, Sergey D.

You forwarded this message on 3/3/2017 2:55 PM.
We removed extra line breaks from this message.

meshplots-Disks-DU.pdf
.pdf File

Power_DU-Disks.xls
.xls File

Phase-2 miniSHINE-MIPS Target report 07 31 2012.pdf
.pdf File

Jim,

Please see the power deposition results in DU disks. The first plot in "meshplots-Disks-DU.pdf" is the power density. The units are [kW/cc] per 1 kW of beam power. I also included the Excel file which has the total power deposition per disk in kW (per 1 kW beam).

Figure 5 in the Phase-2 miniSHINE report also shows the power density data but in [W/cc] per kW of beam.

Vakho

Vakho Makarashvili, PhD.
Assistant Physicist, CSE Division
Argonne National Laboratory
9700 S. Cass Ave.
Argonne, IL 60439

Tel: 630-252-4538
Fax: 630-252-5246
E-mail: makarashvili@anl.gov

Reference 8

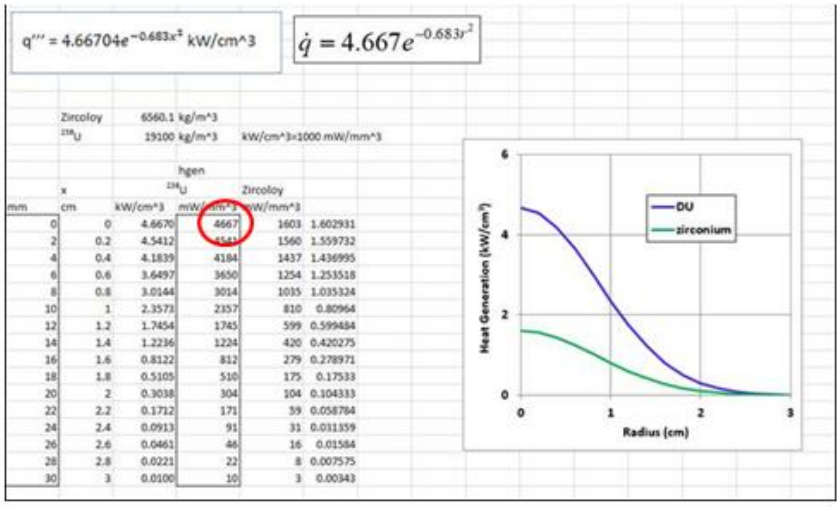
Power deposition for 40Mev electron beam

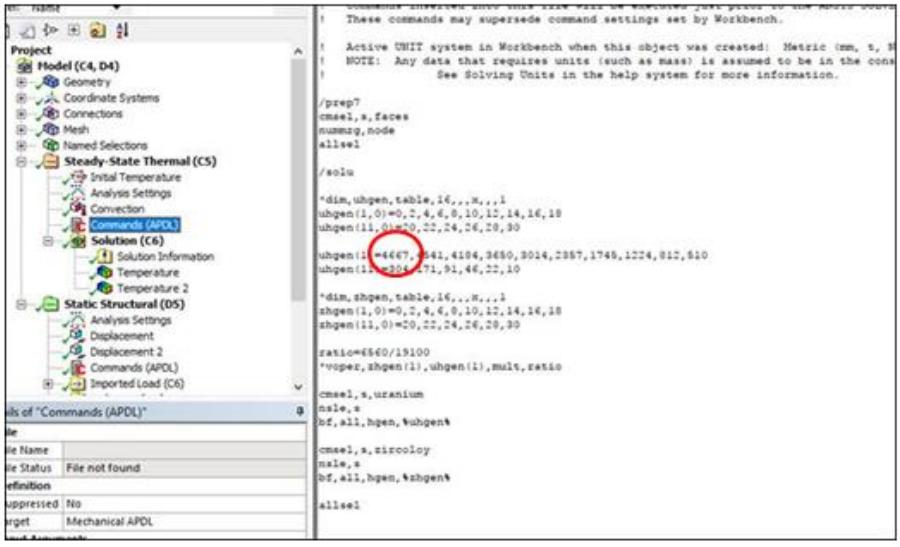
Text file entitled “Power-data-(r,z)_DU-40Mev” by M. Vakho

Reference 9

Email from R. Fischer indicating heat generation density that was used in reference 6.

From: Fischer, Richard
Sent: Thursday, May 25, 2017 2:11 PM
To: Grudzinski, James J. <jjg@anl.gov>; Bailey, James L. <jbailey@anl.gov>
Subject: DU Target





APPENDIX 5

Calculation Note NE-EO-2014-006: "DU Target Disk Clad Analysis"

Title: DU Target Disk Clad Analysis	
Calculation No.: NE-EO-2014-006	Revision Number: 0

CALCULATION COVER SHEET

Supersedes Calculation No.:	Total Number of Attachments:
Analyzed System: Target Cladding Analysis	
Purpose of Revision: Initial Issue	

PREPARER

Richard L. Fischer — Richard L. Fischer — 11/10/2015

Print Name — Signature — Date

REVIEWER

SAURIN MAJUMDAR — [signature] — 11/10/15

Print Name — Signature — Date

VENDOR APPROVER (if vendor-supplied calculation)

n.a.

Print Name — Signature — Date

FINAL APPROVER

Jim Grudzinski — [signature] — 11/10/15

Print Name — Signature — Date

TABLE OF CONTENTS

LIST OF EFFECTIVE PAGES

Pages	Revision
1 to 22	0

1. Objectives

The objective of this analysis was to analyze the Zircaloy-4 cladding on the Short Depleted Uranium (DU) Disk Sub-assembly to determine if the presence of bonding flaws constitutes a failure hazard.

2. Scope

The scope of this analysis was limited to the Short DU Disk Subassembly used in the DU Target Assembly shown in drawing R07844 dated 2/7/2014.

3. Background

The DU disk subassemblies consist of a disk of depleted uranium clad in zirconium alloy. The top and bottom cladding halves are machined from Zircaloy-4 stock. A lump of DU is placed between them and this is heated and compacted in a vacuum. The cladding halves are electron beam welded together and this assembly is then machined to final dimensions. This manufacturing process results in a weld-like bond between the cladding and the DU. Radiographic examination has revealed the prescience of flaws in the bond, which are of random size, shape and location within the disk assembly. There is concern that these flaws will result in localized hot spots due to the lack of heat transfer at these flaws, and that this could lead to higher stress at these locations that could result in fatigue failure. The desired life is thought to be under 1000 cycles.

4. Methodology

The short DU disk subassembly was analyzed with the Ansys finite element program. The thermal loads were based on an estimate of heat generated by the specified electron beam, and the cooling parameters were derived from a previous conjugate heat transfer analysis.

5. Overview of Analysis

A total of eight load cases were analyzed. These load cases represent a variation in the size and location of a small circular flaw in the bonded interface between the cladding and the depleted uranium. A thermal analysis was performed on a half-symmetry model loaded with a thermal flux representative of the heat generated by the electron beam, and cooled by convection with water running over its outside surface. The resultant temperatures were then applied as a structural temperature load in a structural analysis. The cladding was evaluated for failure by short and long cycle fatigue.

6. Assumptions

This analysis is based on the following assumptions:

1. Material response is constant with time (no effects of aging, corrosion, irradiation, etc.).
2. Materials are isotropic and homogeneous.
3. Residual stresses are not included.
4. The flaw results in a perfectly insulated boundary.
5. The cladding is at nominal 0.010" thickness, with no variation due to the final machining operation.

7. Geometry

Geometry is based on drawing number R07844C, DU Target DU Disk Short Sub-assembly, dated 2/7/2014. A solid model based on the drawing was constructed with the Design Modeler module, and consists of a depleted uranium disk between top and bottom Zircaloy-4 claddings. The geometry represents a uniform nominal configuration after final machining. The circular flaw is created by slicing a disk from the top cladding. All parts except this disk are combined as a multi-body part. This part and the disk are imported into the Workbench environment, where all the nodes on the outside diameter of the disk and the mating surface on the top cladding are merged with a command snippet. This results in a free surface between the bottom of the disk and the top of the uranium. This is shown if Figure 1.

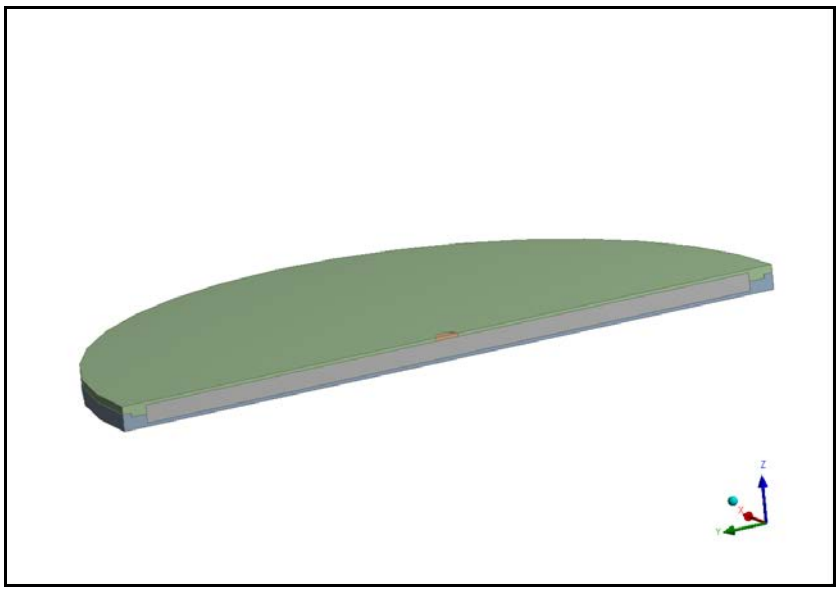


Figure 1
Solid Geometry

The model was meshed with 173,279 quadratic brick and Tet solid elements, as shown in Figure 2. Sliding contact with a thermal conductance of 1E-5 W/mm2 was placed on the flaw surface to enforce dimensional continuity. The low thermal conductance was used to thermally insulate the flaw.

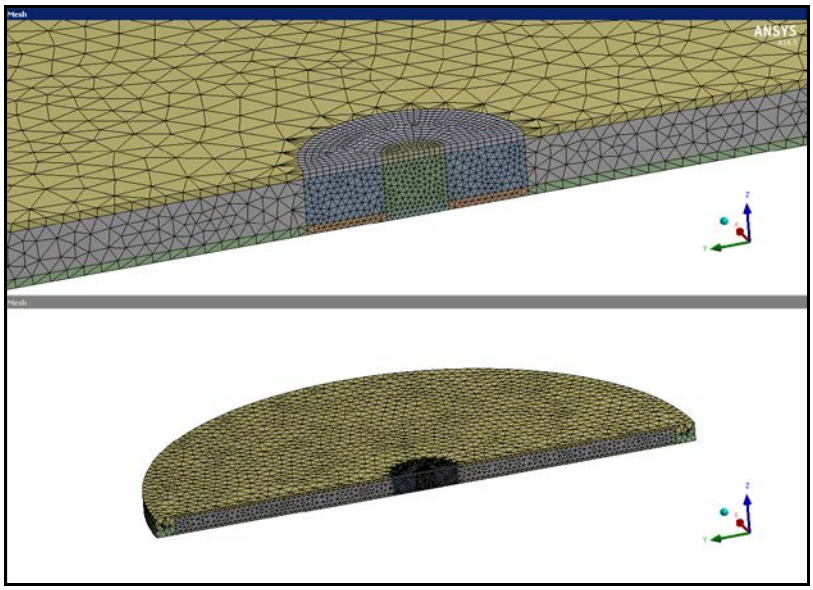


Figure 2
Finite Element Model

8. Materials

Material properties for depleted uranium are based on values given for bulk uranium on Wikipedia. Material properties for Zircaloy-4 are based on Ref. 1. Room temperature data is shown in Table 1.

	DU	Zircoloy-4
ρ (kg/m^3)	19100	6560
E (GPa)	208	92.4
Sy (MPa)	-	381
ν	0.23	0.35
α (C^{-1})	13.9e-6	5.59E-06
k (W/m-K)	27.5	21.5

Table 1
Material Properties for DU

Temperature dependent material data was located for Zircaloy-4 and is show in Figure 3 through Figure 6.

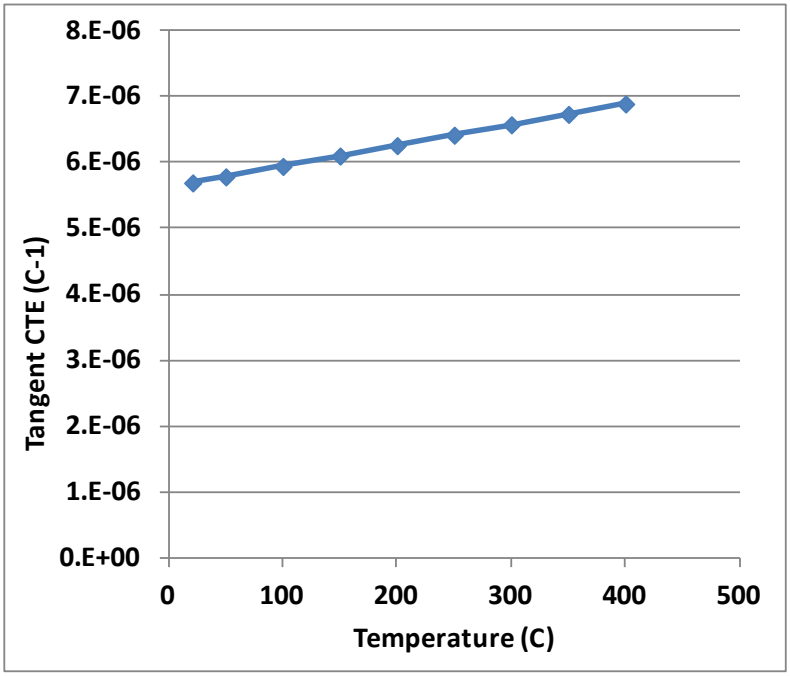


Figure 3
Instantaneous Linear Coefficient of Expansion for Zirconium Alloy from Ref. 1

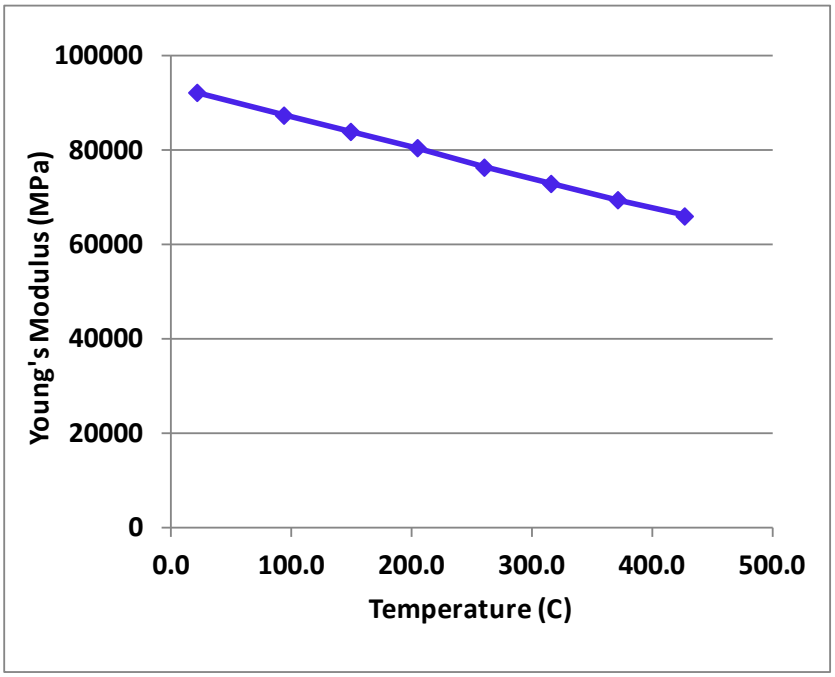


Figure 4
Young's Modulus for Zirconium Alloy from Ref.1

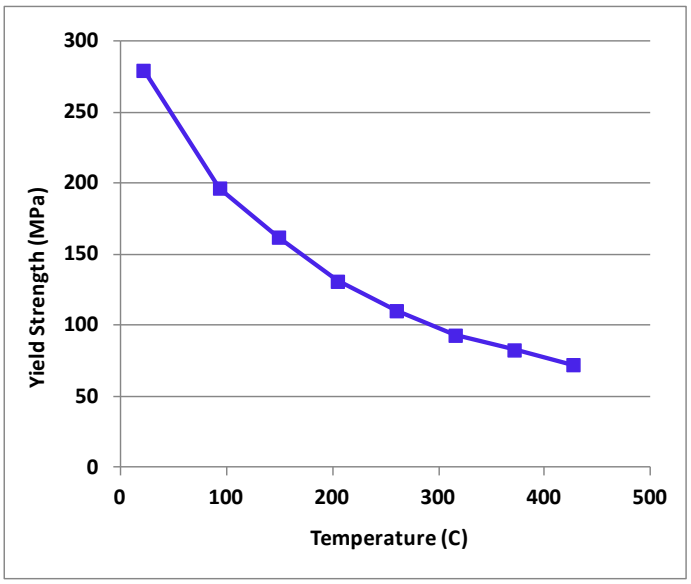


Figure 5
Yield Strength for Zirconium Alloy from Ref. 1

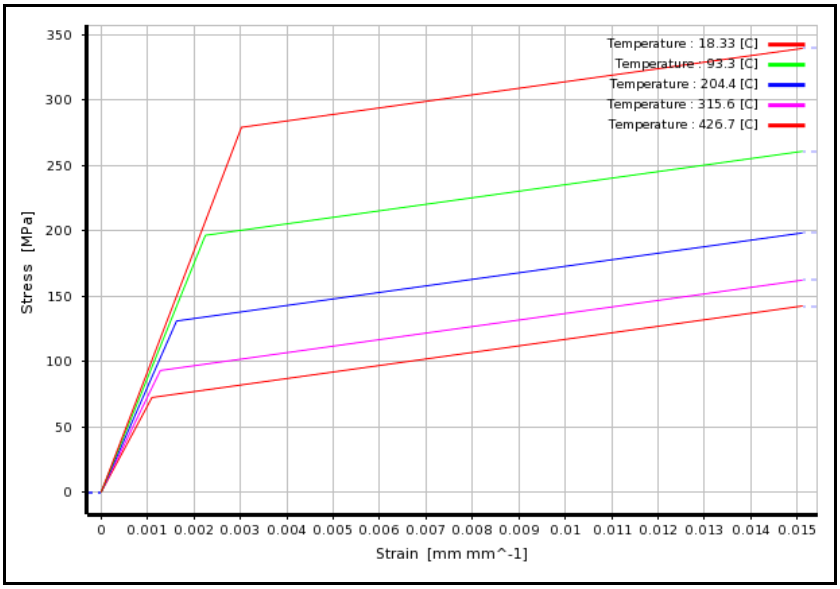


Figure 6
Elastic, Linear Plastic Material Model for Zirconium Alloy

9. Boundary Conditions

The internal heat generated in W/cm^3 by the electron beam was based on an estimate of the maximum heat generated in a DU disk by the electron beam. This value was fitted to the following Gaussian distribution that would produce 95% of that total in the 2" diameter DU disk:

$$\dot{q} = 4.667e^{-0.683r^2}$$

where r is the radius from the beam center. The heat generated in the cladding was found by multiplying this value by the ratio of the density of Zircaloy-4 to the density of DU. These distributions are plotted in Figure 7.

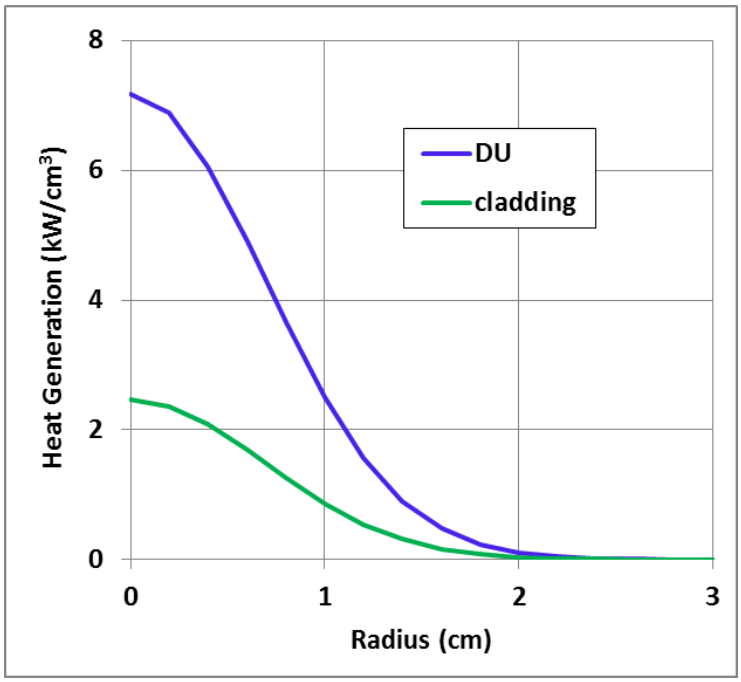


Figure 7
Heat Generation in DU and Cladding

The model was cooled by convection on the top and bottom surfaces of the disk. A film coefficient of 3.93e-2 W/mm2 was used with a water temperature of 18.33 C. The film coefficient value was the average of the calculated values from an unpublished conjugate heat transfer analysis performed by Phil Strons

In the structural analysis, the half symmetry model was restrained as shown in Figure 8.

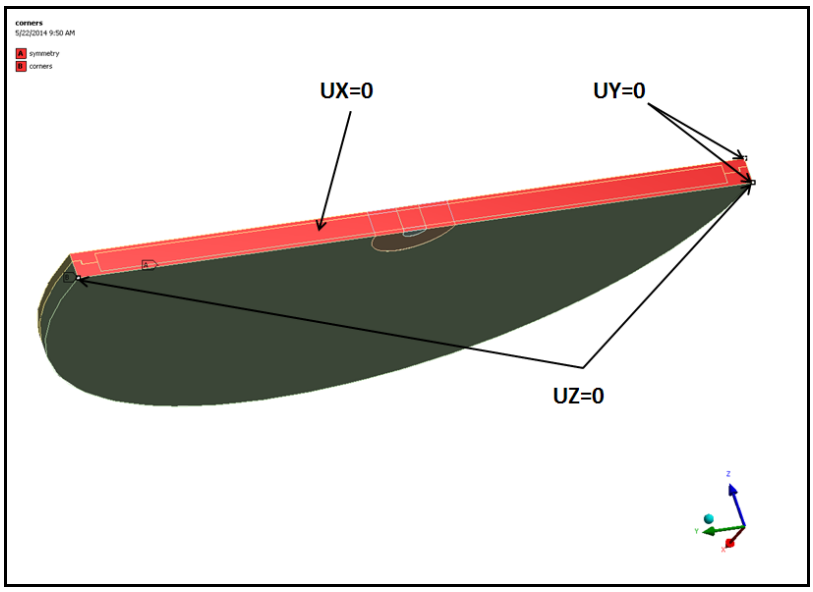


Figure 8
Structural Boundary Conditions

10. Solution and Results

The details of the eight load cases are shown in Table 2. Load case #1 is a baseline run with no flaw. Load Cases #2 through #4 include a 1/16" diameter flaw at various distances from the center of the disk. Load cases #5 through #8 have flaws of varying diameters in the center of the disk.

Load Case	Flaw Parameters		Maximum Temperature (C)		Equivilent Stress (MPa)	Max. Equiv. Total Strain	Cladding Life (cycles)
	Dia. (in)	Offset (in)	DU	Zirconium			
1	0.0000	0.000	180.9	142.1	105.8	0.001217	3.74E+06
2	0.0625	0.000	201.6	148.5	174.2	0.002331	4.20E+05
3	0.0625	0.093	195.0	145.2	169.2	0.002299	4.40E+05
4	0.0625	0.187	180.9	142.2	157.4	0.002117	5.81E+05
5	0.0469	0.000	189.9	144.9	142.4	0.001786	1.03E+06
6	0.0547	0.000	194.9	146.2	153.4	0.002042	6.56E+05
7	0.0703	0.000	208.2	149.9	179.1	0.002358	4.04E+05
8	0.0781	0.000	215.7	152.2	185.8	0.002452	3.54E+05

Table 2
Summary of Results

All analyses were conducted in the Workbench environment. A steady state thermal analysis was performed on the disk with the heat generation and convection boundary condition described above. The thermal result was transferred to a structural analysis where the nodal temperatures were applied as a structural load. A temperature dependent elastic-linear plastic material model shown in Figure 6 was used for the Zircaloy. A linear elastic material was used for the DU.

Plots of temperature and von Mises stress for load case #1 is shown in Figure 9 through Figure 11. Maximum temperature was 180.9 C in the DU and 142.15 C in the cladding. The maximum von Mises stress was 105.8 MPa. Fatigue was evaluated by comparing the maximum total equivalent strain in the cladding to the fatigue curve shown in Figure 14. This curve is based on Figure 13 from Ref. 2. The raw data was generated for Zircaloy-2, but per Ref. 2, Zircaloy-4 is slightly better than Zircaloy-2, so the use of this data is conservative.

Plots of temperature, stress, strain and cycles to failure as a function of flaw size are shown in Figure 15 through Figure 19. Contour plots of temperature, equivalent stress, and equivalent plastic strain for Load Case #2 (r=0) are shown in Figure 20 through Figure 24.

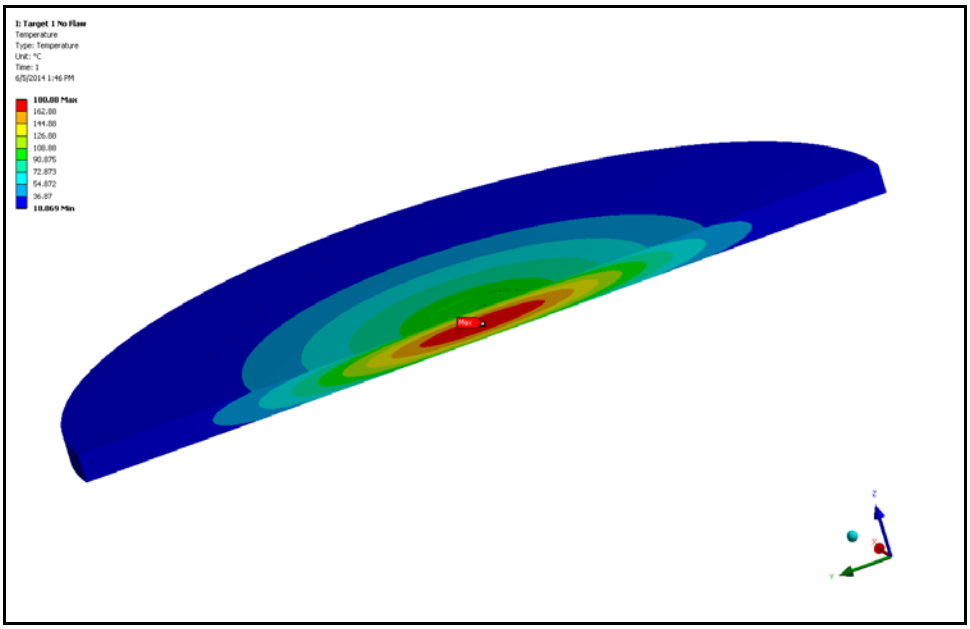


Figure 9
Temperature (C), Load Case 1

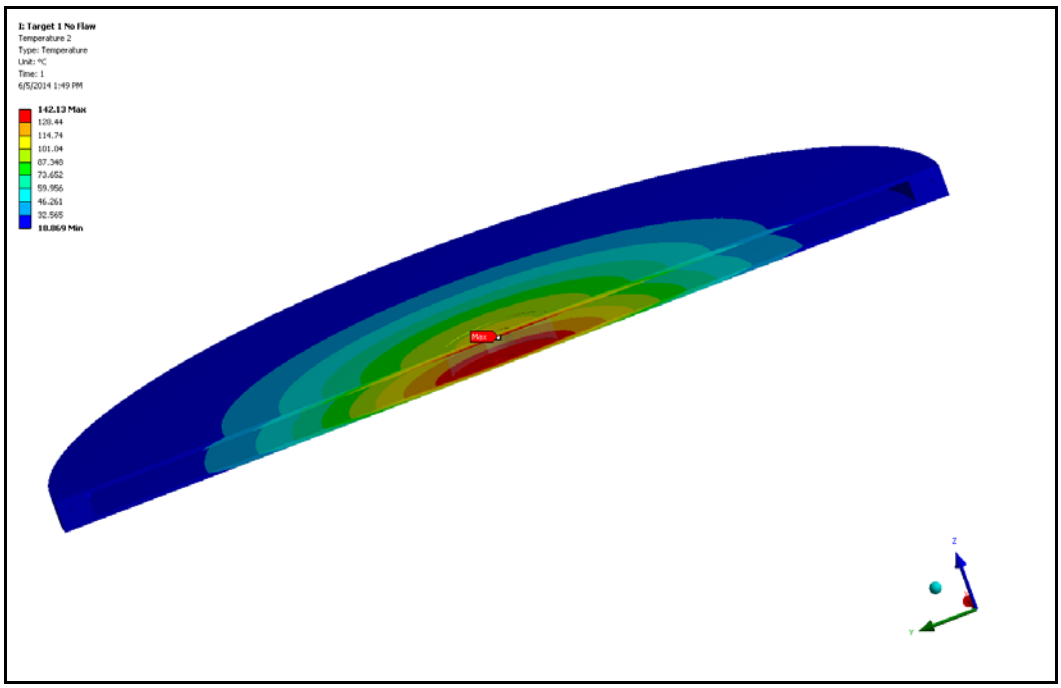


Figure 10
Cladding Temperature, Load Case 1

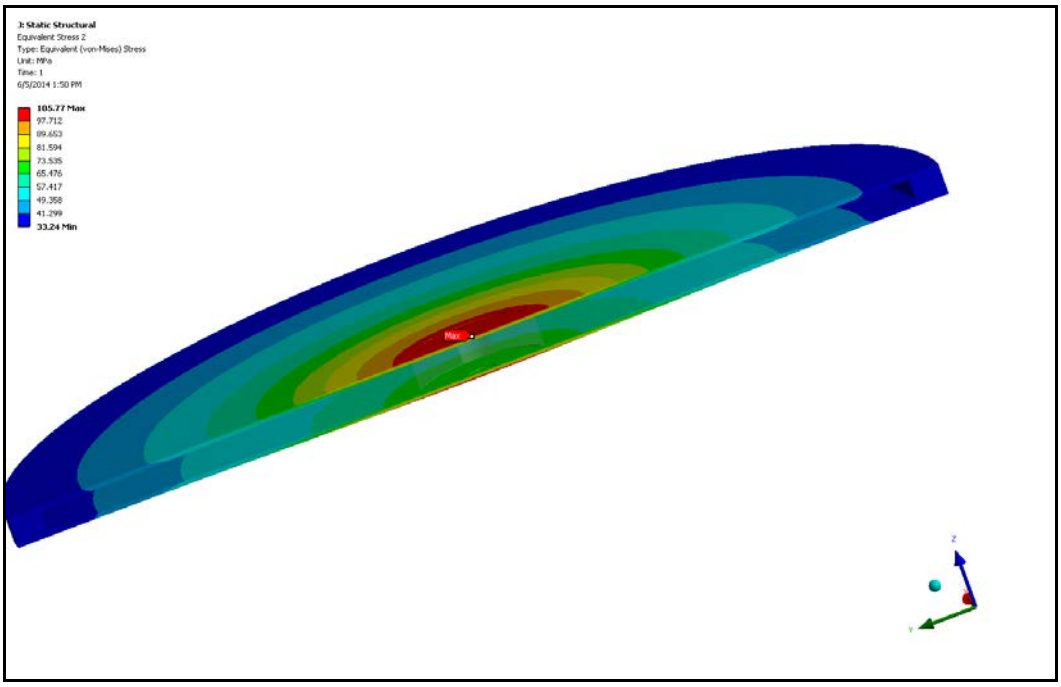


Figure 11
Equivalent Stress in Cladding, Load Case 1

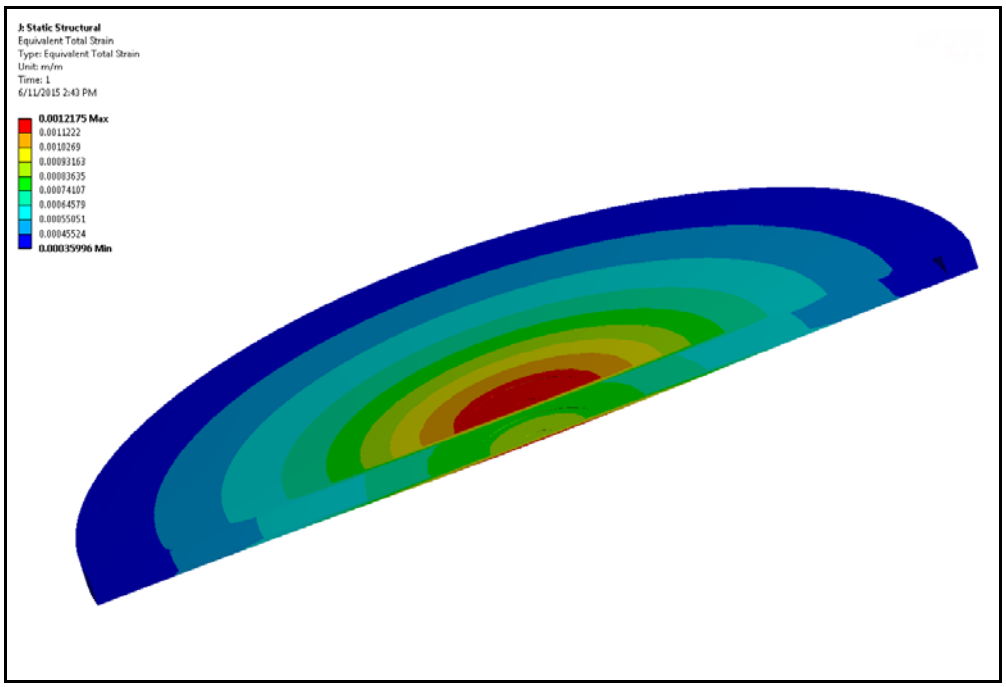


Figure 12
Equivalent Total Strain in Cladding, Load Case 1

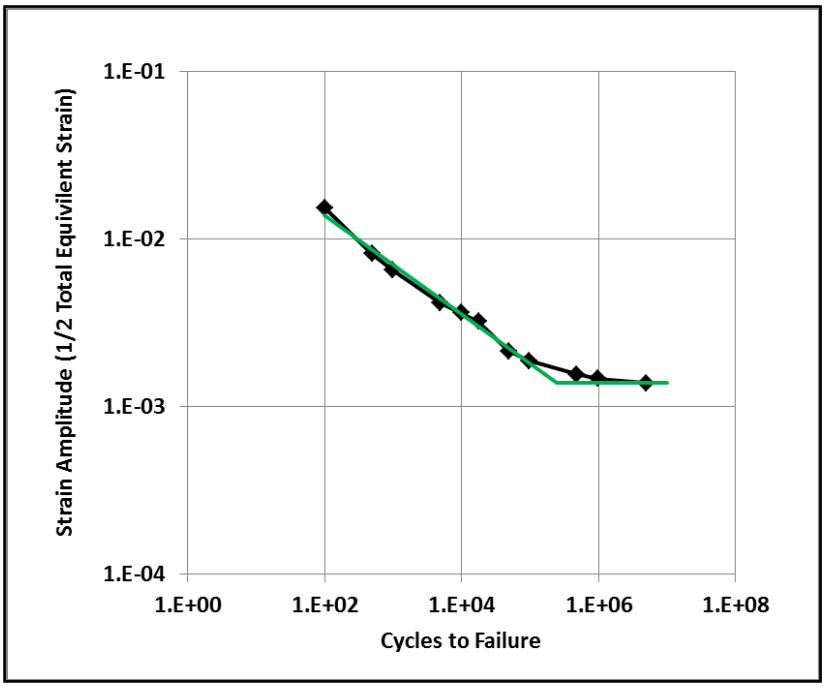


Figure 14
Fatigue Model for Zircaloy-4

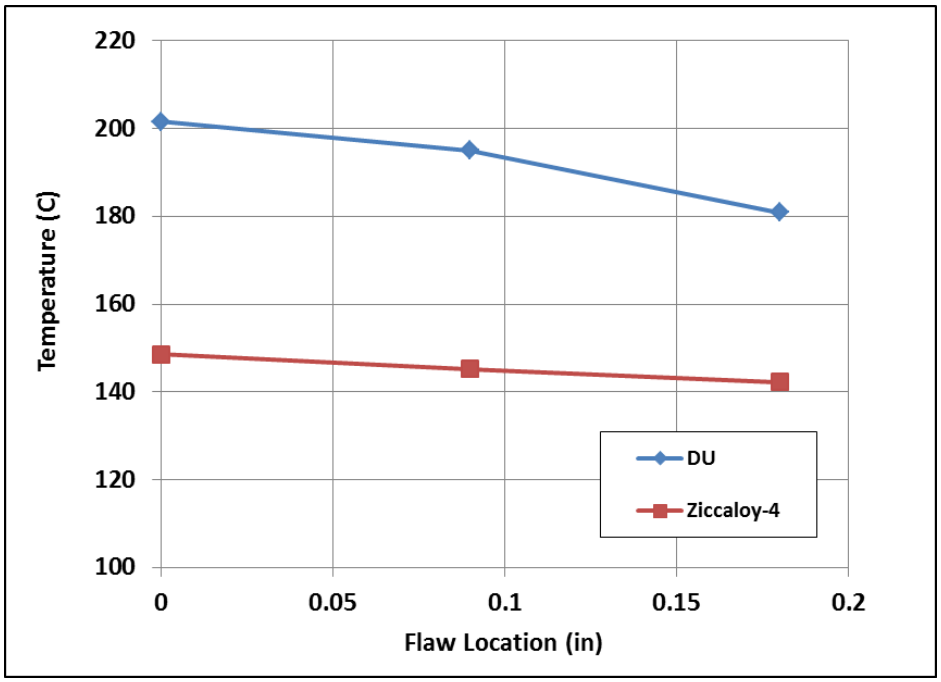


Figure 15
Maximum Temperature vs. Flaw Location

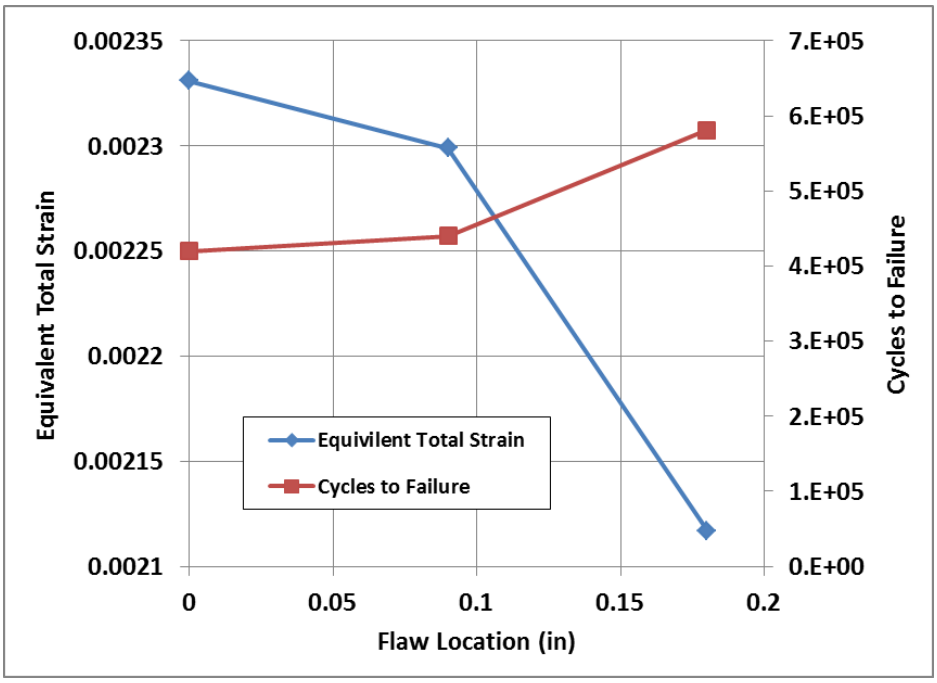


Figure 16
Equivalent Total Strain and Cycles to Failure vs. Flaw Location in Cladding

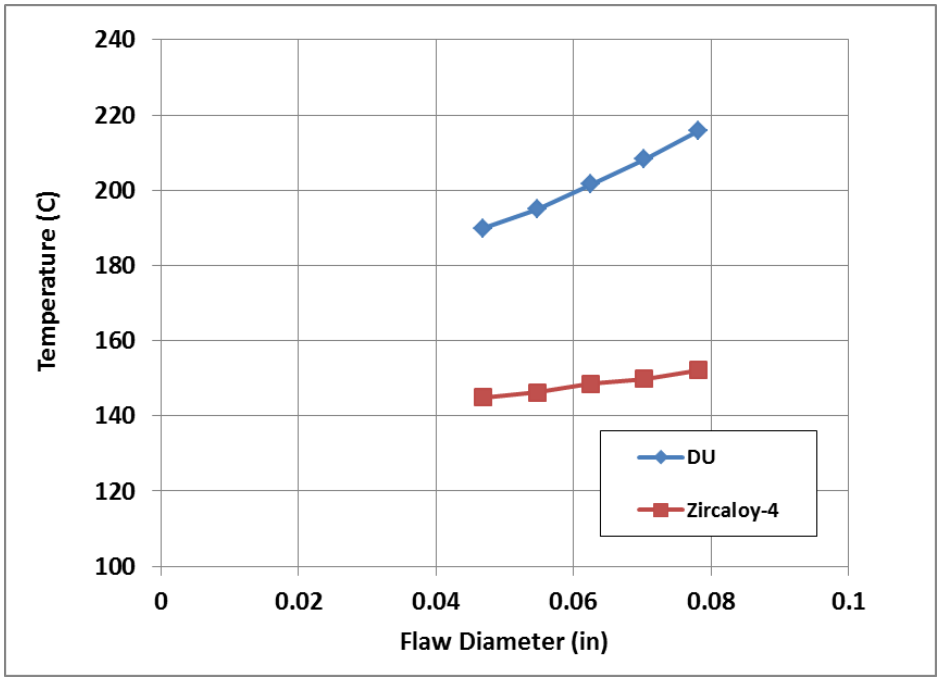


Figure 17
Maximum Temperature vs. Flaw Size

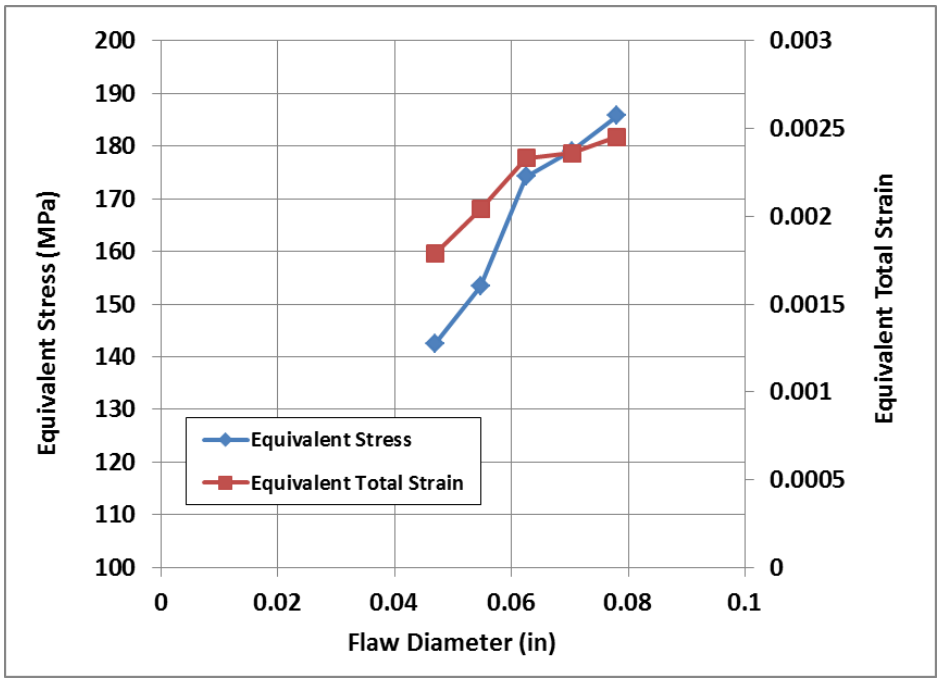


Figure 18
Stress and Strain vs. Flaw Size

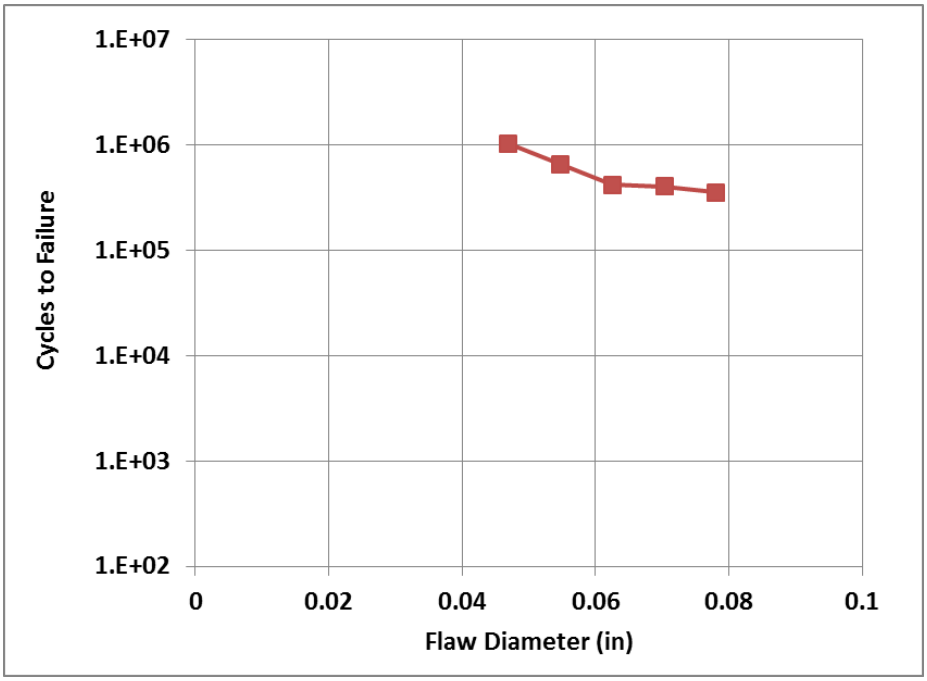


Figure 19
Cycles to Failure vs. Flaw Size

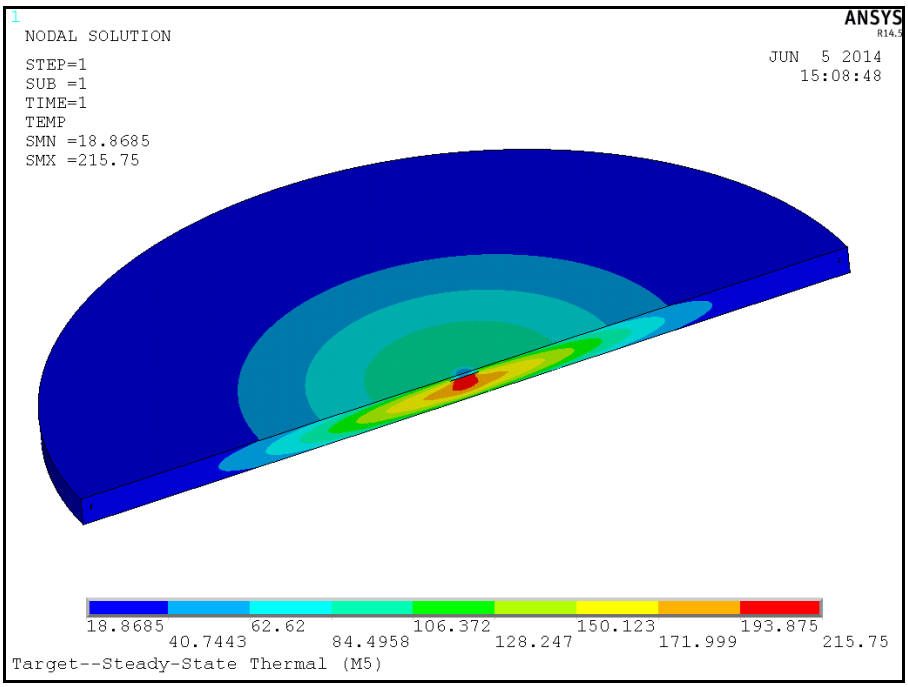


Figure 20
Temperature (C), Load Case 8

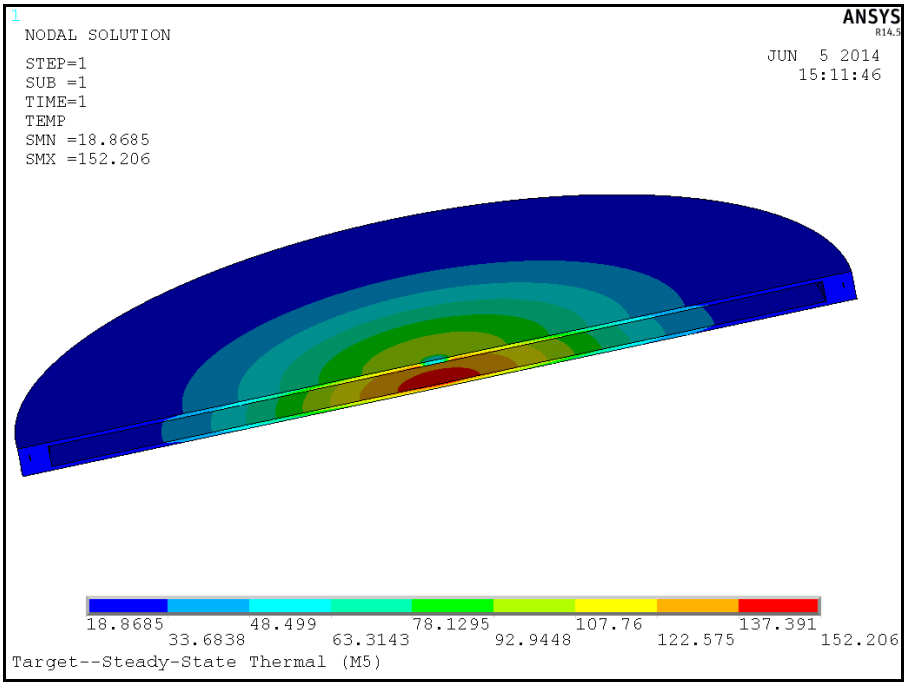


Figure 21
Temperature (C) in Cladding, Load Case 8

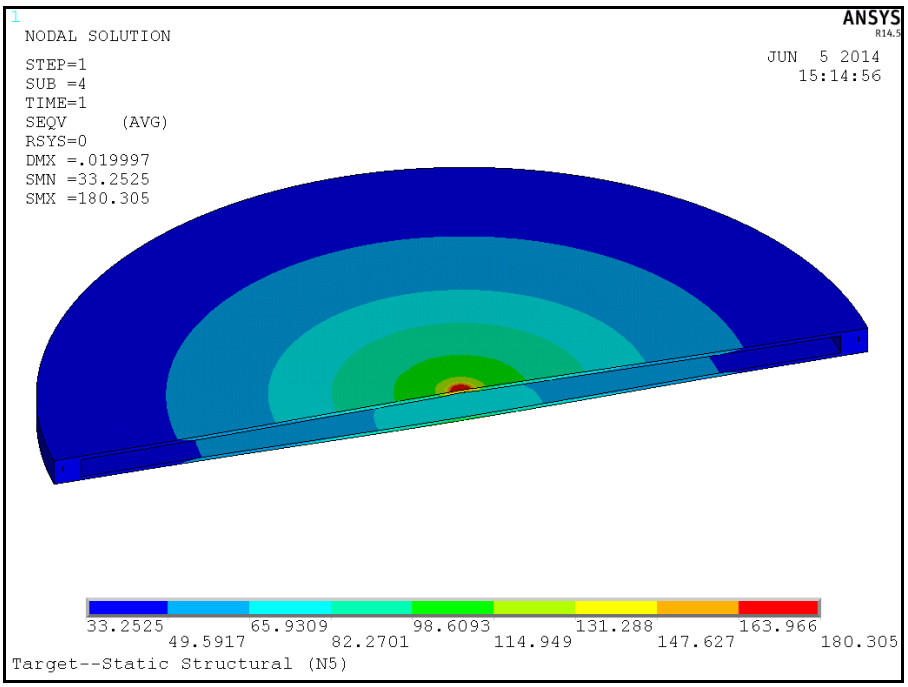


Figure 22
von Mises Stress (MPa) in Cladding, Load Case 8

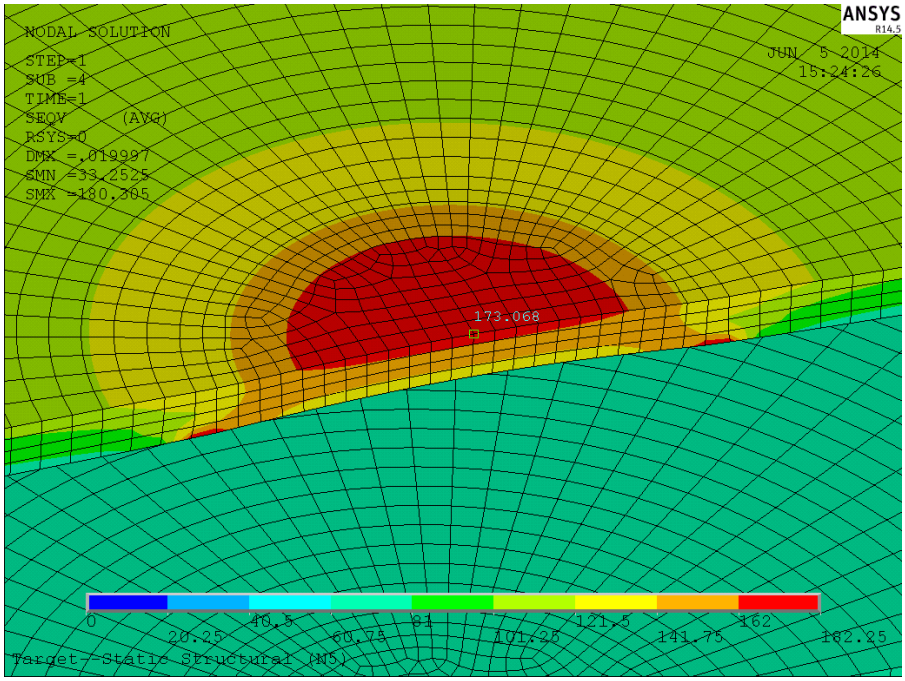


Figure 23
Von Mises Stress (MPa) in Cladding, Load Case 8

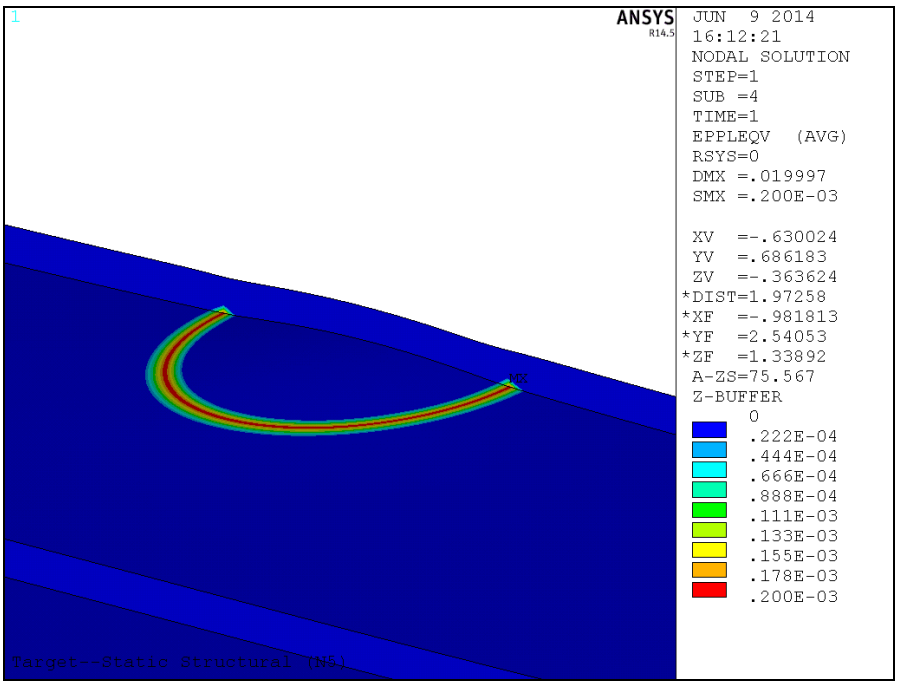


Figure 24
Equivalent Plastic Strain in Cladding, Load Case 8

11. Discussion

The highest stress and strain in the cladding is always at the flaw. Per Figure 16, the highest strain value occurs when the flaw is in the center of the disk. Deformation of the disk is driven by thermal expansion of the DU (see Figure 25), and this plus the effects of the flaw result in the highest stress, and therefore the highest fatigue damage, when the flaw is at the center. Flaws of these magnitudes do not seem to be a problem. Note that the lowest cladding life value in Table 2 is two orders of magnitude higher than the desired life of 1000 cycles.

When the flaw is in the middle and the flaw size varies, temperature, stress, plastic strain and fatigue damage all increase with flaw diameter (Figure 17 through Figure 19). The larger the flaw, the larger the insulating effect. This drives up temperature which increases swelling in the DU and reduces the yield strength of the Zircaloy-4. Thus, plastic strain and fatigue damage increase.

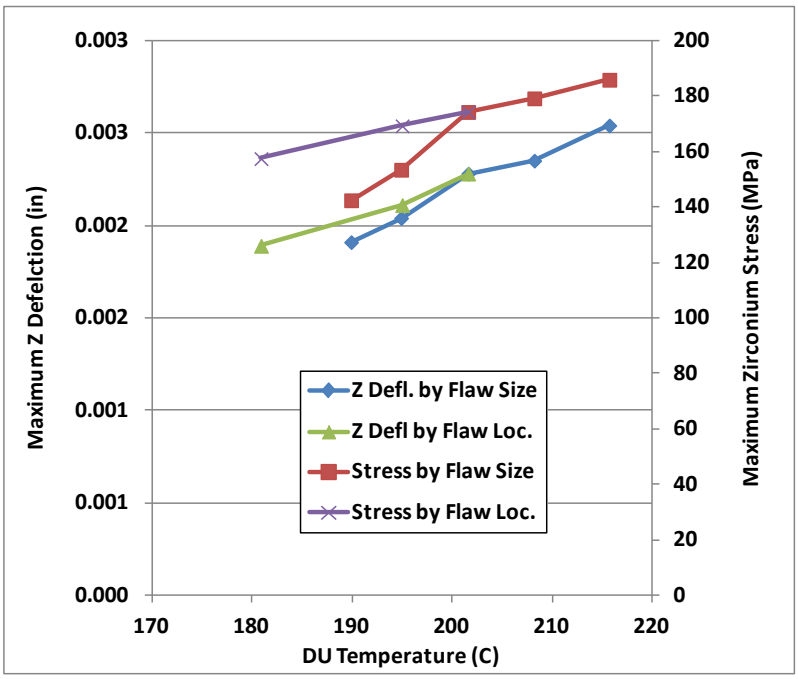


Figure 25
Effect of DU Temperature on Cladding Stress

This analysis was hampered by the inability to locate reliable material data. The materials used to fabricate the disks were identified simply as depleted uranium and Zircaloy-4, without further grade designation, composition, temper, etc. Much of the data also was not clearly identified with grade designation, temper, etc. Furthermore, the lack of quality data over a range of temperatures made it difficult to account for the effect of temperature. These results are believed to be accurate within the limitations imposed by this uncertainty.

12. Conclusions

Based on the results of this analysis the following conclusions are drawn:

1. The highest fatigue damage is present for flaws in the center of the disk.
2. Fatigue damage increases with flaw size.
3. The fatigue life of the Zircaloy-4 cladding is at least 1000 cycles.

13. References

1. Lemoine, P, and Schmuck, J, *Zirconium for Chemical Engineering: Mechanical Properties Useful for Vessel Design*, International Meeting on Chemical Engineering and Biotechnology, Frankfurt, June 9-15, 1991
2. O'Donnell, W.J. and Langer, B.F., *Fatigue Design Basis for Zircaloy Components*, Nuclear Science and Engineering, Col. 20, pgs. 1-12, 1964.

14. Software

- Ansys Mechanical, Version 15.0.7, Build date 4/11/2014, Ansys Inc., Pittsburg, PA.
- Microsoft Windows 7 Enterprise, Service Pack 1, Microsoft Corporation, Redmond WA.
- Microsoft Office Excel Professional Plus2010, (14.0.7015.1000), Microsoft Corporation, Redmond WA.

APPENDIX 1
GENERAL CHECKING CRITERIA SHEET

ANALYSIS CHECKLIST	Yes	No	N/A	Comments
Are analytical methods appropriate?	☐	☐	☐	
Are assumptions appropriate?	☐	☐	☐	
Is the analysis complete?	☐	☐	☐	
Is the source of the input geometry documented?	☐	☐	☐	
Is the source of material properties documented?	☐	☐	☐	
Are the boundary conditions clearly explained?	☐	☐	☐	
Was an applicable and valid computer program used?	☐	☐	☐	
Are the conclusions supported by the results?	☐	☐	☐	
Do the results seem reasonable?	☐	☐	☐	

APPENDIX 2
CRITICAL REVIEW OF REPORT

The report was submitted to Saurin Majumdar, Sr. Mechanical Engineer, who reported his concerns via e-mail. Below are the pertinent excerpts from that e-mail thread.

Saurin Majumdar: I scanned though the report. Figure 12 baffles me. Fatigue involves cyclic stress and creep involves constant stress. How can you plot the two on the same graph and make sense, particularly for tests at various frequencies. First of all what is the ordinate axis? Is this fatigue curve derived from zero to max loading cycles? As it is plotted, the cycle and time correspond only for 1Hz. As the cyclic rate goes down at high temperature, creep-fatigue interaction increases and the cyclic life a drop. One cannot predict the fatigue curve by integrating the creep damage over the cycles, especially if the fatigue cycling is fully reversed. If that was true we could avoid running fatigue tests at high temperature and do all design based on creep rupture data.

Rick Fischer: My intention was to produce a conservative result. As stated on page 11, when the loading frequency is below 1 Hz, creep dominates and the fatigue curve approaches the creep rupture curve. Cycle rate here is measured in hours, so using the creep rupture curve seemed appropriate. I would have to go back and review the data I found, but I'm guessing this was the only data I had at or above the temperature of the cladding.

Rick Fischer: Also, note in Figure 12 that the data at for 0.1 Hz is very close to the creep rupture curve. I believe the cycle is the beam is turned on and the target heats to a steady state condition and is held there for an extended time. At the cycle frequency seen by the targets, i.e. >1 day, it seems this would be very close to a creep rupture scenario.

Saurin Majumdar: I looked at Kim's paper from where you got Fig. 12. I don't know how this paper got published. He doesn't understand that 1000 cycles at 0.1 Hz take 10000 h to complete. In the mean time I found the attached paper by O'Donnell and Langer. However, it is for Zr-2.

Rick Fischer: Figure 8 in O'Donnell's paper has a design curve to be used with unirradiated Zircaloy-2, 3, and 4. On page 4 he states that Zircaloy 4 may be slightly better in fatigue than Zircaloy 2, so the Zircaloy-2 data can be used for Zircaloy 4.

At this point the fatigue calculations were redone using the design curve for unirradiated Zircaloy-2 in O'Donnell's paper, which is now Ref. 2.

APPENDIX 6

Calculation Note NE-EO-doc89: "Structural Analysis of Inconel Window for DU Target Assembly for ^{99}Mo Production"

CALCULATION COVER SHEET

Title: Structural Analysis of Inconel Window for DU Target Assembly for Mo99 Production		
Date: 4/18/2017		
Analyzed System:		
PREPARER Philip Strons	Philip Strons	4/18/17
Print Name	Signature	Date
REVIEWER Rick Fischer	Rick Fischer	4/18/2017
Print Name	Signature	Date
CALCULATION HAND CHECKED BY		
Print Name	Signature	Date
FINAL APPROVER James Grudzinski		4/19/17
Print Name	Signature	Date

TABLE OF CONTENTS

TABLE OF CONTENTS 1

1. Objectives

The objective of this analysis was to determine compliance of the window for the DU target assembly to be used in the SHINE experiment with ASME BPVC Sect. VIII Div. 2.

2. Scope

The scope of this analysis was limited to the Inconel 600 window of the target assembly. The remainder of the target assembly is being built by the vendor in accordance with Sect VIII Div 1 and is not included in this analysis.

3. Background

The Inconel window of the DU target assembly provides a containment boundary between the pressurized cooling water within the assembly and the vacuum of the beam tube upstream from the target.

4. Methodology

The Design by Analysis method is outlined in Section VIII, Division 2, Part 5 of the ASME Boiler & Pressure Vessel Code, which utilizes finite element analysis. A model of the window was created with the ANSYS and subjected to pressure, temperature loading, and a spring force, then analyzed to find component stresses. These stresses were then compared to criteria defined per the ASME BPVC.

5. Overview of Analysis

A total of five analyses were conducted and are summarized in
Table 1. The five analysis cases address possible failure criteria defined in the BPVC.

Analysis Case	Failure Mode	Criteria	Analysis Tool	Material Model
A	Plastic collapse	Sect VIII, Div 2, Part 5.2.3	FEA	Elastic Perfectly Plastic
B	Local Failure	Sect VIII, Div 2, Part 5.3.2	FEA	Linear Elastic
C	Collapse from buckling	Sect VIII, Div 2, Part 5.4.1	FEA	Linear Elastic
D	Fatigue	Sect VIII, Div 2, Part 3, Annex 3-F	FEA	Linear Elastic
E	Ratcheting	Sect VIII, Div 2, Part 5.5.6	FEA	Linear Elastic

Table 1
Analysis Overview

6. Assumptions

This analysis is based on the following assumptions:

1. Loads are steady state or cyclic (no inertial effects are considered).
2. Any loading due to gravity is negligible.

3. The normal operating pressure on the window is 27 psi.
4. The maximum pressure from the cooling system pump is 65 psi.
5. Materials are isotropic and homogeneous.
6. Temperature dependent properties are linearly interpolated between data points.
7. Material response is constant with time (no effects of aging, creep, etc).
8. Residual stresses are not included.

7. Geometry

The window is a machined part. It is attached to the target assembly by welding to the 316 stainless steel tube with an Inconel filler metal. The finite element model was constructed in ANSYS Design Modeler, referencing Drawing R07844A-3-03 and dated 03/20/2015. A half symmetry solid model was created. This model is shown in Figure 1.

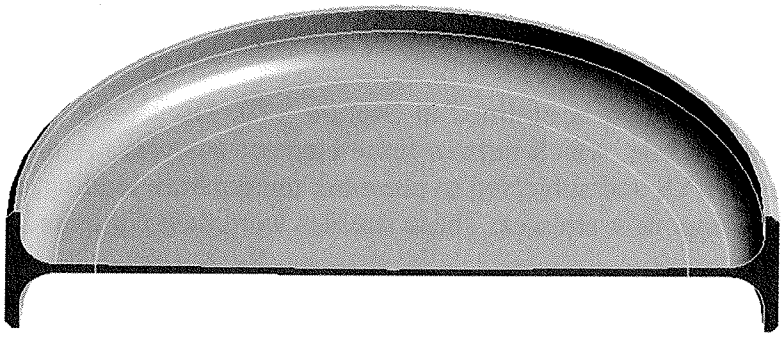


Figure 1
Solid Geometry Model

8. Material Properties

The window is fabricated from Inconel 600 with temperature dependent material properties summarized below in

Inconel 600 Material Properties			
ρ [lb/in^3]	0.304		
v [-]	0.31		
Temperature [°F]	70 - 100	200	300
E [psi]	31.0×10^6	30.3×10^6	29.9×10^6
Su [psi]	80,000	80,000	80,000
Sy [psi]	35,000	32,000	31,200
S [psi]	22,900	21,300	20,800
1.5 S [psi]	34,350	31,950	31,200
4 S [psi]	91,600	85,200	83,200
CTE [in/in/°F]	6.7×10^{-6}	6.9×10^{-6}	7.1×10^{-6}

Table 2. Values for CTE were obtained from Figure 6.3.2.0 of MIL-HDBK-5J, and all other properties were obtained from the ASME BPVC Electronic Stress Tables.

Inconel 600 Material Properties			
ρ [lb/in^3]	0.304		
ν [-]	0.31		
Temperature [°F]	70 - 100	200	300
E [psi]	31.0×10^6	30.3×10^6	29.9×10^6
Su [psi]	80,000	80,000	80,000
Sy [psi]	35,000	32,000	31,200
S [psi]	22,900	21,300	20,800
1.5 S [psi]	34,350	31,950	31,200
4 S [psi]	91,600	85,200	83,200
CTE [in/in/°F]	6.7×10^{-6}	6.9×10^{-6}	7.1×10^{-6}

Table 2 Material Properties

9. Boundary Conditions

The window is restrained by applying a fixed displacement with respect to the Z-axis applied to the surface that is welded to the target housing and another fixed displacement to the center of the window with respect to the Y-axis, as shown in Figure 2. Including symmetry along the YZ plane provides a kinematic restraint for the window.

Loading comes from multiple sources. The combination of pressurized cooling water on one side of the window and a full vacuum on the other side produces a pressure load. There is also the presence of a small force from a compressed spring within the target assembly. These are applied as shown in Figure 4. The loads described above are applied as two analysis load cases. Analysis load case 1 represents pressurizing the coolant to 80 psi (the maximum absolute pressure of the cooling pump) without thermal loading, load case 2 is the same as load case 1 with the addition of thermal loading, and analysis load case 3 represents normal operating conditions (pressure load of 27 psi). Thermal loading (Figure 4) is derived from the results of a previous CFD thermal/hydraulic analysis.

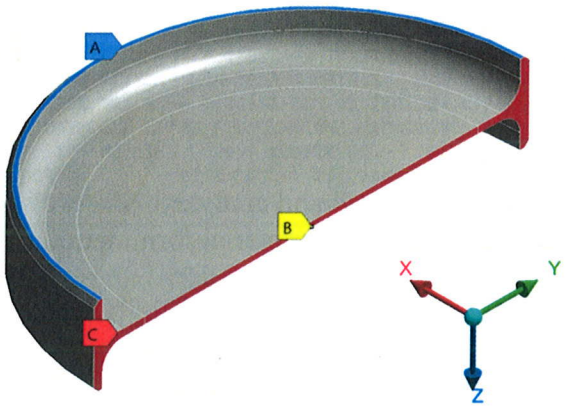


Figure 2 Boundary Conditions

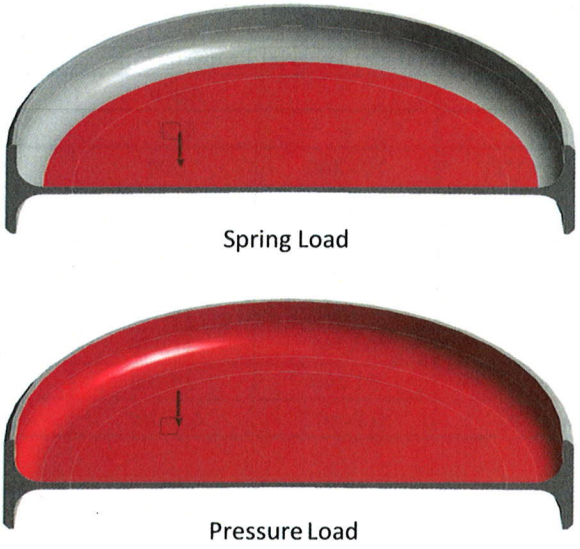


Figure 3 Mechanical Loading

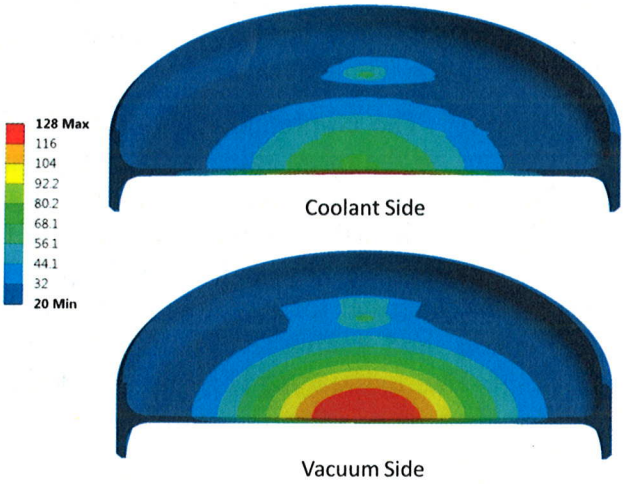


Figure 4 Thermal Loading in degrees Celsius

10. Solution and Results

Three finite element models were constructed and analyzed, and are summarized in Table 3. All are based on the same solid model and differ in material formulation, and load factors.

Finite Element Model	Analysis Result		Material Model	Load Case	Load Factors
1	A	Plastic Collapse	EPP	1,2	1.5,1.3
2	B	Local Failure	LE	2	1
3	C	Buckling	LE	3	1
3	D	Fatigue	LE	3	1
2	E	Ratcheting	LE	2	1

Table 3
Summary of Finite Element Models

A. Protection Against Plastic Collapse

The limit load method was used to check for plastic collapse. This analysis checks for structural instability due to gross plastic deformation. A load factor (1.5 for load case 1 or 1.3 for load case 2) is applied, and structural stability is indicated if the solution converges. This method is outlined at 5.2.3 in Sect VIII, Div 2 of the Code. The load cases, materials and mesh used for this analysis constitute finite element model 1 in Table 3.

The Code specifies that the analysis be run with small displacement theory and an elastic-perfectly plastic (EPP) material model. The yield strength defining the plastic limit is specified as 1.5S. The solid model was meshed with quadratic elements. Ten-node tetrahedral solid elements were used everywhere. Additional refinement was added in areas of the fillets.

Convergence was achieved for Load Case 1, as indicated by the sample from Solution Information shown in Figure 5, indicating compliance with the Code. The requirement for protection against plastic collapse is therefore met.

```
   EQUIL ITER   4 COMPLETED.  NEW TRIANG MATRIX.  MAX DOF INC=  0.1008E-02
    FORCE CONVERGENCE VALUE  =  1.826      CRITERION= 0.3847
    DISP CONVERGENCE VALUE   = 0.9898E-04  CRITERION= 0.8615E-03 <<< CONVERGED
   EQUIL ITER   5 COMPLETED.  NEW TRIANG MATRIX.  MAX DOF INC=  0.9898E-04
    FORCE CONVERGENCE VALUE  = 0.5507E-01  CRITERION= 0.3926     <<< CONVERGED
   >>> SOLUTION CONVERGED AFTER EQUILIBRIUM ITERATION   5
*** LOAD STEP     1   SUBSTEP     4  COMPLETED.    CUM ITER =     10
*** TIME =   1.00000         TIME INC =  0.300000
*** MAX PLASTIC STRAIN STEP = 0.9133E-02   CRITERION = 0.1500
```

```
    FORCE CONVERGENCE VALUE  =  1.155      CRITERION= 0.7474
    DISP CONVERGENCE VALUE   = 0.1901E-02  CRITERION= 0.5172E-01 <<< CONVERGED
   EQUIL ITER   1 COMPLETED.  NEW TRIANG MATRIX.  MAX DOF INC=  0.1901E-02
    FORCE CONVERGENCE VALUE  = 0.2633E-02  CRITERION= 0.7629     <<< CONVERGED
   >>> SOLUTION CONVERGED AFTER EQUILIBRIUM ITERATION   1
*** LOAD STEP     1   SUBSTEP   299  COMPLETED.    CUM ITER =    357
*** TIME =   1.00000         TIME INC =  0.122064E-02
*** MAX PLASTIC STRAIN STEP = 0.7851       CRITERION = 0.1500
```

Figure 5
Solution Convergence

B. Protection Against Local Failure

Protection from local failure was demonstrated with the Elastic Analysis method in 5.3.2 of Sect VIII, Div 2. This method is based on a linear elastic model, and the acceptance criterion is that the sum of the three principal stresses must be less than 4S.

The finite element model used for the Plastic Collapse analysis was copied and modified to use only a linear elastic material. This is finite element model 2 in Table 3.

Plots of the ratio of the sum of the three principle stresses to 4S are shown in Figure 6. Contour levels have been altered so that any value greater than or equal to 1, which indicates failure, is shown as red. There are no locations in the model where the sum of the principle stresses exceeds 4S; therefore, the requirement for protection against local failure is therefore met.

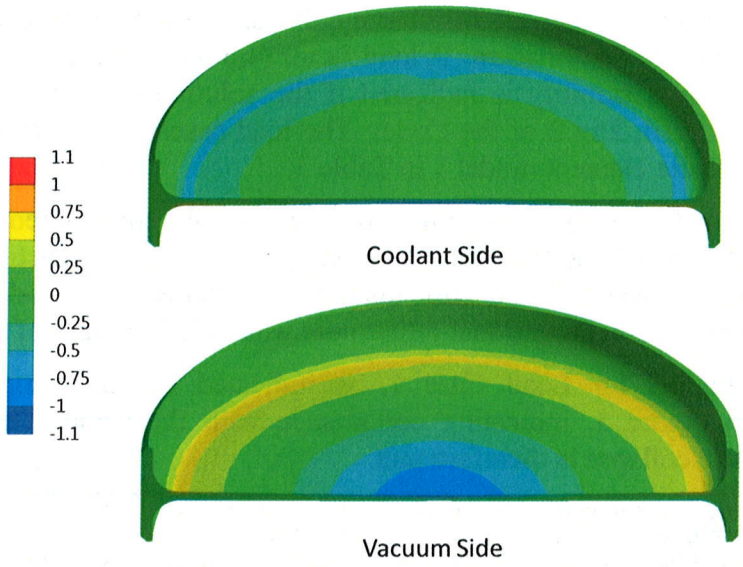


Figure 6 Local Failure, The ratio of the sum of all principal stresses to 4S (83,200 psi).

C. Protection Against Collapse From Buckling

Protection from collapse from buckling was evaluated using the method given at 5.4.1.2 in Sect VIII, Div 2, which specifies a linear elastic pre-stressed eigenvalue buckling analysis. The acceptance criterion is that the buckling load factor Φ_b be greater than $2/\beta_{cr}$, where β_{cr} is the capacity reduction factor, as the window does not fit within any of the geometry categories listed at 5.4.1.3 in the code. The smallest value for β_{cr} produces the highest value for the buckling load factor, so the smallest appropriate value for β_{cr} must be used to ensure compliance. However, XXX 2.3 states that these capacity reduction factors "account for shape imperfections," and so do not address a shape's resistance to buckling, but rather its sensitivity to manufacturing flaws. Since the flat circular window supported on its edge by a cylinder is less sensitive to geometric flaws than a cylinder under external pressure, the value of $\beta_{cr} = 0.80$ was selected. Thus, $2/\beta_{cr} = 0.80$ for a Φ_b of 2.5. The combination of thermal and mechanical loading results in a value for Φ_b of 3.5. For thermal loading only, the result is a value for Φ_b of 3.6. For mechanical loading only, the result is a value for Φ_b of 66. Based on these results, it is determined that the requirement of protection against collapse from buckling is met.

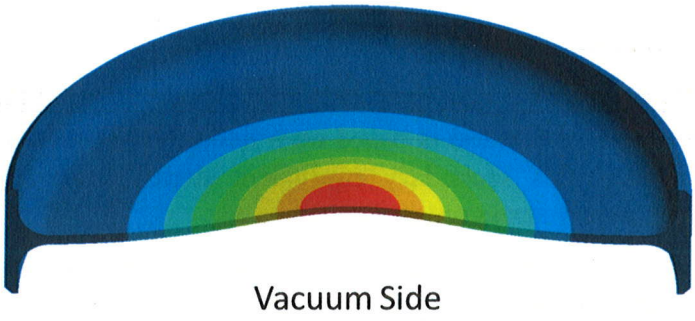


Figure 7
First Mode, Φ_b = 3.5, Combined thermal and mechanical loading

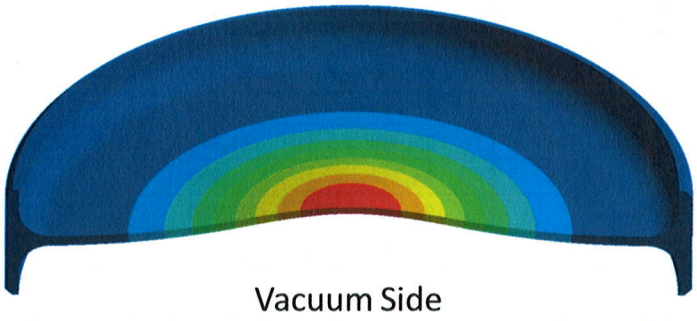


Figure 8 First Mode, Φ_b = 3.6, Thermal loading only

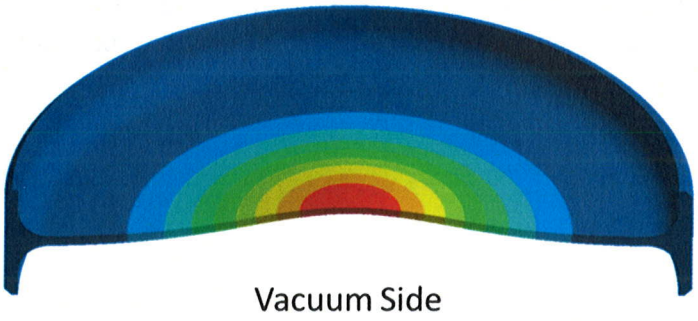


Figure 9 First Mode, Φ_b = 66, Mechanical loading only

D. Protection Against Failure from Cyclic Loading

Protection against failure from cyclic loading (fatigue) was evaluated by calculating the number of allowable cycles in accordance with Sect VIII, Div 2, Part 3, Annex 3-F. The value for stress amplitude was the maximum stress intensity found under normal operating conditions (Load Case 2), which was 28.4 ksi. Using equations 3-F.1, 3-F.2, and 3-F.3 with coefficients for 3-F.2 obtained from Table 3-F.3 resulted in a value of ~22,000 allowable cycles.

E. Ratcheting Assessment

Protection from Ratcheting was demonstrated with the Elastic Ratcheting Elastic Method in 5.5.6.1 of Sect VIII, Div 2. This method is based on a linear elastic model, and the acceptance criterion is that the primary plus secondary equivalent stress range $\Delta S_{n,k}$ is less than the allowable primary plus secondary stress range S_{ps}.

Finite element model 2 in Table 3 was used for this assessment. The maximum equivalent stress was taken as $\Delta S_{n,k}$. The value for S_{PS} in

Inconel 600 Material Properties			
ρ [lb/in^3]	0.304		
ν [-]	0.31		
Temperature [°F]	70 - 100	200	300
E [psi]	31.0×10^6	30.3×10^6	29.9×10^6
Su [psi]	80,000	80,000	80,000
Sy [psi]	35,000	32,000	31,200
S [psi]	22,900	21,300	20,800
1.5 S [psi]	34,350	31,950	31,200
4 S [psi]	91,600	85,200	83,200
CTE [in/in/°F]	6.7×10^{-6}	6.9×10^{-6}	7.1×10^{-6}

Table 2 was found using the method given at 5.5.6.1.d in Sect VIII, Div 2, and is essentially the highest of three times the average of S or two times the average of Sy at the highest and lowest temperatures during the operational cycle. Plots of equivalent stress for the Inconel window are shown in Figure 10Figure 10. Contour levels for these plots have been altered so that all values are divided by S_{PS}. Failure would be indicated by any contour result greater than 1 and appear as red in the plots. Results show that the requirement for protection from ratcheting is met.

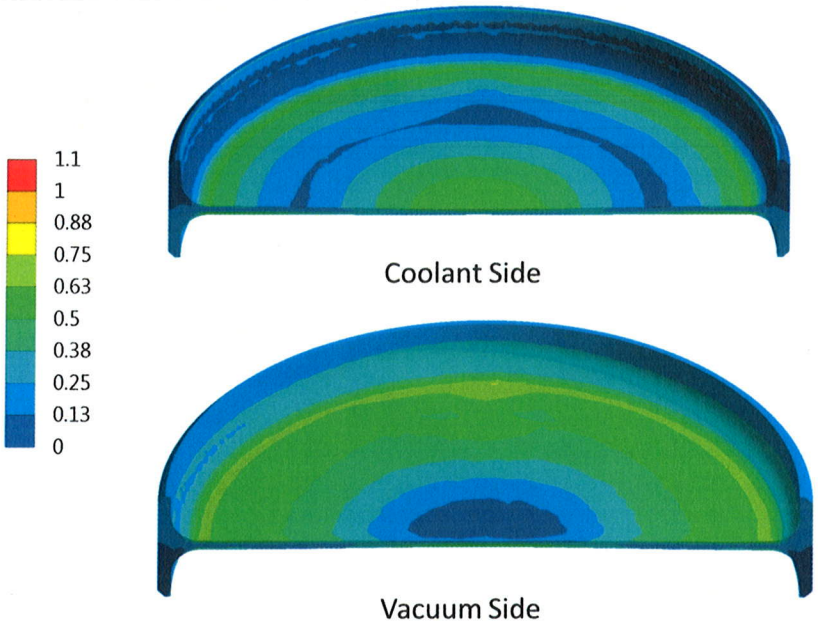


Figure 10 Ratcheting Assessment

11. Conclusions

The results of this analysis presented above show that the requirements for Protection Against Plastic Collapse, Protection Against Local Failure, Protection Against Collapse From Buckling, Protection From Ratcheting, Protection Against Failure from Cyclic Loading, per the ASME BPVC, have been met. Based on this, the following conclusions are drawn:

1. The Inconel 600 window for the DU target assembly is in compliance with Sect VIII, Div 2 when subjected to the loads described in this analysis.
2. From the fatigue analysis, the recommended maximum number of cycles for the assembly is 2,000

12. References

1. *ASME Boiler and Pressure Vessel Code,* American Society of Mechanical Engineers, New York, NY 2015.
2. *Metallic Materials and Elements for Aerospace Vehicle Structures MIL-HDBK-5J,* Department of Defense, 2003

13. Software

- ANSYS Mechanical, Version 17.2, Build date 07/27/2016, Ansys Inc, Pittsburg, PA.
- Microsoft Windows 7 Enterprise, Service Pack 1, 2009, Microsoft Corporation, Redmond WA.

- Microsoft Office Excel 2010, (14.0.7177.5000), 2010, Microsoft Corporation, Redmond WA.

APPENDIX 7

Memo: "Radionuclide inventories and HazCat-3 sum-of-fraction for 35 MeV mini-SHINE irradiations"

Argonne NATIONAL LABORATORY

Intra-Laboratory Memo

14 August 2014

TO: Sergey Chemerisov

FROM: Brad Micklich

SUBJECT: Radionuclide inventories and HazCat-3 sum-of-fractions for 35 MeV mini-SHINE irradiations

As part of Argonne's support for the mini-SHINE experiments, 20 liters of a uranyl sulfate solution (145 g/liter of uranium enriched to just under 20% ^{235}U) will be irradiated in a stainless steel container using neutrons generated by 20kW of 35 MeV electrons incident on a depleted uranium (DU) target. The solution tank will be surrounded by a water reflector. A lead shielding box will be used to contain the irradiation experiments. The box has sides of 4 inches of lead on four sides, four inches of lead with a 4-in thick access door on a fifth side, and eight inches of lead with a 14-inch thick viewing window made of leaded glass on the sixth side. The DU target assembly will be capable of being extracted out of the downstream side of the box into lead shielding. This memo describes the calculation of radionuclide inventories inside the box, the solution, and the target assembly.

Radionuclides in the system are produced by both neutron- and photon-induced reactions. The transport calculations were performed using MCNPX, and the transmutation (buildup and decay) calculations were performed with CINDER08. A special version of MCNPX version 2.6.0 was used that not only calculates the energy-dependent neutron flux for the regions of interest but also calculates radionuclide production for neutrons above the maximum energy in the CINDER data libraries, and due to all other particles (e.g., photons), and prints these rates directly in the MCNPX output file.

Calculations were performed for the electron linac running at 35 MeV, 20 kW for 19.3 hours (the length of time needed to produce 20 Ci of ^{99}Mo in the 20-liter solution) in each irradiation cycle. The complete irradiation history included four runs of 19.3 hours at 4-week periods, followed by a 5th irradiation. Radionuclide inventories were calculated at shutdown and for decay times out to one year following the final irradiation (as well as at intermediate times during the irradiation history). Figure 1 shows the 20-liter solution tank, and Figure 2 shows the MCNPX model for the shielded box and irradiation experiment. The tanks contain much internal structure which is not included in the MCNPX model. The design of these vessels is not yet complete, and the radionuclide inventory of the system is dominated by that in the uranyl nitrate solution, with a much smaller but still significant contribution from the depleted uranium target assembly.

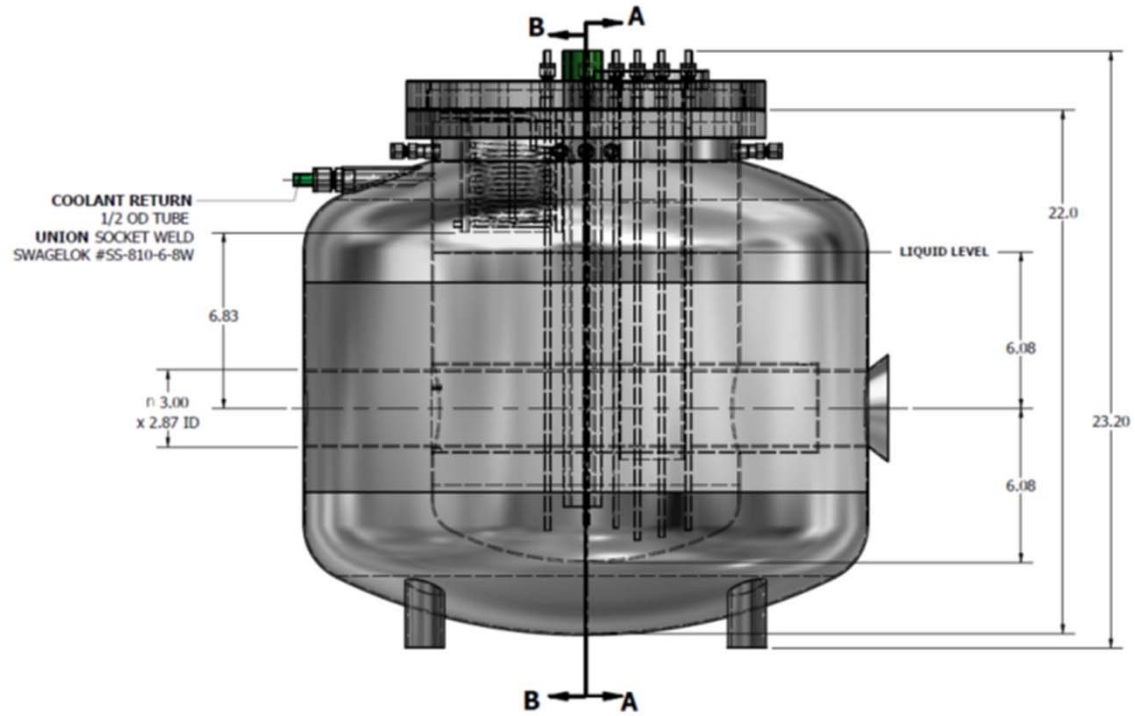


Figure 1. Drawing of the 20-liter uranyl nitrate solution tank surrounded by the reflector tank.

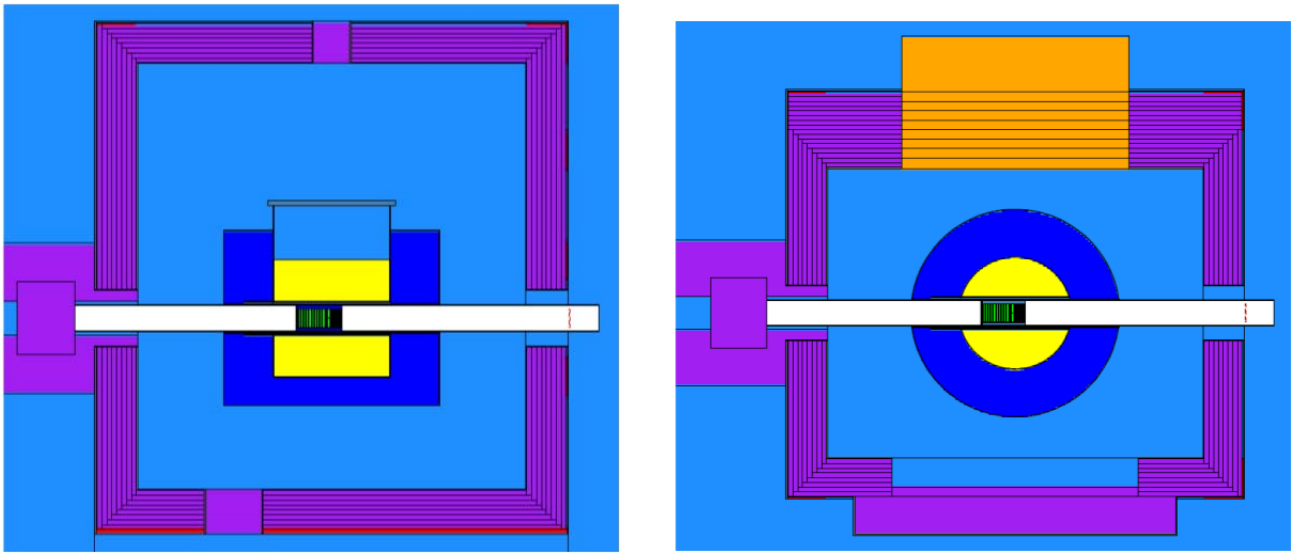


Figure 2. (left) Side view of MCNPX model used for activation calculations. The beam is incident from the right. (right) Top view of the MCNPX model through the beamline. The beam is incident from the right.

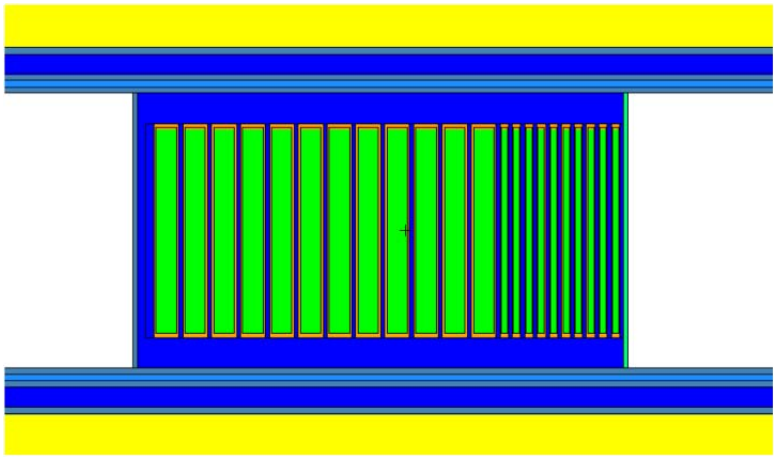


Figure 3. Detailed view of the depleted uranium target assembly from the 20-liter tank model.

First, an optimization study was conducted to find the target position that would maximize the fission rate inside the uranyl nitrate solution. The model for and results of those calculations are shown in Figure 4. The maximum fission rate in the solution occurs when the peak of the neutron source is approximately in the center of the solution tank. This position was used in subsequent calculations for inventories.

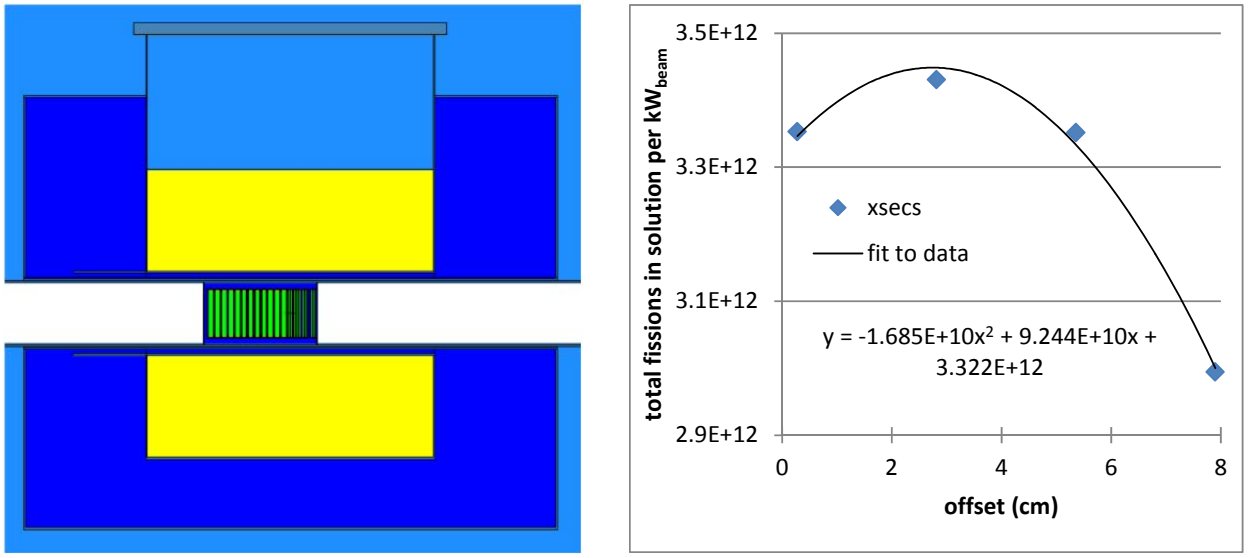


Figure 4. (left) Side view of MCNPX model used for optimizing the source position. The beam is incident from the right. (right) Fission rate in the uranyl sulfate solution as a function of target position. The offset parameter is the difference between the center of the solution container and the front face of the target.

The next step was to determine the running time needed to produce the target quantity (20 Ci) of ^{99}Mo. The buildup curve for ^{99}Mo under the irradiation conditions described above is shown in Figure 5. These data show that the target quantity is reached after 19.3 hours of irradiation. This irradiation length was used for all irradiations in the campaign. Using CINDER90, the time

required to reach the desired activity (20 Ci) is estimated at 17.3 hrs. The difference is due to differences in the ^{235}U thermal fission cross section between the CINDER90 library and the CINDER08 fission-weighted library (see Figure 6). The fission rate calculated using CINDER90 is about 10% higher than that calculated using CINDER08 for a well-thermalized spectrum, due to an apparently high value for the fission cross section in the 58-67 meV group.

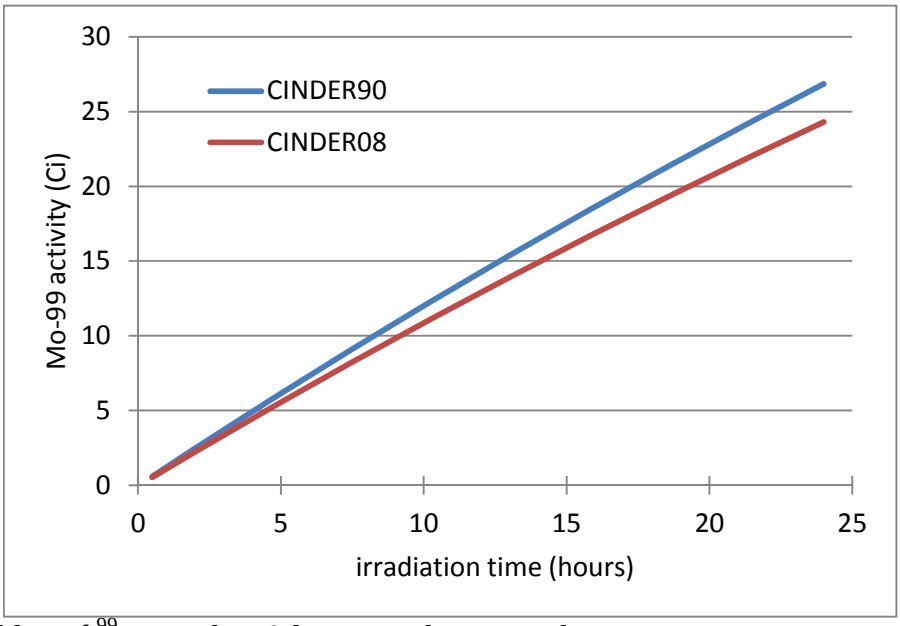


Figure 5. Buildup of ^{99}Mo in the 20-liter uranyl nitrate solution.

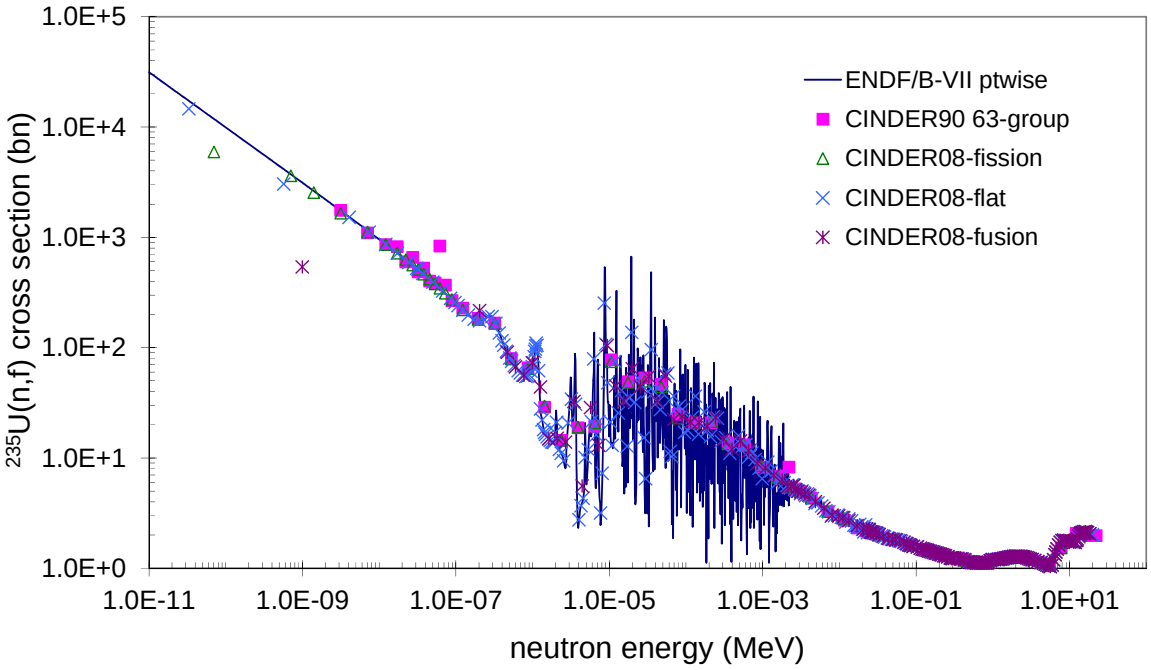


Figure 6. Comparison of the ^{235}U fission cross section in ENDF/B-VII, CINDER90, and CINDER08.

Figure 7 shows the hazard category 3 (HC-3) sum-of-fractions (SOF) in the entire irradiated volume, as well as in selected subsets, for the complete campaign of five irradiations. The percentage of the SOF for the solution is about 91-92% of the total. Table 1 lists the top 50 contributors to the sum-of-fractions at shutdown following the fifth irradiation. The contribution of the target varies between 8-9%, and only a small contribution to SOF comes from the box and vessels. The SOF is also dominated by the fission products ^{131}I and ^{133}I out to several months following the last irradiation, by which time the entire SOF is only about 0.01. Figure 8 shows the activity present in the system (in Ci) as a function of time. The solution contains about 85% of the total activity of the system immediately after irradiation stops, declining to around 60% of the total after about a week of decay, after which the percentage rises again. One year after the last irradiation, the solution contains about 90% of the activity, with the target assembly accounting for about 8% and the balance in the shielding box and stainless steel vessels.

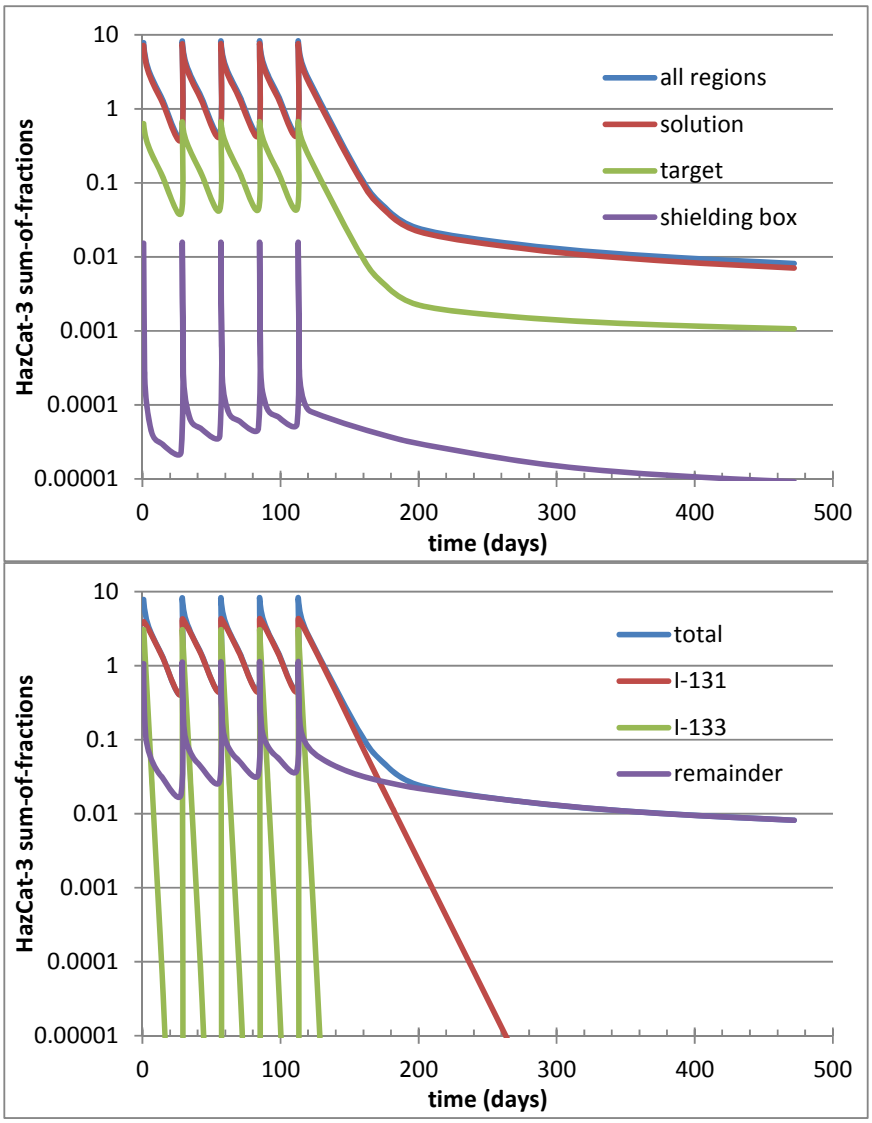


Figure 7. (top) HazCat-3 sum-of-fractions for the entire assembly, the uranyl sulfate solution, the target assembly, and the balance of the system. (bottom) HazCat-3 sum-of-fractions for the entire assembly, the nuclides I-131 and I-133, and the remainder of the nuclides.

Table 1. Top 50 contributors to the HazCat-3 sum-of-fractions in the uranyl sulfate solution at shutdown following the fifth irradiation.

Nuclide	half-life (s)	SOF contribution	Activity (Ci)
I 131	6.93E+05	3.73E+00	3.43E+00
I 133	7.49E+04	2.86E+00	5.55E+01
I 135	2.37E+04	2.34E-01	9.84E+01
Kr 88	1.02E+04	1.57E-01	6.29E+01
Xe138	8.45E+02	1.42E-01	1.13E+02
Sr 91	3.47E+04	4.97E-02	7.86E+01
Kr 87	4.58E+03	4.65E-02	4.65E+01
Zr 97	6.03E+04	4.33E-02	5.97E+01
Sr 92	9.58E+03	3.13E-02	1.06E+02
Xe135	3.29E+04	3.05E-02	6.09E+01
I 134	3.15E+03	2.43E-02	1.41E+02
La142	5.47E+03	2.29E-02	1.05E+02
Te132	2.77E+05	2.06E-02	1.24E+01
Cs138	2.00E+03	1.21E-02	1.21E+02
Ba140	1.10E+06	1.02E-02	6.12E+00
Ce143	1.19E+05	9.26E-03	3.52E+01
Ce144	2.46E+07	8.48E-03	8.48E-01
Sb128	3.24E+04	8.24E-03	4.61E+00
Sr 89	4.37E+06	7.30E-03	2.48E+00
Y 92	1.27E+04	7.18E-03	1.01E+02
Xe135m1	9.17E+02	7.14E-03	1.29E+01
I 132	8.26E+03	6.43E-03	1.07E+01
Y 91	5.06E+06	6.39E-03	2.30E+00
Np239	2.04E+05	6.29E-03	4.91E+01
Te134	2.51E+03	6.28E-03	1.26E+02
Mo 99	2.37E+05	5.93E-03	2.02E+01
La140	1.45E+05	5.70E-03	2.28E+00
Kr 85m1	1.61E+04	5.08E-03	2.03E+01
Y 93	3.66E+04	4.73E-03	8.32E+01
Zr 95	5.53E+06	4.31E-03	3.02E+00
Y 94	1.12E+03	4.15E-03	1.16E+02
Ru105	1.60E+04	4.15E-03	1.66E+01
Sb129	1.57E+04	3.84E-03	8.45E+00
Nb 97	4.33E+03	3.79E-03	5.61E+01
P 32	1.23E+06	3.34E-03	4.01E-02
Rb 89	9.09E+02	3.27E-03	8.50E+01
Ce141	2.81E+06	3.24E-03	3.24E+00
Pr143	1.17E+06	2.17E-03	2.25E+00
Sb130	2.37E+03	1.96E-03	1.33E+01
Sb131	1.38E+03	1.92E-03	4.61E+01
Te131m1	1.08E+05	1.89E-03	1.51E+00
Nd147	9.49E+05	1.86E-03	2.38E+00

Table 1. (continued)

Nuclide	half-life (s)	SOF contribution	Activity (Ci)
Y 95	6.18E+02	1.85E-03	1.15E+02
Nb 95	3.02E+06	1.84E-03	1.77E+00
Sr 90	9.09E+08	1.72E-03	2.75E-02
Y 91m1	2.98E+03	1.56E-03	4.36E+01
Ba142	6.36E+02	1.52E-03	1.04E+02
Ba141	1.10E+03	1.46E-03	1.05E+02
Tc104	1.10E+03	1.31E-03	3.39E+01
Nd149	6.22E+03	1.25E-03	1.95E+01

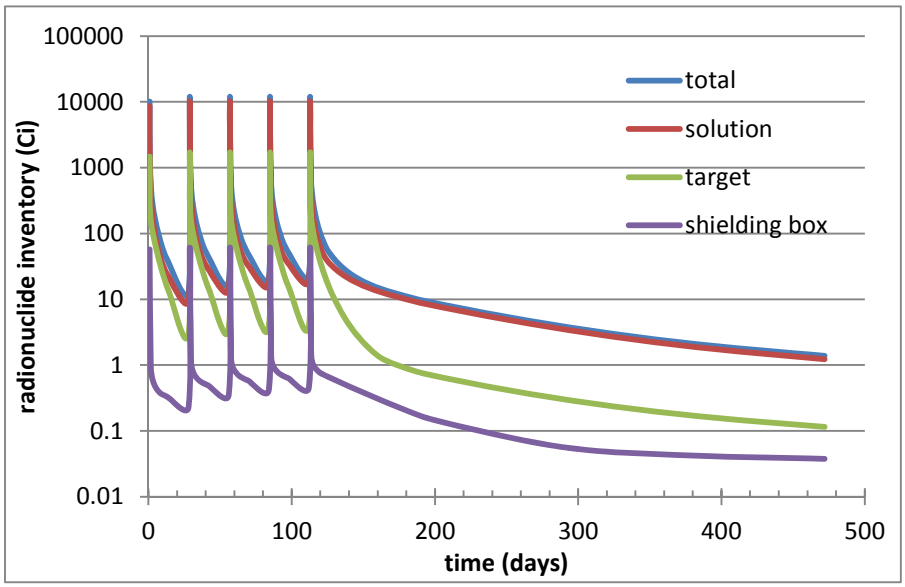


Figure 8. Radionuclide inventory for the entire assembly, the uranyl sulfate solution, the target assembly, and the balance of the system.

The attachments to this report contain printouts of the CINDER08 results. Attachment 1 contains listings by nuclide of the following quantities: total mass (kg), total activity (Ci), total decay power (W), air dilution factor, water dilution factor, total fission power, and fraction of the HazCat-3 threshold. Attachments 2 and 3 contain the same information ranked according to contribution (greatest to least) at the end of the 5th irradiation and 24 hours after the last irradiation ends, respectively.

APPENDIX 8

LEAF-PROC-016, Rev. 3: AMORE Gas Handling Alarm and Interlock Checklist

AMORE Gas Handling Alarm and Interlock Checklist

Low Energy Accelerator Facility, LEAF-PROC-016, Rev. 3

Approved:  Date: 03.23.2021

Sergey Chemerisov, Manager, IVEM/LEAF

Effective Date: 03.24.2021

Experiment _____

1 Purpose

Establish a checklist for the gas handling alarms and interlocks in the AMORE experiment.

2 Scope

This procedure applies to the following Argonne activities and entities.

LMS core processes:	Asset Management
Organizations:	Experimental Operations and Facilities (EOF) Division
Buildings:	211
Specific locations:	LINAC
Other applicability factors:	None
Exclusions:	None
USI applicability:	Yes

3 Work Process

3.1 Introduction

The steps in this procedure are used to ensure the safe operation of the AMORE and Mini-AMORE experiments. These steps must be performed before the experiment.

3.2 Step-by-Step Procedure

The steps below are mandatory unless noted otherwise. This procedure is to be performed by trained personnel.

3.2.1 Test the function of the Gas Collection System [ASE 2.5.2.2]

Step	Action
1	In the LINAC Control Room Chassis #1, set valve control for “D035” _____
2	Open the cylinder and isolation valves on the helium/xenon tank in D032 _____
3	Go to the LINAC Cell D035 Gas Distribution Hub Manifold _____
4	Close valves V-1 ☐ V-6 ☐ V-8 ☐ V-10 ☐ V-15 ☐ V-17 ☐ _____
5	Open Valves V-2 ☐ V-3 ☐ V-4 ☐ V-7 ☐ V-9 ☐ V-12 ☐ _____
6	Open the cylinder and isolation valves on the helium/xenon tank in D032 _____

Step	Action
7	At the Gas Collection System Control Chassis, ensure that the Gas Collection Systems Chamber #1 pump and Chamber #2 compressor function at the designed pressure differential. **[ASE 2.5.2.2]** _____

3.2.2 Flush the Analytical Manifold in D024 with Helium

Step	Action
1	Go to the LINAC Cell D035 Gas Distribution Hub Manifold _____
2	Close valves V-4 ☐ V-7 ☐ V-9 ☐ _____
3	SV-3 "ON"
4	Go to the D024 Analytical Enclosure _____
5	Attach a 1% hydrogen standard to the outside calibration port _____
6	Close Valves A-1 ☐ A-2 ☐ A-10 ☐ _____
7	Open valves A-3 ☐ A-4 ☐ A-5 ☐ A-6 ☐ A-7 ☐ A-8 ☐ A-9 ☐ A-11 ☐ A-12 ☐ _____
8	Turn on the Vacuum Pump _____
9	Flush the lines for about 1 or 2 minutes _____
10	Close Valve A-7 ☐ _____
11	When the pressure is <0.1mbar approximately, Close valves A-3 ☐ A-4 ☐ A-5 ☐ _____

3.2.3 Hydrogen Sensor 1% Alarm [ASE 3.2.1]

Step	Action
1	Open the canister valve to allow standard up to valve A-5. Then close the canister valve. _____
2	Open valve A-5 to introduce the standard into the manifold. Add standard until you reach the calibration pressure for the instrument. Then close valve A-5. Confirm that the Hydrogen Alarm is active. The alarm is on Gas Analysis Chassis #3 (D101 LINAC Control Room). _____

Step	Action	
3	Evacuate the manifold by opening valve A-4. When the pressure is <0.1mbar approximately, close valve A-4.	_____
4	Introduce helium to calibration pressure by opening valve A-7. Close A-7 when complete.	_____
5	Confirm that the alarm is no longer active.	_____

3.2.4 Gas Analyzer # 1 Hydrogen Interlock Test

Step	Action	
1	(Perform these steps only if running the Mini-AMORE Experiment) Analyzer #1 should be running analysis template RGA1_Mini-AMORE_ Analysis.qmt and the inlet valve should be open. Confirm the interlock is satisfied.	_____
2	ION CURRENT to trip the relay/interlock.	_____
3	Attach a 2% hydrogen standard to the outside calibration port.	_____
4	Open valves A-3 ☐ A-4 ☐ A-5 ☐ A-6 ☐ A-7 ☐ A-8 ☐ A-9 ☐ A-11 ☐ A-12 ☐	_____
5	Flush the lines with helium.	_____
6	Close Valve A-7	_____
7	When the pressure is about <0.1mbar, Close valves A-4 ☐ A-5 ☐ A-6 ☐ A-8 ☐	_____
8	Open the canister valve to allow standard up to valve A-5. Then close the canister valve.	_____
9	Open valve A-5 to introduce the standard into the manifold. Add standard until you reach the calibration pressure for the instrument. Then close A-5.	_____
10	Confirm that the interlock is no longer satisfied.	_____
11	Evacuate the standard from Analyzer #1 inlet by opening A-4. Then close A-3 ☐ A-4 ☐	_____
12	Confirm that the interlock is once again satisfied.	_____

3.2.5 Gas Analyzer # 2 Hydrogen Interlock Test

Step	Action
1	Analyzer #2 should be running analysis template RGA#2_ AMORE_Analysis.qmt. The inlet valve should be open. Confirm the RGA interlock is satisfied. _____
2	ION CURRENT to trip the relay/interlock _____
3	Attach a 2% hydrogen standard. _____
4	Introduce to Analyzer # 2 by opening valves A-5 & A-6. Add standard until you reach the calibration pressure then close A-5. _____
5	Confirm that the interlock is no longer satisfied. _____
6	Evacuate the standard by opening valve A-4. Then close A-4. _____
7	Confirm that the Interlock is once again satisfied. _____
8	Close the Analyzer # 2 Inlet valve so as not to interfere with the next test. _____

3.2.6 Hydrogen Sensor - Interlock Test [ASE 3.2.1]

Step	Action
1	Confirm that the Hydrogen Sensor interlock is satisfied. _____
2	Open valve A-8 to the Hydrogen Sensor. _____
3	Introduce the 2% hydrogen standard to Hydrogen Sensor by opening valve A-5. Add standard to the calibration pressure. Then close A-5. _____
4	Confirm that the Hydrogen Sensor interlock is no longer satisfied _____
5	Confirm that the Hydrogen Alarm in the D101 LINAC Control room is active _____
6	Evacuate the standard by opening valve A-4. Then close A-4 _____
7	Add helium to about 1010mbar using valve A-7. Then close A-7 _____
8	Confirm that the interlock is once again satisfied and the alarm is not active. _____
9	Close the cylinder isolation valves on the helium/xenon tank in D032 _____
10	Open Valve A-7 ☐ A-8 ☐ A-9 ☐ A-10 ☐ _____
11	Turn off the Vacuum Pump ☐ Close Valves A-6 ☐ A-11 ☐ _____
12	Go to the LINAC Cell D035 Gas Distribution Hub Manifold _____

Step	Action	
13	Close “de-actuate” valve SV-3 ☐	_____
14	Open valves V-1 ☐ V-6 ☐ V-7 ☐ V-9 ☐ V-10 ☐ V-16 ☐ V-19 ☐	_____

3.2.7 Gas Collection System Alarms & Interlock

Step	Action	
1	**Collection Cylinder** In D032, Confirm the LINAC Interlock for the Gas Collection System is satisfied.	_____
2	Set the alarm-1 (High) setting on the Collection Cylinder controller to a value that is less than the one currently displayed.	_____
3	Confirm the alarm on Chassis #3 in the D-101 LINAC Control Room is active.	_____
4	Confirm LINAC Interlock for the Gas Collection System is no longer satisfied.	_____
5	Reset the alarm-1 (High) to its original value (2000 psi).	_____
6	Confirm the Interlock is once again satisfied and the alarm is no longer active.	_____
7	**Chamber #2** Set the alarm-2 (High) setting on the Chamber #2 controller to a value that is less than the one currently displayed.	_____
8	Confirm the alarm on Chassis #3 in the D-101 LINAC Control Room is active.	_____
9	Confirm the valve GC-SV-1 on the Gas Collection System has closed.	_____
10	Confirm LINAC Interlock for the Gas Collection System is no longer satisfied.	_____
11	Reset the alarm-2 (High) to its original value (1250).	_____
12	Reset valve GC-SV-1 by pressing the OPEN button.	_____
13	Confirm the Interlock is once again satisfied and the alarm is no longer active.	_____
14	Set the alarm-2 (Low) setting on the Chamber #2 controller to a value that is greater than the one currently displayed.	_____
15	Confirm the alarm on Chassis #3 in the D-101 LINAC Control Room is active.	_____
16	Confirm the valve GC-SV-1 on the Gas Collection System has closed.	_____
17	Confirm LINAC Interlock for the Gas Collection System is no longer satisfied.	_____

Step	Action	
18	Reset the alarm-2 (Low) setting to its original value (1000).	_____
19	Reset valve GC-SV-1 by pressing the OPEN button.	_____
20	Confirm the Interlock is once again satisfied and the alarm is no longer active.	_____
21	**Chamber #1** Set the alarm-2 (High) setting on the Chamber #1 controller to a value that is less than the one currently displayed.	_____
22	Confirm the alarm on Chassis #3 in the D-101 LINAC Control Room is active.	_____
23	Confirm LINAC Interlock for the Gas Collection System is no longer satisfied.	_____
24	Reset the alarm-2 (High) to its original value (990).	_____
25	Confirm the Interlock is once again satisfied and the alarm is no longer active.	_____

3.2.8 Solution Vessel Pressure Alarm

Step	Action	
1	On the Gas Analysis Chassis #1 in D-101, set switch to “Upstairs”.	_____
2	Set the alarm-2 (High) setting on the Solution Vessel Pressure meter on Chassis #4 to a value that is less than the one currently displayed.	_____
3	Confirm the Solution Vessel Pressure Alarm is active.	_____
4	Set the alarm-2 (High) setting back to its original value (990mbar)	_____
5	Confirm the Solution Vessel Pressure Alarm is no longer active.	_____

3.2.9 Catalyst Pump Low Flow Alarm and Interlock

Step	Action	
1	Turn ON the catalyst pump switch on Chassis #1	_____
2	Turn OFF the Catalyst Pump Alarm Bypass on Chassis #4.	_____
3	Turn OFF the catalyst pump switch on Chassis #1	_____
4	Confirm the Catalyst Pump Alarm on Chassis #4 is active	_____
5	Confirm the Catalyst Pump interlock is not satisfied	_____
6	Turn ON the catalyst pump switch on Chassis #1.	_____

Step	Action	
7	Confirm the alarm is no longer active.	_____
8	Confirm the Catalyst Pump interlock is satisfied	_____
9	Turn ON the Alarm Bypass.	_____
10	Turn OFF the catalyst pump switch on Chassis #1	_____

Sampling Pump Low Flow Alarm

Step	Action	
1	Turn OFF the Sampling Pump Alarm Bypass on Chassis #3.	_____
2	Confirm the Sampling Pump alarm is active.	_____
3	Turn ON the Sampling Pump Chassis #1.	_____
4	Confirm the alarm is no longer active.	_____
5	Turn OFF the Sampling Pump.	_____
6	Turn ON the Sampling Pump Alarm Bypass on Chassis #3.	_____

4 Records Created by Work Process

The records listed below must be retained as indicated.

Description of Record (include form number if applicable)	Active Records Custodian	Active Records Retention	Indexing Method, Storage Medium	Federal Retention Requirements*
Completed AMORE Gas Handling Alarm and Interlock Checklist	Facility Manager	3 years	Index by job date and name, store on paper or electronically	Destroy 75 years after the date of the permit (DOE ADM 18.37)

*If records are maintained in a business information system that is not currently programmed to purge digital records based on age, the records may be retained in that system past the indicated destruction date.

5 Related Documents

This procedure implements requirements established by the following safety basis documents:

- LEAF-SAD-100, Linac Safety Assessment Document
- LEAF-ASE-100, Linac Accelerator Safety Envelope

This procedure implements requirements established by the following Argonne policies and procedures:

- LMS-PROC-188, *Accelerator Safety*
- EGS-PP-100, *Configuration Management Program Plan for Accelerators*

6 Definitions

None

7 About this Procedure

Issuing organization:	Low Energy Accelerator Facility
Procedure owner:	M. Kalensky
Point of contact:	M. Kalensky
Review cycle (months):	36
Date last revised:	3.18.2021
Date last reviewed:	3.23.2021

8 Summary of Changes in This Version

Change to Section 3.2.1 This Section has been revised to “Test the Function of the Gas Collection System [ASE 2.5.2.2]”. This created an extra part to this section “3.2”, “3.2.9”. The previous version contained up to section “3.2.8”. There is no longer a section named “Start the flow of helium to D024”, instead it has been replace by Section 3.2.2, “Flush the Analytical Manifold in D024 with helium”.

New valves were added to the Gas Distribution Hub Manifold “V-15, V-16, V-17, V-18 and V-19” and a new connection was made from valve V-8 to the sampling pump. These changes affect Sections “3.2.1 Test the Function of the Gas Collection System - STEP 4”, “3.2.2 Flush the Analytical Manifold with Helium - STEP 9” and “3.2.6 Test the Hydrogen Interlock - STEP 13”.

APPENDIX 9

LEAF-PROC-017, Rev. 1: Monitoring the Gas Handling System during the AMORE Experiment

Monitoring the Gas Handling System during the AMORE Experiment

Low Energy Accelerator Facility, LEAF-PROC-017, Rev. 1

Approved:  Date: 04.19.2019

Sergey Chemerisov, Manager, IVEM/LEAF

Effective Date: 04.22.2019

1 Purpose

Establish the process for monitoring the Gas Collection System during AMORE experiment.

2 Scope

This procedure applies to the following Argonne activities and entities.

LMS core processes:	Asset Management
Organizations:	Experimental Operations and Facilities (EOF) Division
Buildings:	211
Specific locations:	LINAC
Other applicability factors:	None
Exclusions:	None
USI applicability:	Yes

3 Work Process

3.1 Introduction

The AMORE experiment is equipped with several systems to monitor gas generation and composition.

3.1.1 Gas Handling System

The **Gas Handling System** was designed to monitor the hydrogen concentration in the Target Solution Vessel (TSV) and to minimize the risk of releasing radioactive gases by keeping the TSV and chemical processes sub-atmospheric.

During the AMORE experiment, hydrogen and oxygen are generated by the radiolysis of water. A Catalyst Pump, located on top of the D035 Hot Cell, circulates the TSV headspace gases through a Pt-Pd Couderite Catalyst, which re-combines the radiolytically generated hydrogen and oxygen. There is also a Sampling Pump, located in the Gas Distribution Hub, which circulates the headspace gas of the TSV to the D024 Analytical Enclosure, where it is analyzed by a H2 Scan HY-OPTIMA 2700 hydrogen sensor (Hydrogen Sensor) and a Pfeiffer Omni-Star Residual Gas Analysis System (Analyzer #2). The following paragraphs discuss these four components of the Gas Handling System in more detail.

The **Catalyst Pump** is monitored using the Gas Hub Pressure on Chassis #4. The Catalyst Pump causes a pressure decrease, as read on transducer PT-1. The decrease in pressure is a positive indication that the Catalyst Pump is in operation. The process controller on Chassis #4 is interlocked with LINAC power.

The **Sampling Pump** flow is monitored using a flow meter. In the event that the pump flow becomes too low, an alarm will sound on Chassis #3 in the LINAC Control Room.

Analyzer #2 also provides continuous monitoring of the gas composition of the TSV headspace. It has a mass selective detector. Analyzer #2 is interlocked with the LINAC accelerator power. If hydrogen concentration of the TSV reaches 2%, the LINAC will automatically shut down. The Analyzer monitors the ion current of mass 2

for hydrogen and mass 32 for oxygen. When the instrument is calibrated, the ion current for hydrogen at 2% is logged. That value is entered into the analysis template RGA-2_AMORE_ANALYSIS.

The **Hydrogen Sensor** provides continuous monitoring of the hydrogen concentration of the TSV. It has two alarm states. The first alarm state is at 1% hydrogen concentration. At this point, an alarm sounds on Chassis #3 in the LINAC Control Room, warning the operators that hydrogen has reached 1%. The second alarm state is interlocked with LINAC accelerator power. In the event hydrogen concentration reaches 2%, the LINAC is automatically shut down. **[ASE 3.2.1]**

3.1.2 Gas Collection System

The Gas Collection System is connected to all parts of the experiment through the Gas Distribution Hub and directly to the D024 Hot Cell. The Gas Collection System stores radioactive gases generated from the fissioning of uranium during the experiment in Collection Cylinders. It also serves to keep the TSV and the various chemical processing sub-atmospheric. It is interlocked with the LINAC main power. If the pressure in any part of the experiment rises to 900 mbar, the interlock will shut down the LINAC.

Tables at the end of this document provide a quick reference to settings, actions to take, and descriptions of various parts of the experimental system. Gas Handling Chassis in the LINAC control room is used to control the operation of solenoid valves, pumps, and additions of oxygen and helium.

3.1.3 Mini-AMORE Experiment

The **Phase II Mini-AMORE experiment** is monitored using another Pfeiffer Omni-Star Gas Analysis System (Analyzer #1). This instrument monitors gases generated during the Mini-AMORE experiment. Through a series of tubing, Analyzer #1 is attached to a capsule containing 2 mL of uranium sulfate solution. Helium flows into the capsule, sweeping headspace gas from the capsule. Analyzer #1 samples the gas stream and monitors the hydrogen and oxygen concentration. The outlet of the gas stream flows into the Gas Collection System. Analyzer #1 is interlocked with the LINAC accelerator power supply such that if the hydrogen concentration reaches 2%, the LINAC is automatically shut down. The value the Analyzer monitors is the ion current of mass 2 for hydrogen and mass 32 for oxygen. When the instrument is recalibrated, the ion current for hydrogen at 2% is logged. That value is entered into the analysis template RGA-1_Mini_AMORE_ANALYSIS. The administrative limit for hydrogen in this experiment is 1%. If hydrogen concentration gets to 1%, inform the LINAC operator to reduce power. To reduce the concentration of hydrogen in the capsule, increase the helium flow though the capsule.

3.2 Step-by-Step Procedure

The steps below are mandatory unless noted otherwise. This procedure is to be performed by trained personnel.

3.2.1 Log Entries

Step	Action
1	In the Gas Analysis Logbook, log in: • Date • Experiment Title • Gas Analyzer Templates (Methods) • Calibration Check Standards and Recovery The Analyzers should be started and running the analysis templates. Pre-experiment checks and interlock/alarm checks should already be complete.
2	Monitor Gas Constituents: The experimenter should monitor the gas constituents continually by observing concentration displayed on the Analyzer computers and the H2Scan hydrogen sensor displays.
3	During the experiment: 3.1 Use the notebook to log: • Log actions such as adding oxygen, purging with helium, increasing LINAC power • Log problems or interesting occurrences • Log the time and duration. Add notes for more information. 3.2 Be mindful of the hydrogen concentration in the AMORE Target Solution Vessel (Analyzer #2 and Hydrogen Sensor) and in the Mini-AMORE Experiment (Analyzer #1). • The administrative limit for this experiment is 1% hydrogen. • If the hydrogen concentration reaches 1%, the LINAC operator needs to be notified and the beam power should be reduced. **[ASE 3.2.1]**
4	• For the AMORE experiment, add oxygen to the Solution Vessel to reduce the hydrogen concentration. • For the Mini-AMORE Experiment, increase the helium flow to reduce the hydrogen concentration. If the hydrogen concentration reaches 2% the experiment needs to be shut down.

Step	Action
5	Monitor alarms for: • Gas Collection System • Sampling Pump flow, • Catalyst Pump pressure, and • Solution Vessel Pressure. SEE TABLE 1, ALARM CONDITIONS, for actions that need to be taken in the event of an alarm state.

3.2.2 Adding Oxygen to Reduce the Concentration of Hydrogen

Step	Action
1	A 60/40 helium/oxygen mixture is added to the Target Solution Vessel. It is best to add oxygen well before hydrogen reaches 1%. There is a three-minute delay from the time you begin adding oxygen till a reduction in hydrogen is observed. When hydrogen is approximately 0.3%, it is time to add oxygen: 1.1 Actuate (open) SV-5 on Chassis #1 1.2 Use the oxygen flow control knob on Chassis #2 to set the desired gas flow.
2	When a sufficient amount of gas is delivered: 2.1 Close SV-5 2.2 Stop the gas flow using the Stop Flow switch or by turning down the control knob on chassis #2

3.2.3 Increasing the Helium Flow to Reduce Hydrogen Concentration in the Mini-AMORE Experiment

Step	Action
1	Use the Mini-AMORE Flow Control knob on Chassis #2 to increase the flow of helium through the capsule. Turn the knob to the right and observe the Flow readout. Observe Analyzer #1 data. Adjust flow until Hydrogen concentration is below 1%.

3.2.4 Post Irradiation

Step	Action
1	Add Helium to Reduce Radiation Levels in D024 It will be necessary to decrease radiation levels in D024 to allow access. This is done by purging the Analytical Manifold with helium. SV-1 will isolate the Vessel and SV-3 allows helium into the Analytical Manifold. 1.1 Put the Catalyst Pump and Sampling Pump alarms in bypass mode. 1.2 Actuate (close) SV-1 1.3 Actuate (open) SV-3
2	When radiation in D024 reaches acceptable levels 2.1 De-actuate (close) SV-3 2.2 Turn off the Gas Sampling Pump 2.3 Actuate SV-6 (close) 2.4 Stop the flow of helium to the Mini-AMORE capsule. 2.5 Close the valves on the three cylinders and their isolation valves in D032. Oxygen and the two helium/xenon cylinders.
3	Put the System in Standby Mode (usually the next day) 3.1 De-actuate (open) SV-1 and SV-6 3.2 On the Analytical Manifold, close valves A-1, A-2, A-7, A-8, A-9 and A-10. 3.3 Turn off the catalyst heater.
4	Save Analyzer Data (Perform when no more data needs to be collected) 4.1 Stop the analysis template 4.2 Close the inlet valve 4.3 Turn off the filaments and SEM 4.4 Save the data file 4.5 Copy Data Files to the GTRI shared drive. This will ensure the data is backed up.

3.3 Reference Tables (see Exhibit A)

Table 1. Alarm and Interlock Conditions and Action: Gives a brief description of the alarms and interlocks that may occur during an experiment and the actions that need to be taken by the operator.

Table 2. Hub Manifold Solenoid Valves: Gives a brief description of the solenoid valves on the Hub Manifold in the Gas Distribution Hub Enclosure in D035. The solenoid valves are remotely actuated from Chassis #1.

Table 3. Switches and Controls on Gas Handling Chassis: Located in the LINAC Control Room: Gives a brief description of the function of other switches and control knobs on the Gas Handling Chassis. Included are oxygen and mini-AMORE, flow control and ON/OFF switches for the Catalyst and Sampling pumps.

Table 4. Hub Manifold Manual Valves: Located in the Gas Distribution Hub Enclosure (D035): Gives a brief description of the manually operated valve located on the Hub Manifold.

Table 5. Gas Collection System Valves: Located in the Gas Collection Enclosure in (D035): Gives a brief description of the function of valves in the Gas Collection System.

Table 6. Dump Tank Valves: Located in D035 beneath the D035 Hot Cell: Gives a brief description of the function of valves on the Dump Tank.

Table 7. Gas Collection System Chassis Settings: Located in (D032) shows Control settings to actuate pumps, alarms and interlocks in the Gas Collection System.

Table 8. Analytical Manifold Valves: located in the D024 Analytical Enclosure, it gives a brief description of the manually operated valves.

4 Records Created by Work Process

The records listed below must be retained as indicated.

Description of Record (include form number if applicable)	Active Records Custodian	Active Records Retention	Indexing Method, Storage Medium	Federal Retention Requirements*
Lab notebook	Facility Manager	3 years	Index by job date and name, store on paper or electronically	Destroy 75 years after the date of the permit (DOE ADM 18.37)

*If records are maintained in a business information system that is not currently programmed to purge digital records based on age, the records may be retained in that system past the indicated destruction date.

5 Related Documents

This procedure implements requirements established by the following safety basis documents:

- LEAF-SAD-100, Linac Safety Assessment Document
- LEAF-ASE-100, Linac Accelerator Safety Envelope

This procedure implements requirements established by the following Argonne policies and procedures:

- LMS-PROC-188, *Accelerator Safety*
- EGS-PP-100, *Configuration Management Program Plan for Accelerator*

6 Definitions

None

7 About this Procedure

Issuing organization:	Low Energy Accelerator Facility
Procedure owner:	M. Kalensky
Point of contact:	M. Kalensky
Review cycle (months):	36
Date last revised:	04.19.2019
Date last reviewed:	04.22.2019

8 Summary of Changes in This Version

Initial release.

Rev. 1. Addition of the references to the ASE controlled parameters.

Exhibit A: Reference Tables

TABLE 1. Alarm and Interlock Conditions and Action

Condition	Action
AMORE Hydrogen concentration 1% (Alarm on Chassis #3) [ASE 3.2.1]	Warn LINAC Operator to reduce beam power. Add oxygen to re-combine the excess hydrogen to <1%.
AMORE Hydrogen Concentration 2% (Alarm on Chassis #3) [ASE 3.2.1]	Interlocked. Warn LINAC Operator to Stop the Experiment if Interlock fails. Add oxygen to re-combine the excess hydrogen.
Gas Collection Alarm. (Alarm on Chassis #3)	Interlocked. Warn LINAC Operator to Stop the experiment. It indicates that the pressure in chambers 1, 2 or the Collection Cylinder is too high. Monitored on LabView.
Sampling Pump Alarm (Alarm on Chassis #3) [ASE 3.2.1]	Warn LINAC Operator to STOP the experiment. This indicates a problem with the Gas Sampling Pump.
Catalyst Pump Alarm (Alarm on Chassis #4)	Warn LINAC Operator. STOP the experiment. This is interlocked
Solution Vessel Pressure (Alarm on Chassis #4)	Warn LINAC Operator. Shut Down the experiment. This may be an indication of a Gas Collection System problem. This is Interlocked.

TABLE 2. Hub Manifold Solenoid Valves – Located in the Gas Distribution Hub Enclosure (D035) - (Controls on Chassis 1) & (Gas Distribution Hub Enclosure)

Valve	Description	Function
SV-1	**Gas Sampling Isolation**	**Normally Open** – Allows the gas stream from the Target Solution Vessel into the Hub Manifold. Purge the Analytical Manifold in D024 post irradiation by actuating SV-3 and SV-1.
SV-2	**Sampling Pump Bypass**	**Normally Closed** – Should the sampling pump fail, the Analytical Manifold in D024 can be purged by actuating SV-1, SV-2 and SV-3.
SV-3	**Isolation Valve/Helium Purge**	**Normally Closed** – Isolates the Gas Sample Path from the helium purge line during an experiment. Open it to purge the Analytical Manifold for calibration and pre-run. You can purge the Analytical Manifold post irradiation by actuating SV-3 and SV-1.
SV-4	**Helium purge**	**Normally Closed** – Open it to purge the Target Solution Vessel.
SV-5	**Oxygen Addition**	**Normally Closed** – Open it to add oxygen to the Target Solution Vessel to reduce hydrogen concentration. Use in conjunction with the Oxygen Flow potentiometer on Chassis 2.
SV-6	**Sampling Pump Isolation**	**Normally Open** – Isolates the Sampling Pump from the Solution Vessel
SV-7	**Gas Sampling Isolation**	**Normally Open** – Allows the analytical gas stream from the Solution Vessel into the Condenser. Close when removing condensate from the condenser.

TABLE 3. Switches and Controls on Gas Handling Chassis – Located in the LINAC Control Room

Switch	Description	Function
Catalyst Pump (Chassis 1)	**Catalyst Pump Switch ON/OFF**	Turns ON and OFF the Catalyst Pump that circulates Target Solution Vessel headspace gas through the catalyst to recombine hydrogen and oxygen. This should be kept ON during an AMORE irradiation.
Sampling Pump (Chassis 1)	**Sampling Pump Switch ON/OFF**	Turns ON and OFF the Sampling Pump that circulates the Target Solution Vessel headspace gas to the Analytical Manifold in D024. This should be kept ON during an AMORE irradiation.
Oxygen Flow Control (Chassis 2)	**Potentiometer which remotely adjusts the flow controller on the oxygen cylinder**	Regulates Oxygen flow to the Target Solution Vessel to reduce the level of hydrogen. Turn right to increase flow. The meter will read out in mL/min. The maximum flow is 50mL/min. Use in conjunction with SV-5 on chassis 1.
Oxygen OFF (Chassis 2)	**Internal valve on the oxygen mass flow controller**	Stops the flow of oxygen without using the potentiometer. A flow setting can be maintained even when no gas is being added. SV-5 needs to be closed to use this function.
Mini-AMORE Flow Control (Chassis 2)	**Potentiometer which remotely adjusts the flow controller on the Mini-AMORE helium cylinder**	Regulates the helium flow in the mini-AMORE experiment. Turn right to increase flow

TABLE 4. Hub Manifold Manual Valves – Located in the Gas Distribution Hub Enclosure (D035)

Valve	Description	Function
V-1	**Condenser Isolation**	Isolates the Condenser from the Hub Manifold
V-2 & V-10	**Analytical Manifold Isolation**	Closing V-2 & V-10 isolate the Analytical Manifold in D024 from the Hub Manifold.
V-3	**Helium Isolation Valve**	Allows helium in to the Hub Manifold for purging the Solution Vessel, Dump Tank and the Analytical Manifold.
V-4	**Main Isolation**	Isolates sampling and purge connections from the Gas Collection System connections on the Hub Manifold.
V-5	**Vacuum Inlet**	Opens the manifold to Vacuum Pump inlet.
V-6	**Target Solution Vessel Head Space Isolation**	Isolates the headspace of the Solution Vessel from the Gas Collection System and Vacuum Pump. Opening V-6, V-7 & V-9 opens the Target Solution Vessel headspace to the Gas Collection System.
V-7	**Target Solution Vessel Head Space Isolation**	Opening V-6, V-7 & V-9 opens the Target Solution Vessel headspace to the Gas Collection System.
V-8	**Vacuum Exhaust to Gas Collection**	Opens the vacuum pump exhaust to the Gas Collection System
V-9	**Gas Collection Isolation**	Isolates the Hub Manifold from the Gas Collection System
V-10	**Analytical Manifold Isolation**	Closing V-2 & V-10 isolate the Analytical Manifold in D024 from the Hub Manifold. (Attached to the Sampling Pump)
V-11	**Primary Recovery Glovebox Isolation**	Isolates the Primary Recovery Glovebox from the Gas Collection System
V-12	**Analyzer Exhaust Isolation**	Isolates the Analyzer Exhaust from the Gas Collection System
V-13	**Condensate Collection Isolation**	Isolates the Condenser from the condensate collection canisters valve.
V-14	**Condensate Collection Canister valve**	Valve on the Condensate Collection Canister

TABLE 5. Gas Collection System Valves – Located in the Gas Collection System Enclosure (D035)

Valve	Description	Function
GC-1	**D024 Hot Cell Isolation**	Isolates the Gas Collection System from the D024 Hot Cell
GC-2	**Open Only For Non-Rad Experiments**	This valve can be open during the Commissioning Tests. When sulfuric acid solution is in the Vessel. Keep valves GC-3 and GC-4 closed to protect the zeolite cartridge from moisture.
GC-3 and GC-4	**Silver Zeolite Isolation valves**	Open during AMORE Experiments. Valve GC-2 is kept closed.
GC-5 and GC-6	**Condensate Drain Valves**	Open when draining condensate
GC-7	**Port**	Port for maintenance. Capped
CSV-1	**High Pressure Isolation**	Part of the interlocks. Closed when the Gas Collection interlock is tripped

TABLE 6. Dump Tank Valves – Located beneath the D035 Hot Cell

Valve	Description	Function
D-1A & D-1B	**Pick-up line to Primary Recovery Glovebox**	Siphons solution from the bottom of the Dump Tank to transport it to the Primary Recovery Glovebox.
D-2A & D-2B	**Return Line from Primary Recovery Glovebox**	Returns solution to the top of the Tank from the Primary Recovery Glovebox
D-3A & D-3B	**Dump Tank headspace gas to Gas Distribution Hub Manifold**	Allows for gas displacement when solution is entering the Dump Tank

TABLE 7. Gas Collection System Chassis Control Settings – Located in D032

Chamber	Set point - 1	Alarm-2 High	Dead Band
Chamber #1	**900 to 960 mbar**	**990 mbar**	**10 mbar**
Chamber #2	**1030 mbar**	**1300 mbar**	**110 mbar**
Collection Cylinders	**2000 psig**	**2000 psig**	**N/A**

TABLE 8. Analytical Manifold Valves – Located in the D024 Analytical Enclosure

Valve	Description	Function
A-1	**Mini-AMORE isolation**	Isolates Mini-AMORE from the manifold
A-2	**Gas Collection System Isolation**	Close to isolate the manifold and Mini-AMORE from Gas Collection System
A-3	**Analyzer #1 Isolation**	Isolates Analyzer #1 from the manifold
A-4	**Vacuum Inlet**	Opens the manifold to vacuum.
A-5	**Calibration isolation**	Isolate calibration gas
A-6	**Analyzer #2 Isolation**	Isolates Analyzer #2 from the manifold
A-7	**Hub Manifold isolation**	Isolates the Hub Manifold in the D-035 Gas Distribution Hub Enclosure from the Analytical Manifold
A-8	**Analyzer #2 Hydrogen Sensor isolation**	Isolates Analyzer #2 from the Hydrogen Sensor
A-9 and A-10	**Hydrogen Sensor Isolation**	Isolates the Hydrogen Sensor for removal
A-11	**Vacuum Exhaust**	Isolates the Vacuum Pump exhaust from the Gas Collection System
A-12	**Calibration Gas**	Allows calibration gas up to A-5

APPENDIX 10

LEAF-PROC-018, Rev.3: AMORE Gas Handling Pre-Run Checklist

AMORE Gas Handling Pre-Run Checklist

Low Energy Accelerator Facility, LEAF-PROC-018, Rev.3

Approved:  Date: 03.23.2021

Sergey Chemerisov, Manager, IVEM/LEAF

Effective Date: 03.24.2021

Experiment_____

NOTE: A second person should verify the steps on this checklist

1 Purpose

Establish a pre-run checklist for gas handling in the AMORE experiment.

2 Scope

This procedure applies to the following Argonne activities and entities.

LMS core processes:	Asset Management
Organizations:	Experimental Operations and Facilities (EOF) Division
Buildings:	211
Specific locations:	LINAC
Other applicability factors:	None
Exclusions:	None
USI applicability:	Yes

3 Work Process

3.1 Introduction

This procedure established the order in which pre-run checks of the AMORE gas handling system shall be performed.

3.2 Step-by-Step Procedure

The steps below are mandatory unless noted otherwise. This procedure is to be performed by trained personnel.

3.2.1 Gas Collection System Chassis Settings in D032

Step	Action		
		Initials	**Verified**
1	Collection Cylinders should be less than 1600psi.	_____	_____
2	Chamber #1: Controller on far right – Set Point-1: between 900 and 960 mbar Alarm-2 High setting: 990 mbar Dead-Band 10mbar.	_____	_____

Step	Action		
3	Chamber #2: Controller in the middle – Set Point-1: 1020mbar Alarm-2 Low: 1000mbar Alarm-2 High: 1250mbar Dead-Band: 100 mbar.	_____	_____
4	Collection Cylinders: Controller on the left – Alarm-2 High: 2000 psi Alarm-1 High: 2000 psi.	_____	_____
5	RESET the Catalyst heater. Set to 130 ºC. Turn the dial to the second mark.	_____	_____

3.2.2 Gas Distribution Hub Manifold in D035 LINAC Cell [ASE2.5.2.1]

Step	Action	Initials	Verified
1	Close Valves V-4 ☐☐ V-8 ☐☐ V-15 ☐☐ V-17 ☐☐ V-18 ☐☐ SV-3 OFF ☐☐	_____	_____
2	Open Valves V-1 ☐☐ V-2 ☐☐ V-3 ☐☐ V-6 ☐☐ V-7 ☐☐ V-9 ☐☐ V-10 ☐☐ V-11 ☐☐ V-12 ☐☐ V-16 ☐☐ V-19 ☐☐	_____	_____
3	Replace the Gas Distribution Hub Door.	_____	_____
4	If installed, remove the meter for measuring manifold pressure.	_____	_____
5	Turn the Meter Switch OFF.	_____	_____

3.2.3 Dump Tank in D035 LINAC CELL [ASE2.5.2.1]

Step	Action	Initials	Verified
1	Open all valves D-1A ☐☐ D-2A ☐☐ D-3A ☐☐	_____	_____

3.2.4 Gas Collection Enclosure D035 LINAC Cell

Step	Action	Initials	Verified
1	Verify the presence of a backflow orifice **[ASE 2.5.2.3]**	_____	_____
2	Open valve GC-1 **[ASE2.5.2.1]**	_____	_____

3.2.5 D024 Analytical Enclosure [ASE2.5.2.1]

Step	Action	Initials	Verified
1	**When Performing the AMORE Experiment ONLY** (If performing Mini-AMORE, go to step 5) Close Valves A-1 ☐☐ A-2 ☐☐ A-3 ☐☐ A-4 ☐☐ A-5 ☐☐ A-6 ☐☐ A-11 ☐☐ A-12 ☐☐	_____	_____
2	Open Valves A-7 ☐☐ A-8 ☐ ☐ A-9 ☐☐ A-10 ☐☐	_____	_____
3	Ensure the Vacuum Pump is OFF	_____	_____
4	Open inlet valve on Analyzer #2.	_____	_____
5	**When performing Mini-AMORE and AMORE Experiments together** (If not applicable N/A) Close Valves A-3 ☐☐ A-4 ☐☐ A-5 ☐☐ A-6 ☐☐ A-11 ☐☐ A-12 ☐☐	_____	_____
6	Open Valves A-1 ☐☐ A-2 ☐☐ A-7 ☐☐ A-8 ☐☐ A-9 ☐☐ A-10 ☐☐	_____	_____
7	Ensure the Vacuum Pump is OFF	_____	_____
8	Open the inlet valve on Analyzer #1 & #2.	_____	_____

3.2.6 Gas Cylinders in D032 [ASE2.5.2.1]

Step	Action	Initials	Verified
1	Open Isolation and cylinder valves on the Oxygen Tank.	_____	_____
2	Open Isolation and cylinder valves on Helium/Xenon Tank.	_____	_____
3	Open Isolation and cylinder valves on Mini-AMORE Helium/Xenon Tank when preforming that experiment. (If not applicable "N/A)	_____	_____

3.2.7 Cell # 2

Step	Action	Initials	Verified
1	Chiller ON **[ASE2.5.2.1]**	_____	_____

3.2.8 Gas Analysis Chassis in the D-101 LINAC Control Room

Step	Action	Initials	Verified
1	Catalyst Pump ON	_____	_____
2	Sampling Pump ON	_____	_____
3	Catalyst Pump Alarm Bypass OFF	_____	_____
4	Sampling Pump Alarm Bypass OFF	_____	_____
5	Solution Vessel Pressure alarm high setting 990mbar	_____	_____
6	Catalyst Pump Pressure alarm high setting < Gas Collection System	_____	_____

4 Records Created by Work Process

The records listed below must be retained as indicated.

Description of Record (include form number if applicable)	Active Records Custodian	Active Records Retention	Indexing Method, Storage Medium	Federal Retention Requirements*

Description of Record (include form number if applicable)	Active Records Custodian	Active Records Retention	Indexing Method, Storage Medium	Federal Retention Requirements*
Completed LEAF-PROC-018	Facility Manager	3 years	Index by job date and name, store on paper or electronically	Destroy 75 years after the date of the permit (DOE ADM 18.37)

*If records are maintained in a business information system that is not currently programmed to purge digital records based on age, the records may be retained in that system past the indicated destruction date.

5 Related Documents

This procedure implements requirements established by the following safety basis documents:

- LEAF-SAD-100, Linac Safety Assessment Document
- LEAF-ASE-100, Linac Accelerator Safety Envelope

This procedure implements requirements established by the following Argonne policies and procedures:

- LMS-PROC-188, *Accelerator Safety*
- EGS-PP-100, *Configuration Management Program Plan for Accelerators*

6 Definitions

None

7 About this Procedure

Issuing organization:	Low Energy Accelerator Facility
Procedure owner:	M. Kalensky
Point of contact:	M. Kalensky
Review cycle (months):	36
Date last revised:	3.18.2020
Date last reviewed:	3.23.2020

8 Summary of Changes in This Version

Change in Section 3.2.2: Step 1 “Turn off the Pump” has been removed since the “Pump” is no longer in use. There are only 5 Steps in this version so they have been re-numbered.

In Section 3.2.2: Step 1: “Close Valves” V-15, V-17 and V-18 have been added. “Close Valves” V-5 has been deleted. (This reflects changes made to the manifold to accommodate new experiments)

In Section 3.2.2: Step 2: “Open Valves” V-16 and V-19 has been added. . (This reflects changes made to the manifold to accommodate new experiments)

APPENDIX 11

LEAF-PROC-020, Rev. 2: Maintenance and Leak Testing in Catalyst Pump Enclosure

Maintenance and Leak Testing in Catalyst Pump Enclosure

Low Energy Accelerator Facility, LEAF-PROC-020, Rev. 2

Approved: _____ Date: _____

Sergey Chemerisov, Manager, IVEM/LEAF

Effective Date: _____

1 Purpose

Establish the process for . . .

2 Scope

This procedure applies to the following Argonne activities and entities.

LMS core processes:	Asset Management
Organizations:	Experimental Operations and Facilities (EOF) Division
Buildings:	211
Specific locations:	LINAC
Other applicability factors:	None
Exclusions:	None
USI applicability:	Yes

3 Work Process

3.1 Introduction

The following applies to maintenance and replacement of parts in the Catalyst Pump Enclosure

3.2 Step-by-Step Procedure

The steps below are mandatory unless noted otherwise. This procedure is to be performed by <leave blank>.

NOTE: *Do Not apply a vacuum of <800 mbar to the system.

NOTE: *Do Not apply a pressure of >1345 mbar to the system.

NOTE: *Do not set the Setpoint 1 of Chamber #1 on the Gas Collection System to <800 mbar.

3.2.1 Purging and Maintenance

Step	Action
1	Ensure that solution from the Solution Vessel has been transferred to the Dump Tank or Verification Tank.
2	In the LINAC Control Room, turn off the Catalyst Pump and Sampling Pump. Set Chassis #1 for control in D-035 (downstairs).
3	In D-032, open the helium cylinder and isolation valves.
4	In the Gas Collection System Enclosure, Close valve GC-1.
5	On the Dump Tank, close valves D-1A, D2A and D-3A to isolate it.
6	On the Gas Collection System Chassis for Chamber #1 control, set the Setpoint-1 to 940 mbar. Then set the Dead-band to 10mbar.

Step	Action
7	Install the pressure meter and turn on the switch
8	On the Hub Manifold (see Figure 1), open valve V-3 to allow helium into the manifold.
9	Close valves to isolate the various parts of the AMORE system. Close V-2 and V-10 to isolate the Analytical Manifold in D-024. Close V-11 to isolate the Primary Recovery Glovebox.
10	Open valves V-1, V-6, V-7, V-9 and V-19.
11	Actuate SV-3 to purge with helium for about 5 minutes.
12	De-actuate SV-3 and allow the Gas Collection System to evacuate.
13	Perform maintenance.

3.2.2 Leak Test

Step	Action
1	On the Hub Manifold, Close V-6 to isolate the Gas Collection System.
2	Pressurize the Catalyst Pump fittings to 1100 mbar by actuating SV-3 (open). Close when complete.
3	Use the Leak Detector sniffer to test fittings.
4	When complete, on the Hub Manifold, open valve V-6.
5	Close valve V-3
6	Reset the settings on the Gas Collection System Chassis controller for Chamber #1 Setpoint-1 anywhere from 900 to 960mbar and Dead-band to 10.

4 Records Created by Work Process

The records listed below must be retained as indicated.

Description of Record (include form number if applicable)	Active Records Custodian	Active Records Retention	Indexing Method, Storage Medium	Federal Retention Requirements*
<….>	Facility Manager	3 years	Index by job date and name, store on paper or electronically	Destroy 75 years after the date of the permit (DOE ADM 18.37)

*If records are maintained in a business information system that is not currently programmed to purge digital records based on age, the records may be retained in that system past the indicated destruction date.

5 Related Documents

This procedure implements requirements established by the following safety basis documents:

- LEAF-SAD-100, Linac Safety Assessment Document
- LEAF-ASE-100, Linac Accelerator Safety Envelope

This procedure implements requirements established by the following Argonne policies and procedures:

- LMS-PROC-188, *Accelerator Safety*
- EGS-PP-100, *Configuration Management Program Plan for Accelerators*

6 Definitions

None

7 About this Procedure

Issuing organization:	Low Energy Accelerator Facility
Procedure owner:	S. Chemerisov
Point of contact:	S. Chemerisov
Review cycle (months):	36
Date last revised:	TBD
Date last reviewed:	TBD

8 Summary of Changes in This Version

Section 3.2.1 Step 7 Setpoint-1 Changed from 900 to 940 mbar and Then Dead-band from 50 to 10mbar.

Exhibit A: Figure

Figure 1: Diagram of the Hub Manifold in the Gas Distribution Hub Enclosure.

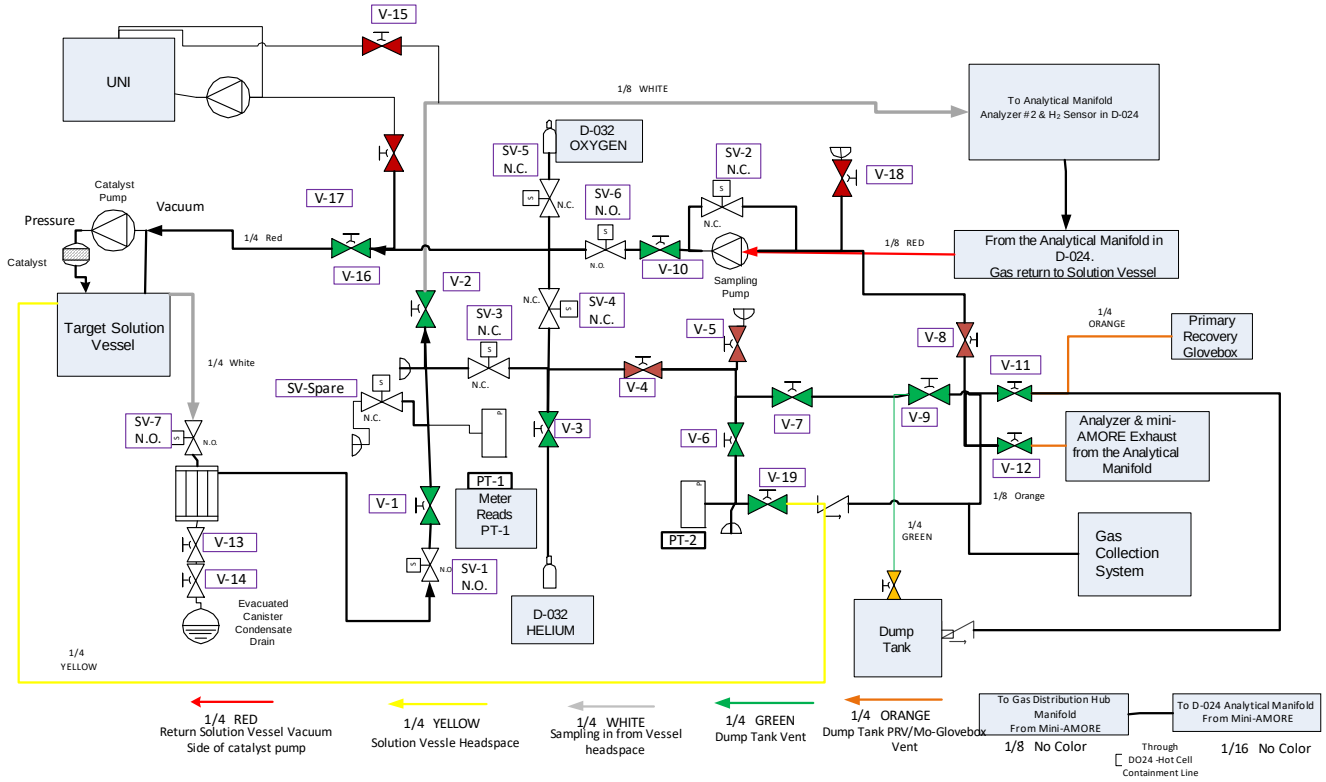


Exhibit B: Tables

TABLE 1. Hub Manifold Manual Valves – Located in the Gas Distribution Hub Enclosure (D-035)

Valve	Description	Function
V-1	**Condenser Isolation**	Isolates the Condenser from the Hub Manifold
V-2 and V-10	**Analytical Manifold Isolation**	Closing V-2 and V-10 isolate the Analytical Manifold in D-024 from the Hub Manifold.
V-3	**Helium Isolation Valve**	Allows helium in to the Hub Manifold for purging the Target Solution Vessel, Dump Tank and the Analytical Manifold.
V-4	**Main Isolation**	Isolates sampling and purge connections from the Gas Collection System connections on the Hub Manifold.
V-5	**Vacuum Inlet**	Opens the manifold to Vacuum Pump inlet.
V-6	**Target Solution Vessel Head Space Isolation**	Isolates the headspace of the Solution Vessel from the Gas Collection System and Vacuum Pump. Opening V-6, V-7 and V-9 opens the Solution Vessel headspace to the Gas Collection System.
V-7	**Target Solution Vessel Head Space Isolation**	Opening V-6, V-7, and V-9 opens the Solution Vessel headspace to the Gas Collection System.
V-8	**Vacuum Exhaust to Gas Collection**	Opens the vacuum pump exhaust to the Gas Collection System
V-9	**Gas Collection Isolation**	Isolates the Hub Manifold from the Gas Collection System
V-10	**Analytical Manifold Isolation**	Closing V-2 and V-10 isolate the Analytical Manifold in D-024 from the Hub Manifold. (Attached to the Sampling Pump)
V-11	**Primary Recovery Glovebox Isolation**	Isolates the Primary Recovery Glovebox from the Gas Collection System
V-12	**Analyzer Exhaust Isolation**	Isolates the Analyzer Exhaust from the Gas Collection System
V-13	**Condensate Collection Isolation**	Isolates the Condenser from the condensate collection canisters valve.
V-14	**Condensate Collection Canister valve**	Valve on the Condensate Collection Canister
V-15	**UNI to Analysis**	UNI to D024 Analytical Hub
V-16	**Return to TSV**	Return to TSV from D024 Analytical
V-17	**UNI return**	UNI return from D024 Analytical Hub
V-18	**Spare (Capped)**	

V-19	**Vessel Headspace to Gas Collection**	Vessel to Gas Collection

TABLE 2. Hub Manifold Solenoid Valves – Located in the Gas Distribution Hub Enclosure (D035) - (Controls on Chassis 1)

Valve	Description	Function
SV-1	**Gas Sampling Isolation**	**Normally Open** – Allows the gas stream from the Solution Vessel into the Hub Manifold. Purge the Analytical Manifold in D-024 post irradiation by actuating SV-3 and SV-1.
SV-2	**Sampling Pump Bypass**	**Normally Closed** – Should the sampling pump fail, the Analytical Manifold in D-024 can be purged by actuating SV-1, SV-2 and SV-3.
SV-3	**Isolation Valve/Helium Purge**	**Normally Closed** – Isolates the Gas Sample Path from the helium purge line during an experiment. Open it to purge the Analytical Manifold for calibration and pre-run. You can purge the Analytical Manifold post irradiation by actuating SV-3 and SV-7.
SV-4	**Helium purge**	**Normally Closed** – Open it to purge the Solution Vessel.
SV-5	**Oxygen Addition**	**Normally Closed** – Open it to add oxygen to the Solution Vessel to reduce hydrogen concentration. Use in conjunction with the Oxygen Flow potentiometer on Chassis 2.
SV-6	**Sampling Pump Isolation**	**Normally Open** – Isolates the Sampling Pump from the Solution Vessel
SV-7	**Gas Sampling Isolation**	**Normally Open** – Allows the analytical gas stream from the Solution Vessel into the Condenser.

TABLE 3. Dump Tank Valves – Located beneath the D-035 Hot Cell

Valve	Description	Function
D-1A and D-1B	**Pick-up line to Primary Recovery Glovebox**	Siphons solution from the bottom of the Dump Tank to transport it to the Primary Recovery Glovebox.
D-2A and D-2B	**Return Line from Primary Recovery Glovebox**	Returns solution to the top of the DumpTank from the Primary Recovery Glovebox
D-3A and D-3B	**Dump Tank headspace gas to Gas Distribution Hub Manifold**	Allows for gas displacement when solution is entering the Dump Tank

APPENDIX 12

LEAF-PROC-021, Rev. 2: Maintenance and Leak Testing in D-024 Analytical Enclosure

Maintenance and Leak Testing in D-024 Analytical Enclosure

Low Energy Accelerator Facility, LEAF-PROC-021, Rev. 2

Approved: _____ Date: _____

Sergey Chemerisov, Manager, IVEM/LEAF

Effective Date: _____

1 Purpose

Establish the process for maintenance and leak testing of the D-024 Analytical Enclosure

2 Scope

This procedure applies to the following Argonne activities and entities.

LMS core processes:	Asset Management
Organizations:	Experimental Operations and Facilities (EOF) Division
Buildings:	211
Specific locations:	LINAC
Other applicability factors:	None
Exclusions:	None
USI applicability:	Yes

3 Work Process

3.1 Introduction

The following applies to maintenance of equipment located in the D024 Analytical Enclosure

3.2 Step-by-Step Procedure

The steps below are mandatory unless noted otherwise. This procedure is to be performed by qualified personel.

NOTE: *Do Not apply a vacuum of <800 mbar to the system.

NOTE: *Do Not apply a pressure of >1345 mbar to the system.

NOTE: *Do not set the Setpoint 1 of Chamber #1 on the Gas Collection System to <800 mbar.

3.2.1 Purging and Maintenance

Step	Action
1	In the LINAC Control Room, turn off the Catalyst Pump and Sampling Pump. Set Chassis #1 for control in D-035 (downstairs).
2	In D-032, open the helium cylinder and isolation valves. The regulator should be set to less than 5psig.
3	Turn off both Analyzers.
4	On the Gas Collection System, Close GC-1.
5	On the Dump Tank, close valves D-1A, D-2A and D-3A to isolate it.
6	On the Gas Collection System Chassis for Chamber #1 control, set the Setpoint-1 to 900 mbar. Then set the Dead-band to 50 mbar.

Step	Action
7	On the Gas Distribution Hub Enclosure install the pressure meter and turn on the switch.
8	On the Hub Manifold (see Figure 1), open valve V-3 to allow helium into the manifold
9	Close V-1, V-4, V-6, V-8, V-10 and V-11.
10	Actuate SV-3 (Open) to allow helium to the Analytical Manifold in D024
11	On the Analytical Manifold in the D-024 Analytical Enclosure (See Figure 2), valve A-7 allows helium into the manifold. Close valves A-1, A-4, A-5, A-11, and A-12. Purge the manifold by opening A-2, A-3, A-6, A-7, A-8, A-9, and A-10. Purge for about a minute then close valve A-7.
12	Allow the manifold to evacuate. Perform maintenance on valves or devices.

3.2.2 Leak Test

Step	Action
1	On the Gas Distribution Hub Manifold in D035, Close valve V-12.
2	On the D024 Analytical manifold, close valve A-10. Open A-7 to pressurize the manifold to about 1100 mbar as read on the manometer. Close A-7 when complete.
3	Use the Leak Detector sniffer to check fittings.
4	When complete, on the Gas Distribution Hub Manifold open V-12 to relieve manifold pressure then open V-6. De-actuate (close) SV-3 and close V-3.
5	On the D024 Analytical Manifold, close valves A-2 and A-3.
6	De-actuate (close) SV-2 and SV-3.
7	Reset the settings on the Gas Collection System Controller (Chamber #1 settings Setpoint-1 anywhere from 900 to 960 mbar and Dead-band to 10).
8	Restart the Analyzers.

4 Records Created by Work Process

The records listed below must be retained as indicated.

Description of Record (include form number if applicable)	Active Records Custodian	Active Records Retention	Indexing Method, Storage Medium	Federal Retention Requirements*
<….>	Facility	3 years	Index by job date and name, store on	Destroy 75 years after the date of the permit

Description of Record (include form number if applicable)	Active Records Custodian	Active Records Retention	Indexing Method, Storage Medium	Federal Retention Requirements*
	Manager		paper or electronically	(DOE ADM 18.37)

*If records are maintained in a business information system that is not currently programmed to purge digital records based on age, the records may be retained in that system past the indicated destruction date.

5 Related Documents

This procedure implements requirements established by the following safety basis documents:

- LEAF-SAD-100, Linac Safety Assessment Document
- LEAF-ASE-100, Linac Accelerator Safety Envelope

This procedure implements requirements established by the following Argonne policies and procedures:

- LMS-PROC-188, *Accelerator Safety*
- EGS-PP-100, *Configuration Management Program Plan for Accelerators*

6 Definitions

None

7 About this Procedure

Issuing organization:	Low Energy Accelerator Facility
Procedure owner:	S. Chemerisov
Point of contact:	S. Chemerisov
Review cycle (months):	36
Date last revised:	TBD
Date last reviewed:	TBD

8 Summary of Changes in This Version

Section 3.2.1 Step 1 “Ensure that solution from the Solution Vessel has been transferred to the Dump Tank or Verification Tank.” This has been removed because it is unnecessary.

Section 3.2.1 Step 7 Setpoint-1 change from 850 to 900 mbar. Then set the Dead-band from 100 to 50 mbar.

Section 3.2.2 Has been edited because the previous versions steps were too complex and unnecessary.

Exhibit A Figure has been updated.

Exhibit B Table 1 has been updated. New valves V-15, V-16, V-17, V-18 & V-19.

Exhibit A: Figures

Figure 1. Diagram of the Hub Manifold.

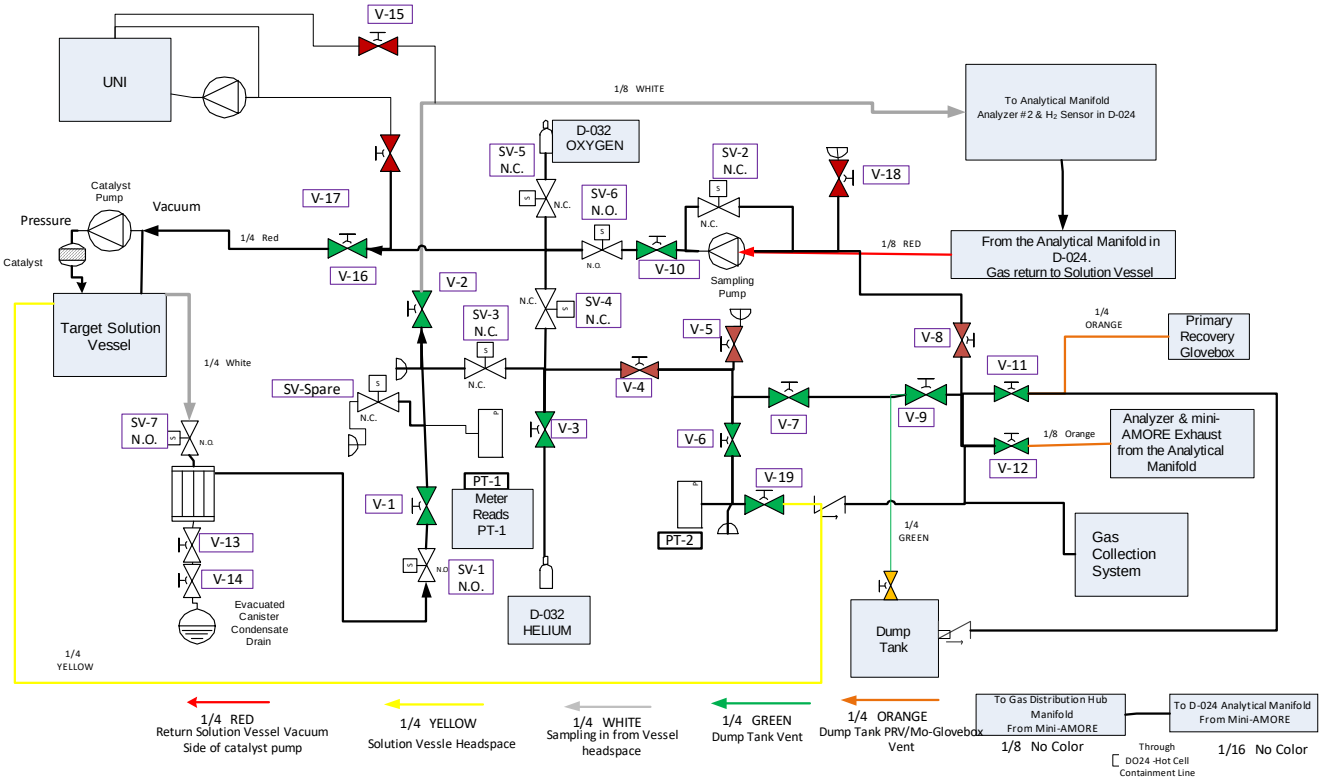


Figure 2. Diagram of the D-024 Analytical Manifold.

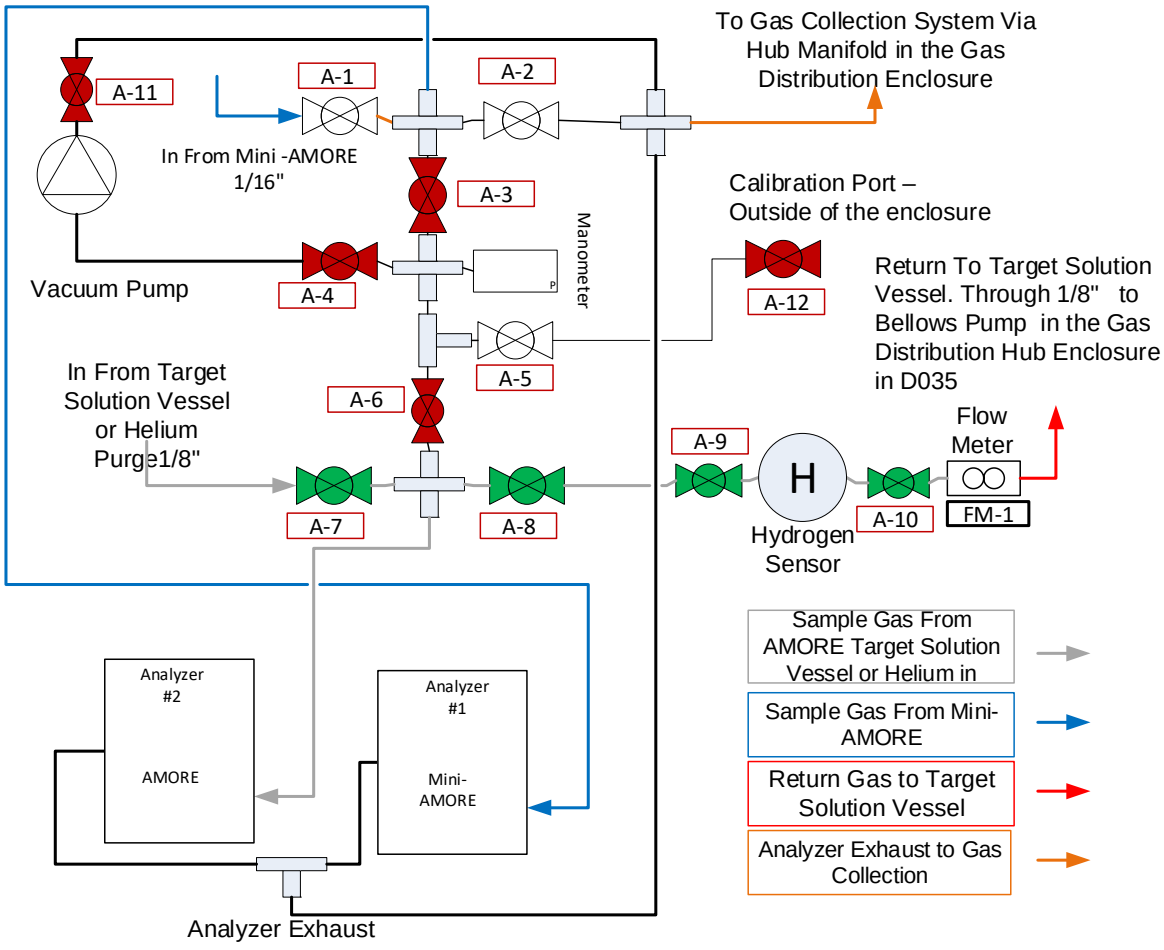


Exhibit B: Tables

TABLE 1. Hub Manifold Manual Valves – Located in the D-035 Gas Distribution Hub Enclosure

Valve	Description	Function
V-1	**Condenser Isolation**	Isolates the Condenser from the Hub Manifold
V-2 and V-10	**Analytical Manifold Isolation**	Closing V-2 and V-10 isolate the Analytical Manifold in D-024 from the Hub Manifold.
V-3	**Helium Isolation Valve**	Allows helium in to the Hub Manifold for purging the Target Solution Vessel, Dump Tank and the Analytical Manifold.
V-4	**Main Isolation**	Isolates sampling and purge connections from the Gas Collection System connections on the Hub Manifold.
V-5	**Vacuum Inlet**	Opens the manifold to Vacuum Pump inlet.
V-6	**Target Solution Vessel Head Space Isolation**	Isolates the headspace of the Solution Vessel from the Gas Collection System and Vacuum Pump. Opening V-6, V-7 and

		V-9 opens the Solution Vessel headspace to the Gas Collection System.
V-7	**Target Solution Vessel Head Space Isolation**	Opening V-6, V-7, and V-9 opens the Solution Vessel headspace to the Gas Collection System.
V-8	**Vacuum Exhaust to Gas Collection**	Opens the vacuum pump exhaust to the Gas Collection System
V-9	**Gas Collection Isolation**	Isolates the Hub Manifold from the Gas Collection System
V-10	**Analytical Manifold Isolation**	Closing V-2 and V-10 isolate the Analytical Manifold in D-024 from the Hub Manifold. (Attached to the Sampling Pump)
V-11	**Primary Recovery Glovebox Isolation**	Isolates the Primary Recovery Glovebox from the Gas Collection System
V-12	**Analyzer Exhaust Isolation**	Isolates the Analyzer Exhaust from the Gas Collection System
V-13	**Condensate Collection Isolation**	Isolates the Condenser from the condensate collection canisters valve.
V-14	**Condensate Collection Canister valve**	Valve on the Condensate Collection Canister
V-15	**UNI to Analysis**	UNI gas to D024 Analytical Manifold
V-16	**Return to TSV**	TVS return from D024 Analytical Manifold
V-17	**Return to UNI**	UNI gas return from D024 Analytical Manifold
V-18	**Spare (Capped)**	
V-19	**TSV Headspace to Gas Collection**	TSV Headspace to Gas Collection

TABLE 2. Hub Manifold Solenoid Valves – Located in the D-035 Gas Distribution Hub Enclosure - (Controls on Chassis 1)

Valve	Description	Function
SV-1	**Gas Sampling Isolation**	**Normally Open** – Allows the gas stream from the Target Solution Vessel into the Hub Manifold. Purge the Analytical Manifold in D-024 post irradiation by actuating SV-3 and SV-1.
SV-2	**Sampling Pump Bypass**	**Normally Closed** – Should the sampling pump fail, the Analytical Manifold in D-024 can be purged by actuating SV-1, SV-2, and SV-3.
SV-3	**Isolation Valve/Helium Purge**	**Normally Closed** – Isolates the Gas Sample Path from the helium purge line during an experiment. Open it to purge the Analytical Manifold for calibration and pre-run. You can purge the Analytical Manifold post irradiation by actuating SV-3 and SV-7.
SV-4	**Helium purge**	**Normally Closed** – Open it to purge the Target Solution Vessel.
SV-5	**Oxygen Addition**	**Normally Closed** – Open it to add oxygen to the Solution Vessel to reduce hydrogen concentration. Use in conjunction with the Oxygen Flow potentiometer on Chassis 2.
SV-6	**Sampling Pump Isolation**	**Normally Open** – Isolates the Sampling Pump from the Solution Vessel
SV-7	**Gas Sampling Isolation**	**Normally Open** – Allows the analytical gas stream from the Target Solution Vessel into the Condenser.

TABLE 3. Analytical Manifold Valves – Located in the D-024 Analytical Enclosure

Valve	Description	Function
A-1	**Mini-AMORE isolation**	Isolates Mini-AMORE from the manifold
A-2	**Gas Collection System Isolation**	Close to isolate the manifold and Mini-AMORE from Gas Collection System
A-3	**Analyzer #1 Isolation**	Isolates Analyzer #1 from the manifold
A-4	**Vacuum Inlet**	Opens the manifold to vacuum.
A-5	**Calibration isolation**	Isolate calibration gas
A-6	**Analyzer #2 Isolation**	Isolates Analyzer #2 from the manifold
A-7	**Hub Manifold isolation**	Isolates the Hub Manifold in the D-035 Gas Distribution Hub Enclosure from the Analytical Manifold
A-8	**Analyzer #2 Hydrogen Sensor isolation**	Isolates Analyzer #2 from the Hydrogen Sensor
A-9 and A-10	**Hydrogen Sensor Isolation**	Isolates the Hydrogen Sensor for removal
A-11	**Vacuum Exhaust**	Isolates the Vacuum Pump exhaust from the Gas Collection System
A-12	**Calibration Gas**	Allows calibration gas up to A-5

APPENDIX 13

LEAF-PROC-022, Rev. 2: Maintenance and Leak Testing in D-035 Gas Distribution Hub Enclosure

Maintenance and Leak Testing in D-035 Gas Distribution Hub Enclosure

Low Energy Accelerator Facility, LEAF-PROC-022, Rev. 2

Approved: _____ Date: _____

Sergey Chemerisov, Manager, IVEM/LEAF

Effective Date: _____

1 Purpose

Establish the process for maintenance and leak testing in the D-035 Gas Distribution Hub enclosure.

2 Scope

This procedure applies to the following Argonne activities and entities.

LMS core processes:	Asset Management
Organizations:	Experimental Operations and Facilities (EOF) Division
Buildings:	211
Specific locations:	LINAC
Other applicability factors:	None
Exclusions:	None
USI applicability:	Yes

3 Work Process

3.1 Introduction

The following pertains to equipment removal and installation in the Gas Distribution Hub enclosure.

3.2 Step-by-Step Procedure

The steps below are mandatory unless noted otherwise. This procedure is to be performed by qualified.

NOTE: *Do Not apply a vacuum of <800 mbar to the system.

NOTE: *Do Not apply a pressure of >1345 mbar to the system.

NOTE: *Do not set the Setpoint 1 of Chamber #1 on the Gas Collection System to <800 mbar.

3.2.1 Purge the Hub Manifold and perform maintenance

See Exhibit A, Hub Manifold Diagram; and Exhibit B, Table 1, Hub Manifold Manual Valves; Table 2, Hub Manifold Solenoid Valves; and Table 3. Dump Tank Valves.

Step	Action
1	In the LINAC Control Room, turn off the Catalyst Pump and Sampling Pump. Set Chassis #1 for control in D-035 (downstairs).
2	In D-032, open the helium cylinder and isolation valves. The regulator should be set to less than 5 psig.
3	Turn off both Analyzers.
4	On the Gas Collection System, Close GC-1.
5	On the Dump Tank, close valves D-1A, D-2A and D-3A to isolate the Dump Tank.

Step	Action
6	On the Gas Collection System Chassis for Chamber #1 control, set the Setpoint-1 to 900 mbar. Then, set the Dead-band to 50 mbar.
7	On the D-035 Gas Distribution Hub Enclosure, install the pressure meter and turn on the switch.
8	On the Hub Manifold (see Figure 1), open valve V-3 to allow helium into the manifold.
9	Close valves V-2 V-11, V-12, V-15, V-16, V-17, V-19 and SV-7 (actuate)
10	Open valves V-1, V-4, V-6, V-7, V-8, V-9 and V-10
11	To purge, open V-3 and actuate SV-3. Purge for about a minute.
12	Close valve V-3 and allow the manifold to evacuate.
13	Perform Maintenance

3.2.2 Leak Test

Step	Action
1	Close valves V-8 and V-9.
2	Open valve SV-4 (actuate).
3	Pressurize to about 1100 mbar by opening V-3. Close V-3 when complete.
4	Use the Leak Detector sniffer to test.
5	When complete, open valves V-11, V-12, V-16 and V-19.
6	Close valves V-4, V-8 and de-actuate (close) SV-7, SV-4 and SV-3
7	Reset the settings on the Gas Collection System Chassis control for Chamber #1 (Setpoint-1 anywhere from 900 to 940 mbar and Dead-band to 10).
8	Restart the Analyzers.

4 Records Created by Work Process

The records listed below must be retained as indicated.

Description of Record (include form number if applicable)	Active Records Custodian	Active Records Retention	Indexing Method, Storage Medium	Federal Retention Requirements*
<enter if needed>	Facility Manager	3 years	Index by job date and name, store on paper or electronically	Destroy 75 years after the date of the permit (DOE ADM 18.37)

*If records are maintained in a business information system that is not currently programmed to purge digital records based on age, the records may be retained in that system past the indicated destruction date.

5 Related Documents

This procedure implements requirements established by the following safety basis documents:

- LEAF-SAD-100, Linac Safety Assessment Document
- LEAF-ASE-100, Linac Accelerator Safety Envelope

This procedure implements requirements established by the following Argonne policies and procedures:

- LMS-PROC-188, *Accelerator Safety*
- EGS-PP-100, *Configuration Management Program Plan for Accelerators*

6 Definitions

None

7 About this Procedure

Issuing organization:	Low Energy Accelerator Facility
Procedure owner:	S. Chemerisov
Point of contact:	S. Chemerisov
Review cycle (months):	36
Date last revised:	TBD
Date last reviewed:	TBD

8 Summary of Changes in This Version

Section 3.2.1 Step 1 "Ensure that solution from the Target Solution Vessel has been transferred to the Dump Tank or Verification Tank." Has been removed since it is unnecessary.

Section 3.2.1 Step 7 set the Setpoint-1 changed from 850 to 900 mbar. Then, set the Dead-band changed from 100 to 50 mbar.

Section 3.2.2 Has been changed to reflect new valves and reduce complexity.

Exhibit A Hub Manifold Diagram has been updated to show new valves.

Exhibit B Table 1 Hub Manifold Manual Valves has been updated to show new valves.

Exhibit A: Diagram of the Hub Manifold

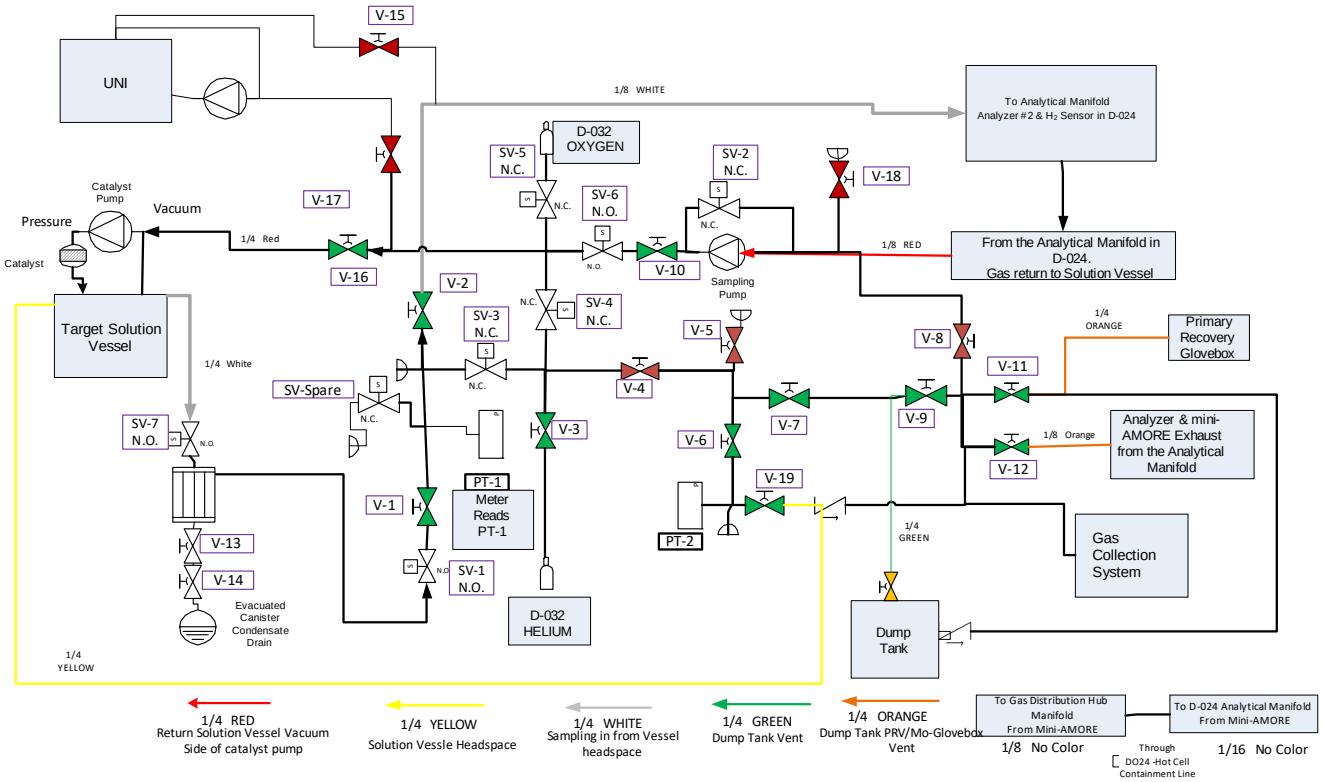


Exhibit B: Tables

TABLE 1. Hub Manifold Manual Valves – Located in the D-035 Gas Distribution Hub Enclosure

Valve	Description	Function
V-1	**Condenser Isolation**	Isolates the Condenser from the Hub Manifold
V-2 and V-10	**Analytical Manifold Isolation**	Closing V-2 and V-10 isolate the Analytical Manifold in D-024 from the Hub Manifold.
V-3	**Helium Isolation Valve**	Allows helium in to the Hub Manifold for purging the Target Solution Vessel, Dump Tank and the Analytical Manifold.
V-4	**Main Isolation**	Isolates sampling and purge connections from the Gas Collection System connections on the Hub Manifold.
V-5	**Vacuum Inlet**	Opens the manifold to Vacuum Pump inlet.
V-6	**Target Solution Vessel Head Space Isolation**	Isolates the headspace of the Solution Vessel from the Gas Collection System and Vacuum Pump. Opening V-6, V-7 and V-9 opens the Solution Vessel headspace to the Gas Collection System.
V-7	**Target Solution Vessel Head Space Isolation**	Opening V-6, V-7, and V-9 opens the Solution Vessel headspace to the Gas Collection System.
V-8	**Vacuum Exhaust to Gas Collection**	Opens the vacuum pump exhaust to the Gas Collection System
V-9	**Gas Collection Isolation**	Isolates the Hub Manifold from the Gas Collection System
V-10	**Analytical Manifold Isolation**	Closing V-2 and V-10 isolate the Analytical Manifold in D-024 from the Hub Manifold. (Attached to the Sampling Pump)
V-11	**Primary Recovery Glovebox Isolation**	Isolates the Primary Recovery Glovebox from the Gas Collection System
V-12	**Analyzer Exhaust Isolation**	Isolates the Analyzer Exhaust from the Gas Collection System
V-13	**Condensate Collection Isolation**	Isolates the Condenser from the condensate collection canisters valve.
V-14	**Condensate Collection Canister valve**	Valve on the Condensate Collection Canister
V-15	**UNI to Analysis**	UNI to Analysis
V-16	**Return to TSV**	Return to TSV from analytical
V-17	**Return to UNI**	Return to UNI from analytical
V-18	**Spare (capped)**	

V-19	**TSV Headspace to Gas Collection**	TSV Headspace to Gas Collection

TABLE 2. Hub Manifold Solenoid Valves – Located in the D-035 Gas Distribution Hub Enclosure - (Controls on Chassis 1)

Valve	Description	Function
SV-1	**Gas Sampling Isolation**	**Normally Open** – Allows the gas stream from the Target Solution Vessel into the Hub Manifold. Purge the Analytical Manifold in D-024 post irradiation by actuating SV-3 and SV-1.
SV-2	**Sampling Pump Bypass**	**Normally Closed** – Should the sampling pump fail, the Analytical Manifold in D-024 can be purged by actuating SV-1, SV-2 and SV-3.
SV-3	**Isolation Valve/Helium Purge**	**Normally Closed** – Isolates the Gas Sample Path from the helium purge line during an experiment. Open it to purge the Analytical Manifold for calibration and pre-run. You can purge the Analytical Manifold post irradiation by actuating SV-3 and SV-7.
SV-4	**Helium purge**	**Normally Closed** – Open it to purge the Target Solution Vessel.
SV-5	**Oxygen Addition**	**Normally Closed** – Open it to add oxygen to the Target Solution Vessel to reduce hydrogen concentration. Use in conjunction with the Oxygen Flow potentiometer on Chassis 2.
SV-6	**Sampling Pump Isolation**	**Normally Open** – Isolates the Sampling Pump from the Target Solution Vessel
SV-7	**Gas Sampling Isolation**	**Normally Open** – Allows the analytical gas stream from the Target Solution Vessel into the Condenser.

TABLE 3. Dump Tank Valves – Located beneath the D035 Hot Cell

Valve	Description	Function
D-1A and D-1B	**Pick-up line to Primary Recovery Glovebox**	Siphons solution from the bottom of the Dump Tank to transport it to the Primary Recovery Glovebox.
D-2A and D-2B	**Return Line from Primary Recovery Glovebox**	Returns solution to the top of the DumpTank from the Primary Recovery Glovebox
D-3A and D-3B	**Dump Tank headspace gas to Gas Distribution Hub Manifold**	Allows for gas displacement when solution is entering the Dump Tank

APPENDIX 14

LEAF-PROC-023, Rev. 2: Maintenance and Leak Testing of the Gas Collection System Enclosure

Maintenance and Leak Testing of the Gas Collection System Enclosure

Low Energy Accelerator Facility, LEAF-PROC-023, Rev. 2

Approved:  Date: 03.23.2021

Sergey Chemerisov, Manager, IVEM/LEAF

Effective Date: 03.24.2021

1 Purpose

Establish the process for maintenance and leak testing of the Gas Collection System Enclosure.

2 Scope

This procedure applies to the following Argonne activities and entities.

LMS core processes:	Asset Management
Organizations:	Experimental Operations and Facilities (EOF) Division
Buildings:	211
Specific locations:	LINAC
Other applicability factors:	None
Exclusions:	None
USI applicability:	Yes

3 Work Process

3.1 Introduction

The following pertains to maintenance activities on equipment in the Gas Collection System Enclosure.

3.2 Step-by-Step Procedure

The steps below are mandatory unless noted otherwise. This procedure is to be performed by qualified personnel.

NOTE: *Do Not apply a vacuum of <800 mbar to the system.

NOTE: *Do Not apply a pressure of >1340 mbar to the system.

NOTE: *Do not set the Setpoint 1 of Chamber #1 on the Gas Collection System to <800 mbar.

3.2.1 Purging and Maintenance

Step	Action
1	Inform LINAC Facility and AMORE personnel that the Gas Collection System will be out of service.
2	In the LINAC Control Room, turn off the Catalyst Pump and Sampling Pump. Set Chassis #1 for control in D-035 (downstairs).
3	Turn off both Analyzers.
4	On the Gas Collection System Enclosure, Close GC-1.
5	On the Dump Tank, close valves D-1A, D-2A and D-3A to isolate the tank.
6	On the Hub Manifold, open valve V-3, V-4, V-7 and V-9 to allow helium to the System.

Step	Action
8	Close valves to isolate the various parts of the AMORE system. Close V-1, V-16 and V-19 to isolate the Target Solution Vessel. Close V-2 and V-8 to isolate Analytical Manifold and UNI. Close V-11 to isolate the Primary Recovery Glovebox. Close V-12 to isolate the Analyzer Exhaust.
9	On the Gas Collection System Chassis for Chamber #1 control, set the Setpoint-1 to 940 mbar and Dead-band to 10 mbar.
10	Open the helium cylinder and isolation valves to purge the Gas Collection System. Allow Chamber #2 to cycle at least three times.
11	Close the Helium Cylinder and Isolation Valves to stop the purge. Allow the system to evacuate. Close GC-SV-1. Turn OFF the Gas Collection System by pushing in the red button below the controllers. Close the valves on the Collection Cylinder.
12	Perform maintenance.

3.2.2 Leak Test Chamber #1 inside Enclosure and attached to the system

Step	Action
1	On the Chamber #1 controller, set the Setpoint-1 to 1100 mbar. Set Alarm-2 to 1200 mbar.
2	Open the helium cylinder and isolation valves to add pressure to Chamber #1 to 1050mbar.
3	Close the helium cylinder and isolation valves.
4	Use the Leak Detector sniffer to test Chamber #1 fittings.

3.2.3 Leak Test Chamber #2 inside Enclosure and attached to the system

Step	Action
1	Open a Collection Cylinder
2	On the Gas Collection System Chassis for Chamber #1 controller, set Setpoint-1 to 950 mbar. Then, set the Dead-band to 10 mbar. Start the Gas collection System by pulling out the red button. Open GC-SV-1
3	Open the helium cylinder and isolation valves in D032.
4	Let Chamber #1 cycle at least once. Then close the helium cylinder and isolation valves.
5	Use the Leak Detector sniffer to test Chamber #2 fittings.

3.2.4 Leak Test the Collection Cylinders

Step	Action
1	Briefly open one of the collection cylinders to pressurize the fittings.
2	Use the Leak Detector sniffer to test Collection Cylinder fittings.

3.2.5 Leak testing either chamber outside of the Enclosure and detached from the system

Step	Action
1	Attach a 5 psig PRV 37 SCFM and a 0.01” orifice to a helium cylinder and regulator. Set the regulator to 1 psig.
2	Attach the cylinder to the Chamber and pressurize to 1 psig,
3	Use the Leak Detector sniffer to test Chambers fittings and gasket.
4	When complete, release the gas into the Gas Collection Enclosure up the vent.

3.2.6 Restart the Gas Collection System

Step	Action
1	In D-035 Gas Distribution Hub Manifold, close valve V-3 and V-4. Open valves V-1, V-6, V-12 and V-19
2	In the Gas Collection Enclosure Open a Collection Cylinder
3	In D-032 close the helium cylinder and isolation valves.
4	On the Gas Collection System Chassis for Chamber #1 control, set the Setpoint-1 to between 900-950 mbar. Then, set the Dead-band to 10 mbar. Turn on the Gas Collection System by pulling out the red button. Open GC-SV-1.
5	Restart the Gas Analyzers

3.2.7 Monthly Check Valve Test GC-CK-2 (Between Collection Cylinders and Chamber #2)

Step	Action
1	Turn off the Gas Analyzers
2	On the D035 Hub Manifold Close valves V-1, V-4, V-8, V-9, V-11, V-12, V-15, V-16 and V-17.
3	In the Gas Collection Enclosure, Close both Collection Cylinder valves.
4	Briefly open and then close the valve on the cylinder with the highest pressure.
5	Observe the pressure drop in the collection cylinders for at least an hour

Step	Action
6	Record the pressure drop in the logbook.
7	Acceptance criteria for Check Valve GC-CK-2 is (Pressure Drop < 12 psig/hour)
8	If the acceptance criteria is not met, the Check Valve should be replaced.

3.2.8 Monthly Check Valve Test GC-CK-1 (Between Chamber #1 and Chamber #2)

Step	Action
1	Turn off the Gas Analyzers
2	On the D035 Hub Manifold Close valves V-1, V-4, V-8, V-9, V-11, V-12, V-15, V-16 and V-17.
3	In the Gas Collection Enclosure, Close both Collection Cylinder valves.
4	Observe the pressure rise in Chamber #1 for at least an hour
5	Record the pressure rise in the logbook.
6	Acceptance criteria for Check Valve GC-CK-1 is (Pressure Rise < 4.9 mbar/hour)
7	If the acceptance criteria is not met, the Check Valve should be replaced.

3.2.9 Monthly test of the UPS Battery Backup

Step	Action
1	In D032 locate the Battery Backup that the controllers for the Gas Collection System are plugged into.
2	Unplug the Battery Backup from the line voltage (wall outlet)
3	Wait 5 minutes.
4	Ensure that the Controllers for the Gas Collection System stay active. Record the available battery time and percent battery life in the logbook.
5	Acceptance criteria for the battery life should be >50% and Controllers stay active.
6	If the acceptance criteria is not met, the battery or the UPS should be replaced.

3.2.10 Response to Gas Collection System Alarms. After the initial assessment and actions, see Exhibit B: Table 2. Alarm Investigation

Step	Action
1	Assess which alarm is active, Chamber #1, Chamber #2 or Collection Cylinders
2	If "Collection Cylinders". This indicates that the "in use" Cylinder is Full. The action is to close the full cylinder and switch to a new Cylinder.
3	If "Chamber #1 or Chamber #2" Isolate the Gas Collection System from gas sources. In the Gas Collection Enclosure, close GC-1. In the D024 Analytical Enclosure, turn of the Gas Analyzers. In D032, Close all cylinder and isolation valves. On the D035 Gas Hub Manifold, Close valves V-1, V-4, V-8, V-9, V-11, V-12, V-15, V-16 and V-17. On the Gas Collection System Chassis Close GC-SV-1. In the Gas Collection System Enclosure, close the Collection Cylinder Valves.

4 Records Created by Work Process

The records listed below must be retained as indicated.

Description of Record (include form number if applicable)	Active Records Custodian	Active Records Retention	Indexing Method, Storage Medium	Federal Retention Requirements*
<….>	Facility Manager	3 years	Index by job date and name, store on paper or electronically	Destroy 75 years after the date of the permit (DOE ADM 18.37)

*If records are maintained in a business information system that is not currently programmed to purge digital records based on age, the records may be retained in that system past the indicated destruction date.

5 Related Documents

This procedure implements requirements established by the following safety basis documents:

- LEAF-SAD-100, Linac Safety Assessment Document
- LEAF-ASE-100, Linac Accelerator Safety Envelope

This procedure implements requirements established by the following Argonne policies and procedures:

- LMS-PROC-188, *Accelerator Safety*
- EGS-PP-100, *Configuration Management Program Plan for Accelerators*

6 Definitions

None

7 About this Procedure

Issuing organization:	Low Energy Accelerator Facility
Procedure owner:	S. Chemerisov
Point of contact:	S. Chemerisov
Review cycle (months):	36
Date last revised:	3.18.2021
Date last reviewed:	3.23.2021

8 Summary of Changes in This Version

Inclusion of section 3.2.7 to test the leak rate of Check Valve GC-CK-2

Inclusion of section 3.2.8 to test the leak rate of Check Valve GC-CK-1

Inclusion of section 3.2.9 Response to Alarms

Update of Exhibit A Figure 2 Gas Collection System

Added Exhibit B: Table 2. Alarm Investigation

Exhibit A: Figures

Figure 1. Diagram of the Hub Manifold in the D-035 Gas Distribution Hub Enclosure.

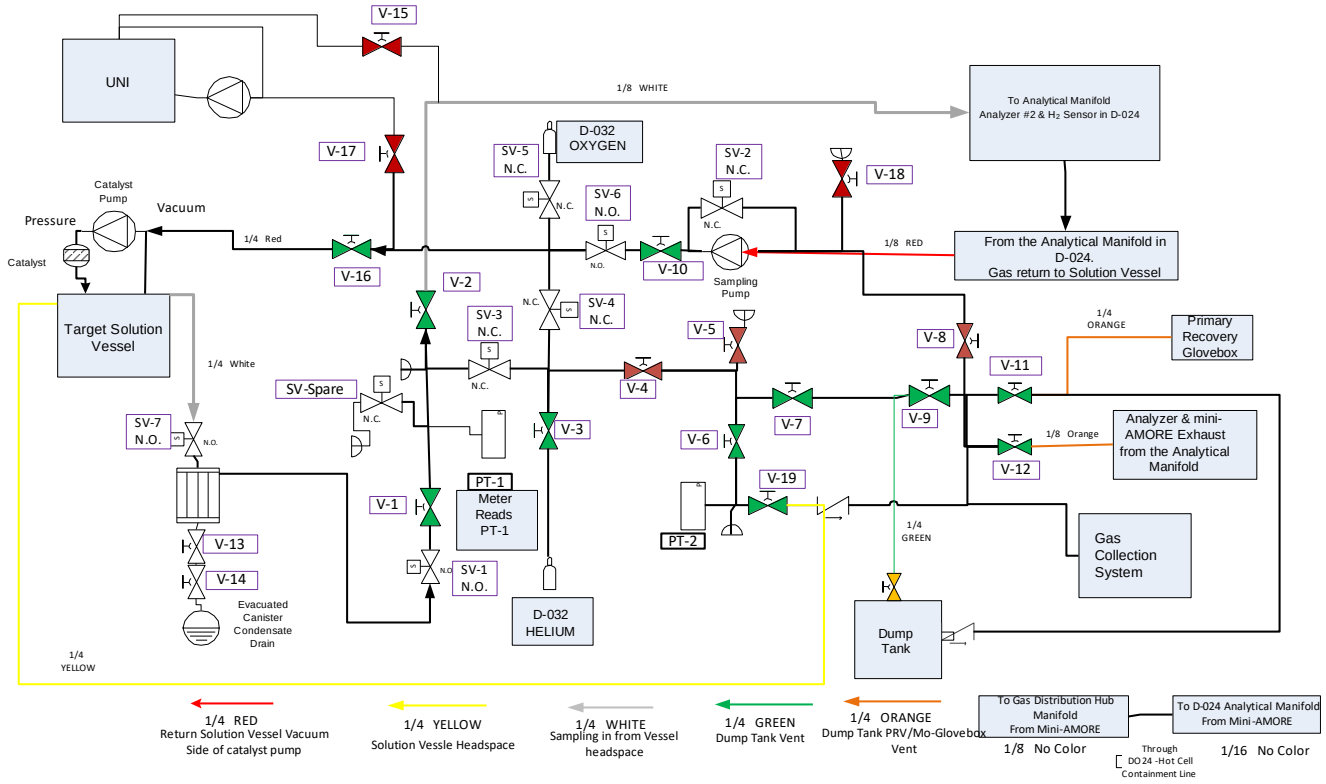
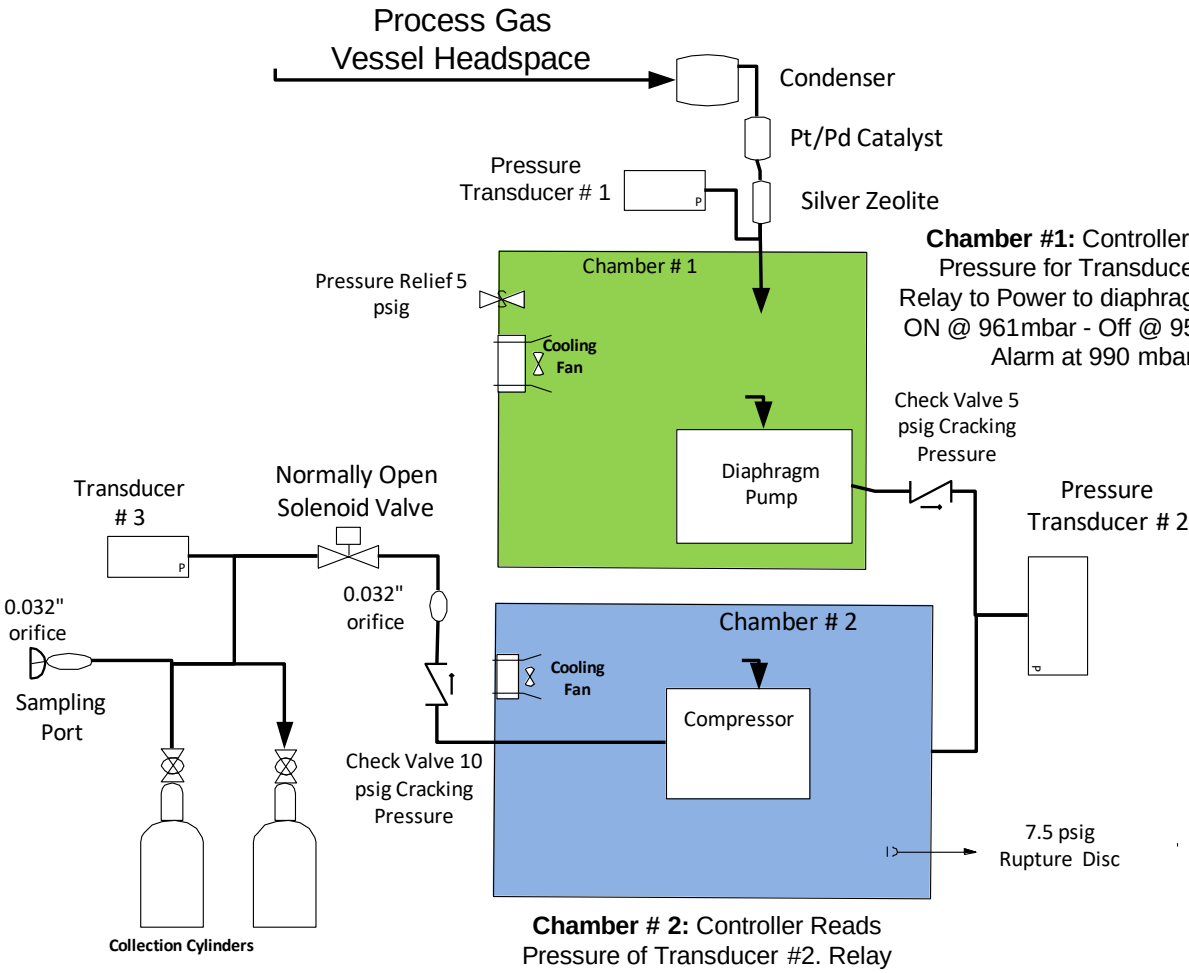


Figure 2. Diagram of the Gas Collection System



Chamber #1: Controller reads Pressure for Transducer #1. Relay to Power to diaphragm pump ON @ 961mbar - Off @ 950 mbar. Alarm at 990 mbar

Collection Cylinder: Controller reads Pressure for Transducer #3. Alarm at 2000 psig

Chamber # 2: Controller Reads Pressure of Transducer #2. Relay To Power to the Compressor ON @ 1126 mbar – Off @ 1015 mbar. Alarm at 1250 mbar

Exhibit B: Tables

TABLE 1. Hub Manifold Manual Valves – Located in the D-035 Gas Distribution Hub Enclosure

Valve	Description	Function
V-1	**Condenser Isolation**	Isolates the Condenser from the Hub Manifold
V-2 and V-10	**Analytical Manifold Isolation**	Closing V-2 and V-10 isolate the Analytical Manifold in D-024 from the Hub Manifold.
V-3	**Helium Isolation Valve**	Allows helium in to the Hub Manifold for purging the Target Solution Vessel, Dump Tank and the Analytical Manifold.
V-4	**Main Isolation**	Isolates sampling and purge connections from the Gas Collection System connections on the Hub Manifold.
V-5	**Vacuum Inlet**	Opens the manifold to Vacuum Pump inlet.
V-6	**Target Solution Vessel Head Space Isolation**	Isolates the headspace of the Solution Vessel from the Gas Collection System and Vacuum Pump. Opening V-6, V-7 and V-9 opens the Solution Vessel headspace to the Gas Collection System.
V-7	**Target Solution Vessel Head Space Isolation**	Opening V-6, V-7, and V-9 opens the Solution Vessel headspace to the Gas Collection System.
V-8	**Vacuum Exhaust to Gas Collection**	Opens the vacuum pump exhaust to the Gas Collection System
V-9	**Gas Collection Isolation**	Isolates the Hub Manifold from the Gas Collection System
V-10	**Analytical Manifold Isolation**	Closing V-2 and V-10 isolate the Analytical Manifold in D-024 from the Hub Manifold. (Attached to the Sampling Pump)
V-11	**Primary Recovery Glovebox Isolation**	Isolates the Primary Recovery Glovebox from the Gas Collection System
V-12	**Analyzer Exhaust Isolation**	Isolates the Analyzer Exhaust from the Gas Collection System
V-13	**Condensate Collection Isolation**	Isolates the Condenser from the condensate collection canisters valve.
V-14	**Condensate Collection Canister valve**	Valve on the Condensate Collection Canister
V-15	**UNI to Analysis**	UNI to Gas Analysis
V-16	**Return to TSV**	Analytical gas returns to TSV
V-17	**UNI Return**	Analytical gas returns to UNI
V-18	**Spare (capped)**	

V-19	**TSV Headspace to Gas Collection**	TSV Headspace to Gas Collection

TABLE 2. Alarm Investigation

Chamber #1 Alarm	Chamber #2 Alarm	Possible Items to Investigate
High	None	Chamber #1 Pump - Check Valve GC-CK-1
High	Low (see also below)	Chamber #2 Compressor - Check Valves
High	High	Chamber #2 Compressor - Check Valves
None	High	Chamber #2 Compressor - Check Valve GC-CK-2
High (>1350mbar)		Possible of Gas Release through Chamber #1 PRV
	Low	Possible of Gas Release through Chamber #2 Rupture Disc

TABLE 2. Hub Manifold Solenoid Valves – Located in the D-035 Gas Distribution Hub Enclosure - (Controls on Chassis 1)

Valve	Description	Function
SV-1	**Gas Sampling Isolation**	**Normally Open** – Allows the gas stream from the Target Solution Vessel into the Hub Manifold. Purge the Analytical Manifold in D-024 post irradiation by actuating SV-3 and SV-1.
SV-2	**Sampling Pump Bypass**	**Normally Closed** – Should the sampling pump fail, the Analytical Manifold in D-024 can be purged by actuating SV-1, SV-2 and SV-3.
SV-3	**Isolation Valve/Helium Purge**	**Normally Closed** – Isolates the Gas Sample Path from the helium purge line during an experiment. Open it to purge the Analytical Manifold for calibration and pre-run. You can purge the Analytical Manifold post irradiation by actuating SV-3 and SV-7.
SV-4	**Helium purge**	**Normally Closed** – Open it to purge the Target Solution Vessel.
SV-5	**Oxygen Addition**	**Normally Closed** – Open it to add oxygen to the Target Solution Vessel to reduce hydrogen concentration. Use in conjunction with the Oxygen Flow potentiometer on Chassis 2.
SV-6	**Sampling Pump Isolation**	**Normally Open** – Isolates the Sampling Pump from the Target Solution Vessel
SV-7	**Gas Sampling Isolation**	**Normally Open** – Allows the analytical gas stream from the Target Solution Vessel into the Condenser.

TABLE 3. Dump Tank Valves – Located beneath the D035 Hot Cell

Valve	Description	Function
D-1A and D-1B	**Pick-up line to Primary Recovery Glovebox**	Siphons solution from the bottom of the Dump Tank to transport it to the Primary Recovery Glovebox.
D-2A and D-2B	**Return Line from Primary Recovery Glovebox**	Returns solution to the top of the Tank from the Primary Recovery Glovebox
D-3A and D-3B	**Dump Tank headspace gas to Gas Distribution Hub Manifold**	Allows for gas displacement when solution is entering the Dump Tank

TABLE 4. Gas Collection System Valves – Located in the Gas Collection System Enclosure (D035)

Valve	Description	Function
GC-1	**D024 Hot Cell Isolation**	Isolates the Gas Collection System from the D024 Hot Cell
GC-2	**Open Only For Non-Rad Experiments**	This valve can be open during the Commissioning Tests. When sulfuric acid solution is in the Vessel. Keep valves GC-3 and GC-4 closed to protect the zeolite cartridge from moisture.
GC-3 and GC-4	**Silver Zeolite Isolation valves**	Open during AMORE Experiments. Valve GC-2 is kept closed.
GC-5 and GC-6	**Condensate Drain Valves**	Open when draining condensate
GC-7	**Port**	Port for maintenance. Capped
CSV-1	**High Pressure Isolation**	Part of the interlocks. Closed when the Gas Collection interlock is tripped

APPENDIX 15

Sampling Gas from AMORE Collection Cylinders

Sampling Gas from AMORE Collection Cylinders

1. Scope Summary

The procedure describes the steps necessary to sample gas from the AMORE Collection Cylinders. The sample is taken to determine the amount of radioactive gases that are present in the cylinders to report for release. This is done after most of the radioactive gases have significantly decayed (60 days post irradiation). This procedure occurs in the Gas Collection Enclosure in D035 of the LINAC Facility. Other activities that require operation of the Gas Collection System must be suspended.

The Gas Collection System is isolated from the various parts of the AMORE system. Then a sampling Assembly is attached to the sampling port. The Sample Cylinder is filled and removed. The Gas Collection System is re-started. The sample is analyzed by gamma spectroscopy. The gamma results are reported to QAS.

2.0 Procedure

2.1 Shutdown the Gas Analyzers in the D024 Analytical Enclosure.

2.2 On the Gas Distribution Hub Manifold, close valves V-1, V-5, V-6, V-7, V-8, V-9, V-10, V-11 and V-12. This will isolate the Gas Collection System from other parts of the AMORE Experiment.

2.3 In the Gas Collection System, close valve GC-1 (to isolate the D024 Hot Cell) and close both Collection Cylinder Valves

2.4 On the Gas Collection Control Chassis in D032, Close GC-SV 1. This isolates Chamber 2 from the High Pressure Cylinders. Then turn off the Gas Collection System by pushing the Red Button.

2.5 Using the glove port on the Gas Collection Enclosure, remove the cap from the Gas Sampling Port. A small amount of gas will be released. Wait for a minute so the Ventilation system evacuates the gas from the enclosure.

2.6 Attach a 0.025inch Orifice and a Sampling Cylinder to a dual stage regulator to make a Sampling Assembly. Close all valves on the assembly and close the regulator valve. (Figure 1)

2.7 Attach the Sampling Assembly to the Sampling Port on the Gas Collection System. (Figure 2)

2.8 Open the valve on the Collection Cylinder that is being sampled.

2.9 Open valve #1. The first gauge on the regulator should rise to the pressure in the Collection Cylinder.

2.10 Set the regulator output to about 150 psig.

2.11 Open Valve #2 and the Sample Valve. This will fill the Cylinder.

2.12 Close all valves on the Sampling Assembly.

2.13 Close the valve on the Collection Cylinder.

2.14 Close the door on the enclosure. Break the connection between Sample Valve and the Orifice. Leave the Cylinder attached. Have a Health Physics Tech survey that connection.

2.15 Remove the Sample Cylinder leaving the Orifice attached to the Regulator. Cap the Sample Valve.

2.16 Remove the Regulator and cap the Sampling Port.

2.17 Open the Collection Cylinder valve.

2.18 Re-start the Gas Collection System by pulling out the Red Button.

2.19 Open GC-SV-1.

2.20 On the Gas Distribution Hub Manifold open valves V-6, V-7, V-9 and V-11.

2.21 The gas sample is analyze using gamma spectroscopy.

Figure 1. Sampling Assembly

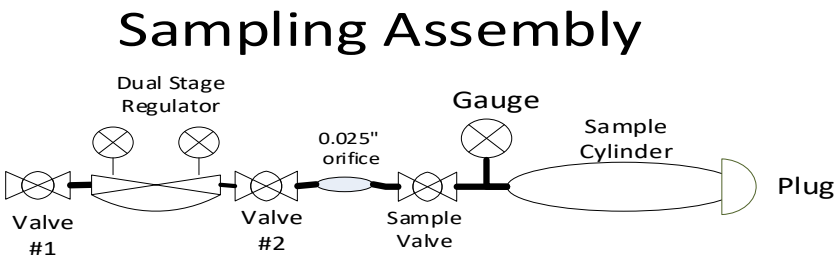
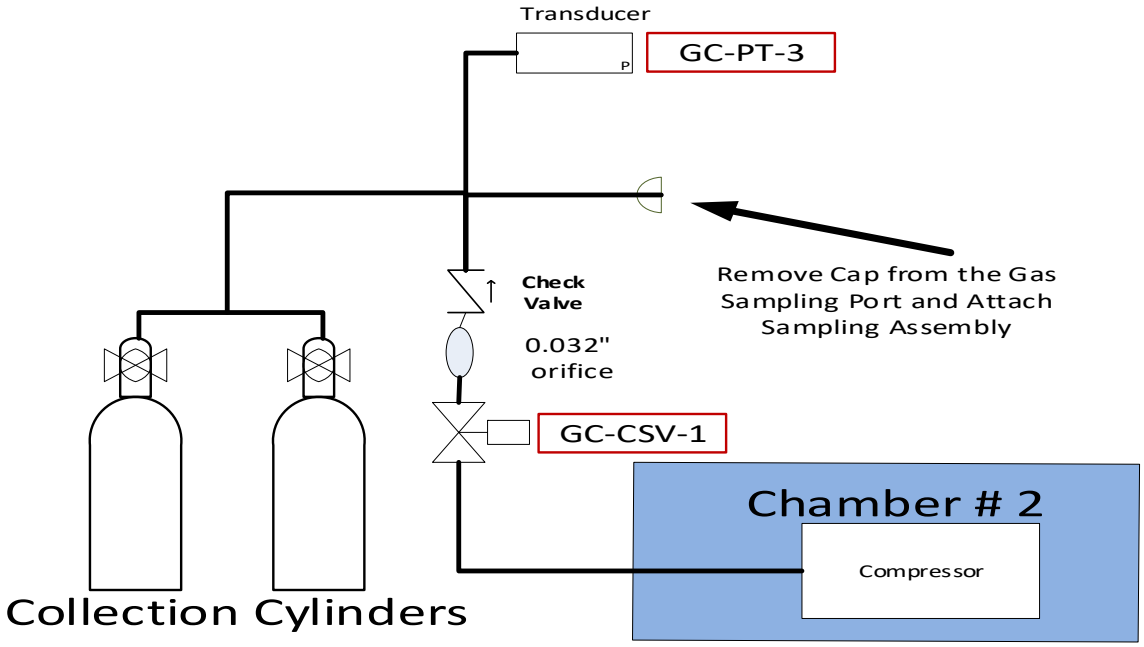


Figure 1. Attachment to Sampling Port in Gas Collection System



APPENDIX 16

Release of Gas from AMORE Collection Cylinders

Release of Gas from AMORE Collection Cylinders

1. Scope Summary

The procedure describes the steps necessary to release gas from the AMORE Collection Cylinders. This procedure is performed only after approval to release the gas has been given by the QAS division. This activity occurs in the Gas Collection Enclosure in D035 of the LINAC Facility. Other activities that require operation of the Gas Collection System must be suspended.

First, the Gas Collection System is isolated from the various parts of the AMORE system. Then a Release Assembly is attached to the sampling port. The gas in the Collection Cylinders is slowly released into the D035 Process Ventilation. The Gas Collection System is re-started.

2.0 Procedure

2.1 Shutdown the Gas Analyzers in the D024 Analytical Enclosure.

2.2 On the Gas Distribution Hub Manifold, close valves V-1, V-5, V-6, V-7, V-8, V-9, V-11 and V-12. This will isolate the Gas Collection System from other parts of the AMORE Experiment.

2.3 In the Gas Collection System Enclosure, close both Collection Cylinder Valves and close valve GC-1 (to isolate the D024 Hot Cell)

2.4 On the Gas Collection Control Chassis in D032, Close GC-SV 1. This isolates Chamber 2 from the High Pressure Cylinders. Then turn off the Gas Collection System by pushing the Red Button.

2.5 Using the glove port on the Gas Collection Enclosure, remove the cap from the Gas Sampling Port. A small amount of gas will be released. Wait for a minute so the Ventilation system evacuates the gas from the enclosure.

2.6 Attach a 0.025inch Orifice, Hepa-filter and Exhaust Tube to a dual stage regulator to make the Release Assembly. Close all valves on the assembly and close the regulator valve. See Figure 1.

2.7 Attach the assembly to the Sampling Port and run Exhaust Tubing from the output of the filter up into the ventilation duct.

2.8 Open the valve on the Collection Cylinder.

2.9 Open valve #1. The first gauge on the regulator should rise to the pressure in the Cylinder.

2.10 Set the regulator output to 10 psig.

2.11 Slowly Open Valve #2 to begin releasing gas.

2.12 Close the door on the enclosure. The cylinder will take several hours to empty. The pressure in the cylinder can be monitored on the Gas Collection System Control Chassis in D032.

2.13 When the Cylinder is empty, HP Tech should survey the enclosure.

2.14 Close the valve on the Collection Cylinder. Remove the assembly and cap the port.

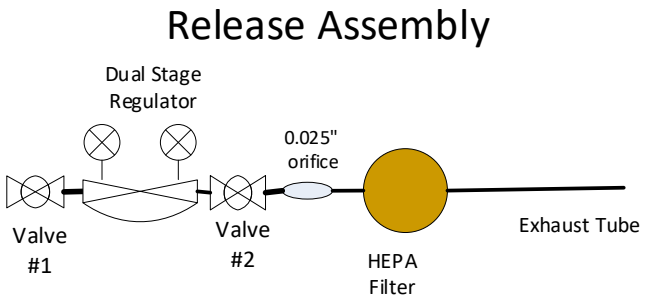
2.15 Re Open the Collection Cylinder valve.

2.16 Re-start the Gas Collection System by pulling out the Red Button.

2.17 Open GC-SV-1.

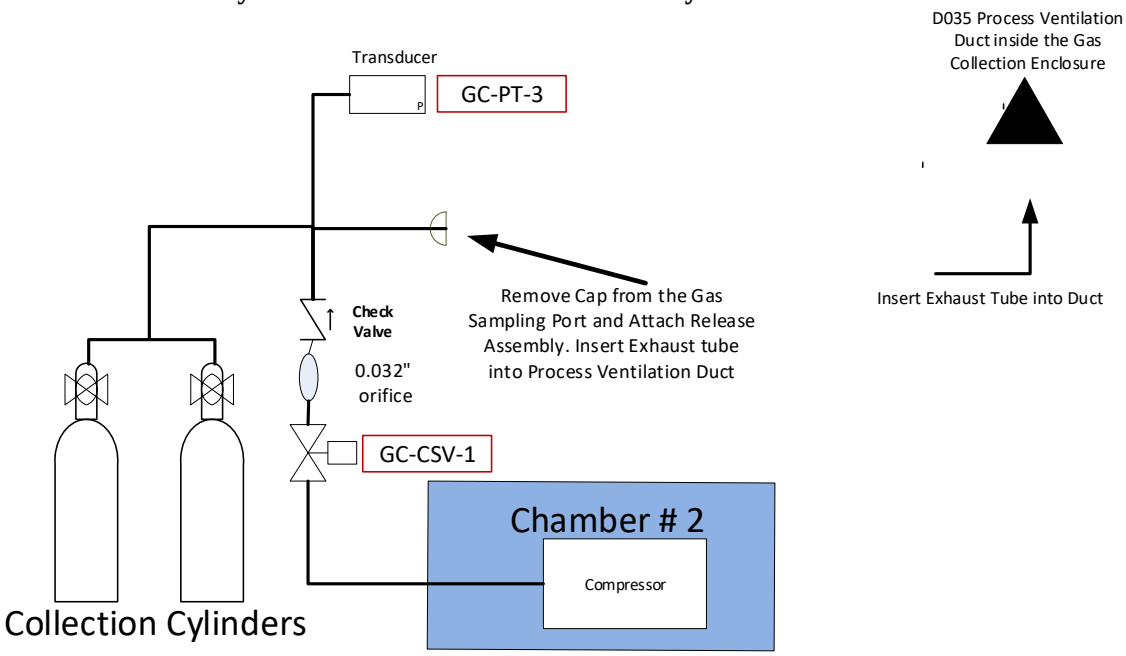
2.18 On the Gas Distribution Hub Manifold Open valve V-6, V-7, V-9 and V-11.

Figure 1. Release Assembly



Attach 0.025" Orifice, Hepa-Filter and Exhaust Tube to a Dual Stage Regulator to form the Release Assembly

Figure 2. Release Assembly Attachment onto Gas Collection System



APPENDIX 17

LEAF-PROC-024, Rev. 3: ^{99}Mo PHASE II Production Tests – LabVIEW ^{99}Mo Remote Recovery Data Acquisition and Control System: Complete Operations Abridged Version

^{99}Mo PHASE II Production Tests – LabVIEW ^{99}Mo Remote Recovery Data Acquisition and Control System: Complete Operations Abridged Version

Low Energy Accelerator Facility, LEAF-PROC-024, Rev. 3

Approved:  Date: 02.25.2020

Sergey Chemerisov, Manager, IVEM/LEAF

Effective Date: 02.26.2020

1 Purpose

Provide complete instructions for glovebox operations for the AMORE experiment in the LEAF facility, including the LabVIEW ^{99}Mo Remote Recovery Data Acquisition and Control System.

2 Scope

This procedure applies to the following Argonne activities and entities.

LMS core processes:	Asset Management
Organizations:	Experimental Operations and Facilities (EOF) Division
Buildings:	211
Specific locations:	LINAC
Other applicability factors:	None
Exclusions:	None
USI applicability:	Yes

3 Work Process

3.1 Introduction

This document combines previous releases of six different work instructions, as listed in Table 1, below. The work instructions are to be used in the order given here:

TABLE 1 Work Instructions in this Procedure

Section	Steps	Work Instruction Name	Previous Release File Name
3.2.1	1-7	Production Feeds Analysis and Process Conditions Summary Sheet	194-v3_AMORE_Glovebox_ FeedsAnalysisProcessCondsSummary Sheet_Rev3.docx
3.2.1	8-24	Installation, Operation, and Removal of Verification Tank	199-v3_AMORE_Glovebox_ InstallOpsRemovalVerifTank_Rev3.docx
3.2.3	25-33	Installation of Feed Bottles, Effluent Cart, Recovery Column, and Priming of Non-Rad Feed Lines	197-v3_AMORE_Glovebox_ InstFeedsEffluCartColumnPrimeLines_ Rev3.docx)
3.2.4	34-69	Solution Irradiation and 99Mo Recovery	195-v4_AMORE_Glovebox_ SolnIrradMo99RecoveryOps_Rev4.docx
3.2.5	70-78	99Mo Recovery Sample Retrieval	219-v4_AMORE_Glovebox_ Mo99RecoverySampRetriev_Rev4.docx
3.2.6	79-92	Washout of 99Mo Recovery System and Sample Retrieval Subsystems	196-v3_AMORE_Glovebox_ Mo99RecoverySystemWashout_ Rev3.docx

The steps in each section have been formatted to be followed in the sequential style presented. The sequential numbering of the steps is used to transcribe information between steps.

When printing this document for use in operations, the user must:

- **PRINT PAGES IN COLOR**
- Hole punch ALL pages and secure in a binder (e.g. 3-ring binder or ¾ in. binding comb) before each experiment.

3.2 Step-by-Step Procedure

The parameters found in step 7 MUST be determined and verified to be within ASE control limits prior to irradiation. All system interface steps (17, 38, 39, 66, 82, and 85) MUST be completed as written. All remaining steps in this procedure are considered work instructions and may be modified to complete the processing steps as long as modifications are documented. This procedure is to be performed by Workers appointed to the AMORE ^{99}Mo Recovery Operations Team.

IMPORTANT NOTES:

- VCR connections with SS gaskets require a 1/8 turn past finger tight
- ALWAYS use a new VCR gasket when making a connection
- When installing/uninstalling/moving shielded containers (verification tank, effluent cart, etc.) ALWAYS wear steel toed shoes

3.2.1 Production Feeds Analysis and Process Conditions Summary Sheet (PFA-PCS Sheet)

LabVIEW – Lab Notebook No.: _____ Pages: _____

Step	Action
1.	**Samples To Be Collected** 1.1. Number of Target Solution Mixing samples pre-irradiation: _____ 1.2. Number of Target Solution Mixing samples during irradiation: _____ 1.3. Number of Pre-Load Acid Wash samples: _____ OR ☐ Skipped 1.4. Number of Column Loading samples: _____ 1.5. Number of Post-Load Acid Wash samples: _____ 1.6. Number of Post-Load H2O Wash samples: _____ 1.7. Number of Post-Load NaOH Wash samples: _____ OR ☐ Skipped 1.8. Number of Column Stripping samples: _____ 1.9. Number of Post-Strip H2O Wash samples: _____
2.	**Select a Column Stripping Path** 2.1. ☐ To transfer cask **OR** 2.2. ☐ To Mo99 processing cell (D024 Hot Cell)
3.	**Fresh Acid – Used for the following processing steps** 3.1. Leak checking the column

Step	Action
	A. Volume to be used: _____ mL i. Default = 5 min. x 167 mL/min = 835 mL ii. Flow rate: _____ mL/min 3.2. Pre-Pre-Load Acid Wash (Acid Pre-heater activation) A. Volume to be used: _____ mL i. Default = 5 min. x 84 mL/min = 420 mL B. Flow rate: _____ mL/min 3.3. Pre-Load Acid Wash A. Volume to be used: _____ mL i. Default = 7.5 min. x 167 mL/min = 1252 mL B. Flow rate: _____ mL/min 3.4. Post-Load Acid Wash A. Volume to be used: _____ mL i. Default = 7.5 min. x 167 mL/min = 1252 mL B. Flow rate: _____ mL/min 3.5. Final Acid Wash A. Volume to be used: _____ mL i. Default = 7.5 min. x 84 mL/min = 630 mL B. Flow rate: _____ mL/min 3.6. Loop rinsing for spike test A. Volume to be used: _____ mL i. Default = 7.5 min. x 84 mL/min = 630 mL B. Flow rate: _____ mL/min 3.7. Acid system rinse out A. Volume to be used: _____ mL i. Default = 7.5 min. x 84 mL/min = 630 mL B. Flow rate: _____ mL/min 3.8. Priming lines A. Volume to be used: _____mL B. Flow rate: varies 3.9. Total Volume to be used: A. Sum of **[step 3.1.A]** + **[step 3.2.A] + [step 3.3.A]** + **[step 3.4.A**] + **[step 3.5.A] + [step 3.6.A] + [step 3.7.A] + [step 3.8.A]** = _____ mL

Step	Action
	3.10. Properties A. ☐ Verify cap and bottle properly labeled B. Lab Notebook No.: _____ Pages: _____ C. Preparation Date: _____ D. Concentration: _____ M (mol/L) *(target range 0.0831- 0.0919 M)* E. pH value: _____ *(target range pH 0.95-1.05)* F. Density: _____ g/mL or g/cc *(target range 0.95-1.05 g/mL)* G. Mass of empty bottle+cap: _____ g H. Mass of bottle+cap+solution: _____ g I. Total volume: _____ mL J. ☐ Verify total volume EQUALS **or** EXCEEDS **line 3.9.A.** K. Enter values in Exhibit B cheat sheet, if desired
4.	**Demineralized Water – Used for the following processing steps** 4.1. Post-Load H_2O Wash A. Volume to be used: _____ mL i. Default = 7.5 min. x 167 mL/min = 1252 mL B. Flow rate: _____ mL/min 4.2. Post-Strip H_2O Wash A. When STRIP to CELL flowpath is chosen a portion of the Post-Strip H_2O Wash is used to flush the transfer line before switching to the waste bottle (Strip path THEN Waste bottle path) B. When STRIP to CASK flowpath is chosen all of the Post-Strip H_2O Wash is sent to the waste bottle (Strip path + Waste bottle path) C. Default = 7.5 min. x 84 mL/min = 630 mL D. Enter Post-Strip H_2O Wash volume to Strip product path i. Volume to be used: _____ mL a. Default = 12.0 min. x 84 mL/min = 1008 mL ii. Flow rate: _____ mL/min E. Enter Post-Strip H_2O Wash volume to waste bottle path i. Volume to be used: _____ mL a. Default = 4.5 min. x 84 mL/min = 378 mL ii. Flow rate: _____ mL/min 4.3. Base System Final Rinse

Step	Action
	A. Volume to be used: _____ mL i. Default = 7.5 min. x 84 mL/min = 630 mL 4.4. Loop rinsing for spike test A. Volume to be used: _____ mL i. Default = 7.5 min. x 84 mL/min = 630 mL B. Flow rate: _____ mL/min 4.5. Base system rinse out A. Volume to be used: _____ i. Default = 7.5 min. x 84 mL/min = 630 mL B. Flow rate: _____ mL/min 4.6. Priming lines A. Volume to be used: _____ B. Flow rate varies 4.7. Total Volume to be used A. Sum of **[step 4.1.A]** + **[step 4.2.D.i]** + **[step 4.2.E.i]** + **[step 4.3.A]** + **[step 4.4.A]** + **[step 4.5.A]** + **[step 4.6.A]** = _____ mL 4.8. Density: _____ g/mL or g/cc *(target range 0.95-1.05 g/mL)* 4.9. Properties A. Mass of empty bottle+cap: _____ g B. Mass of bottle+cap+solution: _____ g C. Total volume: _____ mL D. ☐ Verify total volume EQUALS **or** EXCEEDS line **4.7.A.** E. Enter values in Exhibit B cheat sheet, if desired
5.	**NaOH Wash – Used for the following processing steps** 5.1. Base Pre-heater activation A. Volume to be used: _____ mL *(default 420 mL)* B. Flow rate: _____ mL/min 5.2. Post-Load NaOH Wash A. Volume to be used: _____ mL **OR** ☐ Skipped i. Default = 3.0 min. x 84 mL/min = 252 mL B. Flow rate: _____ mL/min 5.3. Priming Lines A. Volume to be used: _____ mL **OR** ☐ Skipped

Step	Action
	B. Flow rate varies 5.4. Sum **[step 5.1.A]** + **[step 5.2.A]** + **[step 5.3.A]** = _____ mL 5.5. Properties A. ☐ Verify cap and bottle properly labeled B. Lab Notebook No.: _____ Pages: _____ C. Preparation Date: _____ D. Concentration: _____ M (mol/L) *(target range 0.95-1.05 M)* E. Density: _____ g/mL or g/cc *(target range 0.95-1.05 g/mL)* F. Mass of empty bottle+cap: _____ g G. Mass of bottle+cap+solution: _____ g H. Total volume: _____ mL I. ☐ Verify total volume EQUALS **or** EXCEEDS line **5.4.** J. Enter values in Exhibit B cheat sheet, if desired
6.	**NaOH Strip – Used for the following processing steps** 6.1. Column Strip A. Volume to be used: _____ mL i. Default = 30.0 min. x 84 mL/min = 2520 mL B. Flow rate: _____ mL/min 6.2. Priming lines A. Volume to be used: _____ mL B. Flow rate varies 6.3. Sum **[step 6.1.A]** + **[step 6.2.A]** = _____ mL 6.4. Properties A. ☐ Verify cap and bottle properly labeled B. Lab Notebook No.: _____ Pages: _____ C. Preparation Date: _____ D. Concentration: _____ M (mol/L) *(target range 0.95-1.05 M)* E. Density: _____ g/mL or g/cc *(target range 0.95-1.05 g/mL)* F. Mass of empty bottle+cap: _____ g G. Mass of bottle+cap+solution: _____ g H. Total volume: _____ mL I. ☐ Verify total volume EQUALS **or** EXCEEDS line **6.3**

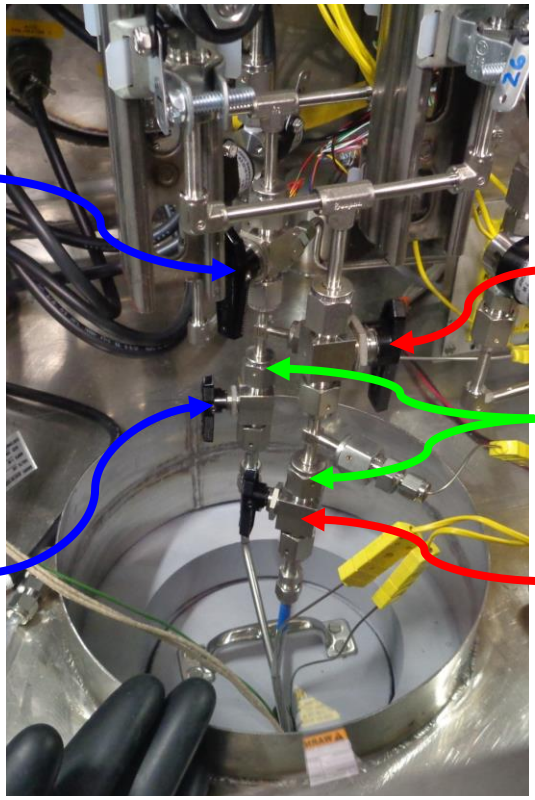
Step	Action
	J. Enter values in Exhibit B cheat sheet, if desired
7.	**<u>Uranyl Sulfate Target Solution – Used for the following processing steps</u>** 7.1. Target Solution Mixing during irradiation 7.2. Column loading A. Volume to be used: _____ mL i. Default = 108 min. x 167 mL/min = 18036 mL B. Flow rate: _____ mL/min 7.3. Properties A. Target solution CURIE No.: _____ B. SPM No.: _____ C. Post-irradiation analysis i. Date of last irradiation: _____ ii. Lab Notebook No.: _____ Pages: _____ iii. Analysis Date: _____ iv. Analysis ID: _____ v. Uranium concentration: _____ g U/L *(target range ≤ 145 g U/L)* a. Must be measured by ACL using High Precision ICP-OES b. **Concentration limit 145 g U/L set in [ASE 2.9.1.1]** vi. Acid concentration: _____ M (mol/L) *(target range 0.0831- 0.0919 M)* vii. pH value: _____ *(target range pH 0.95-1.05)* viii. Density: _____ g/mL or g/cc *(target range 1.14-1.26 g/mL)* ix. Mass in verification tank: _____ g (from step 19.26 on p. 24) x. Calculate volume in verification tank: _____ mL *(target range ≤ 18 L)* a. **[Line 7.3.C.ix]** / **[Line 7.3.C.viii]** = volume in mL b. **Volume limit of 20 L set in [ASE 2.9.1.1]** D. Post-adjustment analysis i. Date of last irradiation: _____ ii. Lab Notebook No.: _____ Pages: _____ iii. Analysis Date: _____ iv. Analysis ID: _____ v. Uranium concentration: _____ g U/L *(target range ≤ 145 g U/L)* a. Must be measured by ACL using High Precision ICP-OES b. **Concentration limit 145 g U/L set in [ASE 2.9.1.1]**

Step	Action
	vi. Acid concentration: _____ M (mol/L) *(target range 0.0831- 0.0919 M)* vii. pH value: _____ *(target range pH 0.95-1.05)* viii. Density: _____ g/mL or g/cc *(target range 1.14-1.26 g/mL)* ix. Mass in verification tank: _____ g (from step 21.2 on p. 26) x. Calculate volume in verification tank: _____ mL *(target range ≤ 18 L)* a. **[Line 7.3.D.ix]** / **[Line 7.3.D.viii]** = volume in mL b. **Volume limit of 20 L set in [ASE 2.9.1.1]** E. Enter values in Exhibit B cheat sheet, if desired

3.2.2 Installation, Operation, and Removal of Verification Tank

Step	Action
8.	**<u>Remove Spent Packed Column from Glovebox Cabinet #1 (Left Cabinet)</u>** 8.1. Verify most current RWP signed by workers A. RWP #: _____ 8.2. Open cabinet #1 door (left most cabinet) 8.3. Have HP Tech perform pre-job survey smears A. Suggested smear locations: interface between lead shielded pig and glove box, exterior of lead shielded pig, LEFT, RIGHT, and BOTTOM sides of Cabinet #1 i. DO NOT remove support table under lead shielded pig to perform smears ii. Smear around support table under lead shielded pig iii. ONLY smear legs of support table iv. DO NOT smear top surface of support table – POTENTIAL PINCH/CRUSH HAZARD v. DO NOT smear back wall of Cabinet #1 – this area can be smeared once the lead shielded pig has been removed 8.4. Close cabinet #1 door and hold for pre-job smears. If HP Tech determines it is required, assist in reducing contamination levels prior to proceeding to step 8.5 8.5. Stage the transport collar for the column/pig assembly, ratcheting transport strap, 4 gallon trash bag and tape, 4-section ramp, and two VCR caps with two NEW VCR gaskets. Stage crescent wrench and ¾ in open-ended wrench in glovebox A. Transport collar has two large eye bolts attached B. DO NOT INSTALL THE RAMP YET

8.6. **Inside the glovebox:**



A. Stage column jumper (see Exhibit B for components)

B. Verify that the VCR 2-way system column connection valves (V-2002 & V-2003) and the VCR 2-way ball valves attached to the column (V-2004 & V-2005) are closed

C. Prepare paper towels or rags to catch residual liquid trapped between system valve/column valve interfaces

D. Disconnect column valves at the VCR fittings shown by green arrows in figure (above)

i. The VCR tees should remain attached to the system valves V-2002 & V-2003

E. Verify connections are FULLY loosened and FREE

F. Use a CLEAN rag or paper towel (may be wetted with Radiac) to wipe down column valves V-2004 & V-2005, the lead shielded pig cover and handle, and exposed surfaces of the connection collar

G. Disconnect column heat tape from receptacle and tuck power cable in the interior ring of the connection collar

H. Disconnect cables from Column CONTROL & OVER-TEMPERATURE thermocouples and tuck cables out of the way, off to the side, away from the column port hole

I. Disconnect leak sensor cable

i. Tuck the end of the leak sensor cable CONNECTED TO THE LEAK SENSOR into the interior ring of the connection collar

Step	Action
	ii. Tuck the end of the leak sensor cable CONNECTED TO THE GLOVEBOX LEAK SENSOR PANEL out of the way, off to the side, away from the column port hole
	8.7. **Inside Cabinet #1 / Outside the Glovebox**
	A. Open cabinet #1 door
	B. Install 4-section ramp from in front of Cabinet #1
	C. DO NOT remove support table under lead shielded pig
	D. Roll pig transport cart into place – near support table under lead shielded pig – and raise platform of pig transport cart EQUAL to height of support table under lead shielded pig
	E. REMOVE support table under lead shielded pig THEN IMMEDIATELY roll pig transport cart under lead shielded pig and center the cart under the pig
	F. Have HP Tech smear TOP surface AND BOTTOM feet of support table once removed and set the table to the side
	G. Ratchet pig transport cart platform until surface is snug against bottom of lead shielded column pig
	H. Chock wheels of pig transport cart to prevent the cart from moving as lead shielded column pig is detached from glovebox mounting ring
	I. **Inside the glovebox** - Verify the following
	i. ☐ Column connection valves are closed
	ii. ☐ Column is freed from system VCR connections
	iii. ☐ Column thermocouples are disconnected
	iv. ☐ Column heat tape plug is disconnected
	v. ☐ Column leak sensor is disconnected
	J. Disengage one toggle clamp from appropriate toggle clamp hook of pig locking ring to lower the column/pig assembly slightly
	K. **Inside the glovebox** - Verify VCR connections are still free
	L. Disengage remaining toggle clamp from appropriate toggle clamp hook of pig locking ring to fully lower the column/pig assembly onto the cart
	M. **Inside the glovebox** – Verify VCR connections are still free
	N. Slowly lower platform of pig transport cart, ensuring cables are free and come down with the column pig
	O. Fully lower platform of pig transport cart
	P. IMMEDIATELY attach VCR caps with NEW gaskets to column valves V-2004 & V-2005
	Q. Have HP Tech smear the column valves (V-2004 & V-2005), column heater power cable, column thermocouple ends, cable attached to column leak sensor
	a. DO NOT pull the leak sensor out

Step	Action
	R. Place 4-gallon plastic trash bag over end of column, tucking cable ends into bag and taping the end of the bag around column tubing i. DO NOT tape bag to column pig  S. Have HP Tech smear the top and exterior surfaces of the column pig and the transport cart wheels T. Hold for HP Tech survey results. If HP Tech determines it is required, assist in reducing contamination levels prior to proceeding to next step (**8.7.U)** i. THE COLUMN PIG REMAINS IN CABINET #1 U. Un-chock column pig transport cart wheels and roll the column pig out of the cabinet i. DO NOT roll the column pig transport cart down the ramp V. Attach the transfer collar and tie-down strap W. Attach transport collar to column pig i. Feed taped trash bag covering column valves through center hole of transport collar ii. Secure collar to pig using the two toggle clamps X. Attach the tie-down strap i. Place one hook of strap into one of the eye-bolts on the transfer collar and run strap underneath the transport cart ii. Attach remaining strap hook to remaining free eye-bolt iii. Ratchet lever MUST face outward to ensure column pig is strapped to transport cart iv. Ratchet tie-down strap until strap is tight a. DO NOT over-tighten the strap v. Stow excess strap around column pig and ensure it does not come loose while wheeling cart to the next location Y. Wheel cart down ramp

Step	Action
	i. It may be necessary to remove 4-section ramp to wheel column pig transport cart out of Cell 1 (D035) Z. Close Cabinet #1 door AA. **Inside Glovebox** i. Cover column port with column port cover ii. Attach column jumper to the column connection fittings iii. Verify V2002 & V-2003 remain closed BB. Have HP Tech survey 4-section ramp before stowing ramp CC. Have HP Tech survey work area i. Excludes the Cabinet #1 interior
9.	**<u>All Mass Measurements are to be Made With the Four Manual 2-Way Ball Valves Closed</u>** 9.1. Pickup line valve V-2034 9.2. Return line valve V-2036 9.3. Sample-pickup line valve V-2037 9.4. Vent line valve V-2035
10.	**<u>There is One Shielded Verification Tank Cart</u>** 10.1. Identified by the serial number of the balance inside A. Ohaus Defender 7000 Model D50QLUS; Serial No. B552895095 10.2. **SECURE BALANCE CABLE AND LIQUID LEAK SENSOR CABLE TO CART BEFORE MOVING CART** A. Prevent cable ends from being crushed by cart casters 10.3. **ONLY MOVE VERIFICATION TANK CART WITH TRAY IN TRANSPORT POSITION – HANDLE IN THE UP POSITION** A. Failure to have the tray above the balance could damage the balance B. Black line on hex head is up  

Step	Action
	10.4. **IF NECESSARY ONLY REMOVE THE RIGHT SIDE OF THE SHIELDED VERIFICATION TANK CART LID** A. Right half of lid weighs over 120 lbs. 10.5. The tank has four ¼ in liquid lines A. Pickup line attached at bottom of tank through 1-1/2 in. tri-clamp fitting (see Exhibit B for list of parts) i. **BALL VALVE HANDLE POINTS AWAY FROM TANK (DIRECTION OF FLOW FROM TANK)** B. Return line attached to tank cover (see Exhibit B for list of parts) i. **BALL VALVE HANDLE POINTS TOWARD TANK (DIRECTION OF FLOW INTO TANK)** C. ⅛ in. sample-pickup & vent lines (see Exhibit B for list of parts) i. **BALL VALVE HANDLE POINTS AWAY FROM TANK (DIRECTION OF FLOW FROM TANK)** D. Sample-pickup line attached to tank cover i. **BALL VALVE HANDLE POINTS TOWARD TANK (DIRECTION OF FLOW TO TANK)**
11.	**Verify Verification Tank Balance is Calibrated** (calibration sticker is affixed to Feed Balance Indicator located on the left side of the LabVIEW control rack) 11.1. Record feed balance calibration date: _____ A. Next calibration due: _____ B. If balance is out of calibration have balance calibrated C. **DO NOT PROCEED IF FEED BALANCE IS NOT CALIBRATED**

12. **Install Verification Tank Cart**

12.1. Open cabinet #1 (left side) door to full open

12.2. Position the 4-section ramp to roll verification tank cart into cabinet #1

12.3. ☐ Verify balance lever in transport positon

12.4. Roll verification tank cart to edge of cabinet #1

12.5. ☐ Attach balance cable

A. Connectors operate smoothly when mated to one another correctly – **DO NOT FORCE CONNECTORS TOGETHER**

12.6. ☐ Verify balance is read by verification balance indicator

A. May need to turn appropriate balance indicator on

B. After startup if balance is read by indicator the indicator will show 0

i. If no reading double check connection

ii. **DO NOT PROCEED UNTIL BALANCE IS READ BY INDICATOR**

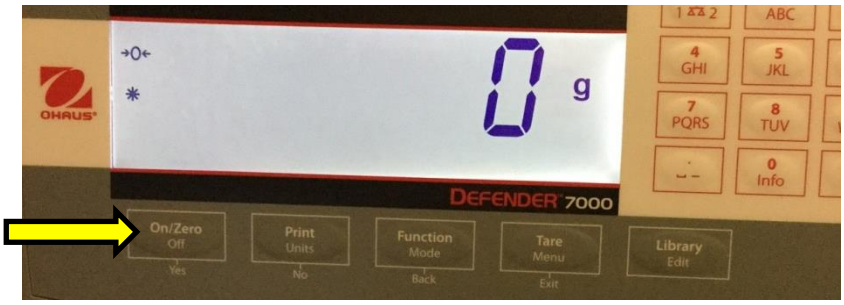
12.7. ☐ Attach liquid leak sensor cable

12.8. Center the four plastic lines on top of verification tank cart lid

12.9. Push the fully connected verification cart into position in cabinet #1

12.10. Remove the handle from the verification tank cart and store in D032 until verification tank cart is removed

12.11. Zero verification balance indicator by pressing the ON/ZERO soft key



12.12. ☐ Turn balance transport handle to weigh (down, with red dot on hex head facing up)

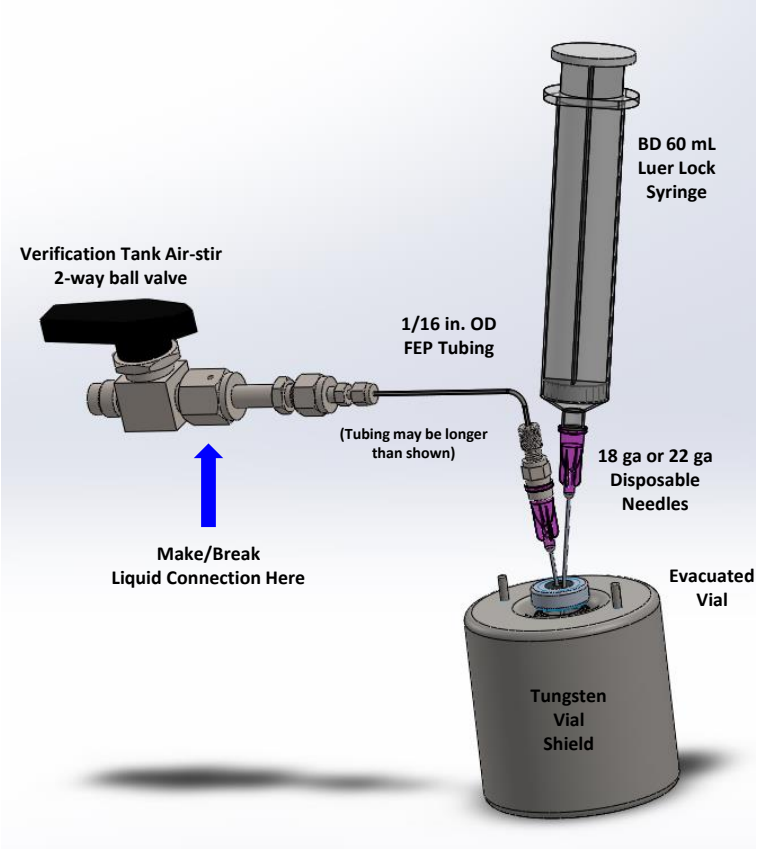
Step	Action
	 
	12.13. ☐ Verify verification balance is read by balance indicator A. Should read a positive, non-zero value 12.14. Remove the 4-section ramp and slowly close cabinet #1 A. **DO NOT STORE 4-SECTION RAMPS IN CELL 1 (D035)**
13.	**<u>Inside the Glovebox – MAKE/BREAK LIQUID CONNECTIONS AT VCR FITTINGS ONLY</u>** 13.1. Identify the feed connection jumper and effluent connection jumper (see Exhibit B items 4-5) 13.2. Extend verification vent line jumper from V-0160 to column/tank port hole 13.3. Attach verification vent line V-2035 to V-0160 vent line jumper (sends verification tank vent to gas collection system) 13.4. Attach verification sample-pickup line to thief vial assembly
14.	**<u>Checking Column Stripping Transfer Cask Installation</u>** 14.1. Outside the white glovebox in Cell 1 (D035) 14.2. ☐ Verify column stripping transfer cask is attached to white glovebox A. Inside the white glovebox in Cell 1 (D035) 14.3. ☐ Verify both 2-way valves for liquid service (on the ¼ in. line) are open A. Handles parallel to the long axis of valve body 14.4. ☐ Verify both 2-way valves for vent service (on the ⅛ in. line) are open A. Handles parallel to the long axis of valve body
15.	**<u>At the Rack – Verify Sample Retrieval Valves Powered Off</u>** 15.1. Verify Sample Retrieve Valve Power to OFF (switch is down)

Step	Action
	A. If the switch is not OFF then gently pull out the switch while moving the handle down to actuate 
16.	**<u>Begin Operation of Mo-99 Remote Recovery Data Acquisition and Control Software</u>** 16.1. Version used: _____ A. Current version: **M3_SHINE_PhaseII_ver06J.vi** (as of 3/27/2018) 16.2. ☐ External computer speakers powered ON A. ☐ Verify speakers work by running "beep10.bat" file from desktop  B. If sound does not come out of speakers DO NOT proceed until speakers are operational 16.3. Start the program using the following parameters of input: A. Manual mode B. Process operation C. Verification tank D. RECORD VERIFICATION BALANCE DATA? i. YES E. Set verification balance type and COM port i. Balance and COM port should match the verification tank and indicator connection

Step	Action
	F. Fresh Acid density → as default value → press OK G. Base Wash density → as default value → press OK H. Base Strip density → as default value → press OK I. Target solution volume i. Enter last target solution volume J. Target solution concentration i. Enter last target solution concentration K. Target solution density i. Enter last target solution density L. Column effluent path i. To Transfer Cask M. Pre-Load Acid Wash processing volume → as default value → press OK N. Column Loading processing volume → as default value → press OK O. Post-Load Acid Wash processing volume → as default value → press OK P. Post-Load Water Wash processing volume → as default value → press OK Q. Use the Post-Load NaOH Wash step? → NO R. Column stripping processing volume → as default value → press OK S. Post-Strip Water Wash To Strip product processing volume → as default value → press OK T. Post-Strip Water Wash To Waste processing volume → as default value → press OK U. Final Base System Water Wash processing volume → as default value → press OK V. Final Acid System Acid Wash processing volume → as default value → press OK W. Record LINAC temperatures? → YES X. Filename prefix: _____ (see **[File Paths]**.tab → File Prefix) Y. ☐ ACID Pump controller powered ON (Rocker switch under front/left of ACID Pump V300 controller) Z. ☐ ACID Pump to **STOP** (display alternates between OFF and current setting) AA. ☐ Verify verification tank balance reading at **[Sensors]**.tab i. Compare LabVIEW value to value on verification tank balance indicator
17.	**Check Manual Dump Tank Valve OPEN** ### SYSTEMS INTERFACE STEP ### 17.1. Contact a Gas Analysis/Collection team member

Step	Action
	17.2. **At the Dump Tank (211-D035)** A. ☐ Verify manual dump tank valve is OPEN B. Recovery team member // Name: _____PRINT Initials: _____ Date: _____ Time: _____ C. Gas Analysis/Collection team member // Name: _____PRINT Initials: _____ Date: _____ Time: _____ 17.3. Recovery personnel continue to **step 0.**
18.	**<u>Pump Target Solution from Dump Tank to Verification Tank</u>** **– YOU ARE OPERATING THE SYSTEM IN MANUAL MODE –** 18.1. ☐ Verify feed pick-up valve V-3003 to verification tank side arm 18.2. ☐ Verify V-3001 directed to feed pick-up valve V-3003 18.3. ☐ Verify verification tank pickup line valve V-2034 open 18.4. ☐ Verify effluent valve V-3002 to verification tank 18.5. ☐ Verify verification tank return line valve V-2036 open 18.6. ☐ Verify verification tank vent line valve V-2035 open 18.7. ☐ Verify verification tank sample-pickup line valve V-2037 closed 18.8. ☐ Open V-0004 (from Dump tank) 18.9. ☐ Open V-0011 (to Verification tank) 18.10. ☐ Open V-0160 (to Verification tank vent) 18.11. ☐ Enter flow rate **<u>100</u>** mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab 18.12. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab 18.13. ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate A. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller 18.14. ☐ ACID Pump to **<u>RUN</u>** 18.15. ☐ Start 5 minute timer 18.16. ☐ Verify flow path is leak free at 5 minute timer end A. If leaks are detected stop pump and fix leaks prior to proceeding 18.17. Enter flow rate **<u>250</u>** mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab 18.18. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab A. **Ensure no cavitation occurs if motor is set above 50% motor power**

Step	Action
	18.19. ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate A. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller 18.20. Monitor FEP tubing from V-0011 for target solution 18.21. ☐ Once FEP tubing from V-0011 is empty, set ACID Pump to **STOP** 18.22. ☐ Close V-0004 (from Dump tank) 18.23. ☐ Close V-0011 (to Verification tank) 18.24. ☐ Close V-0160 (to Verification tank vent) 18.25. ☐ Close verification tank pickup line valve V-2034 18.26. ☐ Close verification tank return line valve V-2036 closed 18.27. ☐ Verify verification tank sample-pickup line valve V-2037 closed 18.28. Record: verification tank balance indicator: _____ grams 18.29. Record verification tank balance value at **[Sensors]**.tab: _____ grams
19.	**Retrieve Analytical Sample** 19.1. ☐ Verify V-0004 closed (from Dump tank) 19.2. ☐ Verify V-0011 closed (to Verification tank) 19.3. ☐ Open V-0160 (to Verification tank vent) 19.4. ☐ Verify verification tank pickup line valve V-2034 closed 19.5. ☐ Verify verification tank return line valve V-2036 closed 19.6. Attach thief vial assembly to verification tank sample-pickup valve V-2037 A. Use disposable needles B. **Do not use all metal needles** – hubs are made of nickel plated brass C. **Step 24, Alternate Sample Retrieval Configuration,** page **29** may be used

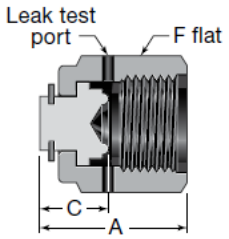
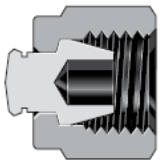
Step	Action
	 19.7. ☐ Open verification tank sample-pickup line valve V-2037 19.8. Retract plunger of a 60 mL Luer lock syringe 10-20 mL to draw sample into thief vial A. Maintain syringe needle in head space of vial (out of liquid) 19.9. Once sample collected THEN CLOSE verification tank sample-pickup line valve V-2037 19.10. Retract plunger of 60 mL Luer lock syringe further to empty liquid in 1/16 in. line 19.11. ☐ Ready clean evacuated vial 19.12. Remove liquid transfer needle from sample vial and insert liquid transfer needle into clean evacuated vial 19.13. Remove syringe needle from sample vial 19.14. Fully retract plunger of 60 mL Luer lock syringe 19.15. Insert syringe needle into clean evacuated vial 19.16. Open verification tank sample-pickup line valve V-2037 19.17. Slowly push plunger of syringe to bottom of syringe body A. Using air in syringe to empty sample-pickup line back into verification tank 19.18. Close verification tank sample-pickup line valve V-2037 19.19. Remove needles and dispose of in sharps container

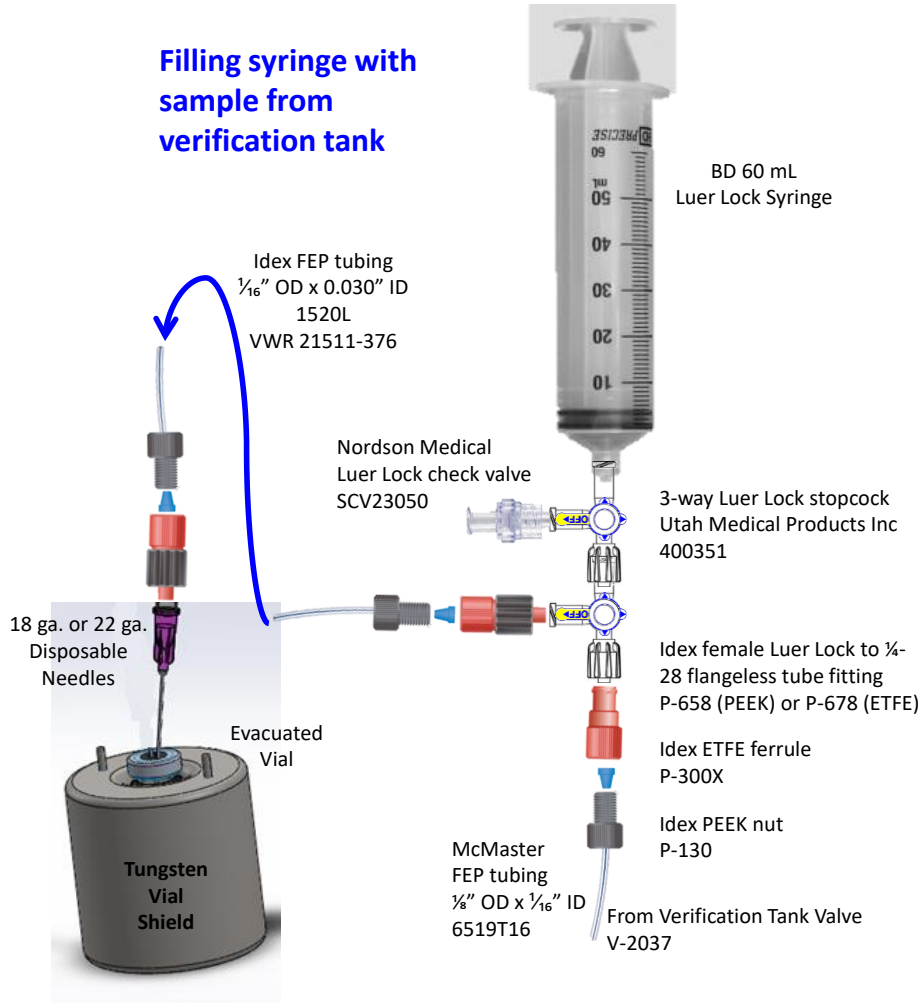
Step	Action
	19.20. Disconnect thief vial assembly from verification tank sample-pickup line valve V-2037 19.21. ☐ Verify V-0160 closed (to Verification tank vent) 19.22. Close verification tank vent line valve V-2035 19.23. Record verification tank balance indicator: _____ grams 19.24. Record verification tank balance value at **[Sensors]**.tab: _____ grams 19.25. Submit sample for analysis (if from step **21.1**, continue to step **21.2**, p **26**) 19.26. Enter analysis values at **step 7.3.C** on page **8** 19.27. Person In Charge to determine if feed adjustment is required (choose ONE) A. ☐ If adjustment is required go to **step 20** **OR** B. ☐ If no adjustment is required go to **step 22**
20.	**Adding Make-up Solution or Mo Spike to Verification Tank** 20.1. Record verification tank balance indicator: _____ grams 20.2. Record verification tank balance value at **[Sensors]**.tab: _____ grams 20.3. If only a small volume needs to be added, a syringe may be used to inject the volume through valve V-2037 using the **Alternative Sample Retrieval Configuration** (**Step 24**, p. **29**) 20.4. Insert tubing from feed valve V-3003 into make-up bottle 20.5. Turn feed valve V-3003 to bottle side-arm port 20.6. ☐ Verify V-3001 directed to feed pick-up valve V-3003 20.7. ☐ Verify verification tank pickup line valve V-2034 open 20.8. ☐ Verify verification tank return line valve V-2036 open 20.9. ☐ Verify verification tank vent line valve V-2035 open 20.10. ☐ Verify verification tank sample-pickup line valve V-2034 closed 20.11. ☐ Open V-0003 (from External vessel) 20.12. ☐ Open V-0011 (to Verification tank) 20.13. ☐ Open V-0160 (to Verification tank vent) 20.14. ☐ Enter flow rate **100** mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab 20.15. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab 20.16. ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate A. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller

Step	Action
	20.17. ☐ ACID Pump to **RUN** 20.18. Monitor feed make-up bottle until contents have been pumped into verification tank 20.19. When feed make-up bottle contents are in system ACID pump to **STOP** 20.20. Turn feed valve V-3003 to verification tank side-arm port 20.21. ☐ ACID Pump to **RUN** A. Using pump to mix contents of verification tank 20.22. ☐ Enter flow rate **300** mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab 20.23. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab A. **Ensure no cavitation occurs if motor is set above 50% motor power** 20.24. ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate 20.25. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller 20.26. ***NOTE – at 300 mL/min it will take 60 minutes to completely circulate 18000 mL*** 20.27. Record start time of mixing: _____ *(LabVIEW time)* 20.28. Record mixing duration: _____ minutes 20.29. Record end time of mixing: _____ *(LabVIEW time)* 20.30. After sufficient mixing time ACID pump to **STOP** 20.31. Insert tubing from feed valve V-3003 bottle side-arm port into empty bottle 20.32. Turn feed valve V-3003 to bottle side-arm port 20.33. ☐ ACID Pump to **RUN** A. Drawing glovebox atmosphere through 3-way ball valve bottle side-arm port B. Pushing solution from tubing into verification tank 20.34. Start 3 minute timer 20.35. At 3 minute timer end ACID pump to **STOP** 20.36. ☐ Close V-0003 (from External vessel) 20.37. ☐ Close V-0011 (to Verification tank) 20.38. ☐ Close V-0160 open (to Verification tank vent) 20.39. ☐ Close verification tank pickup line valve V-2034 20.40. ☐ Close verification tank return line valve V-2036 20.41. ☐ Close verification tank vent line valve V-2035 20.42. ☐ Verify verification tank sample-pickup line valve V-2034 closed 20.43. Record verification tank balance indicator: _____ grams

Step	Action
	20.44. Record verification tank balance value at **[Sensors]**.tab: _____ grams 20.45. Go to **step 21, <u>Retrieve Analytical Sample After Addition of Make-up Solution</u>.**
21.	**<u>Retrieve Analytical Sample After Addition of Make-up Solution or Mo Spike</u>** 21.1. Follow step **19** for retrieval of analytical sample (page **22**), proceeding to step **21.2** after step **19.25** 21.2. Enter analysis values at **step 7.3.D** on **page 8** 21.3. Go to **step 22, <u>Return Target Solution to Target Vessel</u>**
22.	**<u>Return Target Solution to Target Vessel</u>** 22.1. Record verification tank balance indicator: _____ grams 22.2. Record verification tank balance value at **[Sensors]**.tab: _____ grams 22.3. ☐ Verify feed valve V-3003 to verification tank side arm 22.4. ☐ Verify V-3001 directed to feed pick-up valve V-3003 22.5. ☐ Verify verification tank pickup line valve V-2034 open 22.6. ☐ Verify effluent valve V-3002 to verification tank 22.7. ☐ Verify verification tank return line valve V-2036 open 22.8. ☐ Verify verification tank vent line valve V-2035 open 22.9. ☐ Verify verification tank sample-pickup line valve V-2034 closed 22.10. ☐ Open V-0003 (from External vessel) 22.11. ☐ Open V-0009 (to Target Mixing path) 22.12. ☐ Open Target Mixing loop 1 on **[Sample Collection]**.tab → **[Target Mixing]**.tab by pressing the purple 100/102 22.13. ☐ Open V-0151/0152 (to Target vessel) 22.14. ☐ Open V-0160 (to Verification tank vent) 22.15. ☐ Enter flow rate **<u>100</u>** mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab 22.16. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab 22.17. ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate A. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller 22.18. ☐ ACID Pump to **<u>RUN</u>** 22.19. ☐ Start 2 minute timer 22.20. ☐ Verify flow path is leak free at 2 minute timer end A. **IF LEAKS ARE DETECTED STOP PUMP and fix leaks before proceeding**

Step	Action
	22.21. ☐ Enter flow rate **300** mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab 22.22. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab A. **Ensure no cavitation occurs if motor is set above 50% motor power** 22.23. ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate A. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller 22.24. Monitor FEP tubing to V-0003 for target solution 22.25. ☐ Verify FEP tubing from V-0003 is empty 22.26. ☐ Start 3 minute timer 22.27. At 3 minute timer end ACID Pump to **STOP** 22.28. ☐ Close V-0003 (from External vessel) 22.29. ☐ Close V-0009 (to Target Mixing path) 22.30. ☐ Close Target Mixing loop 1 on **[Sample Collection]**.tab → **[Target Mixing]**.tab by pressing the green 100/102 button 22.31. ☐ Close V-0151/0152 (to Target vessel) 22.32. ☐ Close V-0160 (to Verification tank vent) 22.33. ☐ Close verification tank pickup line valve V-2034 22.34. ☐ Close verification tank return line valve V-2036 closed 22.35. ☐ Close verification tank vent line valve V-2035 closed 22.36. ☐ Verify verification tank sample-pickup line valve V-2034 closed 22.37. Record verification tank balance indicator: _____ grams 22.38. Record verification tank balance value at **[Sensors]**.tab: _____ grams 22.39. Go to **step 23, Remove Verification Tank**
23.	**Remove Verification Tank** 23.1. Disconnect verification tank pickup line valve V-2034 from feed jumper 23.2. Cap verification tank pickup line valve V-2034 with ¼ in. VCR cap (part SS-4-VCR-CP) A. **ALL VCR CAPS AND PLUGS REQUIRE A VCR GASKET**

Step	Action
	 23.3. Disconnect verification tank return line valve V-2036 from effluent jumper 23.4. Cap verification tank pickup line valve V-2034 with ¼ in. VCR cap (part SS-4-VCR-CP) 23.5. Disconnect verification tank sample-pickup line valve V-2034 from thief vial assembly A. OR disconnect from 5 psig N_2 source 23.6. Cap verification tank sample-pickup line valve V-2034 with ¼ in. Swagelok cap (part SS-400-P)  23.7. Disconnect verification tank vent line valve V-2035 from vent line jumper 23.8. Cap verification tank vent line valve V-2035 with ¼ in. Swagelok cap (part SS-400-P) 23.9. Wipe down all four lines and insert ends into zip-lock bag 23.10. Pass zip-lock bag with ends through column interface port (hole in bottom of glovebox) so bag is sitting/laying on verification tank cart lid 23.11. Follow steps 8.2 through 8.4 for opening cabinet #1 23.12. Upon cabinet #1 open A. Assemble and place 4-section ramps for removing verification tank cart B. Lower and remove leveling jacks C. Attach handle to verification tank cart D. Put balance in transport mode E. Pull verification tank cart out enough to access balance and leak sensor connections F. Disconnect balance and leak sensor G. Secure interior balance and leak sensor cables on hook on right side of cabinet #1 H. Secure balance and leak sensor cables inside cart to cart handle to avoid being crushed I. Remove verification tank cart J. Follow HP guidelines for smearing cabinet #1, cart, and ramps

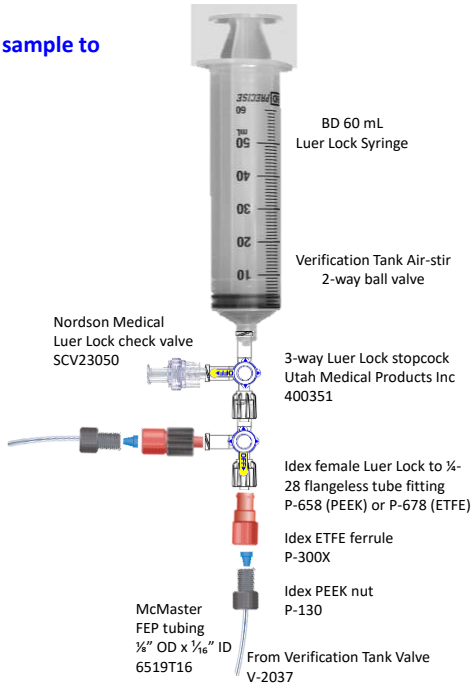
Step	Action
	K. Disassemble and stow 4-section ramps i. **DO NOT** store ramps in Cell 1 (211-D035) L. Close cabinet #1 M. Go to **Section 3.2.3, step 25, Install Feed Bottles**
24.	**Alternate Sample Retrieval Configuration**  24.1. See page for **32** additional figures 24.2. Assemble alternate sample retrieval setup according to the above figure, verifying the following: A. ☐ Plunger of **CLEAN & NEW** 60 mL syringe fully depressed B. ☐ **CLEAN & NEW** upper stopcock attached to Luer Lock fitting of syringe C. ☐ Upper stopcock handle turned to close check valve sidearm D. ☐ **CLEAN & NEW** Luer Lock check valve attached to middle-sidearm of upper stopcock

Step	Action
	E. ☐ **CLEAN & NEW** lower stopcock attached to upper stopcock and tubing from V-2037 (labeled "sparge line") F. ☐ Lower stopcock handle turned to close collection vial sidearm G. ☐ **CLEAN & NEW** 1/16 in. OD FEP tubing attached to middle-sidearm of lower stopcock H. ☐ Other end of 1/16 in. tubing attached to **CLEAN & NEW** disposable needle i. **DO NOT** remove needle shroud at this time I. ☐ **CLEAN & NEW** pre-evacuated vial inserted into a vial shield (shield may be TUNGSTEN **<u>or</u>** STAINLESS STEEL) 24.3. Detach disposable needle shroud THEN insert disposable needle into pre-evacuated vial residing in vial shield, saving the needle shroud 24.4. Attach **CLEAN & NEW** 1/8 in. OD FEP tubing to bottom-sidearm of lower stopcock 24.5. Attach other end of **CLEAN & NEW** 1/8 in. OD FEP tubing to ¼ in. VCR x 1/8 in. Swagelok fitting assembly (see Exhibit B item 6) 24.6. Attach adapter ¼ in. VCR x 1/8 in. Swagelok fitting assembly to V-2037 24.7. ☐ Verify upper stopcock handle turned to close check valve sidearm 24.8. ☐ Verify lower stopcock handle turned to close collection vial middle-sidearm 24.9. Open verification tank sample-pickup line valve V-2037 24.10. Retract plunger of 60 mL Luer lock syringe to draw at least 5 mL target solution (sample) into syringe for measurements and analysis A. If no sample is drawn up check all fittings are tight and sealed, return plunger to syringe bottom, and try again 24.11. When sufficient sample is in syringe close verification tank sample-pickup line valve V-2037 24.12. Turn lower stopcock handle to close V-2037 tubing sidearm, directing flow to the sample collection vial A. **NOTE** – sample may start flowing to the vial as the small vacuum in the vial may draw solution into the vial 24.13. Slowly push the plunger of the syringe to finish delivering the sample to the vial, stopping once a slight resistance is felt A. **DO NOT** attempt to fully push the plunger to the bottom of the syringe as this may over-pressurize the vial 24.14. Turn the upper stopcock handle to the bottom-sidearm

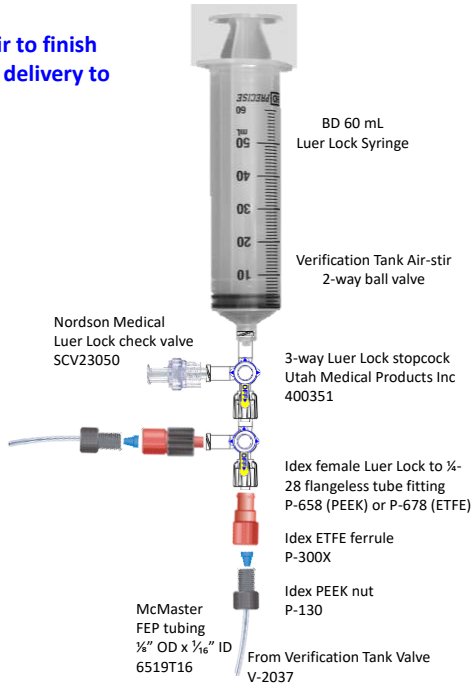
Step	Action
	A. This opens the syringe to the check valve and closes the path to the vial 24.15. Retract plunger of syringe to draw 20-40 mL of air into the syringe 24.16. Turn the upper stopcock handle to the middle-sidearm A. This opens the syringe to the path of the vial and closes the path to the check valve 24.17. Slowly push the syringe plunger to further empty liquid in the 1/16 in. OD FEP line to the vial 24.18. Turn lower stopcock handle to the middle-sidearm to close the path to the vial 24.19. Turn the upper stopcock handle to the bottom-sidearm A. This opens the syringe to the check valve and closes the path to the verification tank 24.20. Retract plunger of syringe to draw 20-40 mL of air into the syringe 24.21. Turn the upper stopcock handle to the middle-sidearm A. This opens the syringe to the path of the verification tank and closes the path to the check valve 24.22. Open verification tank sample-pickup line valve V-2037 24.23. Slowly push plunger of syringe to bottom of syringe body using air in syringe to empty sample pickup line 24.24. Close verification tank sample-pickup line valve V-2037 24.25. Remove needles and dispose of in sharps container 24.26. Go to **step 19.22** on page **24**

Figures for Alternate Sample Retrieval

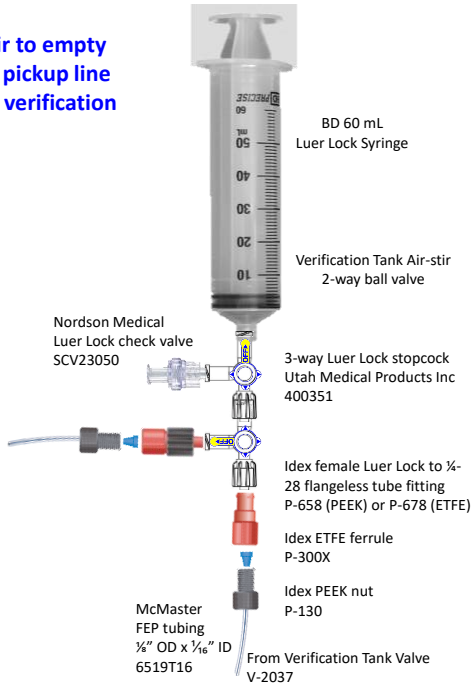
Deliver sample to vial



Draw air to finish sample delivery to vial



Draw air to empty sample pickup line back to verification tank



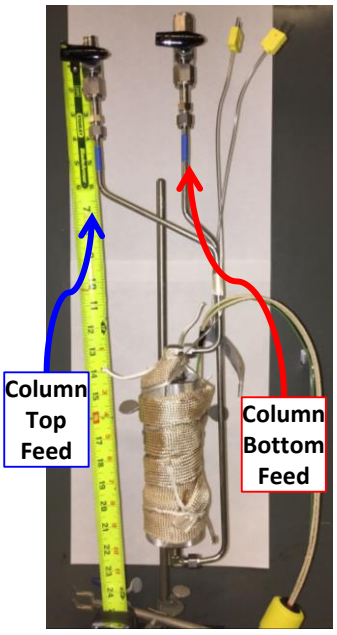
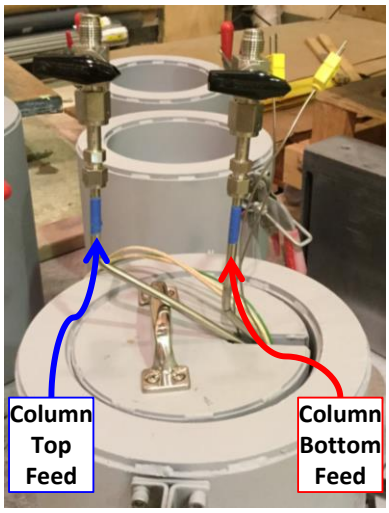
3.2.3 Installation of Feed Bottles, Effluent Cart, and Recovery Column; and Priming of Non-Rad Feed Lines

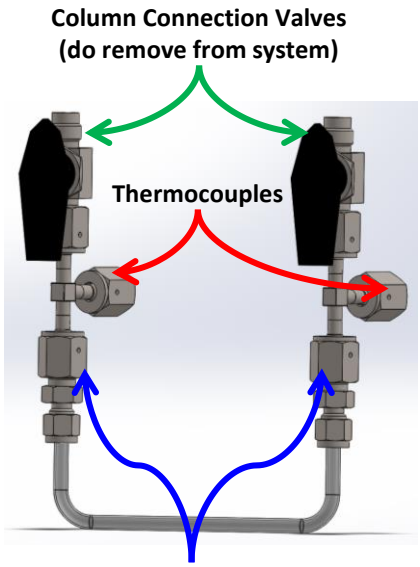
Step	Action
25.	**<u>Install Feed Bottles</u>** **Date:_____**
	25.1. All non-rad feed bottles are to be located in the ***MIDDLE*** cabinet (cabinet #2)
	25.2. Record feed balance calibration date: _____ (calibration sticker is affixed to Feed Balance Indicator on left side of LabVIEW control rack)
	A. Next calibration due: _____
	B. **If balance is out of calibration have balance calibrated**
	C. DO NOT PROCEED IF FEED BALANCE IS NOT CALIBRATED
	25.3. ☐ Remove secondary tray
	25.4. ☐ Ensure nothing is touching feed balance
	25.5. ☐ Tare feed balance
	25.6. ☐ Verify feed bottle secondary tray is in good condition (no cracks or separation)
	A. DO NOT PROCEED WITHOUT A SECONDARY TRAY
	B. If cracks or separations are found replace secondary tray (use those found in Exhibit B or equivalent)
	25.7. ☐ Center secondary tray on feed balance
	25.8. Install feed pickup lines into feed bottles (recommended to use the same type of bottle every time for a given feed bottle)
	A. Leave a lid that fits the appropriate feed bottle in the middle cabinet with the feed line inserted
	B. Verify that each feed line is properly inserted into the appropriate feed bottle to the proper depth
	i. Length of inserted tubing should be ½ in. shorter than overall height of bottle (ensures proper pickup of fluid from bottle)
	ii. Distilled water feed bottle
	a. ☐ Fresh water line from ACID sub-system
	b. ☐ Fresh water line from BASE sub-system
	1. Place this bottle in the center of the balance as it is the largest bottle
	iii. Acid feed bottle
	a. ☐ Acid line from ACID sub-system
	iv. NaOH Wash feed bottle
	a. ☐ NaOH Wash line from BASE sub-system

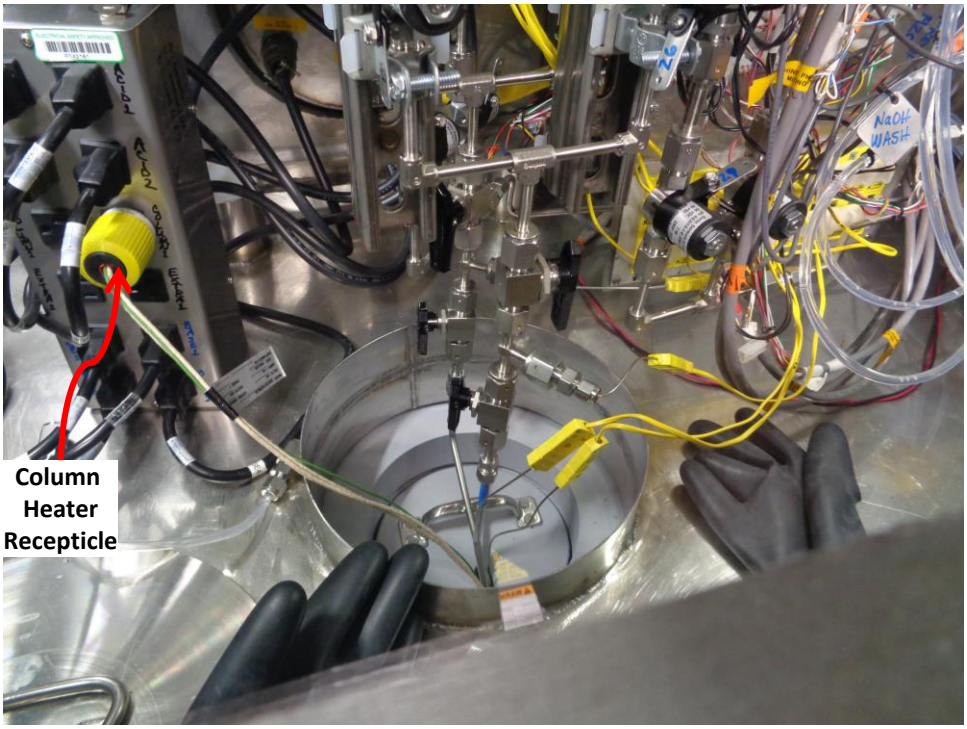
Step	Action
	v. NaOH Strip feed bottle a. ☐ Strip line from BASE sub-system vi. ☐ Verify all feed bottles are located within perimeter of balance pan 25.9. Position lab stand and clamp to help hold liquid transfer lines to keep bottles upright 25.10. ☐ Verify leak sensor in place within secondary
26.	**<u>Install Effluent Bottles Into A Shielded Effluent Bottle Cart</u> Date:_____** 26.1. There are two shielded effluent bottle carts identified by the serial number of the balance inside them A. CART #1: Ohaus Defender 7000 Model D25QRUS; Serial No. B541541414 B. CART #2: Ohaus Defender 7000 Model D25QRUS; Serial No. B541541416 26.2. **SECURE BALANCE CABLE AND LIQUID LEAK SENSOR CABLE TO CART BEFORE MOVING CART** (prevents cable ends from being crushed by cart casters) 26.3. **ONLY MOVE AN EFFLUENT CART WITH TRAY IN TRANSPORT POSITION – HANDLE IN THE DOWN POSITION** (failure to do so could damage the balance) 26.4. Record effluent balance calibration date: _____ (calibration sticker is affixed to appropriate effluent balance indicator located on the left side of LabVIEW control rack) A. Next calibration due: _____ B. If balance is out of calibration have balance calibrated C. **DO NOT PROCEED IF FEED BALANCE IS NOT CALIBRATED** 26.5. **ONLY REMOVE THE RIGHT SIDE OF THE SHIELDED EFFLUENT BOTTLE CART LID** A. Right half of lid weighs over 100 lbs and requires hoisting/rigging to remove B. The left half of the lid has two manifolds attached as well as a secondary tray serving the valve manifolds i. Manifold nearest the handle: effluent bottle // glovebox liquid manifold ii. Manifold furthest from handle: effluent bottle // glovebox vent manifold 26.6. Replace effluent cart bottles as needed. A list of appropriate bottles with silicone seals is found in the Exhibit B. **ALL EFFLUENT BOTTLES MUST BE GAS TIGHT** (prevents escape of fission gases) A. Effluent bottles have ¼ in. Liquid connections and 1/8 in. Vent connections, as described in the Exhibit B 26.7. ☐ Verify liquid leak sensor in plastic secondary inside of cart

Step	Action
	26.8. ☐ Verify liquid leak sensor in stainless steel secondary on left half of lid
27.	**<u>Install Shielded Effluent Bottle Cart</u>** 27.1. Open cabinet #3 (right side) door to full open 27.2. Position the 4-section ramp to roll the shielded effluent bottle cart into cabinet #3 27.3. ☐ Verify balance lever in transport positon 27.4. Roll shield effluent bottle cart to edge of cabinet #3 27.5. ☐ Attach balance cable A. Connectors operate smoothly when mated to one another correctly – **DO NOT FORCE CONNECTORS TOGETHER** 27.6. ☐ Verify balance is read by appropriate balance indicator (should show a 0, may require to be turned on) A. If no reading double check connection 27.7. ☐ Attach liquid leak sensor cable 27.8. ☐ Connect 2-way VCR ball valve from vent line to effluent bottle // glovebox vent manifold connection 27.9. ☐ Double check all vent lines are connected and VCR vent connections from bottles are tight 27.10. Open 2-way VCR ball valves for: A. ☐ Glovebox vent line B. ☐ PRE-LOAD ACID WASH vent line C. ☐ POST-LOAD ACID WASH vent line D. ☐ POST-LOAD H_2O WASH vent line E. ☐ ACID RINSE vent line F. ☐ POST-LOAD NaOH WASH vent line G. ☐ POST-STRIP H_2O WASH vent line H. ☐ BASE RINSE vent line 27.11. It may be necessary to push the effluent cart into cabinet #3 a little further to make liquid connections 27.12. Connect 2-way VCR ball valves on the glovebox liquid manifold from: A. ☐ PRE-LOAD ACID WASH line to effluent bottle B. ☐ POST-LOAD ACID WASH line to effluent bottle C. ☐ POST-LOAD H_2O WASH line to effluent bottle D. ☐ ACID RINSE line to effluent bottle

Step	Action
	E. ☐ POST-LOAD NaOH WASH line to effluent bottle F. ☐ POST-STRIP H_2O WASH line to effluent bottle G. ☐ BASE RINSE line to effluent bottle 27.13. ☐ Double check all liquid lines from bottles are connected and bottle-side VCR liquid connections are tight 27.14. ☐ Double check all liquid lines from glovebox effluent liquid lines are connected and glovebox-side VCR liquid connections are tight 27.15. Open BOTH 2-way VCR ball valves for: A. ☐ PRE-LOAD ACID WASH liquid line B. ☐ POST-LOAD ACID WASH liquid line C. ☐ POST-LOAD H_2O WASH liquid line D. ☐ ACID RINSE liquid line E. ☐ POST-LOAD NaOH WASH liquid line F. ☐ POST-STRIP H_2O WASH liquid line G. ☐ BASE RINSE liquid line 27.16. Fully push the fully connected effluent cart into cabinet #3 27.17. Remove the handle from the effluent cart and store in the instrument room until the cart needs to be removed 27.18. Tare appropriate balance indicator 27.19. ☐ Turn balance transport handle to weigh (up) 27.20. ☐ Verify balance is read by appropriate balance indicator (should be a positive, non-zero value) 27.21. Remove 4-section ramp (**DO NOT STORE 3-SECTION RAMPS IN CELL 1)** 27.22. Close cabinet #3 door slowly
28.	**<u>Install Packed Recovery Column</u> Date:_____** 28.1. Place the column in column pig (see figures on following page) A. Face pig so that toggle clamps are to the left & right (toggle clamp plane) with the pig lid slot facing front (facing user) B. Remove pig lid (if already inserted) C. Insert column so that column bottom feed line faces user / column top feed line is away from user i. Tubing plane is perpendicular to toggle clamp plane D. Insert pig lid such that tubing, heater connection, and thermocouples fit through slot

Step	Action
	i. It may be necessary to partially lift the column straight up while positioning the pig lid, then move both together to lower pig lid in place ii. NOTE – in the picture below the handle of pig lid is crooked relative to toggle clamp plane    28.2. Secure the column pig to the transport cart using a ratcheting strap A. **COLUMN PIG MUST ALWAYS BE STRAPPED DOWN DURING MOVEMENT OF COLUMN PIG CART** B. Wind excess strap to cart to prevent the cart from rolling over it and to remove a potential tripping hazard 28.3. Open door to cabinet #1 28.4. Install 4-section ramp in front of cabinet #1 28.5. Roll column/pig assembly into cabinet #1 28.6. The slot in the pig lid faces the front of the glovebox A. This configuration will orient the toggle clamps on the column/pig assembly left/right 28.7. Remove the ratcheting strap and install the black gasket on the top of the column pig 28.8. Position column/pig assembly under pig locking ring on the top, inside face of cabinet #1 28.9. ☐ Verify that toggle clamps on column/pig assembly align with the two toggle clamp hooks on the pig locking ring 28.10. ☐ Verify that slot of pig lid points to front of glovebox

Step	Action
	28.11. ☐ Verify that the column connection valves (V-2002 & V-2003) are closed 28.12. Remove the column jumper from the column connection fittings  28.13. ☐ Verify the two VCR 2-way ball valves (V-2004 & V-2005) attached to the column are closed 28.14. Remove VCR caps from the two VCR 2-way ball valves 28.15. Use a ratcheting wrench to jack the column/pig assembly toward the pig locking ring 28.16. While raising the column/pig assembly pass the column heater cable into the glovebox and align the two VCR 2-way ball valves to their appropriate ports A. Column top feed is the rear connection B. Column bottom feed is the front connection 28.17. Make the two VCR connections when the jack is nearly fully extended A. The column will hang free inside the pig even though the pig is not locked in place yet 28.18. Plug column heater cable into column heater receptacle 28.19. ☐ Connect column heater control thermocouple 28.20. ☐ Connect column heater over-temperature thermocouple 28.21. ☐ Verify thermocouples reading at Column Heater temperature controller at LabVIEW rack 28.22. Secure one toggle clamp to appropriate toggle clamp hook of pig locking ring A. Clamp will be locked down which slightly lifts the column/pig assembly

Step	Action
	28.23. Secure remaining toggle clamp to appropriate toggle clamp hook of pig locking ring A. Locking the toggle clamp will lift the column/pig assembly off of the cart  28.24. Remove the pig transport cart and insert the pig table under the column/pig assembly A. The black rubber gasket at the glovebox/column pig interface may prevent the ability to remove the transport cart. In this case detach the ratcheting wrench and leave the cart in the cabinet 28.25. Check cables are hung on hook inside of cabinet #1 28.26. Remove 4-section ramp from in front of cabinet #1 A. **4-SECTION RAMP IS NOT TO BE STOWED IN CELL 1** 28.27. Close cabinet #1 door
29.	**<u>Verify Sample Retrieval Valves Powered Off</u>** 29.1. **At the rack** A. Verify Sample Retrieve Valve Power to OFF (switch is down) B. If the switch is not OFF then actuate the switch by gently pulling out while moving the handle down

Step	Action
	
30.	**<u>Begin Operation of Mo-99 Remote Recovery Data Acquisition & Control Software</u>** 30.1. Version used: _____ A. Current version: ***M3_SHINE_PhaseII_ver06J.vi*** (as of 3/27/2018) 30.2. ☐ External computer speakers powered ON A. Verify speakers work B. Run beep10.bat file from desktop  30.3. ☐ Sound came out of speakers A. If sound does not come out of speakers DO NOT proceed until speakers are operational 30.4. Start the program using the following parameters of input: A. Manual mode B. Process operation C. Column D. RECORD EFFLUENT BALANCE DATA? i. YES E. Set effluent balance type and COM port i. Ohaus Defender 7000 (25/50 kg)

Step	Action
	ii. Pick ONE a. Effluent cart #1: ends in 414, set COM 11 b. Effluent cart #1: ends in 416, set COM 10 F. Fresh Acid density → as default value → press OK G. Base Wash density → as default value → press OK H. Base Strip density → as default value → press OK I. Target solution volume i. Enter last target solution volume J. Target solution concentration i. Enter last target solution concentration K. Target solution density i. Enter last target solution density L. Column effluent path i. To Transfer Cask M. Pre-Load Acid Wash processing volume → as default value → press OK N. Column Loading processing volume → as default value → press OK O. Post-Load Acid Wash processing volume → as default value → press OK P. Post-Load Water Wash processing volume → as default value → press OK Q. Use the Post-Load NaOH Wash step? → NO R. Column stripping processing volume → as default value → press OK S. Post-Strip Water Wash To Strip product processing volume → as default value → press OK T. Post-Strip Water Wash To Waste processing volume → as default value → press OK U. Final Base System Water Wash processing volume → as default value → press OK V. Final Acid System Acid Wash processing volume → as default value → press OK W. Record LINAC temperatures? → YES X. Filename prefix: _____ (see **[File Paths]**.tab → File Prefix) Y. ☐ ACID Pump controller powered ON (rocker switch under front/left of ACID Pump V300 controller) Z. ☐ ACID Pump to **STOP** (display alternates between OFF and current setting) AA. ☐ Verify feed and effluent balances are reading at **[Sensors]**.tab and compare to feed balance indicator i. Feed balance

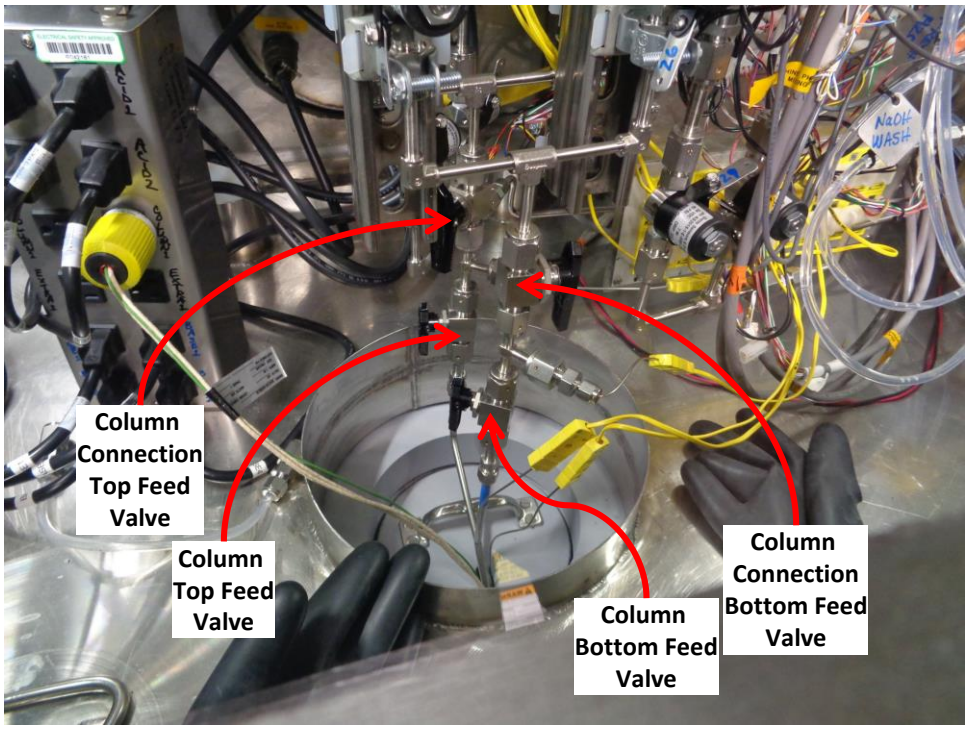
Step	Action
	a. Indicator: _____ grams b. LabVIEW: _____ grams ii. Effluent balance indicator a. Indicator: _____ grams b. LabVIEW: _____ grams 30.5. **DO NOT PROCEED IF FEED OR EFFLUENT BALANCES ARE NOT BEING READ**
31.	**Prime Feed Bottle Lines and Flush Target Mixing Path** **– YOU ARE OPERATING THE SYSTEM IN MANUAL MODE –** 31.1. Prime the acid feed lines through target mixing path A. ☐ Open V-0001 (H_2O feed for acid manifold) B. ☐ Verify flow path through acid flow meter 163/164 (valves should already be open) C. ☐ Open V-0009 (Target Mixing path) D. ☐ Open Target Mixing loop 1 on **[Sample Collection]**.tab → **[Target Mixing]**.tab (press the purple 100/102 button) E. ☐ Open V-0153/0154 (Target Mixing path to Dump Tank path) F. ☐ Open V-0172/0173 (Target Mixing path to Acid Rinse bottle) i. Selecting this valve closes both 147/148 (frit to Dump Tank) and 149/150 (bypass to Dump Tank) G. ☐ Enter flow rate **80** mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab H. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab I. ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate i. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller J. Monitor FEP tubing to V-0001 K. ☐ ACID Pump to **RUN** L. When H_2O is at V-0001 ACID Pump to **STOP** M. ☐ Close V-0001 (H_2O feed) N. ☐ Open V-0002 (Fresh Acid feed) O. Monitor Effluent balance reading @ **[Sensors]**.tab P. ☐ ACID Pump to **RUN**

Step	Action
	Q. When Fresh Acid is at Effluent balance ACID Pump to **STOP** R. ☐ Enter flow rate **300** mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab S. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab i. **Ensure no cavitation occurs if motor is set above 50% motor power** T. ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate i. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller U. ☐ Prepare 2 minute timer V. ☐ ACID Pump to **RUN** and start timer to flush the path of possible residual uranium before performing column leak check W. At 2 minute timer end ACID Pump to **STOP** X. ☐ Verify ACID Pump to **STOP** Y. ☐ Close V-0002 (Fresh Acid feed) Z. ☐ Open V-0171 (Surge Vessel gas) AA. ☐ Open V-0158 (Surge tank vent) BB. ☐ Enter flow rate **300** mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab CC. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab i. **Ensure no cavitation occurs if motor is set above 50% motor power** DD. ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate i. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller EE. Monitor effluent balance **[Sensors]**.tab FF. ☐ ACID Pump to **RUN** to empty the Target Mixing Path of acid rinse solution GG. At stable effluent balance reading (no increase) ACID Pump to **STOP** HH. ☐ Close V-0171 (Surge Vessel gas) II. ☐ Close V-0158 (Surge tank vent) JJ. ☐ Close V-0009 (Target Mixing path) KK. ☐ Close Target Mixing loop 1 on **[Sample Collection]**.tab → **[Target Mixing]**.tab by pressing the purple 100/102 button LL. ☐ Close V-0153/0154 (Target Mixing path to Dump Tank path) MM. ☐ Close V-0172/0173 (Target Mixing path to Acid Rinse bottle)

Step	Action
	NN. ☐ ACID Pump controller powered OFF using rocker switch under front/left of ACID Pump controller 31.2. Priming base feed lines through column stripping path A. ☐ BASE Pump controller powered ON using the rocker switch under front/left of BASE Pump V300 controller B. ☐ Open V-0006 (H_2O feed for base manifold) C. ☐ Verify flow path through base flow meter 167/168 (valves should already be open) D. ☐ Open V-0024/0025 (Base column bypass) using toggle switch at lower left corner of **[System]**.tab E. ☐ Verify flow path through base column stripping filter 28/29 (valves should already be open) F. ☐ Open Column Stripping loop 1 on **[Sample Collection]**.tab → **[Column Stripping]**.tab by pressing the purple 66/68 button G. ☐ Open V-0134 (Base rinse) H. ☐ Enter flow rate **80** mL/min *Base Flow Rate Set Pt* @ **[System]**.tab I. ☐ Record calculated _____ *% Base Motor Power* @ **[System]**.tab J. ☐ Verify/Adjust BASE Pump Controller to *% Base Motor Power* for desired flow rate i. If a lower % motor power value is required due to pressure readings adjust *Base Flow Rate Set Pt* until calculated *% Base Motor Power* matches % motor power reading at controller K. Monitor FEP tubing to V-0006 L. ☐ BASE Pump to **RUN** M. When H_2O is at V-0006 BASE Pump to **STOP** N. ☐ Close V-0006 (H_2O feed for base manifold) O. Will Post-Load NaOH wash be used? (Pick one) i. ☐ NO a. Skip to **step 31.2.P.** ii. ☐ YES a. ☐ Open V-0007 (NaOH wash feed) b. ☐ Enter flow rate **80** mL/min *Base Flow Rate Set Pt* @ **[System]**.tab c. ☐ Record calculated _____ *% Base Motor Power* @ **[System]**.tab d. ☐ Verify/Adjust BASE Pump Controller to *% Base Motor Power* for desired flow rate

Step	Action
	1. If a lower % motor power value is required due to pressure readings adjust *Base Flow Rate Set Pt* until calculated *% Base Motor Power* matches % motor power reading at controller e. Monitor FEP tubing to V-0007 f. ☐ BASE Pump to **RUN** g. When NaOH is at V-0007 BASE Pump to **STOP** h. ☐ Close V-0007 (NaOH wash feed) i. Go to **step 31.2.P** P. ☐ Open V-0008 (NaOH Strip feed) Q. ☐ Enter flow rate **80** mL/min *Base Flow Rate Set Pt* @ **[System]**.tab R. ☐ Record calculated _____ *% Base Motor Power* @ **[System]**.tab S. ☐ Verify/Adjust BASE Pump Controller to *% Base Motor Power* for desired flow rate i. If a lower % motor power value is required due to pressure readings adjust *Base Flow Rate Set Pt* until calculated *% Base Motor Power* matches % motor power reading at controller T. Monitor FEP tubing to V-0008 U. ☐ BASE Pump to **RUN** V. When NaOH Strip is at V-0008 BASE Pump to **STOP** W. ☐ Close V-0008 (NaOH Strip feed) X. ☐ Open V-0006 (H_2O feed for base manifold) Y. ☐ Enter flow rate **200** mL/min *Base Flow Rate Set Pt* @ **[System]**.tab Z. ☐ Record calculated _____ *% Base Motor Power* @ **[System]**.tab AA. ☐ Verify/Adjust BASE Pump Controller to *% Base Motor Power* for desired flow rate i. If a lower % motor power value is required due to pressure readings adjust *Base Flow Rate Set Pt* until calculated *% Base Motor Power* matches % motor power reading at controller BB. ☐ Prepare 3 minute timer CC. ☐ BASE Pump to **RUN** and start timer to flush the column stripping path of NaOH solution DD. At 3 minute timer end BASE Pump to **STOP** EE. ☐ Verify BASE Pump to **STOP** FF. ☐ Close V-0006 (H_2O feed for base manifold)

Step	Action
	GG. ☐ Close Column Stripping loop 1 on **[Sample Collection]**.tab → **[Column Stripping]**.tab by pressing the purple 66/68 button HH. ☐ Close V-0134 (Base rinse) II. ☐ BASE Pump controller powered OFF using rocker switch under front/left of BASE Pump V300 controller
32.	**Column Leak Checking** **– YOU ARE OPERATING THE SYSTEM IN MANUAL MODE –** 32.1. ☐ ACID Pump controller powered ON using rocker switch under front/left of ACID Pump V300 controller 32.2. ☐ ACID Pump to **STOP** (display alternates between OFF and current setting) 32.3. ☐ Open V-0002 (Fresh Acid feed) 32.4. ☐ Open V-0010 (Column path) 32.5. ☐ Verify flow path through acid flow meter 163/0164 (valves should already be open) 32.6. ☐ Open V-0014/0015 (Acid column bypass) using toggle switch at lower left corner of **[System]**.tab A. Going through column bypass to first allow additional rinsing of potential residual uranium before switching to the column 32.7. ☐ Verify flow path through acid column loading filter 18/19 (valves should already be open) 32.8. ☐ Open Column Loading loop 1 on **[Sample Collection]**.tab → **[Column Loading]**.tab by pressing the purple 32/34 button 32.9. ☐ Open V-0139 (Acid rinse) 32.10. ☐ Open V-0156 (Effluent bottle vent) 32.11. ☐ Enter flow rate **200** mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab 32.12. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab 32.13. ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate A. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller 32.14. Monitor effluent balance @ **[Sensors]**.tab 32.15. Prepare 2 minute timer

Step	Action
	32.16. ☐ ACID Pump to **RUN** and start timer to flush the path of possible residual uranium before performing column leak check 32.17. At 2 minute timer end ACID Pump to **STOP** 32.18. ☐ Verify ACID Pump to **STOP**  32.19. ☐ Open column connection bottom feed valve V-2002 32.20. ☐ Open column bottom feed valve V-2004 32.21. ☐ Open column top feed valve V-2005 32.22. ☐ Open column connection top feed valve V-2003 32.23. ☐ Open V-0012/0013 + V-0016/0017 (Column loading bottom feed) using toggle switch at lower left corner of **[System]**.tab 32.24. ☐ Verify flow path through acid column loading post-column filter 18/19 (valves should already be open) 32.25. ☐ Verify Column Loading loop 1 open on **[Sample Collection]**.tab → **[Column Loading]**.tab by checking purple 32/34 button 32.26. ☐ Verify V-0139 open (Acid rinse) 32.27. ☐ Enter flow rate **80** mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab 32.28. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab 32.29. ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate

Step	Action
	A. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller 32.30. Prepare 2 minute timer 32.31. ☐ ACID Pump to **RUN** and start timer 32.32. Monitor for leaks at column connections A. If leaks are detected, stop pump and fix the leaks before finishing the 2 minute timer and proceeding to step **32.33** B. **DO NOT PROCEED TO NEXT STEPS IF LEAKS ARE OBSERVED** 32.33. At 2 minute timer end ACID Pump to **STOP** 32.34. ☐ Enter flow rate **167** mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab 32.35. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab 32.36. ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate A. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller 32.37. Prepare 2 minute timer 32.38. ☐ ACID Pump to **RUN** and start timer 32.39. Monitor for leaks at column connections A. If leaks are detected, stop pump and fix the leaks before finishing the 2 minute timer and proceeding to **step 32.40** 32.40. **DO NOT PROCEED TO NEXT STEPS IF LEAKS ARE OBSERVED** 32.41. At 2 minute timer end ACID Pump to **STOP** 32.42. ☐ Close V-0002 (Fresh acid feed) 32.43. ☐ Close V-0010 (Column loading path) 32.44. ☐ Close V-0139 (Acid Rinse bottle) 32.45. ☐ Close V-0156 (Effluent bottle vent) 32.46. ☐ ACID Pump controller powered OFF using rocker switch under front/left of ACID Pump V300 controller
33.	**End Operation of Mo-99 Remote Recovery Data Acquisition & Control Software** 33.1. Press MASTER EXIT button @ **[System]**.tab

3.2.4 Solution Irradiation and ^{99}Mo Recovery

Linac – Lab Notebook No.: _____ Pages: _____

GC/MS-RGA – Lab Notebook No.: _____ Pages: _____

LabVIEW – Lab Notebook No.: _____ Pages: _____

Step	Action
34.	**<u>Ensure Production Feeds Analysis and Process Conditions Summary Sheet is Completed and Enter Information Into Section</u>** \\ **Initials: _____ Date: _____ Time: _____** 34.1. ☐ Obtain a copy of Production Feeds Analysis and Process Conditions Summary (PFA-PCS) or associated cheat sheet 34.2. Enter all pertinent information from PFA-PCS Sheet in steps **44.8 – 44.23, 53.8, 54.7, 55.7, 56.7, 58.1, 58.8, 58.9, 59.5, 59.6, 59.12, 60.7.A,** and **61.10**. 34.3. Shade in appropriate sample loops to be used in steps **45.8.B, 47.3.D, 53.10.A, 54.11.E, 55.9.B, 56.9.C, 58.10.G, 59.14.G,** and **60.9.A**
35.	**<u>Checking J-Kem Temperature Controllers Are Powered On</u>** \\ **Initials: _____ Date: _____ Time: _____** 35.1. Controllers must be ON and OPERABLE for software to run correctly 35.2. BASE Pre-Heater controller (top unit) A. ☐ **Output Power Level** control knob to OFF B. ☐ Check **Power Toggle** switch is in the ON position (up) C. ☐ LED displays on i. Control temperature (left): _____ °C ii. Over-temperature (right): _____ °C D. ☐ Press red RESET button to turn off Orange **Limit Controller** light if on 35.3. COLUMN Heater controller (middle unit) A. ☐ **Output Power Level** control knob to OFF B. ☐ Check **Power Toggle** switch is in the ON position (up) C. ☐ LED displays on i. Control temperature (left): _____ °C ii. Over-temperature (right): _____ °C D. ☐ Press red RESET button to turn off Orange **Limit Controller** light if on 35.4. ACID Pre-Heater controller (bottom unit) A. ☐ **Output Power Level** control knob to OFF B. ☐ Check **Power Toggle** switch is in the ON position (up)

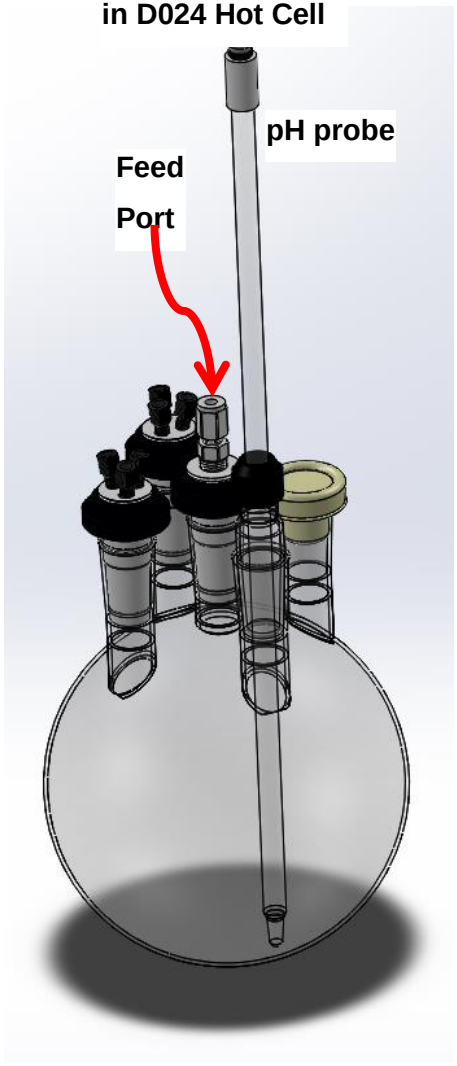
Step	Action
	C. ☐ LED displays on i. Control temperature (left): _____ °C ii. Over-temperature (right): _____ °C D. ☐ Press red RESET button to turn off Orange **Limit Controller** light if on 35.5. If LED display shows alternating **"inPt"** and **"FAiL"** A. Thermocouple path is broken or the thermocouple is bad and must be repaired before proceeding B. Record repair in lab notebook, then record readings in appropriate step 35.6. ☐ Turn on electrical box for heaters
36.	**Checking Leak Sensors Active** \\ **Initials: _____ Date: _____ Time: _____** 36.1. Leak sensor panel is located below display panel for load cells 36.2. ☐ Verify leak sensor power panel is on (if not, turn it on) 36.3. ☐ Verify leak sensor audible alarm mute is off A. The mute audible alarm switch should be in the down position B. If it is in the up position, gently pull the switch out so that it can be moved into the down position
37.	**Verify Power To Sampling Valves is OFF** 37.1. See figure below, toggle points down 
38.	**Checking Manual Dump Tank Valve OPEN**

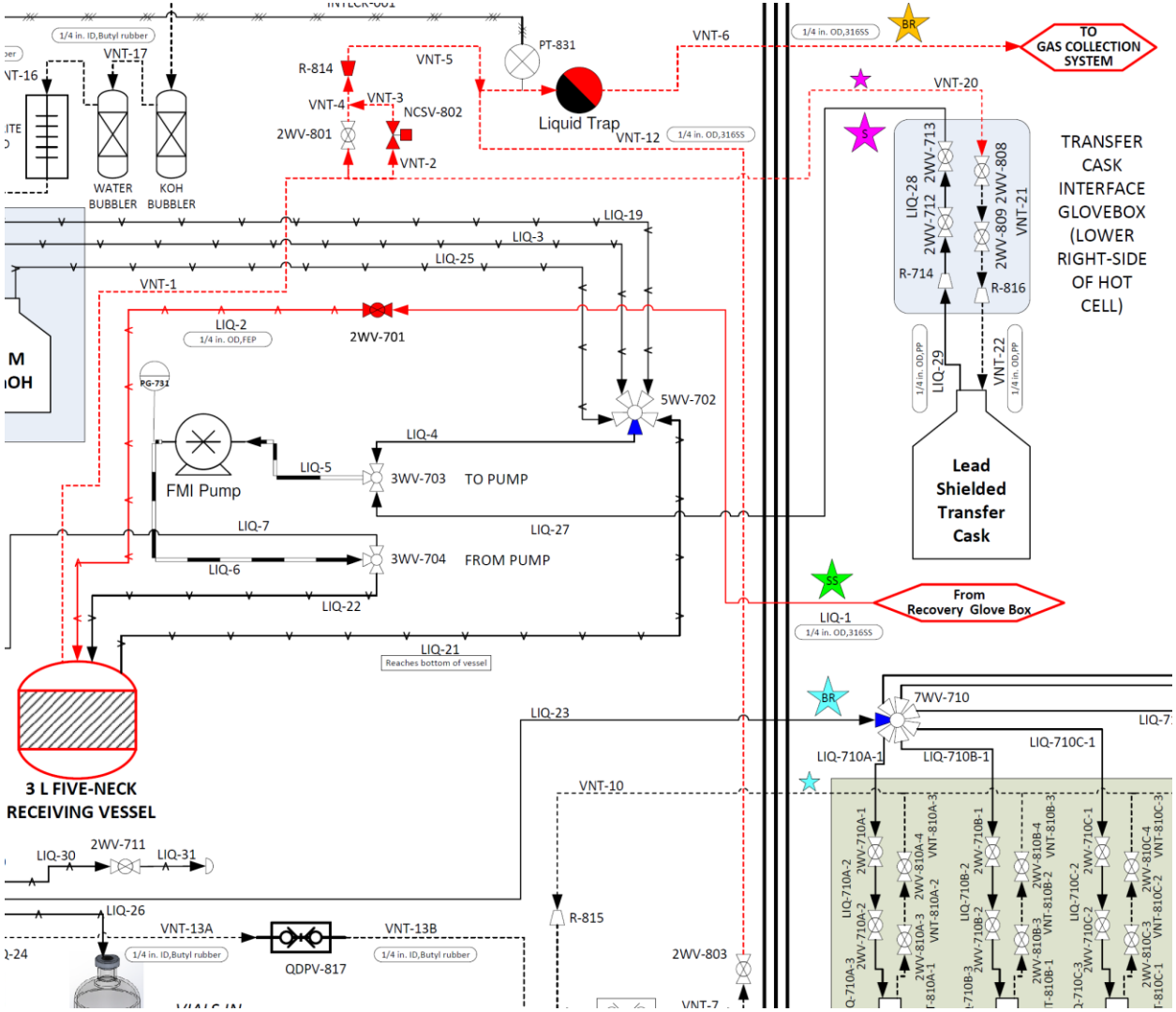
Step	Action
	### SYSTEMS INTERFACE STEP ### 38.1. Contact a Gas Analysis/Collection team member 38.2. **At the Dump Tank (211-D035)** A. ☐ Verify manual dump tank valve is OPEN B. Recovery team member // Name: _____PRINT Initials: _____ Date: _____ Time: _____ C. Gas Analysis/Collection team member // Name: _____PRINT Initials: _____ Date: _____ Time: _____ 38.3. Recovery personnel continue to **step 39**
39.	**<u>Checking D024 Hot Cell 3-L/5-Neck Flask Installation</u>** \\ **Recovery Member Name: _____PRINT Date: _____ Time: _____** **D024 Hot Cell Ops Member Name: _____PRINT Date: _____ Time: _____** ### SYSTEMS INTERFACE STEP ### 39.1. Contact a D024 Hot Cell Operations team member 39.2. Appropriate team member **<u>INITIALIZES</u>** every step in this section 39.3. Refer to figures on page **55** 39.4. **Inside D024 Hot Cell** A. ☐ Recovery ☐ HotCellOps Verify 3-L/5-neck flask in place B. ☐ Recovery ☐ HotCellOps Verify pH probe inserted and not broken C. ☐ Recovery ☐ HotCellOps Verify plastic feed line attached to center neck D. ☐ Recovery ☐ HotCellOps Verify septum in un-used neck and secured E. ☐ Recovery ☐ HotCellOps Verify multi-port neck adapters inserted and attached to other two ports F. ☐ Recovery ☐ HotCellOps Verify flask on balance

Step	Action
	G. ☐ Recovery ☐ HotCellOps Verify balance is ON H. Record balance reading: _____ grams I. ☐ Recovery ☐ HotCellOps Verify 2WV-701 liquid valve OPEN i. Handle parallel to the long axis of valve body J. ☐ Recovery ☐ HotCellOps Verify plastic line attached between 2WV-701 and center neck of flask K. ☐ Recovery ☐ HotCellOps Verify 2WV-801 vent valve OPEN i. Handle perpendicular to the long axis of valve body L. Verify liquid trap to Gas Collection System is: i. ☐ Recovery ☐ HotCellOps Attached ii. ☐ Recovery ☐ HotCellOps Good condition (not cracked/broken), lines attached iii. ☐ Recovery ☐ HotCellOps Empty 39.5. Recovery personnel continue to **step 40**

3-L / 5-neck Flask

in D024 Hot Cell





Step	Action
40.	**<u>Checking Column Stripping Transfer Cask Installation</u>** **\\ Initials: _____ Date: _____ Time: _____** 40.1. Outside the white Transfer Cask Interface glovebox in Cell 1 (D035) A. ☐ Verify column stripping transfer cask is attached to white glovebox 40.2. Inside the white Transfer Cask Interface glovebox in Cell 1 (D035) A. ☐ Verify ¼ in. liquid line connections secure (segment between V-2007 & V-2009) B. ☐ Verify both 2-way valves for liquid service (on the ¼ in. line) are open i. Handles parallel to the long axis of valve body C. ☐ Verify ⅛ in. liquid line connections secure i. Segment between V-2006 & V-2008 D. ☐ Verify both 2-way valves for vent service (on the ⅛ in. line) are open i. Handles parallel to the long axis of valve body E. ☐ Verify Transfer Cask Interface glovebox leak sensor attached F. ☐ Verify Transfer Cask leak sensor attached 40.3. IF **Section 3.2.3 (Installation of Feed Bottles, Effluent Cart, and Recovery Column)** was completed directly before irradiation, operators may skip to **step 44**, otherwise proceed to step **41**
41.	**<u>Checking Column Installed and Manual Valves Open</u> \\ Initials: _____ Date: _____ Time: _____** 41.1. In Cabinet #1 A. ☐ Verify column pig in place and latches are locked B. ☐ Verify that Mytec support table is under column pig 41.2. Inside Mo99 Recovery Glovebox A. ☐ Verify that all four (4x) manual 2-way valves are open to the column i. Handles parallel to the long axis of valve body ii. ☐ V-2002 iii. ☐ V-2004 iv. ☐ V-2005 v. ☐ V-2003 B. ☐ Verify that the liquid pickup line from the Pressure Relief Valve surge tank is attached to side-arm of V-3001 C. ☐ Verify center arm of V-3001 is attached to V-0003

Step	Action
	D. ☐ Verify point of handle of V-3001 points to side-arm attached to pickup line from the Pressure Relief Valve surge tank
42.	**<u>Checking Feed Bottles in Place</u> \\ Initials: _____ Date: _____ Time: _____** 42.1. Perform if step **25** was completed more than 1 week ago: In Cabinet #2 A. ☐ Verify feed bottles are in secondary B. ☐ Verify that pickup tubes are properly inserted into bottles i. Tubes opening should just touch bottom of feed bottle C. ☐ Verify leak sensor lays flat in secondary tray
43.	**<u>Checking Effluent Cart in Place and Manual Valves Open</u>** **\\ Initials: _____ Date: _____ Time: _____** 43.1. Perform if step **26** was completed more than 1 week ago: In Cabinet #3 A. Record last three digits of balance serial number: _____ B. ☐ Verify tare handle is in the up READ position C. Verify BOTH 2-way liquid valves (¼ in.) open for each process stream (handles parallel to the long axis of the valve body) i. Pre-load acid wash a. ☐ V-2012 b. ☐ V-2031 ii. Post-load acid wash a. ☐ V-2013 b. ☐ V-2029 iii. Post-load H_2O wash a. ☐ V-2014 b. ☐ V-2027 iv. Acid rinse a. ☐ V-2018 b. ☐ V-2019 v. Post-load NaOH wash a. ☐ V-2015 b. ☐ V-2025 vi. Post-strip H_2O wash a. ☐ V-2016

Step	Action
	b. ☐ V-2023 vii. Base rinse a. ☐ V-2017 b. ☐ V-2021 D. Verify single 2-way vent valves (⅛ in.) for each process stream and main 2-way vent valve are open (handles parallel to the long axis of the valve body) i. ☐ V-2032 Pre-load acid wash ii. ☐ V-2030 Post-load acid wash iii. ☐ V-2028 Post-load H_2O wash iv. ☐ V-2020 Acid rinse v. ☐ V-2026 Post-load NaOH wash vi. ☐ V-2024 Post-strip H_2O wash vii. ☐ V-2022 Base rinse viii. ☐ V-2011 Main 2-way vent valve
44.	**<u>Begin Operation of Mo-99 Remote Recovery Data Acquisition & Control Software</u>** \\ **Initials: _____ Date: _____ Time: _____** 44.1. Version used: _____ A. Current version: **M3_SHINE_PhaseII_ver06J.vi** (as of 3/27/2018) 44.2. ☐ External computer speakers powered ON 44.3. Verify speakers work A. Run beep10.bat file from desktop  B. ☐ Sound came out of speakers i. If sound does not come out of speakers DO NOT proceed until speakers are operational 44.4. Start the program 44.5. Mode (PICK ONLY ONE) A. ☐ Automated **<u>OR</u>**

Step	Action
	B. ☐ Manual i. Select Operation a. ☐ Process Operation 1. ☐ Recovery Column **OR** 2. ☐ Verification Tank **OR** b. ☐ Cleanout Operation 44.6. Prepare Feed Balance & COM port for data acquisition A. ☐ Yes i. ☐ Ohaus Defender 7000 (25/50 kg) ii. ☐ COM 9 B. ☐ No i. No additional information 44.7. Prepare Effluent Balance & COM port for data acquisition A. ☐ Yes i. There are TWO effluent bottle carts SELECT the correct cart a. ☐ 414 - Ohaus Defender 7000 (25/50 kg) 1. ☐ COM 11 b. ☐ 416 - Ohaus Defender 7000 (25/50 kg) 1. ☐ COM 10 ii. ☐ Verify the EFFLUENT BALANCE COM cable is connected to the correct LCD display at the rack B. ☐ No i. No additional information 44.8. Enter density of fresh acid solution in g/ml A. ☐ _____ g/mL 44.9. Enter density of base wash solution in g/ml A. ☐ _____ g/mL 44.10. Enter density of base strip solution in g/ml A. ☐ _____ g/mL 44.11. Enter post-adjustment volume of target solution in mL

Step	Action
	A. ☐ _____ mL 44.12. Enter post-adjustment target solution concentration in g U/L A. ☐ _____ g U/L 44.13. Enter post-adjustment density of target solution in g/ml A. ☐ _____ g/mL 44.14. Please select column effluent path for Mo-99 strip A. ☐ To transfer cask B. ☐ To Mo99 processing cell (Hot Cell / Big foot) 44.15. Enter Pre-Load Acid Wash volume A. ☐ _____ mL 44.16. Enter Column Loading volume A. ☐ _____ mL 44.17. Enter Post-Load Acid Wash volume A. ☐ _____ mL 44.18. Enter Post-Load H_2O Wash volume A. ☐ _____ mL 44.19. Enter Post-Load NaOH Wash volume A. ☐ Skipped **OR** B. ☐ _____ mL 44.20. Enter Column Strip volume A. ☐ _____ mL 44.21. Enter Post-Strip H_2O Wash A. Enter Post-Strip H_2O Wash volume to Strip product path (to hot cell) i. ☐ _____ mL B. Enter Post-Strip H_2O Wash volume to waste bottle path (to waste) i. ☐ _____ mL 44.22. Enter Final Base System H_2O Wash volume A. ☐ _____ mL 44.23. Enter Final Acid System Acid Wash volume A. ☐ _____ mL 44.24. Record LINAC temperatures? *(LINAC temperatures must be recorded)*

A. ☐ Yes

44.25. Filename prefix: _____ (see **[File Paths]**.tab → File Prefix)

44.26. Verify gas collection system pressure readings

A. Record gas collection system pressure readings from LED displays

i. 0-2000 mbar gauge (Chamber 1) = _____ mbar *(RIGHT)*

ii. 0-2000 mbar gauge (Chamber 2) = _____ mbar *(MIDDLE)*

iii. 0-5000 psig gauge (High Pressure Cyl.) = _____ psig *(LEFT)*

B. Record gas collection system pressure readings from LabVIEW displays at

i. 0-2000 mbar gauge (Chamber 1) = _____ mbar *(RIGHT)*

ii. 0-2000 mbar gauge (Chamber 2) = _____ mbar *(MIDDLE)*

iii. 0-5000 psig gauge (High Pressure Cyl.) = _____ psig *(LEFT)*

44.27. Verify balance readings

A. Record balance readings from balance displays

i. Feed balance: _____ g

ii. Effluent cart balance: _____ g

44.28. ☐ ACID Pump controller powered ON using rocker switch under front/left of ACID Pump V300 controller

44.29. ☐ ACID Pump to **STOP** (display alternates between OFF and current setting)

44.30. ☐ BASE Pump controller powered OFF using rocker switch under front/left of BASE Pump V300 controller

44.31. ☐ Verify dial @ *Start*

44.32. ☐ Enter desired flow rate _____ mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab (default this step is 167 mL/min)

44.33. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab

44.34. ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate

A. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller

44.35. Press *{NEXT STEP}*

A. ☐ Answer OK

B. ☐ Answer OK

C. ☐ Press *{NEXT SAMPLE}* on **[Sample Collection]**.tab → **[Target Mixing]**.tab

44.36. ☐ Verify dial @ *Target Solution Mixing*

A. Path reference information

Step	Action
	i. Uses ACID path pump ii. V-0005 – from target vessel iii. V-0163/0164 – Acid Flow Meter iv. V-0009 – Mixing Path Sampling Assembly v. Loop 1 on target solution monitoring sample collection – V-0100/0102 vi. V-0151/0152 – to target vessel vii. Valve text: **5 > 163/164 > 9 > 100/102 Loop 1 > 151/152 > Target Vessel** 44.37. ☐ ACID Pump to **RUN** @ _____ *(LabVIEW clock time)* 44.38. ☐ Ensure flow path bypasses the flow meter (open valves V-0165/0166) 44.39. ☐ Verify flow path is leak free _____ *(LabVIEW clock time)* A. If leaks are observed STOP the ACID pump and report leaks to Linac Operator B. Address leaks i. If leaks CANNOT be successfully addressed/fixed notify Linac Operator do not continue this procedure ii. If leaks are successfully addressed/fixed continue to **step 45**
45.	**Prepare to Report System Ready to Linac Operator** \\ **Initials: _____ Date: _____ Time: _____** 45.1. Record temperature DU Target Cooling Water in and out before beam is put on target 45.2. Prepare to collect samples of pre-irradiation target solution 45.3. ☐ Enter desired flow rate _____ mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab (default this step is 300 mL/min) 45.4. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab i. **Ensure no cavitation occurs if motor is set above 50% motor power** 45.5. ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate A. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller 45.6. ☐ Verify ACID Pump to **RUN** 45.7. ☐ Verify ***DUMP TANK*** manual vent valve is ***OPEN*** 45.8. ☐ Collect samples of target solution pre-irradiation A. Press *{NEXT SAMPLE}* on **[Sample Collection]**.tab → **[Target Mixing]**.tab i. Wait a minimum of 90 sec. between samples B. Samples Collected (*all times LabVIEW computer time*)

Step	Action
	i. Fill in dot patterns for those loops to be collected ii. ▒ Loop 1 \\ Time: _____ Initials: _____ a. Comment: _____ iii. ▒ Loop 2 \\ Time: _____ Initials: _____ a. Comment: _____ iv. ▒ Loop 3 \\ Time: _____ Initials: _____ a. Comment: _____ v. ▒ Loop 4 \\ Time: _____ Initials: _____ a. Comment: _____ vi. ▒ Loop 5 \\ Time: _____ Initials: _____ a. Comment: _____ vii. ▒ Loop 6 \\ Time: _____ Initials: _____ a. Comment: _____ viii. ▒ Loop 7 \\ Time: _____ Initials: _____ a. Comment: _____ ix. ▒ Loop 8 \\ Time: _____ Initials: _____ a. Comment: _____ 45.9. At LabVIEW computer (211-D032) A. Start iSpy camera software  i. Two camera views should be immediately visible B. Two cameras are used to monitor the system during target solution irradiation i. Camera #0 – pointed at ACID pump controller ii. Camera #1 – pointed at leak sensor panel C. ☐ Verify Camera #0 is pointed at ACID pump controller D. ☐ Adjust Camera #0 until LCD of ACID pump controller is in view i. View should clearly see the LCD display E. ☐ Verify Camera #1 – pointed at leak sensor panel F. ☐ Adjust Camera #1 until leak sensor panel lights are in view i. View should clearly see all leak sensor panel lights

Step	Action
46.	**<u>Startup Remote Monitoring Computer (211-B121) and Access Downstairs</u>** 46.1. ☐ Use Remote Desktop to access downstairs Mo99 Recovery Process primary computer 46.2. ☐ Verify that Remote Desktop access is working A. If Remote desktop access is not working properly notify Mo99 Recovery Team Lead B. DO NOT proceed to line **46.3** until Remote Desktop access is established and stable 46.3. ☐ Report to Linac Operator that **<u>Cell 1 (D035)</u>** door ready to be closed
47.	**<u>Irradiation Started & Target Solution Mixing Sample Collection During Irradiation</u>** \\ **Initials: _____ Date: _____ Time: _____** 47.1. Energy A. All times are LabVIEW times B. Time 0 (_____): _____ MeV _____ kW C. Time (_____): _____ MeV _____ kW D. Time (_____): _____ MeV _____ kW E. Time (_____): _____ MeV _____ kW F. Time (_____): _____ MeV _____ kW G. Time (_____): _____ MeV _____ kW H. Time (_____): _____ MeV _____ kW I. Time (_____): _____ MeV _____ kW J. Time (_____): _____ MeV _____ kW K. Time END (_____): _____ MeV _____ kW 47.2. Monitor LINAC thermocouples and pressure for total time of irradiation – report any issues to LINAC operator 47.3. Target Solution Mixing Sample Collection During Irradiation A. Samples to be collected: ☐YES ☐NO *(if YES fill out information below)* B. Number of samples to collect [(max. of 8) – (no. of pre-irradiation samples taken, **45.8.B.i**)] = _____ C. Calculate time between samples (y) = _____ minutes i. $y = \left(\frac{Target\ Solution\ Volume\ (mL)}{Flow\ rate\ (\frac{mL}{min})}\right) \Big/ No.of\ samples$ ii. Target solution volume: _____ mL iii. Flow rate: _____ mL/min D. Press *{NEXT SAMPLE}* on **[Sample Collection]**.tab → **[Target Mixing]**.tab i. Start from empty loop after step **45.8.B**

Step	Action
	ii. Samples Collected (all times LabVIEW computer time) a. Fill in dot patterns for those loops to be collected b. ☐ Set timer to value of (y) from **47.3.C** c. ▒ Loop 1 (V-0032/0034) \\ Time: _____ Initials: _____ 1. Comment: _____ d. ▒ Loop 2 (V-0036/0038) \\ Time: _____ Initials: _____ 1. Comment: _____ e. ▒ Loop 3 (V-0040/0042) \\ Time: _____ Initials: _____ 1. Comment: _____ f. ▒ Loop 4 (V-0044/0046) \\ Time: _____ Initials: _____ 1. Comment: _____ g. ▒ Loop 5 (V-0048/0050) \\ Time: _____ Initials: _____ 1. Comment: _____ h. ▒ Loop 6 (V-0052/0054) \\ Time: _____ Initials: _____ 1. Comment: _____ i. ▒ Loop 7 (V-0056/0058) \\ Time: _____ Initials: _____ 1. Comment: _____ j. ▒ Loop 8 (V-0060/0062) \\ Time: _____ Initials: _____ 1. Comment: _____
48.	**Irradiation Stopped \\ Initials: _____ Date: _____ Time: _____**
49.	**Gas Collection Lines Purged \\ Initials: _____ Date: _____ Time: _____**
50.	**HP Tech Cleared D024/D032 \\ Initials: _____ Date: _____ Time: _____** 50.1. Close Remote Desktop session at Mo99 Recovery Process remote monitoring computer (211-B121) 50.2. Return to LabVIEW computer in D032
51.	**Emptying Target Solution Mixing Flowpath Back Into Target Vessel** **\\ Initials: _____ Date: _____ Time: _____** 51.1. ☐ ACID Pump to **STOP**

Step	Action
	51.2. Turn off flow meter bypass to isolate a sample in the bypass loop (valves V-0165/0166) 51.3. Record balance readings A. From LabVIEW or balance LCD display B. Feed balance: _____ grams C. Effluent balance: _____ grams 51.4. ☐ Enter desired flow rate _____ mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab (default this step is 167 mL/min) 51.5. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab 51.6. ☐ Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate A. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller 51.7. Press *{NEXT STEP}* 51.8. ☐ Verify Dial @ *Target Return Line Purge* A. Path reference information i. V-0171 – from surge vessel gas ii. V-0163/0164 – Acid Flow Meter iii. V-0009 – Mixing Path Sampling Assembly iv. Current target solution mixing sample loop a. One of the following pairs (mark correct pair): 1. ▒ Loop 1, V-0100/0102 or 2. ▒ Loop 2, V-0104/0106 or 3. ▒ Loop 3, V-0108/0110 or 4. ▒ Loop 4, V-0112/0114 or 5. ▒ Loop 5, V-0116/0118 or 6. ▒ Loop 6, V-0120/0122 or 7. ▒ Loop 7, V-0124/0126 or 8. ▒ Loop 8, V-0128/0130 or 9. ▒ Bypass, V-0132/0133 v. V-0151/0152 – to target vessel

Step	Action
	vi. Valve text: **171 > 163/164 > 9 > *{current target mixing sample loop}* > 151/152 > Target Vessel** 51.9. ☐ ACID Pump to **RUN** 51.10. ☐ Hold for approximately 5 minutes 51.11. ☐ Hold time ended 51.12. ☐ ACID Pump to **STOP** 51.13. Record balance readings A. From LabVIEW or balance LCD display B. Feed balance: _____ grams C. Effluent balance: _____ grams
52.	**Pre-Pre-Load Acid Wash \\ Initials: _____ Date: _____ Time: _____** 52.1. Primes lines to column and turns on acid solution pre-heater 52.2. ☐ ACID Pump to **STOP** 52.3. ☐ Enter desired flow rate _____ mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab (default this step is 84 mL/min) 52.4. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab 52.5. ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate A. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller 52.6. Press *{NEXT STEP}* A. ☐ Answer OK B. ☐ Answer OK C. ☐ Answer OK D. ☐ Press *{NEXT SAMPLE}* on **[Sample Collection]**.tab → **[Column Loading]**.tab 52.7. Verify Dial @ *Pre-Pre-Load Acid Wash* A. Path reference information i. V-0002 – Fresh Acid ii. V-0163/0164 – Acid Flow Meter iii. V-0010 – Acid Column Path iv. V-0014/0015 – Acid Column Bypass v. V-0018/0019 – Acid Column Post-Column Filter vi. Open Loop 1 on column loading sample collection – V-0032/0034

Step	Action
	vii. V-0139 – Acid Rinse viii. Valve text: **2 > 163/164 > 10 > 14/15 > 18/19 > 32/34 Loop 1 > 139 > Pre-Load Acid Wash** 52.8. ACID Pump to **RUN** 52.9. ☐ Hold for approximately 5 minutes 52.10. ☐ Verify effluent balance reading increasing @ **[Sensors]**.tab → **[Balances]**.tab 52.11. Activate Acid Pre-Heater A. ☐ Adjust Acid Pre-Heater ***OUTPUT POWER LEVEL*** control knob to 300 mL – 2 L setting B. ☐ Press red RESET button to turn Acid Pre-Heater orange ***OVER-TEMP*** light off 52.12. ☐ Hold time ended 52.13. ☐ ACID Pump to **STOP** 52.14. Record balance readings A. From LabVIEW or balance LCD display B. Feed balance: _____ grams C. Effluent balance: _____ grams
53.	**Pre-Load Acid Wash** \\ **Initials: _____ Date: _____ Time: _____** 53.1. ☐ Verify ACID Pump to **STOP** 53.2. ☐ Enter desired flow rate _____ mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab (default this step is 167 mL/min) 53.3. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab 53.4. ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate A. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller 53.5. Press *{NEXT STEP}* 53.6. Verify Dial @ *Pre-Load Acid Wash* A. Path reference information i. V-0002 – Fresh Acid ii. V-0163/0164 – Acid Flow Meter iii. V-0010 – Acid Column Path iv. V-0012/0013 – Acid Column Bottom Feed v. V-0016/0017 – Acid column Top Exit

Step	Action
	vi. V-0018/0019 – Acid Column Post-Column Filter vii. Current column loading sample loop – V-0032/0034 viii. V-0142 – Pre-Load Acid Wash ix. Valve text: **2 > 163/164 > 10 > 12/13 > 16/17 > 18/19 > *{current column loading sample loop}* > 142 > Pre-Load Acid Wash** 53.7. Activate Column Heater A. ☐ Adjust Column Heater ***OUTPUT POWER LEVEL*** control knob to 300 mL-2 L setting B. ☐ Press red RESET button to turn Column Heater orange ***OVER-TEMP*** light off 53.8. ☐ Hold for approximately: _____ seconds (**53.8.B x 60**) A. Read acid flow rate from **[Sensors]**.tab → **[Flow Meters]**.tab B. Calculate: _____ mL (from **44.15.A**) / _____ mL/min = _____ min. 53.9. ☐ ACID Pump to **RUN** 53.10. Samples to be collected: ☐YES ☐NO (*if YES fill out information below*) A. Press *{NEXT SAMPLE}* on **[Sample Collection]**.tab → **[Target Mixing]**.tab i. Samples Collected (all times LabVIEW computer time) a. Fill in dot patterns for those loops to be collected b. ▒ Loop 1 (V-0032/0034) \\ Time: _____ Initials: _____ 1. Comment: _____ c. ▒ Loop 2 (V-0036/0038) \\ Time: _____ Initials: _____ 1. Comment: _____ d. ▒ Loop 3 (V-0040/0042) \\ Time: _____ Initials: _____ 1. Comment: _____ e. ▒ Loop 4 (V-0044/0046) \\ Time: _____ Initials: _____ 1. Comment: _____ f. ▒ Loop 5 (V-0048/0050) \\ Time: _____ Initials: _____ 1. Comment: _____ g. ▒ Loop 6 (V-0052/0054) \\ Time: _____ Initials: _____ 1. Comment: _____ h. ▒ Loop 7 (V-0056/0058) \\ Time: _____ Initials: _____ 1. Comment: _____

Step	Action
	i. ☐ Loop 8 (V-0060/0062) \\ Time: _____ Initials: _____ **1.** Comment: _____ 53.11. ☐ Hold time ended 53.12. ☐ ACID Pump to **STOP** 53.13. Record balance readings A. From LabVIEW or balance LCD display B. Feed balance: _____ grams C. Effluent balance: _____ grams
54.	**Column Loading & Column Loading Sample Collection** \\ **Initials: _____ Date: _____ Time: _____** 54.1. ☐ Verify ACID Pump to **STOP** 54.2. ☐ Enter desired flow rate _____ mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab (default this step is 167 mL/min) 54.3. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab 54.4. ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate A. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller 54.5. Press *{NEXT STEP}* 54.6. Verify Dial @ *Column Loading* A. Path reference information i. V-0005 – Target Solution ii. V-0163/0164 – Acid Flow Meter iii. V-0010 – Acid Column Path iv. V-0012/0013 – Acid Column Bottom Feed v. V-0016/0017 – Acid column Top Exit vi. V-0018/0019 – Acid Column Post-Column Filter vii. Current column loading sample loop – V-0032/0034 viii. V-0145/0146 – Dump Tank ix. V-0147/0148 – Dump Tank Filter x. Valve text: **5 > 163/164 > 10 > 12/13 > 16/17 > 18/19 > *{current column loading sample loop}* > 145/146 > 147/148 > Dump Tank**

Step	Action
	54.7. Hold pump running for approximately: _____ seconds (**54.7.B x 60**)
	A. Read acid flow rate from **[Sensors]**.tab → **[Flow Meters]**.tab
	B. Calculate: _____ mL (from **44.16.A**) / _____ mL/min = _____ min.
	C. LabVIEW time = _____ + **54.7.B** = _____
	D. LabVIEW time = _____ + **54.7.B** – 5 min = _____
	54.8. ☐ Enter value from line **54.7.D** TO line **54.12**
	54.9. ☐ ACID Pump to **RUN**
	54.10. Monitor pressure reading at Post-ACID Pump Pressure Gauge
	A. Pressure should be ~4-6 psig @ 167 mL/min
	B. Monitor at **[System]**.tab - OR - Monitor at **[Sensors]**.tab → **[Pressures]**.tab
	54.11. Samples to be collected: ☐YES ☐NO (*if YES fill out information below*)
	A. Total sample loops = 8 (loops: V-0032/0034 to V-0060/0062)
	B. Loop 9 cannot be used to trap a sample as it is used to maintain a flow path through the sample assembly when all other samples have been collected
	C. Column loading sample collection calculations
	i. Target solution volume = _____ mL
	ii. Flow rate = _____ mL/min
	iii. Number of samples to collect (*max. of 8*) = _____
	iv. Time between samples (*y*) = _____ min
	a. $y = \left(\frac{Volume\ (mL)}{Flow\ rate\ (\frac{mL}{min})}\right) \Big/ No.of\ samples$
	D. ***All times LabVIEW computer time***
	E. Fill in dot patterns for those loops to be collected
	i. Start from empty loop after step **53.10.A.i.a**
	F. ☐ Verify flow path is to DUMP TANK before collecting samples – goes to pre-load acid wash before dump tank to purge line and not dilute target solution – post pump transducer drops to 1.8-1.9 psig when solution is basically gone from the target vessel – 17960 mL loading time was 101-102 min
	G. ▒ Loop 1 (V-0032/0034) \\ Time: _____ Initials: _____
	i. Comment: _____
	H. ▒ Loop 2 (V-0036/0038) \\ Time: _____ Initials: _____
	i. Comment: _____

Step	Action
	I. ☐ Loop 3 (V-0040/0042) \\ Time: _____ Initials: _____ i. Comment: _____ J. ☐ Loop 4 (V-0044/0046) \\ Time: _____ Initials: _____ i. Comment: _____ K. ☐ Loop 5 (V-0048/0050) \\ Time: _____ Initials: _____ i. Comment: _____ L. ☐ Loop 6 (V-0052/0054) \\ Time: _____ Initials: _____ i. Comment: _____ M. ☐ Loop 7 (V-0056/0058) \\ Time: _____ Initials: _____ i. Comment: _____ N. ☐ Loop 8 (V-0060/0062) \\ Time: _____ Initials: _____ i. Comment: _____ 54.12. ☐ @ LabVIEW time _____ (**54.7.D**) monitor pressure reading at Pre-ACID Pump Pressure Gauge A. LabVIEW time from STEP **54.7.D** 54.13. ☐ Hold time ended 54.14. ☐ ACID Pump to **STOP** 54.15. Record balance readings A. From LabVIEW or balance LCD display B. Feed balance: _____ grams C. Effluent balance: _____ grams
55.	**Post-Load Acid Wash** \\ **Initials: _____ Date: _____ Time: _____** 55.1. ☐ Verify ACID Pump to **STOP** 55.2. ☐ Enter desired flow rate _____ mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab (default this step is 167 mL/min) 55.3. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab 55.4. ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate A. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller 55.5. Press *{NEXT STEP}*

Step	Action
	55.6. Verify Dial @ *Post-Load Acid Wash* A. Path reference information i. V-0002 – Fresh Acid Feed ii. V-0163/0164 – Acid Flow Meter iii. V-0010 – Acid Column Path iv. V-0012/0013 – Acid Column Bottom Feed v. V-0016/0017 – Acid column Top Exit vi. V-0018/0019 – Acid Column Post-Column Filter vii. Current column loading sample loop – V-0032/0034 viii. V-0141 – Post-Load Acid Wash ix. Valve text: **2 > 163/164 > 10 > 12/13 > 16/17 > 18/19 > *{current column loading sample loop}* > 141 > Post-Load Acid Wash** 55.7. Hold pump running for approximately: _____ seconds (**55.7.B x 60**) A. Read acid flow rate from **[Sensors]**.tab → **[Flow Meters]**.tab B. Calculate: _____ mL (from **44.17.A**) / _____ mL/min = _____ min. 55.8. ☐ ACID Pump to **RUN** 55.9. Samples to be collected: ☐YES ☐NO (*if YES fill out information below*) A. Press *{NEXT SAMPLE}* on **[Sample Collection]**.tab → **[Column Loading (ACIDIC)]**.tab B. Fill in dot patterns for those loops to be collected i. Start data entry from last loop @ line **54.11** C. ☐ Verify flow path is to POST-LOAD ACID WASH bottle before collecting samples D. ▒ Loop 1 (V-0032/0034) \\ Time: _____ Initials: _____ i. Comment: _____ E. ▒ Loop 2 (V-0036/0038) \\ Time: _____ Initials: _____ i. Comment: _____ F. ▒ Loop 3 (V-0040/0042) \\ Time: _____ Initials: _____ i. Comment: _____ G. ▒ Loop 4 (V-0044/0046) \\ Time: _____ Initials: _____ i. Comment: _____ H. ▒ Loop 5 (V-0048/0050) \\ Time: _____ Initials: _____ i. Comment: _____

Step	Action
	I. ☐ Loop 6 (V-0052/0054) \\ Time: _____ Initials: _____ i. Comment: _____ J. ☐ Loop 7 (V-0056/0058) \\ Time: _____ Initials: _____ i. Comment: _____ K. ☐ Loop 8 (V-0060/0062) \\ Time: _____ Initials: _____ i. Comment: _____ 55.10. ☐ Hold time ended 55.11. ☐ ACID Pump to **STOP** 55.12. Record balance readings A. From LabVIEW or balance LCD display B. Feed balance: _____ grams C. Effluent balance: _____ grams
56.	**Post-Load H_2O Wash \\ Initials: _____ Date: _____ Time: _____** 56.1. ☐ Verify ACID Pump to **STOP** 56.2. ☐ Enter desired flow rate _____ mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab (default this step is 167 mL/min) 56.3. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab 56.4. ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate A. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller 56.5. Press *{NEXT STEP}* 56.6. Verify Dial @ *Post-Load H_2O Wash* A. Path reference information i. V-0001 – Fresh H_2O ii. V-0163/0164 – Acid Flow Meter iii. V-0010 – Acid Column Path iv. V-0012/0013 – Acid Column Bottom Feed v. V-0016/0017 – Acid column Top Exit vi. V-0018/0019 – Acid Column Post-Column Filter vii. Current column loading sample loop – V-0032/0034 viii. V-0140 – Post-Load Water Wash

Step	Action
	ix. Valve text: **1 > 163/164 > 10 > 12/13 > 16/17 > 18/19 > *{current column loading sample loop}* > 140 > Post-Load H_2O Wash**
	56.7. ☐ Hold pump running for approximately: _____ seconds (**56.7.B x 60**)
	A. Read acid flow rate from **[Sensors]**.tab → **[Flow Meters]**.tab
	B. Calculate: _____ mL (from **44.18.A**) / _____ mL/min = _____ min.
	56.8. Pump to **RUN**
	56.9. Collect samples
	A. Samples to be collected: ☐YES ☐NO (*if YES fill out information below*)
	B. Press *{NEXT SAMPLE}* on **[Sample Collection]**.tab → **[Column Loading (ACIDIC)]**.tab
	C. Fill in dot patterns for those loops to be collected
	i. Start data entry from last loop @ line **55.9.B**
	D. ☐ Verify flow path is to POST-LOAD H_2O WASH bottle before collecting samples
	E. ▒ Loop 1 (V-0032/0034) \\ Time: _____ Initials: _____
	i. Comment: _____
	F. ▒ Loop 2 (V-0036/0038) \\ Time: _____ Initials: _____
	i. Comment: _____
	G. ▒ Loop 3 (V-0040/0042) \\ Time: _____ Initials: _____
	i. Comment: _____
	H. ▒ Loop 4 (V-0044/0046) \\ Time: _____ Initials: _____
	i. Comment: _____
	I. ▒ Loop 5 (V-0048/0050) \\ Time: _____ Initials: _____
	i. Comment: _____
	J. ▒ Loop 6 (V-0052/0054) \\ Time: _____ Initials: _____
	i. Comment: _____
	K. ▒ Loop 7 (V-0056/0058) \\ Time: _____ Initials: _____
	i. Comment: _____
	L. ▒ Loop 8 (V-0060/0062) \\ Time: _____ Initials: _____
	i. Comment: _____
	56.10. ☐ Hold time ended
	56.11. ☐ ACID Pump to **STOP**

Step	Action
	56.12. Record balance readings A. From LabVIEW or balance LCD display B. Feed balance: _____ grams C. Effluent balance: _____ grams 56.13. ☐ ACID Pump power to **OFF** A. Rocker switch under front/left of ACID Pump V300 controller 56.14. ☐ Acid Pre-Heater ***OUTPUT POWER LEVEL*** knob from **300 mL – 2 L** → TO → **OFF**
57.	**Activate Base Pre-heater** \\ **Initials: _____ Date: _____ Time: _____** 57.1. ☐ BASE Pump controller power to **ON** using rocker switch under front/left of BASE Pump V300 controller 57.2. ☐ BASE Pump to **STOP** 57.3. Record Recovery System balance readings A. From LabVIEW or balance LCD display B. Feed balance: _____ grams C. Effluent balance: _____ grams 57.4. Record 3-L/5-neck flask balance reading (D024 Hot Cell): _____ grams 57.5. ☐ Enter desired flow rate _____ mL/min *Base Flow Rate Set Pt* @ **[System]**.tab (default this step is 84 mL/min) 57.6. ☐ Record calculated _____ *% Base Motor Power* @ **[System]**.tab 57.7. ☐ Verify/Adjust BASE Pump Controller to *% Base Motor Power* for desired flow rate A. If a lower % motor power value is required due to pressure readings adjust *Base Flow Rate Set Pt* until calculated *% Base Motor Power* matches % motor power reading at controller 57.8. Press *{NEXT STEP}* A. ☐ Answer OK B. ☐ Answer OK C. ☐ Answer OK D. ☐ Press *{NEXT SAMPLE}* on **[Sample Collection]**.tab → **[Column Stripping]**.tab 57.9. Verify Dial @ *Post-Load NaOH Wash* A. Path reference information i. V-0007 – NaOH Wash

Step	Action
	ii. V-0167/168 – Base Flow Meter iii. V-0024/25 – Base Column Bypass iv. V-0028/29 – Base Column Post-Column Filter v. Open Loop 1 on column stripping sample collection – V-0066/0068 vi. V-0138 – NaOH Wash vii. Valve text: **7 > 167/168 > 24/25 > 28/29 > 66/68 Loop 1 > 138 > Post-Load NaOH Wash** 57.10. BASE Pump to **RUN** 57.11. ☐ Verify effluent balance reading increasing **[Sensors]**.tab → **[Balance]**.tab 57.12. Adjust BASE Pre-heater settings A. ☐ Base Pre-Heater to ***OUTPUT POWER LEVEL*** knob to 300 mL – 2 L B. ☐ Press red RESET button to turn Base Pre-Heater orange ***OVER-TEMP*** light off 57.13. ☐ Confirm Column Heater ***OUTPUT POWER LEVEL*** knob to 300 mL-2L 57.14. ☐ Hold for approximately 5 minutes 57.15. ☐ Hold time ended 57.16. ☐ BASE pump to **STOP** 57.17. Record balance readings A. From LabVIEW or balance LCD display B. Feed balance: _____ grams C. Effluent balance: _____ grams 57.18. Record 3-L/5-neck flask balance reading (D024 Hot Cell): _____ grams
58.	**Post-Load NaOH Wash** \\ **Initials: _____ Date: _____ Time: _____** 58.1. Select one of the following: A. ☐ Operate → continue to **58.2** **OR** B. ☐ Skipped → continue to **59.1** 58.2. ☐ Verify BASE Pump to **STOP** 58.3. ☐ Enter desired flow rate _____ mL/min *Base Flow Rate Set Pt* @ **[System]**.tab (default this step is 84 mL/min) 58.4. ☐ Record calculated _____ *% Base Motor Power* @ **[System]**.tab 58.5. ☐ Verify/Adjust BASE Pump Controller to *% Base Motor Power* for desired flow rate

Step	Action
	A. If a lower % motor power value is required due to pressure readings adjust *Base Flow Rate Set Pt* until calculated *% Base Motor Power* matches % motor power reading at controller 58.6. Press *{NEXT STEP}* 58.7. Verify Dial @ *Post-Load NaOH Wash* A. Path reference information i. V-0007 – NaOH Wash ii. V-0167/168 – Base Flow Meter iii. V-0022/23 – Base Column Bottom Feed iv. V-0026/27 – Base column Top Exit v. V-0028/29 – Base Column Post-Column Filter vi. Open Loop 1 on column stripping sample collection – V-0066/0068 vii. V-0138 – NaOH Wash viii. Valve text: **7 > 167/168 > 22/23 > 27/26 > 28/29 > 66/68 Loop 1 > 138 > Post-Load NaOH Wash** 58.8. Hold running for approximately: _____ seconds (**58.8.B x 60**) A. Read base flow rate from **[Sensors]**.tab → **[Flow Meters]**.tab B. Calculate: _____ mL (from **44.19.B**) / _____ mL/min = _____ min. 58.9. ☐ BASE Pump to **RUN** 58.10. Samples to be collected: ☐YES ☐NO (*if YES fill out information below*) A. Total loops = 9 (loops: V-0066/0068– V-0098/99) B. Total sample loops = 8 (loops: V-0066/0068– V-0094/96) C. Loop 8 cannot be used to trap a sample as it is used to maintain a flow path through the sample assembly when all other samples have been collected D. NaOH Wash = _____ no. samples E. **Total = _____ no. samples (*max. of 8*)** F. NaOH Wash sample collection calculations i. $NaOH/NH_4OH$ strip volume = _____ mL ii. Flow rate = _____ mL/min iii. Time to deliver volume = _____ min iv. Number of samples to collect (*max. of 7*) = _____ v. Time between samples (*y*) = _____ min

Step	Action
	a. $y = \left(\frac{Volume\ (mL)}{Flow\ rate\ (\frac{mL}{min})}\right) \Big/ No.of\ samples$ G. Press *{NEXT SAMPLE}* on **[Sample Collection]**.tab → **[Column Stripping (BASIC)]**.tab i. ***All times LabVIEW computer time*** ii. Fill in dot patterns for those loops to be collected iii. ☐ Verify flow path is to POST-LOAD NaOH WASH bottle before collecting samples iv. ⁞⁞ Loop 1 (V-0066/0068) \\ Time: _____ Initials: _____ a. Comment: _____ v. ⁞⁞ Loop 2 (V-0070/0072) \\ Time: _____ Initials: _____ a. Comment: _____ vi. ⁞⁞ Loop 3 (V-0074/0076) \\ Time: _____ Initials: _____ a. Comment: _____ vii. ⁞⁞ Loop 4 (V-0078/0080) \\ Time: _____ Initials: _____ a. Comment: _____ viii. ⁞⁞ Loop 5 (V-0082/0084) \\ Time: _____ Initials: _____ a. Comment: _____ ix. ⁞⁞ Loop 6 (V-0086/0088) \\ Time: _____ Initials: _____ a. Comment: _____ x. ⁞⁞ Loop 7 (V-0090/0092) \\ Time: _____ Initials: _____ a. Comment: _____ xi. ⁞⁞ Loop 8 (V-0094/0096) \\ Time: _____ Initials: _____ a. Comment: _____ 58.11. ☐ Hold time ended 58.12. ☐ BASE Pump to **STOP** 58.13. Record balance readings A. From LabVIEW or balance LCD display B. Feed balance: _____ grams

Step	Action
	C. Effluent balance: _____ grams 58.14. Record 3-L/5-neck flask balance reading (D024 Hot Cell): _____ grams
59.	**<u>Column Stripping</u> \\ Initials: _____ Date: _____ Time: _____** 59.1. ☐ Verify BASE Pump to **<u>STOP</u>** 59.2. ☐ Record feed balance value: _____ grams 59.3. ☐ At **[Solutions]**.tab copy strip mass from *{proc06_column strip mass}*: _____ grams 59.4. ☐ Calculate strip end feed balance value (**59.2 – 59.3**): _____ grams 59.5. OPTIONAL - To double check the strip mass value A. At **[Solutions]**.tab copy strip volume (LabVIEW label: proc06_column strip vol 2) : _____ mL i. Should match line **44.20.A** on page **60** B. At **[System]**.tab Copy strip density from (Base Strip Density): _____ g/mL (or g/cc) i. Should match line **44.10.A** on page **59** C. Calculate: **59.5.A** x **59.5.B** = _____ grams i. Should match line **59.3** 59.6. Verify Strip flow path (check only ONE) A. ☐ To TRANSFER CASK **OR** B. ☐ To HOT CELL i. ☐ Record Hot Cell balance value: _____ grams ii. Calculate Hot Cell receipt vessel strip end mass (**59.6.B.i + 59.3**): _____ grams 59.7. ☐ Enter desired flow rate _____ mL/min *Base Flow Rate Set Pt* @ **[System]**.tab (default this step is 84 mL/min) 59.8. ☐ Record calculated _____ *% Base Motor Power* @ **[System]**.tab 59.9. ☐ Verify/Adjust BASE Pump Controller to *% Base Motor Power* for desired flow rate A. If a lower % motor power value is required due to pressure readings adjust *Base Flow Rate Set Pt* until calculated *% Base Motor Power* matches % motor power reading at controller 59.10. Press *{NEXT STEP}*

Step	Action
	59.11. Verify Dial @ *Hydroxide strip* A. Path reference information i. V-0008 – NaOH Strip ii. V-0167/168 – Base Flow Meter iii. V-0022/23 – Base Column Bottom Feed iv. V-0026/27 – Base column Top Exit v. V-0028/29 – Base Column Post-Column Filter vi. Current column stripping sample loop – V-0066/0068 vii. V-0136 – Strip to Transfer Cask viii. Valve text: **8 > 167/168 > 22/23 > 27/26 > 28/29 > *{current column stripping sample loop}* > 136 > CASK (or HOT CELL)** 59.12. Hold pump running for approximately: **_____** seconds (**59.12.B x 60**) A. Read balance base flow rate @ **[Sensors]**.tab → **[Flow Meters]**.tab B. Calculate: _____ mL (from **44.20.A**) / _____ mL/min = _____ min. 59.13. ☐ BASE Pump to **RUN** 59.14. Samples to be collected: ☐YES ☐NO (*if YES fill out information below*) A. Total loops = 9 (loops: V-0066/0068– V-00098/99) B. Total sample loops = 8 (loops: V-0066/0068– V-00094/96) C. Loop 8 cannot be used to trap a sample as it is used to maintain a flow path through the sample assembly when all other samples have been collected D. NaOH Strip = **_____** no. samples E. **Total = _____ no. samples** (***max. of 8***) F. NaOH strip sample collection calculations i. NaOH strip volume = _____ mL ii. Flow rate = **_____** mL/min iii. Time to deliver volume = **_____** min iv. Number of samples to collect (*max. of 8*) = **_____** v. Time between samples (y) = **_____** min a. $y = \left(\frac{Volume\ (mL)}{Flow\ rate\ (\frac{mL}{min})}\right) \Big/ No.of\ samples$ G. Press *{NEXT SAMPLE}* on **[Sample Collection]**.tab → **[Column Stripping (BASIC)]**.tab i. Start data entry from last loop @ line **58.9**

Step	Action
	ii. ***All times LabVIEW computer time***
	iii. ☐ Verify flow path is to correct STRIP path before collecting samples
	iv. Fill in dot patterns for those loops to be collected
	v. ⁘ Loop 1 (V-0066/0068) \\ Time: _____ Initials: _____
	a. Comment: _____
	vi. ⁘ Loop 2 (V-0070/0072) \\ Time: _____ Initials: _____
	a. Comment: _____
	vii. ⁘ Loop 3 (V-0074/0076) \\ Time: _____ Initials: _____
	a. Comment: _____
	viii. ⁘ Loop 4 (V-0078/0080) \\ Time: _____ Initials: _____
	a. Comment: _____
	ix. ⁘ Loop 5 (V-0082/0084) \\ Time: _____ Initials: _____
	a. Comment: _____
	x. ⁘ Loop 6 (V-0086/0088) \\ Time: _____ Initials: _____
	a. Comment: _____
	xi. ⁘ Loop 7 (V-0090/0092) \\ Time: _____ Initials: _____
	a. Comment: _____
	xii. ⁘ Loop 8 (V-0094/0096) \\ Time: _____ Initials: _____
	a. Comment: _____
	59.15. ☐ Hold time ended
	59.16. ☐ BASE Pump to **STOP**
	59.17. Record balance readings
	A. From LabVIEW or balance LCD display
	B. Feed balance: _____ grams
	C. Effluent balance: _____ grams
	59.18. Record 3-L/5-neck flask balance reading (D024 Hot Cell): _____ grams
60.	**Post-Strip H_2O Wash \\ Initials: _____ Date: _____ Time: _____**
	60.1. ☐ Verify BASE Pump to **STOP**
	60.2. ☐ Enter desired flow rate _____ mL/min *Base Flow Rate Set Pt* @ **[System]**.tab (default this step is 84 mL/min)

Step	Action
	60.3. ☐ Record calculated _____ *% Base Motor Power* @ **[System]**.tab 60.4. ☐ Verify/Adjust BASE Pump Controller to *% Base Motor Power* for desired flow rate A. If a lower % motor power value is required due to pressure readings adjust *Base Flow Rate Set Pt* until calculated *% Base Motor Power* matches % motor power reading at controller 60.5. Press *{NEXT STEP}* 60.6. Verify Dial @ *Post-Strip H_2O Wash* A. Path reference information i. V-0006 – Fresh H_2O ii. V-0167/168 – Base Flow Meter iii. V-0022/23 – Base Column Bottom Feed iv. V-0026/27 – Base column Top Exit v. V-0028/29 – Base Column Post-Column Filter vi. Current column loading sample loop – V-0066/0068 vii. V-0135 – Post-Strip H_2O Wash viii. Valve text: **6 > 167/168 > 22/23 > 27/26 > 28/29 > *{current column stripping sample loop}* > 135 > Post-Strip H_2O Wash** 60.7. HOLD TIME (pick one: **60.7.A *OR* 60.7.B**) A. ☐ STRIP to HOT CELL i. ☐ H_2O to HOT CELL hold for approximately: _____ minutes a. Read balance base flow rate @ **[Sensors]**.tab → **[Flow Meters]**.tab b. Calculate: _____ mL (from **44.21.A.i**) / _____ mL/min = _____ min. ii. ☐ H_2O to WASTE BOTTLE hold for approximately: _____ minutes a. Read base flow rate from **[Sensors]**.tab → **[Flow Meters]**.tab b. Calculate: _____ mL (from **44.21.B.i**) / _____ mL/min = _____ min. 1. Portion of H_2O used to flush transfer line **OR** B. ☐ STRIP to TRANSPORT CASK i. ☐ H_2O to WASTE BOTTLE hold for approximately: _____ minutes a. Read balance base flow rate @ **[Sensors]**.tab → **[Flow Meters]**.tab

Step	Action
	b. Calculate: _____ mL (from **44.21.A.i + 44.21.B.i**) / _____ mL/min = _____ min.
	60.8. ☐ BASE Pump to **RUN**
	60.9. Samples to be collected: ☐YES ☐NO (*if YES fill out information below*)
	A. Press *{NEXT SAMPLE}* on **[Sample Collection]**.tab → **[Column Stripping (BASIC)]**.tab
	i. Start data entry from last loop @ line **59.13**
	ii. ***All times LabVIEW computer time***
	iii. ☐ Verify flow path is to POST-STRIP H_2O WASH bottle before collecting samples – going to bottle comes after the timer
	iv. Fill in dot patterns for those loops to be collected
	v. ⁘ Loop 1 (V-0066/0068) \\ Time: _____ Initials: _____
	a. Comment: _____
	vi. ⁘ Loop 2 (V-0070/0072) \\ Time: _____ Initials: _____
	a. Comment: _____
	vii. ⁘ Loop 3 (V-0074/0076) \\ Time: _____ Initials: _____
	a. Comment: _____
	viii. ⁘ Loop 4 (V-0078/0080) \\ Time: _____ Initials: _____
	a. Comment: _____
	ix. ⁘ Loop 5 (V-0082/0084) \\ Time: _____ Initials: _____
	a. Comment: _____
	x. ⁘ Loop 6 (V-0086/0088) \\ Time: _____ Initials: _____
	a. Comment: _____
	xi. ⁘ Loop 7 (V-0090/0092) \\ Time: _____ Initials: _____
	a. Comment: _____
	xii. ⁘ Loop 8 (V-0094/0096) \\ Time: _____ Initials: _____
	a. Comment: _____
	60.10. ☐ Hold time ended
	60.11. ☐ BASE pump to **STOP**
	60.12. Record balance readings
	A. From LabVIEW or balance LCD display

Step	Action
	B. Feed balance: _____ grams C. Effluent balance: _____ grams 60.13. Record 3-L/5-neck flask balance reading (D024 Hot Cell): _____ grams 60.14. ☐ BASE Pre-Heater ***OUTPUT POWER LEVEL*** knob from **300 mL – 2 L** → TO → **OFF** 60.15. ☐ Column Heater ***OUTPUT POWER LEVEL*** knob from **> 2 L** → TO → **OFF** 60.16. **Turn OFF electrical box on side of computer area for heaters**
61.	**Final Base Wash** \\ **Initials: _____ Date: _____ Time: _____** 61.1. Washing BASE flow path through: A. Column bypass B. Column stripping sampling assembly last loop C. To Base Rinse 61.2. ☐ BASE Pump to **STOP** 61.3. Record balance readings A. From LabVIEW or balance LCD display B. Feed balance: _____ grams C. Effluent balance: _____ grams 61.4. Record 3-L/5-neck flask balance reading (D024 Hot Cell): _____ grams A. Verifying final mass of solution delivered to D024 Hot Cell 3-L/5-neck flask 61.5. ☐ Enter desired flow rate _____ mL/min *Base Flow Rate Set Pt* @ **[System]**.tab (default this step is 84 mL/min) 61.6. ☐ Record calculated _____ *% Base Motor Power* @ **[System]**.tab 61.7. ☐ Verify/Adjust BASE Pump Controller to *% Base Motor Power* for desired flow rate A. If a lower % motor power value is required due to pressure readings adjust *Base Flow Rate Set Pt* until calculated *% Base Motor Power* matches % motor power reading at controller 61.8. Press *{NEXT STEP}* 61.9. Verify Dial @ *Final Base Wash* A. Path reference information i. V-0006 – Fresh H_2O ii. V-0167/168 – Base Flow Meter iii. V-0024/25 – Base Column Bypass

Step	Action
	iv. V-0028/29 – Base Column Post-Column Filter v. Current column stripping sample loop – V-00098/99 vi. V-0134 – Base Rinse vii. Valve text: **6 > 167/168 > 24/25 > 28/29 > *{current column stripping sample loop}* > 134 > Base Rinse** 61.10. ☐ Hold pump running for approximately: _____ seconds (**61.10.B x 60**) A. Read base flow rate from **[Sensors]**.tab → **[Flow Meters]**.tab B. Calculate: _____ mL (from **44.22.A**) / _____ mL/min = _____ min. 61.11. ☐ BASE Pump to **RUN** 61.12. ☐ Hold time ended 61.13. ☐ BASE Pump to **STOP** 61.14. Record balance readings A. From LabVIEW or balance LCD display B. Feed balance: _____ grams C. Effluent balance: _____ grams 61.15. ☐ BASE Pump controller power OFF using rocker switch under front/left of BASE Pump V300 controller
62.	**Final Acid Wash \\ Initials: _____ Date: _____ Time: _____** 62.1. Washing ACID flow path through: A. Column bypass B. Column loading sampling assembly last loop C. To Acid Rinse 62.2. ☐ ACID Pump controller power ON using rocker switch under front/left of ACID Pump V300 controller 62.3. ☐ ACID Pump to **STOP** 62.4. ☐ Enter desired flow rate _____ mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab (default this step is 84 mL/min) 62.5. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab 62.6. ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate A. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller 62.7. Press *{NEXT STEP}*

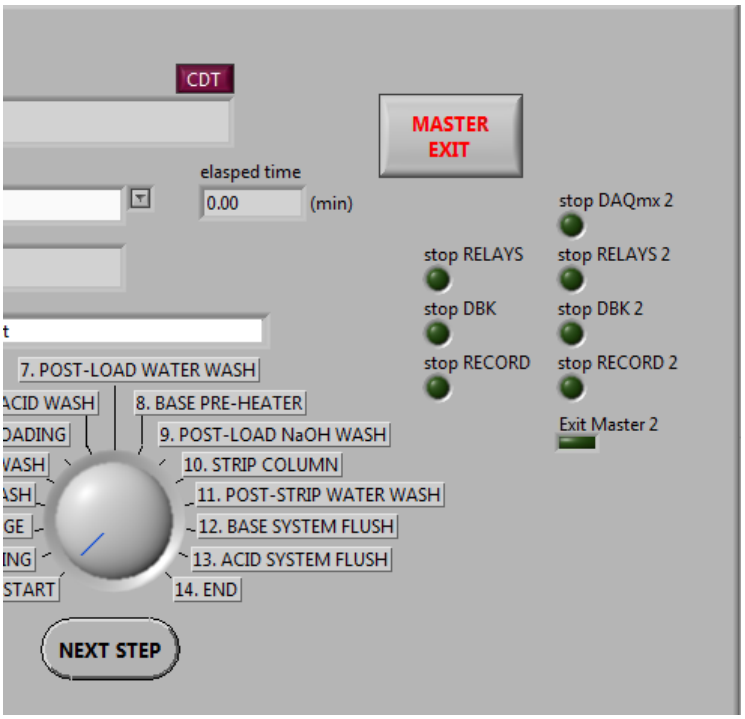
Step	Action
	62.8. Verify Dial @ *Final Acid Wash* A. Path reference information i. V-0002 – Fresh Acid ii. V-0163/0164 – Acid Flow Meter iii. V-0014/0015 – Acid Column Bypass iv. V-0018/0019 – Acid Column Post-Column Filter v. Current column loading sample loop – V-0064/0065 vi. V-0139 – Acid Rinse vii. Valve text: **2 > 163/164 > 14/15 > 18/19 > *{current column loading sample loop}* > 139 > Acid Rinse** 62.9. ☐ Hold for approximately: _____ seconds (**62.9.B x 60**) A. Read acid flow rate from **[Sensors]**.tab → **[Flow Meters]**.tab B. Calculate: _____ mL (from **44.23.A**) / _____ mL/min = _____ min. 62.10. ☐ ACID Pump to **RUN** 62.11. ☐ Hold time ended 62.12. ☐ ACID Pump to **STOP** 62.13. Record balance readings A. From LabVIEW or balance LCD display B. Feed balance: _____ grams C. Effluent balance: _____ grams 62.14. ☐ ACID Pump controller power OFF A. Rocker switch under front/left of ACID Pump V300 controller
63.	**Calculate Feeds Delivered and Effluents Received** 63.1. Complete entries in the following table 63.2. Enter value of balance reading from indicated step 63.3. Calculate difference and record in space provided 63.4. Refer to this table when conducting washout work instructions

	Feed Balance	Effluent Balance	D024 Balance	Feed Used
Emptying Target Path	_____ - _____ = _____ 51.13.B 51.3.B	_____ - _____ = _____x,y 51.13.C 51.3.C	Not Applicable	Acid Feed Used (sum a) _____
Pre-Pre Load Acid Wash	_____ - _____ = _____a 52.14.B 51.13.B	_____ - _____ = _____x 52.14.C 51.13.C	Not Applicable	Base Wash Used (sum b) _____
Pre-Load Acid Wash	_____ - _____ = _____a 53.13.B 52.14.B	_____ - _____ = _____ 53.13.C 52.14.C	Not Applicable	Base Strip Used (sum c) _____
Column Loading	_____ - _____ = _____ 54.15.B 53.13.B	_____ - _____ = _____ 54.15.C 53.13.C	Not Applicable	Water Wash Used (sum d) _____
Post-Load Acid Wash	_____ - _____ = _____a 55.12.B 54.15.B	_____ - _____ = _____ 55.12.C 54.15.C	Not Applicable	
Post-Load H2O Wash	_____ - _____ = _____d 56.12.B 55.12.B	_____ - _____ = _____ 56.12.C 55.12.C	Not Applicable	Acid Rinse Rec. V. Left _____ (8000 – sum x)
Base Heater On	_____ - _____ = _____b 57.17.B 57.3.B	_____ - _____ = _____ 57.17.C 57.3.C	_____ - _____ = _____ 57.18 57.4	Base Rinse Rec. V. Left _____(8000 – sum y)
Post-Load NaOH Wash	_____ - _____ = _____b 58.13.B 57.17.B	_____ - _____ = _____ 58.13.C 57.17.C	_____ - _____ = _____ 58.14 57.18	
Column Strip	_____ - _____ = _____c 59.17.B 58.13.B	_____ - _____ = _____ 59.17.C 58.13.C	_____ - _____ = _____ 59.18 58.14	
Post-Strip H2O Wash	_____ - _____ = _____d 60.12.B 59.17.B	_____ - _____ = _____ 60.12.C 59.17.C	_____ - _____ = _____ 60.13 59.18	
Base System Wash	_____ - _____ = _____d 61.14.B 60.12.B	_____ - _____ = _____y 61.14.C 60.12.C	Not Applicable	
Acid System Wash	_____ - _____ = _____a 62.13.B 61.14.B	_____ - _____ = _____x 62.13.C 61.14.C	Not Applicable	

Values can also be obtained from data file.

Step	Action
64.	☐ **Turn Off the Relays to The Glovebox Heaters**
65.	☐ **Hang Reminder Sign to LabVIEW Rack to Turn OFF Manual Dump Tank Valve** 65.1. After sign is posted continue to **step 66** (if the manual dump tank valve cannot be accessed yet, proceed to **step 67**)
66.	**Closing Manual Dump Tank Valve** ### SYSTEMS INTERFACE STEP ### 66.1. When D035 released to personnel 66.2. Contact a Gas Analysis/Collection team member 66.3. **At the Dump Tank (211-D035)** A. ☐ Close manual dump tank valve B. Recovery team member // Name: _____PRINT Initials: _____ Date: _____ Time: _____ C. Gas Analysis/Collection team member // Name: _____PRINT Initials: _____ Date: _____ Time: _____ D. Recovery personnel continue to step **67**
67.	**End** \\ **Initials: _____ Date: _____ Time: _____** 67.1. ☐ Confirm ACID Pump controller powered OFF 67.2. ☐ Confirm BASE Pump controller powered OFF 67.3. Press *{NEXT STEP}* 67.4. Verify Dial @ *End*
68.	**End of Run** \\ **Initials: _____ Date: _____ Time: _____** 68.1. ☐ Enter any final comments 68.2. ☐ Press the **MASTER EXIT** button at **[System]**.tab A. Properly stops Mo99 Remote Recovery Data Acquisition & Control Software

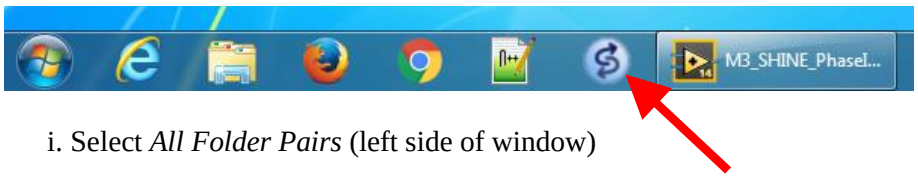
Step	Action



68.3. ☐ Data saved to USB stick \\ **Initials: _____ Date: _____ Time: _____**

A. Insert USB stick into computer

B. Use SyncToy 2.1 to backup files



i. Select *All Folder Pairs* (left side of window)

ii. ONLY check-mark the following items (in the *Active* column)

a. C_to_USB_Labuser_docs

b. C_to_USB_Public_docs

c. C_to_USB_temp1

iii. Press the **Run All** button (bottom right corner of window)

iv. Wait for backup to complete

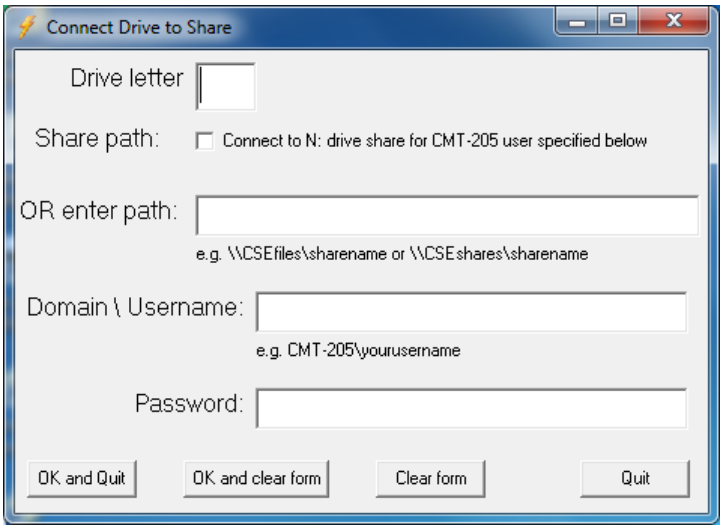
Step	Action
	C. Left click the *Safely Remove Hardware* icon and select ***Safely Remove G: drive***

68.4. ☐ Data saved to GTRI Mo99 Production Tests Share Drive

\\ Initials: _____ Date: _____ Time: _____

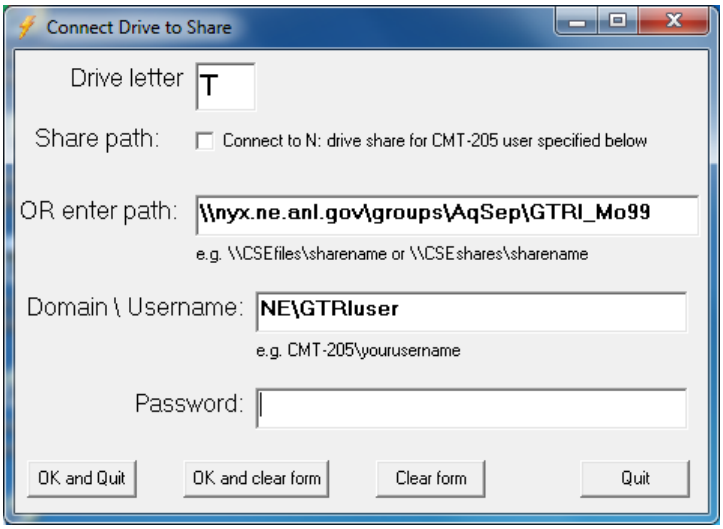
A. Open Connect Share

i. At "Drive Letter" enter T



ii. Then click the "Password" field

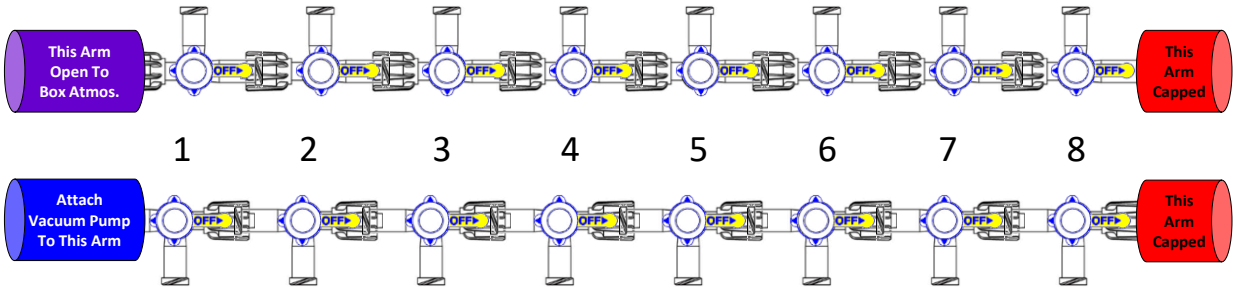
a. The screen will change to

Step	Action
	 iii. In the "Password" field type: Gtri#fy17q3 iv. Press the "OK and Quit" button v. The message "T drive is connected." should appear, click **OK** B. Use SyncToy 2.1 to backup files  i. Select *All Folder Pairs* (left side of window) ii. **ONLY** check-mark the following items (in the *Active* column) a. C_to_Tshare_labuser b. C_to_Tshare_public c. C_to_Tshare_temp1 iii. Press the **Run All** button (bottom right corner of window) iv. Wait for backup to complete C. Open Windows Explorer (folder view) D. Right click the T: drive and select ***Disconnect***
69.	☐ **Verify Step 66 Has Been Completed**

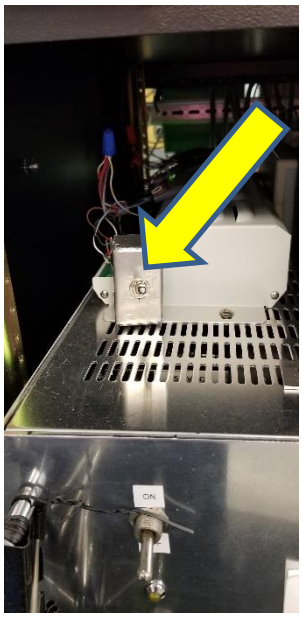
3.2.5 ^{99}Mo Recovery Sample Retrieval

Step	Action
70.	**Check Most Current RWP** 70.1. ☐ Sign most current RWP 70.2. ☐ Check most current RWP for PPE requirements Obtain Production Feeds Analysis and Process Conditions Summary Sheet
71.	**It is Recommended that Two People Retrieve Samples** 71.1. Person A collects the samples in the Mo Recovery glovebox 71.2. Person B actuates the solenoid valves via computer and double checks vial labels prior to retrieving each sample
72.	**Prepare the Following Items** 72.1. Up to 24 labeled 10 mL pre-evacuated vials (number should be ≥ # of samples taken). Part number can be found in Exhibit B A. ☐ Vials for Target Solution Mixing B. ☐ Vials for Column Loading C. ☐ Vials for Column Stripping D. Labels should have the following information i. RMS number ii. Lab notebook number and page number(s) iii. Date of irradiation iv. Date samples retrieved v. Process step identification vi. Approximate solution composition vii. Valve identification 72.2. ☐ Twenty-four (24x) vial holders with covers A. Tungsten holders i. ☐ Set of eight for Target Solution samples B. 304 stainless steel i. ☐ Set of eight for Column Loading samples ii. ☐ Set of eight for Column Stripping samples

Step	Action
	C. **ALL HOLDERS MUST HAVE COVERS THAT STAY SEATED WITH THE TWO PINS ON SHIELD BODY – IF THE PINS ARE BROKEN DO NOT USE THAT SHIELD BODY**
73.	**Materials at the Ready** 73.1. Parts for the sampling retrieval assemblies can be found in Exhibit B
74.	**Staging Collection Vials with Vial Shields** 74.1. The following operational checklist uses the Target Solution Sampling Assembly as an example 74.2. **YOU WILL BE PASSING THROUGH THESE STEPS THREE TIMES** A. First pass (Target Solution samples) mark the black boxes ☐ B. Second pass (Column Loading samples) mark the red boxes ☐ C. Third pass (Column Stripping samples) mark the blue boxes ☐ 74.3. At the Mo Recovery Glovebox A. Stage eight (8x) TUNGSTEN shielded vial holders (may not be possible to stage all 24 vials at one time i. Use 316 STAINLESS STEEL vial holders for Column Loading and Column Stripping samples 74.4. ☐ ☐ ☐ Remove the cover from the vial shield body 74.5. ☐ ☐ ☐ Insert the individual appropriately numbered concentric needle assembly into the appropriately numbered individual evacuated vial 74.6. ☐ ☐ ☐ Leave the vials in the vial holders when inserting the needles to minimize risk of glove puncture 74.7. ☐ ☐ ☐ Set the 3-way Luer Lock valves into the following position A. Top-row: all handles pointing to the right i. Check valves are only on the top row B. Bottom row: all handles pointing to the right

Step	Action
	VENT LINES TO INDIVIDUAL SAMPLING LOOP SOLENOID VENT VALVES (check valves not shown)  VENT LINES FROM CONCENTRIC NEEDLE ASSEMBLIES
75.	**<u>Retrieving Samples</u>** 75.1. The following operational checklist uses the Target Solution Sampling Assembly as an example – open the valve BEFORE the vacuum pump is turned on 75.2. **YOU WILL BE PASSING THROUGH THESE STEPS THREE TIMES** A. First pass (Target Solution samples) mark the black boxes ☐ B. Second pass (Column Loading samples) mark the red boxes ☐ C. Third pass (Column Stripping samples) mark the blue boxes ☐ 75.3. **IT IS STRONGLY RECOMMENDED THAT SAMPLES BE COLLECTED SEQUENTIALLY** A. It is NOT recommended to collect all eight (8x) samples of a given set at the same time 75.4. **At the rack** A. Switch the Sample Retrieve Valve Power to ON by gently pulling out the switch while moving the handle up to actuate i. Turns on the 24VDC power to sample retrieval valves

Step	Action



75.5. **Inside the glovebox**

A. Verify that the long tubing from vacuum pump is connected to solenoid valve V-0155

i. Vents vacuum pump outlet to gas collection system

B. Check that power supply for small vacuum pump is plugged in to a receptacle (pump part number can be found in Exhibit B)

i. As of 7/3/2017 the power supply is plugged into the receptacle controlled by Glovebox Power Panel rocker switch #6

C. Verify that the vacuum pump operates by turning pump power on/off

i. If vacuum pump does not operate

a. Verify that correct rocker switch was operated to turn pump on

b. Verify 12VDC is outputting correct voltage

75.6. Choose a computer to use (PICK ONE)

A. Desktop

i. Open **{M3_SHINE_sample retrieval control_ver03.vi}** to control the Solution Sampling Assembly Valves

OR

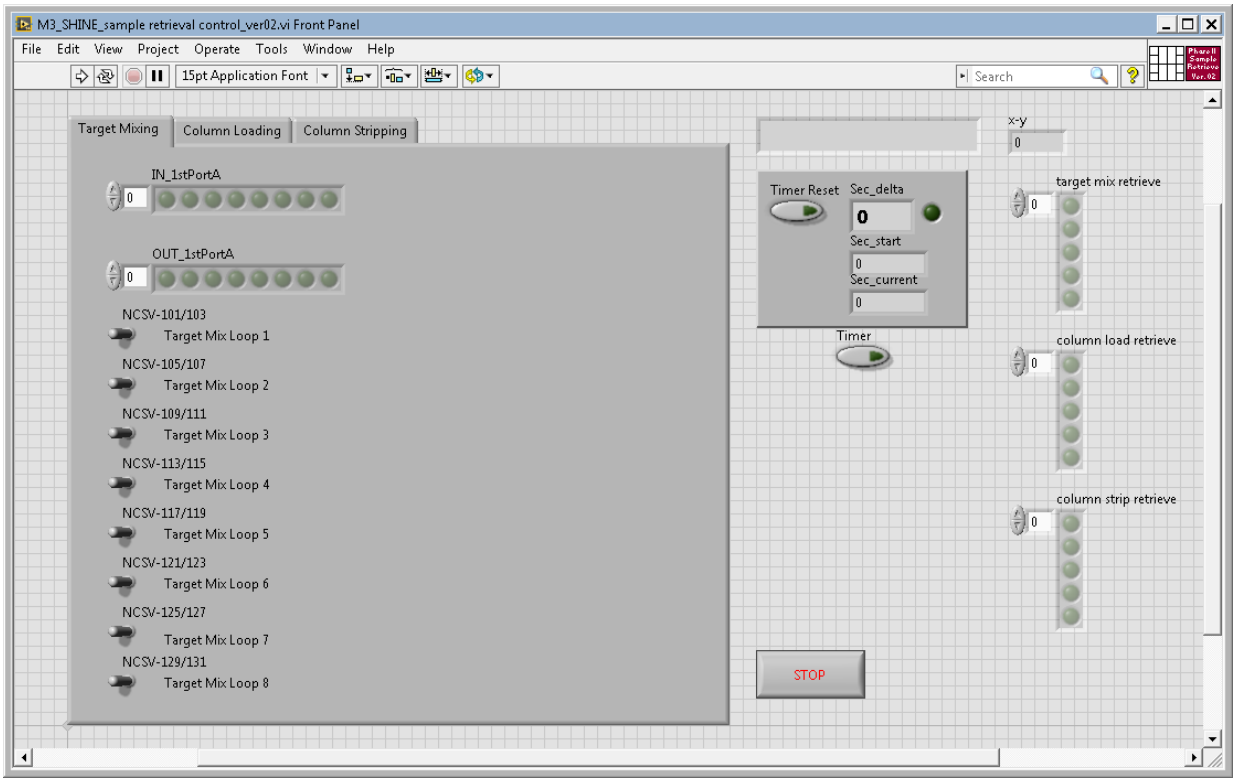
B. Laptop

i. Position laptop cart near glovebox

ii. Uncoil orange Ethernet cable and attach to Ethernet port of laptop

iii. HDMI cable from large monitor should be plugged into HDMI port of laptop

Step	Action
	iv. Turn laptop on v. Use Remote Desktop to connect to LabVIEW computer vi. In the Remote Desktop window open **{M3_SHINE_sample retrieval control_ver03.vi}** to control the Solution Sampling Assembly Valves



75.7. Press the LabVIEW play button to activate the program

75.8. Select the **[Target Mixing]** tab

A. Choose the appropriate tab for the samples to be retrieved

75.9. ☐ ☐ ☐ Attach vacuum tube inlet to left side, bottom row of 3-way Luer valve manifold to be collected

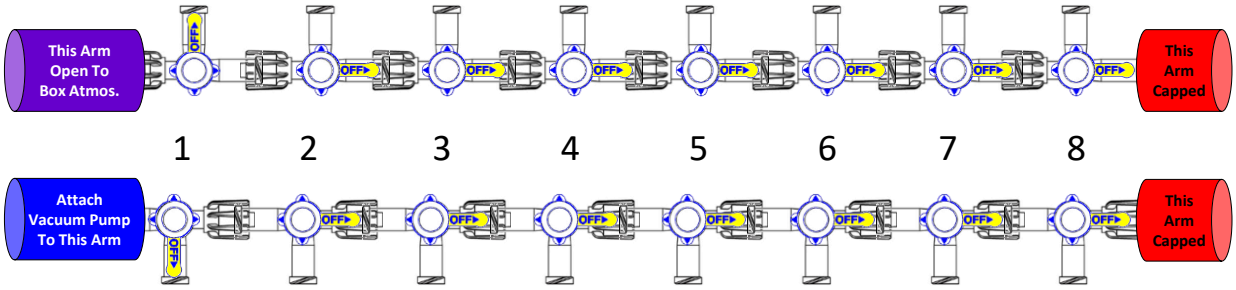
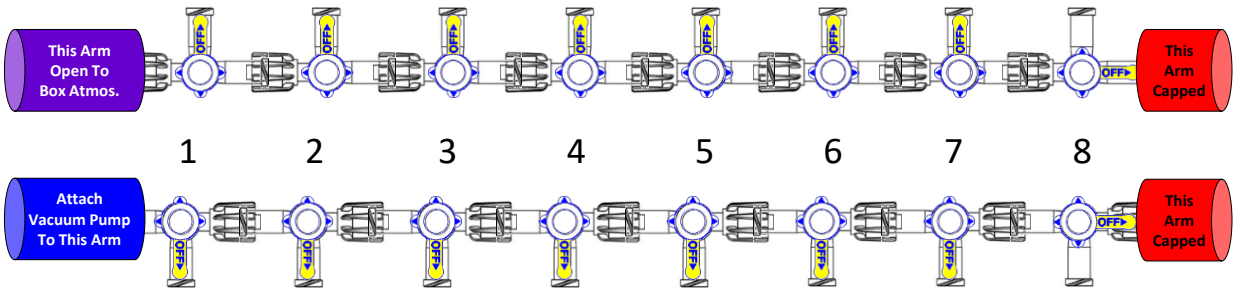
75.10. **DOSE HAZARD!! — KEEP HANDS AWAY FROM 1/16 in. LIQUID LINE WHILE TRANSFFERING LIQUID**

75.11. ☐ ☐ ☐ Open Loop 1 using LabVIEW program BEFORE turning on vacuum pump to ensure liquid can be retrieved

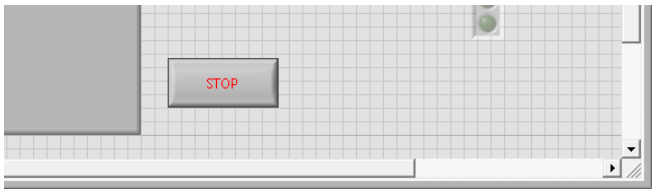
A. This opens the liquid and vent valves associated with a given loop and valve V-0155 to gas collection system

75.12. Watch for liquid in 1/16 in. line while switching vacuum pump power switch #6

75.13. When liquid appears to have been transferred turn off rocker switch #6

Step	Action
	75.14. ☐ ☐ ☐ Close Loop 1 using LabVIEW program 75.15. ☐ ☐ ☐ Turn handles of the Sample #1 3-way Luer valves to the following positions A. Top row 3-way valve #1 handle points up to close the vent valve to glovebox atmosphere B. Bottom row 3-way valve #1 handle points down to close the vent to the vacuum pump VENT LINES TO INDIVIDUAL SAMPLING LOOP SOLENOID VENT VALVES (check valves not shown)  VENT LINES FROM CONCENTRIC NEEDLE ASSEMBLIES 75.16. Repeat **steps 75.11 – 75.15.B** for each of the loops A. After collecting Loops 1-7 but before collecting Loop 8 the manifold should look like the following VENT LINES TO INDIVIDUAL SAMPLING LOOP SOLENOID VENT VALVES (check valves not shown)  VENT LINES FROM CONCENTRIC NEEDLE ASSEMBLIES 75.17. After collecting the last sample the 3-way Luer valves for each of the loops collected will be pointing up for the top row and down for the bottom row

Step	Action
	A. Verify the vacuum pump is turned off at switch #6 B. Verify all loops are closed in the LabVIEW program 75.18. **DOSE HAZARD!! — DO NOT PLACE YOUR HAND ON TOP OF ANY OF THE LOADED VIALS** 75.19. Using long tweezers/forceps to hold vial in vial shield and pull concentric needle assembly from vial 75.20. Insert free concentric needle assembly into storage vessel 75.21. Place vial shield cover on vial shield body 75.22. Repeat **steps 75.18 – 75.21** for each collected sample 75.23. Retrieve the sample from the flow meter bypass sample loop A. Ensure the evacuated sample collection vial and needle setup (Appendix B item 11, but with 1/8" lines) is attached to the BOTTOM manual valve B. Open the bottom manual valve to the sample collection bottle C. Turn on the small vacuum pump using Glovebox Power Panel rocker #6 D. Slowly open the top valve to allow solution to flow out into the vial E. When the sample has been collected, close BOTH manual valves and turn off the small vacuum pump 75.24. **DOSE HAZARD!! — ALL SAMPLES SHOULD BE COVERED BEFORE COLLECTING ANY OTHER SAMPLES OR DOING ANY OTHER WORK TO MINIMIZE EXPOSURE**
76.	**If Collecting Samples from other Sampling Assemblies** 76.1. Move the vacuum pump inlet tubing to the appropriate left, bottom row 3-way Luer valve manifold (see **step 75.9**) 76.2. Repeat **steps 75.10** – **75.24.**
77.	**Effluent Cart Sample Collection** 77.1. Follow the instructions outlined in WCD-EZ "Sampling AMORE Effluent Bottles Using a Syringe" appended to WCD 56833.1 77.2. Proceed to the following step.
78.	**When All Samples Have Been Collected** 78.1. Press **[STOP]** button in **{M3_SHINE_sample retrieval control_ver03.vi}**

Step	Action
	 78.2. Close the **{M3_SHINE_sample retrieval control_ver03.vi}** program 78.3. Close LabVIEW 78.4. If using the laptop A. Close the Remote Desktop connection B. Disconnect the orange Ethernet cable, neatly coil it up and hang it on the lower right side of the main LabVIEW rack C. Turn off the laptop and roll it back into the Instrument Room (D027)

3.2.6 Washout of ^{99}Mo Recovery System and Sample Retrieval Subsystems

Step	Action
79.	**Begin Operation of Mo-99 Remote Recovery Data Acquisition & Control Software**

79.1. Version used: _____

A. Current version: **{M3_SHINE_PhaseII_ver06J.vi}** (as of 3/27/2018)

79.2. ☐ External computer speakers powered ON

A. Verify speakers work

B. Run beep10.bat file from desktop

79.3. ☐ Sound came out of speakers

A. If sound does not come out of speakers DO NOT proceed until speakers are operational

79.4. Start the program inputting the following parameters:

A. Manual mode

B. Process operation

C. Column

D. RECORD EFFLUENT BALANCE DATA?

i. YES

E. Set effluent balance type and COM port

i. Ohaus Defender 7000 (25/50 kg)

ii. Pick ONE

a. Effluent cart #1: ends in 414, set COM 11

b. Effluent cart #1: ends in 416, set COM 10

F. Fresh Acid density → as default value → press OK

G. Base Wash density → as default value → press OK

H. Base Strip density → as default value → press OK

I. Target solution volume

i. Enter last target solution volume

J. Target solution concentration

i. Enter last target solution concentration

K. Target solution density

i. Enter last target solution density

Step	Action
	L. Column effluent path i. To Transfer Cask M. Pre-Load Acid Wash processing volume → as default value → press OK N. Column Loading processing volume → as default value → press OK O. Post-Load Acid Wash processing volume → as default value → press OK P. Post-Load Water Wash processing volume → as default value → press OK Q. Use the Post-Load NaOH Wash step? → NO R. Column stripping processing volume → as default value → press OK S. Post-Strip Water Wash To Strip product processing volume → as default value → press OK T. Post-Strip Water Wash To Waste processing volume → as default value → press OK U. Final Base System Water Wash processing volume → as default value → press OK V. Final Acid System Acid Wash processing volume → as default value → press OK W. Record LINAC temperatures? → YES X. Filename prefix: _____ (see **[File Paths]**.tab → File Prefix) Y. ☐ ACID Pump controller powered ON i. Rocker switch under front/left of ACID Pump V300 controller Z. ☐ ACID Pump to **STOP** i. Display alternates between OFF and ##.# (current setting) AA. ☐ Verify feed and effluent balances are reading at **[Sensors]**.tab i. Compare LabVIEW value to value on feed balance indicator a. Indicator: _____ grams b. LabVIEW: _____ grams ii. Compare LabVIEW value to value on effluent balance indicator a. Indicator: _____ grams b. LabVIEW: _____ grams BB. **DO NOT PROCEED IF FEED OR EFFLUENT BALANCES ARE NOT BEING READ**
80.	**Acid System Rinse Out With Acid Solution** 80.1. At feed bottle cabinet A. ☐ Verify >3000 mL of pH 1 H_2SO_4 is in the Fresh Acid feed bottle **AND** ≥3000 mL free volume in Acid Rinse effluent bottle (use table on page 88) i. If sufficient volume, continue to step **80.2**, otherwise proceed directly to step **81**

Step	Action
	80.2. **At LabVIEW computer** **– YOU ARE OPERATING THE SYSTEM IN MANUAL MODE –** A. ☐ Open V-0003 (Fresh Acid) B. ☐ Verify flow path through acid flow meter 163/164 (valves should already be open) C. ☐ Open V-0009 (Target Mixing path) D. ☐ Open Target Mixing loop 1 on **[Sample Collection]**.tab → **[Target Mixing]**.tab by pressing the purple 100/102 button E. ☐ Open V-0153/0154 (Target Mixing path to Dump Tank path) F. ☐ Open V-0172/0173 (Target Mixing path to Acid Rinse bottle) i. Selecting this valve closes both 147/148 (frit to Dump Tank) and 149/150 (bypass to Dump Tank) G. ☐ Open V-0156 (Effluent bottle vent) i. **V-0156 MUST BE OPEN DURING OPERATIONS – WHEN OPERATIONS ARE NOT BEING CONDUCTED CLOSE THE VALVE** H. ☐ Enter flow rate **100** mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab I. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab J. ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate i. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller K. ☐ Prepare 3 minute timer L. ☐ ACID Pump to **RUN** and start timer M. At 3 minute timer end ACID Pump to **STOP** N. ☐ Open Target Mixing loop 2 on **[Sample Collection]**.tab → **[Target Mixing]**.tab by pressing the purple 104/106 button i. The previous loop valves will automatically close when another loop is opened O. ☐ Prepare 1 minute 15 second timer (75 seconds total) P. ☐ ACID Pump to **RUN** and start timer Q. At 75 second timer end ACID Pump to **STOP** R. Repeat steps **80.2.N – 80.2.Q** for each Target Mixing loop, actuating the loop valves using the following purple buttons on **[Sample Collection]**.tab → **[Target Mixing]**.tab i. ☐ Loop 3: 108/110

Step	Action
	ii. ☐ Loop 4: 112/114 iii. ☐ Loop 5: 118/118 iv. ☐ Loop 6: 120/122 v. ☐ Loop 7: 124/126 vi. ☐ Loop 8: 128/130 vii. ☐ Loop 9: 132/133 S. ☐ Verify ACID Pump to **STOP** T. ☐ Close V-0009 (Target Mixing path) U. ☐ Close V-0153/0154 (Target Mixing path to Dump Tank path) V. ☐ Close V-0172/0173 (Target Mixing path to Acid Rinse bottle) W. ☐ Verify V-0003 open (Fresh acid) X. ☐ Verify flow path through acid flow meter 163/164 (valves should already be open) Y. ☐ Open V-0010 (Column Loading path) Z. ☐ Open Column Loading loop 1 on **[Sample Collection]**.tab → **[Column Loading]**.tab by pressing the purple 32/34 button AA. ☐ Open V-0014/0015 (Acid column bypass) using toggle switch at lower left corner of **[System]**.tab (using column bypass in case column still attached to system) BB. ☐ Verify flow path through acid column loading filter 18/19 (valves should already be open) CC. ☐ Open V-0139 (Acid rinse) DD. ☐ Verify V-0156 (Effluent bottle vent) EE. ☐ Enter flow rate **100** mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab FF. ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab GG. ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate i. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller HH. ☐ Prepare 6 minute timer II. ☐ ACID Pump to **RUN** and start timer JJ. At 6 minute timer end ACID Pump to **STOP** KK. ☐ Open Column Loading loop 2 on **[Sample Collection]**.tab → **[Column Loading]**.tab by pressing the purple 36/38 button i. The previous loop valves will automatically close when another loop is opened

Step	Action
	LL. ☐ Prepare 75 second timer (1 min: 15 sec) MM. ☐ ACID Pump to **RUN** and start timer NN. At 75 second timer end ACID Pump to **STOP** OO. Repeat steps **80.2.KK – 80.2.NN** for each Column Loading loop, actuating the loop valves using the following purple buttons on **[Sample Collection]**.tab →**[Column Loading]**.tab i. ☐ Loop 3: 40/42 ii. ☐ Loop 4: 44/46 iii. ☐ Loop 5: 48/50 iv. ☐ Loop 6: 52/54 v. ☐ Loop 7: 56/58 vi. ☐ Loop 8: 60/62 vii. ☐ Loop 9: 64/65 PP. ☐ Verify ACID Pump to **STOP** QQ. ☐ Close V-0003 (Fresh acid) RR. ☐ Close V-0010 (Column Loading path) SS. ☐ Close V-0139 (Acid rinse) TT. ☐ Close V-0156 (Effluent bottle vent) UU. ☐ ACID Pump controller powered OFF using rocker switch under front/left of ACID Pump V300 controller VV. Proceed to **step 81**
81.	**Acid System Purge with 5 psig N_2** 81.1. Outside of recovery glovebox **THE 2-WAY BALL VALVE (V-2038) FOR NITROGEN SERVICE IS TO REMAIN ATTACHED TO THE GLOVEBOX – DO NOT REMOVE THIS VALVE** A. Verify V-2038 for nitrogen service is closed B. Attach nitrogen cylinder to V-2038 on right side of recovery glovebox, above white transfer cask glovebox C. Set regulator to 5 psig D. **DO NOT OPEN V-2038 FOR NITROGEN SERVICE** 81.2. Inside of recovery glovebox

Step	Action
	A. Verify acid injection port V-2001 closed (valve is after acid pump and before acid flow meter) B. If present remove needle port guide from acid injection port valve V-2001 i. Needle port guide is three pieces a. Needle port guide nut, Septum, White ¼ in. Teflon one-piece ferrule C. Verify check valve V-0406 attached to end of ¼ in. FEP tubing line from V-2038 i. Prevents potential of glovebox atmosphere exiting V-2038 D. Attach existing ¼ in. FEP tubing line to acid injection port valve V-2001 via check valve V-0406 i. FEP tubing is attached to a stainless steel line that passes across glovebox wall and ends near the solenoid vent valves manifold 81.3. Outside of recovery glovebox A. Open V-2038 for nitrogen service 81.4. At LabVIEW computer AND inside recovery glovebox A. ☐ Verify flow path through acid flow meter 163/164 (valves are already open) B. ☐ Open V-0009 (Target Mixing path) C. ☐ Open Target Mixing loop 1 on **[Sample Collection]**.tab → **[Target Mixing]**.tab by pressing the purple 100/102 button D. ☐ Open V-0153/0154 (Target Mixing path to Dump Tank path) E. ☐ Open V-0172/0173 (Target Mixing path to Acid Rinse bottle) i. Selecting this valve closes both 147/148 (frit to Dump Tank) and 149/150 (bypass to Dump Tank) F. ☐ Open V-0156 (Effluent bottle vent) i. **V-0156 MUST BE OPEN DURING OPERATIONS – WHEN OPERATIONS ARE NOT BEING CONDUCTED CLOSE THE VALVE** G. Prepare 3 minute timer for loop 1 H. Open acid injection port valve V-2001 and start timer I. At 3 minute timer end close injection port valve V-2001 J. ☐ Open Target Mixing loop 2 on **[Sample Collection]**.tab → **[Target Mixing]**.tab by pressing the purple 104/106 button i. The previous loop valves will automatically close when another loop is opened K. Prepare 1 minute timer L. Open acid injection port valve V-2001 and start timer

Step	Action
	M. At 1 minute timer timer end close injection port valve V-2001 N. Repeat steps **81.4.J – 81.4.M** for each Target Mixing loop, actuating the loop valves using the following purple buttons on **[Sample Collection]**.tab → **[Target Mixing]**.tab i. ☐ Loop 3: 108/110 ii. ☐ Loop 4: 112/114 iii. ☐ Loop 5: 118/118 iv. ☐ Loop 6: 120/122 v. ☐ Loop 7: 124/126 vi. ☐ Loop 8: 128/130 vii. ☐ Loop 9: 132/133 O. ☐ Verify injection port valve V-2001 is closed P. ☐ Close V-0009 (Target Mixing path) Q. ☐ Close V-0153/0154 (Target Mixing path to Dump Tank path) R. ☐ Close V-0172/0173 (Target Mixing path to Acid Rinse bottle) S. ☐ Verify flow path through acid flow meter 163/164 (valves already open) T. ☐ Open V-0010 (Column Loading path) U. ☐ Open Column Loading loop 1 on **[Sample Collection]**.tab → **[Column Loading]**.tab by pressing the purple 32/34 V. ☐ Open V-0014/0015 (Acid column bypass) using toggle switch at lower left corner of **[System]**.tab (using column bypass in case column still attached to system) W. ☐ Verify flow path through acid column loading filter 18/19 (valves already open) X. ☐ Open V-0139 (Acid rinse) Y. ☐ Verify V-0156 (Effluent bottle vent) Z. Prepare 3 minute timer for loop 1 AA. Open acid injection port valve V-2001 and start timer BB. At 3 minute timer end close injection port valve V-2001 CC. ☐ Open Column Loading loop 2 on **[Sample Collection]**.tab → **[Column Loading]**.tab by pressing the purple 36/38 button i. The previous loop valves will automatically close when another loop is opened DD. Prepare 1 minute timer timer EE. Open acid injection port valve V-2001 and start timer FF. At 1 minute timer timer end close injection port valve V-2001

Step	Action
	GG. Repeat steps **81.4.CC – 81.4.FF** for each Column Loading loop, actuating the loop valves using the following purple buttons on **[Sample Collection]**.tab →**[Column Loading]**.tab i. ☐ Loop 3: 40/42 ii. ☐ Loop 4: 44/46 iii. ☐ Loop 5: 48/50 iv. ☐ Loop 6: 52/54 v. ☐ Loop 7: 56/58 vi. ☐ Loop 8: 60/62 vii. ☐ Loop 9: 64/65 HH. ☐ Verify injection port valve V-2001 is closed II. ☐ Close V-0139 (Acid rinse) JJ. ☐ Open V-0142 (Pre-Load acid wash) KK. ☐ Open injection port valve V-2001 LL. Hold purge for at least one minute or until pre-load acid wash line is empty MM. ☐ Close V-0142 (Pre-Load acid wash) NN. ☐ Open V-0141 (Post-Load acid wash) OO. Hold purge for at least one minute or until post-load acid wash line is empty PP. ☐ Close V-0141 (Post-Load acid wash) QQ. ☐ Open V-0140 (Post-Load water wash) RR. Hold purge for at least one minute or until post-load water wash line is empty SS. ☐ Close V-0140 (Post-Load water wash) TT. ☐ Close V-0010 (Column Loading path) UU. ☐ Close V-0156 (Effluent bottle vent) VV. ☐ Close injection port valve V-2001 81.5. Outside of recovery glovebox A. ☐ Close V-2038 for nitrogen service
82.	**<u>Checking D024 Hot Cell 3-L/5-Neck Flask Installation</u>** \\ **Recovery Member Name: _____PRINT Date: _____ Time: _____** **D024 Hot Cell Ops Member Name: _____PRINT Date: _____ Time: _____** ### SYSTEMS INTERFACE STEP ###

Step	Action
	82.1. Contact a D024 Hot Cell Operations team member 82.2. Appropriate team member **INITIALIZES** every step in this section 82.3. Refer to figures on page 55 82.4. **Inside D024 Hot Cell** A. ☐ Recovery ☐ HotCellOps Verify 3-L/5-neck flask in place B. ☐ Recovery ☐ HotCellOps Verify pH probe inserted and not broken C. ☐ Recovery ☐ HotCellOps Verify plastic feed line attached to center neck D. ☐ Recovery ☐ HotCellOps Verify septum in un-used neck and secured E. ☐ Recovery ☐ HotCellOps Verify multi-port neck adapters inserted and attached to other two ports F. ☐ Recovery ☐ HotCellOps Verify flask on balance G. ☐ Recovery ☐ HotCellOps Verify adequate free volume in Product receipt vessel i. Product receipt vessel will receive ~1000 mL of washout solution H. ☐ Recovery ☐ HotCellOps Verify balance is ON I. Record balance reading: _____ grams J. ☐ Recovery ☐ HotCellOps Verify 2WV-701 liquid valve OPEN i. Handle parallel to the long axis of valve body K. ☐ Recovery ☐ HotCellOps Verify plastic line attached between 2WV-701 and center neck of flask L. ☐ Recovery ☐ HotCellOps Verify 2WV-801 vent valve OPEN i. Handle perpendicular to the long axis of valve body M. Verify liquid trap to Gas Collection System is: i. ☐ Recovery ☐ HotCellOps Attached ii. ☐ Recovery ☐ HotCellOps Good condition (not cracked/broken), lines attached iii. ☐ Recovery ☐ HotCellOps Verify empty 82.5. Recovery personnel continue to **Step 83**

Step	Action
83.	**<u>Base System Rinse Out with Distilled Water</u>** 83.1. At feed bottle cabinet A. ☐ Verify >2000 mL of distilled H_2O is in the Fresh H_2O feed bottle **AND** ≥ 1500 mL free volume in Base Rinse effluent bottle (see table on page 88) i. If sufficient volume, continue to step **83.2**, otherwise proceed directly to step **84** 83.2. At LabVIEW computer A. ☐ BASE Pump controller powered ON using rocker switch under front/left of BASE Pump V300 controller **– YOU ARE OPERATING THE SYSTEM IN MANUAL MODE –** B. ☐ Open V-0006 (Fresh H_2O, base manifold) C. ☐ Verify flow path through base flow meter 167/168 (valves are already open) D. ☐ Open V-0024/0025 (Base column bypass) using toggle switch at lower left corner of **[System]**.tab (using column bypass in case column still attached to system) E. ☐ Verify flow path through base column loading filter 28/29 (valves are already open) F. ☐ Open Column stripping loop 1 on **[Sample Collection]**.tab → **[Column Stripping]**.tab by pressing the purple 66/68 button G. ☐ Open V-0134 (Base rinse) H. ☐ Open V-0156 (Effluent bottle vent) i. **V-0156 MUST BE OPEN DURING OPERATIONS – WHEN OPERATIONS ARE NOT BEING CONDUCTED CLOSE THE VALVE** I. ☐ Enter flow rate **<u>100</u>** mL/min *Base Flow Rate Set Pt* @ **[System]**.tab J. ☐ Record calculated _____ *% Base Motor Power* @ **[System]**.tab K. ☐ Verify/Adjust BASE Pump Controller to *% Base Motor Power* for desired flow rate i. If a lower % motor power value is required due to pressure readings adjust *Base Flow Rate Set Pt* until calculated *% Base Motor Power* matches % motor power reading at controller L. ☐ Prepare 6 minute timer M. ☐ BASE Pump to **<u>RUN</u>** and start timer N. At 6 minute timer end BASE Pump to **<u>STOP</u>** O. ☐ Open Column Stripping loop 2 on **[Sample Collection]**.tab → **[Column Stripping]**.tab by pressing the purple 70/72 i. The previous loop valves will automatically close when another loop is opened P. ☐ Prepare 75 second timer (1 min: 15 sec)

Step	Action
	Q. ☐ BASE Pump to **RUN** and start timer R. At 75 second timer end BASE Pump to **STOP** S. Repeat steps **83.2.O – 83.2.R** for each Column Stripping loop, actuating the loop valves using the following purple buttons on **[Sample Collection]**.tab → **[Column Stripping]**.tab i. ☐ Loop 3: 74/76 ii. ☐ Loop 4: 78/80 iii. ☐ Loop 5: 82/84 iv. ☐ Loop 6: 86/88 v. ☐ Loop 7: 90/92 vi. ☐ Loop 8: 94/96 vii. ☐ Loop 9: 98/99 T. ☐ Verify BASE Pump to **STOP** U. ☐ Close V-0134 (Base rinse) V. ☐ Close V-0156 (Effluent bottle vent) 83.3. **Washout of ^{99}Mo product transfer line to Bigfoot** A. ☐ Open V-0137 (Product to Hot Cell) B. ☐ Open V-0159 (Cell Receipt Vessel vent) C. ☐ Prepare 5 minute timer D. ☐ BASE Pump to **RUN** and start timer E. At 5 minute timer end BASE Pump to **STOP** F. ☐ Close V-0006 (Fresh H_2O, base feed manifold) G. ☐ Close V-0137 (Product to Hot Cell) H. ☐ Close V-0159 (Cell Receipt Vessel vent) I. ☐ BASE Pump controller powered OFF using rocker switch under front/left of BASE Pump V300 controller J. Proceed to **step 84**
84.	**Base System Purge with 5 psig N_2** 84.1. Outside of recovery glovebox **THE V-2038 for nitrogen service IS TO REMAIN ATTACHED TO THE GLOVEBOX – DO NOT REMOVE THIS VALVE** A. Verify V-2038 for nitrogen service is closed

Step	Action
	B. Verify N_2 tank attached and set to 5 psig i. If not attached see **step 81.1** then return to this step C. **DO NOT OPEN V-2038 for nitrogen service** 84.2. Inside of recovery glovebox A. Verify base system purge port valve V-2033 closed (valve is after base pump and before base flow meter) B. If present remove needle port guide from base system purge port valve V-2033 i. Needle port guide is three pieces a. Needle port guide nut, Septum, and White ¼ in. Teflon one-piece ferrule C. Verify check valve V-0406 attached to end of ¼ in. FEP tubing line from V-2038 i. Prevents potential of glovebox atmosphere exiting V-2038 D. Attach existing ¼ in. FEP tubing line to base system purge port valve V-2033 via check valve V-0406 i. FEP tubing is attached to a stainless steel line that passes across glovebox wall and ends near the solenoid vent valves manifold 84.3. Outside of recovery glovebox A. Open V-2038 for nitrogen service 84.4. At LabVIEW computer AND inside recovery glovebox A. ☐ Verify flow path through base flow meter 167/168 (valves are already open) B. ☐ Open V-0024/0025 (Base column bypass) using toggle switch at lower left corner of **[System]**.tab (using column bypass in case column still attached to system) C. ☐ Verify flow path through base column loading filter 28/29 (valves are already open) D. ☐ Open Column stripping loop 1 on **[Sample Collection]**.tab → **[Column Stripping]**.tab by pressing the purple 66/68 button E. ☐ Open V-0134 (Base rinse) F. ☐ Open V-0156 (Effluent bottle vent) i. **V-0156 MUST BE OPEN DURING OPERATIONS – WHEN OPERATIONS ARE NOT BEING CONDUCTED CLOSE THE VALVE** G. Prepare 3 minute timer for loop 1 H. Open base system purge port valve V-2033 and start timer I. At 3 minute timer end close base system purge port valve V-2033 J. ☐ Open Column Stripping loop 2 on **[Sample Collection]**.tab → **[Column Stripping]**.tab by pressing the purple 70/72 button

Step	Action
	i. The previous loop valves will automatically close when another loop is opened K. Prepare 1 minute timer L. Open base system purge port valve V-2033 and start timer M. At 1 minute timer end close base system purge port valve V-2033 N. Repeat steps **84.4.J – 84.4.M** for each Column Stripping loop, actuating the loop valves using the following purple buttons on **[Sample Collection]**.tab → **[Column Stripping]**.tab i. ☐ Loop 3: 74/76 ii. ☐ Loop 4: 78/80 iii. ☐ Loop 5: 82/84 iv. ☐ Loop 6: 86/88 v. ☐ Loop 7: 90/92 vi. ☐ Loop 8: 94/96 vii. ☐ Loop 9: 98/99 O. ☐ Verify base system purge port valve V-2033 is closed P. ☐ Close V-0134 (Base rinse) Q. ☐ Open V-0135 (post-strip water wash) R. ☐ Open base system purge port valve V-2033 S. Leave purge for at least one minute to flush out post strip water wash line T. ☐ Close V-0135 (post-strip water wash) U. ☐ Close V-0156 (Effluent bottle vent) V. ☐ Close base system purge port valve V-2033 84.5. **Purge out of ^{99}Mo product transfer line to Bigfoot** A. ☐ Open V-0137 (Product to Hot Cell) B. ☐ Open V-0159 (Cell Receipt Vessel vent) C. ☐ Prepare 1 minute timer D. Open base system purge port valve V-2033 and start timer E. At 1 minute timer end close base system purge port valve V-2033 F. ☐ Close V-0137 (Product to Hot Cell) G. ☐ Close V-0159 (Cell Receipt Vessel vent) 84.6. Outside of recovery glovebox A. ☐ Close V-2038 for nitrogen service
85.	**Report RecoveryOps Done with D024 Hot Cell 3-L/5-Neck Flask**

Step	Action
	\\ **Recovery Member Name: _____**PRINT **Date: _____ Time: _____** **D024 Hot Cell Ops Member Name: _____**PRINT **Date: _____ Time: _____** ### SYSTEMS INTERFACE STEP ### 85.1. Contact a D024 Hot Cell Operations team member 85.2. Appropriate team member **INITIALIZES** every step in this section 85.3. **Inside D024 Hot Cell** A. ☐Recovery ☐HotCellOps Verify 3-L/5-neck flask in place B. ☐Recovery ☐HotCellOps Verify pH probe inserted and not broken C. ☐Recovery ☐HotCellOps Verify plastic feed line attached to center neck D. ☐Recovery ☐HotCellOps Verify septum in un-used neck and secured E. ☐Recovery ☐HotCellOps Verify multi-port neck adapters inserted and attached to other two ports F. ☐Recovery ☐HotCellOps Verify 2WV-701 liquid valve CLOSED i. Handle perpendicular to the long axis of valve body G. ☐Recovery ☐HotCellOps Verify plastic line attached between 2WV-701 and center neck of flask H. ☐Recovery ☐HotCellOps Verify 2WV-801 vent valve CLOSED i. Handle perpendicular to the long axis of valve body I. Verify liquid trap to Gas Collection System is: i. ☐Recovery ☐HotCellOps Attached ii. ☐Recovery ☐HotCellOps Good condition (not cracked/broken), lines attached iii. ☐Recovery ☐HotCellOps Empty 85.4. Recovery personnel continue to **Step 86**
86.	**Stage the Following Items at the Glovebox** 86.1. Place bottles for receiving loop washings inside of glovebox (bottle descriptions and part numbers can be found in the Exhibit B)

Step	Action
	A. Three bottles are required i. One for target mixing washes ii. One for column loading washes iii. One for column stripping washes a. Insert 18 gauge disposable needle into each bottle to vent purge N_2 into glovebox iv. DO NOT COMBINE TARGET MIXING AND COLUMN LOADING a. Having separate bottles will keep the needles as their respective sets B. NOTE – for very first time through these washout steps you may want to consider using two 60 mL bottles for each individual retrieval needle washout. This would allow for analysis to verify that amounts coming out of second pass through the procedure are very low and would help determine if a third pass is required. Once this information is known then washings of the eight retrieval needles for a given set can be combined into a 125 mL bottle. 86.2. Ensure there is enough solution for wash out in feed bottles in middle cabinet (cabinet #2) A. Deionized water feed bottle contains 10 L of deionized water B. Fresh Acid Feed bottle of contains 4 L of pH 1 H_2SO_4 86.3. Ensure there is enough empty space to receive wash solution in effluent bottles (cabinet #3) A. Effluent bottle 10 L empty → to Acid Rinse line (from valve #139) B. Effluent bottle 10 L empty → to Base Rinse line (from valve #134) 86.4. If insufficient empty volume in the effluent bottles perform the following, otherwise continue to step **86.5** A. Remove effluent bottle cart following Steps **91** and **92** B. Install new Acid Rinse and Base Rinse bottles in cabinet #3, making the following connections: i. ☐ Acid Rinse Bottle connected to Acid Rinse line (from valve #139 via 2-way ball valve #2018) ii. ☐ Acid Rinse Bottle connected to Acid Rinse vent line (to 2-way ball valve #2020) iii. ☐ Base Rinse Bottle connected to Base Rinse line (from valve #134 via 2-way ball valve #2017) iv. ☐ Base Rinse Bottle connected to Base Rinse vent line (to 2-way ball valve #2022) 86.5. ☐ Verify all effluent cart (or newly installed effluent bottle) liquid lines are connected and open

Step	Action
	86.6. ☐ Verify all effluent cart (or newly installed effluent bottle)vent lines are connected and open
87.	**<u>Washout of Acid Sample Loops</u>** **– YOU ARE OPERATING THE SYSTEM IN MANUAL MODE –** 87.1. **Outside of recovery glovebox** **THE V-2038 for nitrogen service IS TO REMAIN ATTACHED TO THE GLOVEBOX – DO NOT REMOVE THIS VALVE** A. Verify V-2038 for nitrogen service is closed B. Attach nitrogen cylinder to V-2038 on right side of recovery glovebox, above white transfer cask glovebox C. Set regulator to 5 psig D. **<u>DO NOT</u> OPEN V-2038 for nitrogen service** 87.2. **Inside of recovery glovebox** A. Verify acid injection port valve V-2001 closed (valve is after acid pump and before acid flow meter) B. If present remove needle port guide from acid injection port valve V-2001 i. Needle port guide is three pieces a. Needle port guide nut, Septum, and White ¼ in. Teflon one-piece ferrule C. Verify check valve V-0406 attached to end of ¼ in. FEP tubing line from V-2038 i. Prevents potential of glovebox atmosphere exiting V-2038 D. Attach existing ¼ in. FEP tubing line to acid injection port valve V-2001 via check valve V-0406 i. FEP tubing is attached to a stainless steel line that passes across glovebox wall and ends near the solenoid vent valves manifold 87.3. **Outside of recovery glovebox** A. Open V-2038 for nitrogen service 87.4. **YOU WILL BE PASSING THROUGH THESE STEPS AT LEAST TWICE** A. First pass mark the black boxes ☐ B. Second pass mark the red boxes ☐ C. Third pass mark the blue boxes ☐ D. Fourth pass (if required) mark the green boxes ☐

Step	Action
	i. NOTE – only steps **87.6.A** through **87.6.EEE** have the green boxes 87.5. At LabVIEW computer AND inside recovery glovebox 87.6. Removing residual liquid from purged sample loops A. ☐ ☐ ☐ ☐ Verify flow path through acid flow meter V-0163/0164 (valves are open) B. ☐ ☐ ☐ ☐ Open V-0009 (Target Mixing path) C. ☐ ☐ ☐ ☐ Open Target Mixing loop 1 on **[Sample Collection]**.tab → **[Target Mixing]**.tab by pressing the purple 100/102 button D. ☐ ☐ ☐ ☐ Open V-0153/0154 (Target Mixing path to Dump Tank path) E. ☐ ☐ ☐ ☐ Open V-0172/0173 (Target Mixing path to Acid Rinse bottle) i. Selecting this valve closes both 147/148 (frit to Dump Tank) and 149/150 (bypass to Dump Tank) F. ☐ ☐ ☐ ☐ Open V-0156 (Effluent bottle vent) i. **V-0156 MUST BE OPEN DURING OPERATIONS – WHEN OPERATIONS ARE NOT BEING CONDUCTED CLOSE THE VALVE** G. ☐ ☐ ☐ ☐ Insert needle for Target Mixing loop 1into 60 mL square PETG bottle with septum closure H. ☐ ☐ ☐ ☐ Open Target Mixing loop 1 retrieval valves on **[Sample Collection]**.tab → **[Target Mixing]**.tab by pressing the purple 101/103 button I. ☐ ☐ ☐ ☐ Close V-0153/0154 (Target Mixing path to Dump Tank path) J. ☐ ☐ ☐ ☐ Prepare 10 second timer K. Open acid injection port valve V-2001 and start timer (pre and post-acid pump pressures will rise) L. At 10 second timer end close injection port valve V-2001 M. ☐ ☐ ☐ ☐ Close Target Mixing loop 1 retrieval valves on **[Sample Collection]**.tab → **[Target Mixing]**.tab by pressing the green 101/103 button N. ☐ ☐ ☐ ☐ Open V-0153/0154 (Target Mixing path to Dump Tank path) O. ☐ ☐ ☐ ☐ Remove and stow needle for Target Mixing loop 1 P. ☐ ☐ ☐ ☐ Open Target Mixing loop 2 on **[Sample Collection]**.tab → **[Target Mixing]**.tab by pressing the purple 104/106 i. The previous loop valves will automatically close when another loop is opened

Step	Action
	Q. ☐ ☐ ☐ ☐ Insert needle for Target Mixing loop 2 into 60 mL square PETG bottle with septum closure R. ☐ ☐ ☐ ☐ Open Target Mixing loop 2 retrieval valves on **[Sample Collection]**.tab → **[Target Mixing]**.tab by pressing the purple 105/107 button S. ☐ ☐ ☐ Close V-0153/0154 (Target Mixing path to Dump Tank path) T. ☐ ☐ ☐ ☐ Prepare 10 second timer U. Open acid injection port valve V-2001 and start timer V. At 10 second timer end close injection port valve V-2001 W. ☐ ☐ ☐ ☐ Close Target Mixing loop 2 retrieval valves on **[Sample Collection]**.tab → **[Target Mixing]**.tab by pressing the green 105/107 button X. ☐ ☐ ☐ ☐ Open V-0153/0154 (Target Mixing path to Dump Tank path) Y. ☐ ☐ ☐ ☐ Remove and stow needle for Target Mixing loop 2 Z. Repeat steps **87.6.P – 87.6.Y** for each Target Mixing loop, actuating the loop valves using the following purple/green buttons on **[Sample Collection]**.tab → **[Target Mixing]**.tab i. ☐ ☐ ☐ ☐ Loop 3: Valves 108/110 & 109/111 ii. ☐ ☐ ☐ ☐ Loop 4: Valves 112/114 & 113/115 iii. ☐ ☐ ☐ ☐ Loop 5: Valves 116/118 & 117/119 iv. ☐ ☐ ☐ ☐ Loop 6: Valves 120/122 & 121/123 v. ☐ ☐ ☐ ☐ Loop 7: Valves 124/126 & 125/127 vi. ☐ ☐ ☐ ☐ Loop 8: Valves 128/130 & 129/131 AA. ***NOTE – loop 9 does not get washed as it is not used to collect a sample*** BB. ☐ ☐ ☐ ☐ Close V-0009 (Target Mixing path) CC. ☐ ☐ ☐ ☐ Close V-0153/0154 (Target Mixing path to Dump Tank path) DD. ☐ ☐ ☐ ☐ Close V-0172/0173 (Target Mixing path to Acid Rinse bottle) EE. ☐ ☐ ☐ ☐ Verify flow path through acid flow meter 163/164 (valves are already open) FF. ☐ ☐ ☐ ☐ Open V-0010 (Column Loading path)

Step	Action
	GG. ☐ ☐ ☐ ☐ Open V-0014/0015 (Acid column bypass) using toggle switch at lower left corner of **[System]**.tab (using column bypass in case column still attached to system) HH. ☐ ☐ ☐ ☐ Verify flow path through acid column loading filter 18/19 (valves already open) II. ☐ ☐ ☐ ☐ Open Column Loading loop 1 on **[Sample Collection]**.tab → **[Column Loading]**.tab by pressing the purple 32/34 button JJ. ☐ ☐ ☐ ☐ Open V-0139 (Acid rinse) KK. ☐ ☐ ☐ ☐ Verify V-0156 open (Effluent bottle vent) i. **V-0156 MUST BE OPEN DURING OPERATIONS – WHEN OPERATIONS ARE NOT BEING CONDUCTED CLOSE THE VALVE** LL. ☐ ☐ ☐ ☐ Insert needle for Column Loading loop 1into 60 mL square PETG bottle with septum closure MM. ☐ ☐ ☐ ☐ Open Column Loading loop 1 retrieval valves on **[Sample Collection]**.tab → **[Column Loading]**.tab by pressing the purple 33/35 button NN. ☐ ☐ ☐ ☐ Close V-0139 (Acid rinse bottle) OO. ☐ ☐ ☐ ☐ Prepare 10 second timer PP. Open acid injection port valve V-2001 and start timer (pre- and post-acid pump pressures will rise) QQ. At 10 second timer end close injection port valve V-2001 RR. ☐ ☐ ☐ ☐ Close Column Loading loop 1 retrieval valves on **[Sample Collection]**.tab → **[Target Mixing]**.tab by pressing the green 33/35 button SS. ☐ ☐ ☐ ☐ Open V-0139 (Acid rinse bottle) TT. ☐ ☐ ☐ ☐ Remove and stow needle for Column Loading loop 1 UU. ☐ ☐ ☐ ☐ Open Column Loading loop 2 on **[Sample Collection]**.tab → **[Column Loading]**.tab by pressing the purple 36/38 button i. The previous loop valves will automatically close when another loop is opened VV. ☐ ☐ ☐ ☐ Insert needle for Column Loading loop 2 into 60 mL square PETG bottle with septum closure WW. ☐ ☐ ☐ ☐ Open Column Loading loop 2 retrieval valves on **[Sample Collection]**.tab → **[Column Loading]**.tab by pressing the purple 37/39 button

Step	Action
	XX. ☐ ☐ ☐ ☐ Close V-0139 (Acid rinse bottle) YY. ☐ ☐ ☐ ☐ Prepare 10 second timer ZZ. Open acid injection port valve V-2001 and start timer AAA. At 10 second timer end close injection port valve V-2001 BBB. ☐ ☐ ☐ ☐ Close Column Loading loop 2 retrieval valves on **[Sample Collection]**.tab → **[Column Loading]**.tab by pressing the green 37/39 button CCC. ☐ ☐ ☐ ☐ Open V-0139 (Acid rinse bottle) DDD. ☐ ☐ ☐ ☐ Remove and stow needle for Column Loading loop 2 EEE. Repeat steps **87.6.UU – 87.6.DDD** for each Column Loading loop, actuating the loop valves using the following purple/green buttons on **[Sample Collection]**.tab → **[Column Loading]**.tab i. ☐ ☐ ☐ ☐ Loop 3: Valves 40/42 & 41/43 ii. ☐ ☐ ☐ ☐ Loop 4: Valves 44/46 & 45/47 iii. ☐ ☐ ☐ ☐ Loop 5: Valves 48/50 & 49/51 iv. ☐ ☐ ☐ ☐ Loop 6: Valves 52/54 & 53/55 v. ☐ ☐ ☐ ☐ Loop 7: Valves 56/58 & 57/59 vi. ☐ ☐ ☐ ☐ Loop 8: Valves 60/62 & 61/63 FFF. ***NOTE – loop 9 does not get washed as it is not used to collect a sample*** GGG. Was this the third pass for this section? (blue squares ☐ checked) **PICK ONE** i. NO – go to step **87.7.A** ***OR*** ii. YES – go step **88** 87.7. Filling all sample loops with fresh acid A. Verify ACID Pump controller powered ON i. Rocker switch under front/left of ACID Pump V300 controller B. Verify ACID Pump to **STOP** i. Display alternates between OFF and ##.# (current setting) C. ☐ ☐ ☐ Open V-0003 (Fresh Acid)

Step	Action
	D. ☐ ☐ ☐ Verify flow path through acid flow meter 163/164 (valves are already open) E. ☐ ☐ ☐ Open V-0009 (Target Mixing path) F. ☐ ☐ ☐ Open Target Mixing loop 1 on **[Sample Collection]**.tab → **[Target Mixing]**.tab by pressing the purple 100/102 button G. ☐ ☐ ☐ Open V-0153/0154 (Target Mixing path to Dump Tank path) H. ☐ ☐ ☐ Open V-0172/0173 (Target Mixing path to Acid Rinse bottle) i. Selecting this valve closes both 147/148 (frit to Dump Tank) and 149/150 (bypass to Dump Tank) I. ☐ ☐ ☐ Open V-0156 (Effluent bottle vent) i. **V-0156 MUST BE OPEN DURING OPERATIONS – WHEN OPERATIONS ARE NOT BEING CONDUCTED CLOSE THE VALVE** J. ☐ ☐ ☐ Enter flow rate **100** mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab K. ☐ ☐ ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab L. ☐ ☐ ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate i. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller M. ☐ ☐ ☐ Prepare 3 minute timer N. ☐ ☐ ☐ ACID Pump to **RUN** and start timer O. At 3 minute timer end ACID Pump to **STOP** P. ☐ ☐ ☐ Open Target Mixing loop 2 on **[Sample Collection]**.tab → **[Target Mixing]**.tab by pressing the purple 104/106 button i. The previous loop valves will automatically close when another loop is opened Q. ☐ ☐ ☐ Prepare 1 minute 15 second timer (75 seconds total) R. ☐ ☐ ☐ ACID Pump to **RUN** and start timer S. At 75 second timer end ACID Pump to **STOP** T. Repeat steps **87.7.P – 87.7.S** for each Target Mixing loop, actuating the loop valves using the following purple buttons on **[Sample Collection]**.tab → **[Target Mixing]**.tab

Step	Action
	i. ☐ ☐ ☐ Loop 3: Valves 108/110 ii. ☐ ☐ ☐ Loop 4: Valves 112/114 iii. ☐ ☐ ☐ Loop 5: Valves 116/118 iv. ☐ ☐ ☐ Loop 6: Valves 120/122 v. ☐ ☐ ☐ Loop 7: Valves 124/126 vi. ☐ ☐ ☐ Loop 8: Valves 128/130 U. ☐ ☐ ☐ Verify ACID Pump to **STOP** V. ***NOTE – loop 9 does not get washed as it is not used to collect a sample*** W. ☐ ☐ ☐ Close V-0009 (Target Mixing path) X. ☐ ☐ ☐ Close V-0153/0154 (Target Mixing path to Dump Tank path) Y. ☐ ☐ ☐ Close V-0172/0173 (Target Mixing path to Acid Rinse bottle) Z. ☐ ☐ ☐ Verify V-0003 open (Fresh acid) AA. ☐ ☐ ☐ Verify flow path through acid flow meter 163/164 (valves already open) BB. ☐ ☐ ☐ Open V-0010 (Column Loading path) CC. ☐ ☐ ☐ Open V-0014/0015 (Acid column bypass) using toggle switch at lower left corner of **[System]**.tab (using column bypass in case column still attached to system) DD. ☐ ☐ ☐ Verify flow path through acid column loading filter 18/19 (valves are already open) EE. ☐ ☐ ☐ Open Column Loading loop 1 on **[Sample Collection]**.tab → **[Column Loading]**.tab by pressing purple 32/34 button FF. ☐ ☐ ☐ Open V-0139 (Acid rinse) GG. ☐ ☐ ☐ Verify V-0156 (Effluent bottle vent) HH. ☐ ☐ ☐ Enter flow rate **100** mL/min *Acid Flow Rate Set Pt* @ **[System]**.tab II. ☐ ☐ ☐ Record calculated _____ *% Acid Motor Power* @ **[System]**.tab JJ. ☐ ☐ ☐ Verify/Adjust ACID Pump Controller to *% Acid Motor Power* for desired flow rate i. If a lower % motor power value is required due to pressure readings adjust *Acid Flow Rate Set Pt* until calculated *% Acid Motor Power* matches % motor power reading at controller

Step	Action
	KK. ☐ ☐ ☐ Prepare 6 minute timer LL. ☐ ☐ ☐ ACID Pump to **RUN** and start timer MM. At 6 minute timer end ACID Pump to **STOP** NN. ☐ ☐ ☐ Open Column Loading loop 2 on **[Sample Collection]**.tab → **[Column Loading]**.tab by pressing the purple 36/38 button i. The previous loop valves will automatically close when another loop is opened OO. ☐ ☐ ☐ Prepare 75 second timer (1 min: 15 sec) PP. ☐ ☐ ☐ ACID Pump to **RUN** and start timer QQ. At 75 second timer end ACID Pump to **STOP** RR. Repeat steps **87.7.NN – 87.7.QQ** for each Column Loading loop, actuating the loop valves using the following purple buttons on **[Sample Collection]**.tab → **[Column Loading]**.tab i. ☐ ☐ ☐ Loop 3: Valves 40/42 ii. ☐ ☐ ☐ Loop 4: Valves 44/46 iii. ☐ ☐ ☐ Loop 5: Valves 48/50 iv. ☐ ☐ ☐ Loop 6: Valves 52/54 v. ☐ ☐ ☐ Loop 7: Valves 56/58 vi. ☐ ☐ ☐ Loop 8: Valves 60/62 SS. ☐ ☐ ☐ Verify ACID Pump to **STOP** TT. ***NOTE – loop 9 does not get washed as it is not used to collect a sample*** 87.8. Purging all liquid from all acid paths A. Outside of recovery glovebox i. Verify V-2038 for nitrogen service is closed ii. Verify regulator set to 5 psig B. Inside of recovery glovebox i. Verify acid injection port valve V-2001 closed (valve is after acid pump and before acid flow meter) ii. Verify existing ¼ in. FEP tubing line attached to acid injection port valve V-2001 a. FEP tubing is attached to stainless steel line that passes across glovebox wall and ends near the solenoid vent valves manifold C. Outside of recovery glovebox

Step	Action
	i. Verify V-2038 is open for nitrogen service D. ☐ ☐ ☐ Verify flow path through acid flow meter 163/164 (valves already open) E. ☐ ☐ ☐ Open V-0009 (Target Mixing path) F. ☐ ☐ ☐ Open Target Mixing loop 1 on **[Sample Collection]**.tab → **[Target Mixing]**.tab by pressing the purple 100/102 button G. ☐ ☐ ☐ Open V-0153/0154 (Target Mixing path to Dump Tank path) H. ☐ ☐ ☐ Open V-0172/0173 (Target Mixing path to Acid Rinse bottle) i. Selecting this valve closes both 147/148 (frit to Dump Tank) and 149/150 (bypass to Dump Tank) I. ☐ ☐ ☐ Open V-0156 (Effluent bottle vent) i. **V-0156 MUST BE OPEN DURING OPERATIONS – WHEN OPERATIONS ARE NOT BEING CONDUCTED CLOSE THE VALVE** J. ☐ ☐ ☐ Prepare 30 second timer K. Open acid injection port valve V-2001 and start timer L. At 30 second timer end close injection port valve V-2001 M. ☐ ☐ ☐ Open Target Mixing loop 2 on **[Sample Collection]**.tab → **[Target Mixing]**.tab by pressing the purple 104/106 button i. The previous loop valves will automatically close when another loop is opened N. ☐ ☐ ☐ Prepare 30 second timer O. Open acid injection port valve V-2001 and start timer P. At 30 second timer end close injection port valve V-2001 Q. Repeat steps **87.8.M – 87.8.P** for each Target Mixing loop, actuating the loop valves using the following purple buttons on **[Sample Collection]**.tab → **[Target Mixing]**.tab i. ☐ ☐ ☐ Loop 3: Valves 108/110 ii. ☐ ☐ ☐ Loop 4: Valves 112/114 iii. ☐ ☐ ☐ Loop 5: Valves 116/118 iv. ☐ ☐ ☐ Loop 6: Valves 120/122 v. ☐ ☐ ☐ Loop 7: Valves 124/126 vi. ☐ ☐ ☐ Loop 8: Valves 128/130

Step	Action
	R. ☐ ☐ ☐ Verify injection port valve V-2001 is closed S. ***NOTE – loop 9 does not get washed as it is not used to collect a sample*** T. ☐ ☐ ☐ Close V-0009 (Target Mixing path) U. ☐ ☐ ☐ Close V-0153/0154 (Target Mixing path to Dump Tank path) V. ☐ ☐ ☐ Close V-0172/0173 (Target Mixing path to Acid Rinse bottle) W. ☐ ☐ ☐ Verify flow path through acid flow meter 163/164 (valves are already open) X. ☐ ☐ ☐ Open V-0010 (Column Loading path) Y. ☐ ☐ ☐ Open V-0014/0015 (Acid column bypass) using toggle switch at lower left corner of **[System]**.tab (using column bypass in case column still attached to system) Z. ☐ ☐ ☐ Verify flow path through acid column loading filter 18/19 (valves are already open) AA. ☐ ☐ ☐ Open Column Loading loop 1 on **[Sample Collection]**.tab → **[Column Loading]**.tab by pressing the purple 32/34 button BB. ☐ ☐ ☐ Open V-0139 (Acid rinse) CC. ☐ ☐ ☐ Verify V-0156 (Effluent bottle vent) DD. ☐ ☐ ☐ Prepare 30 second timer EE. Open acid injection port valve V-2001 and start timer FF. At 30 second timer end close injection port valve V-2001 GG. ☐ ☐ ☐ Open Column Loading loop 2 on **[Sample Collection]**.tab → **[Column Loading]**.tab by pressing the purple 36/38 button i. The previous loop valves will automatically close when another loop is opened HH. ☐ ☐ ☐ Prepare 30 second timer II. Open acid injection port valve V-2001 and start timer JJ. At 30 second timer end close injection port valve V-2001 KK. Repeat steps **87.8.GG – 87.8.JJ** for each Column Loading loop, actuating the loop valves using the following purple buttons on **[Sample Collection]**.tab → **[Column Loading]**.tab i. ☐ ☐ ☐ Loop 3: Valves 40/42 ii. ☐ ☐ ☐ Loop 4: Valves 44/46

Step	Action
	iii. ☐ ☐ ☐ Loop 5: Valves 48/50 iv. ☐ ☐ ☐ Loop 6: Valves 52/54 v. ☐ ☐ ☐ Loop 7: Valves 56/58 vi. ☐ ☐ ☐ Loop 8: Valves 60/62 LL. ☐ ☐ ☐ Verify injection port valve V-2001 is closed MM. ***NOTE – loop 9 does not get washed as it is not used to collect a sample*** NN. ☐ ☐ ☐ Close V-0010 (Column Loading path) OO. ☐ ☐ ☐ Close V-0139 (Acid rinse) PP. ☐ ☐ ☐ Close V-0156 (Effluent bottle vent) 87.9. Return to step **87.6**
88.	**Wash Out of Base Sample Loops** **– YOU ARE OPERATING THE SYSTEM IN MANUAL MODE –** 88.1. Outside of recovery glovebox **THE V-2038 for nitrogen service IS TO REMAIN ATTACHED TO THE GLOVEBOX – DO NOT REMOVE THIS VALVE** A. Verify V-2038 for nitrogen service is closed B. Verify N_2 tank attached and set to 5 psig i. If not attached see **step 81.1** then return to this step C. **DO NOT OPEN V-2038 for nitrogen service** 88.2. Inside of recovery glovebox A. Verify base system purge port valve V-2033 closed (valve is after base pump and before flow meter) B. If present remove needle port guide from base system purge port valve V-2033 i. Needle port guide is three pieces a. Needle port guide nut, Septum, and White ¼ in. Teflon one-piece ferrule C. Verify check valve V-0406 attached to end of ¼ in. FEP tubing line from V-2038 i. Prevents potential of glovebox atmosphere exiting V-2038 D. Attach existing ¼ in. FEP tubing line to base system purge port valve V-2033 via check valve V-0406

Step	Action
	i. FEP tubing is attached to a stainless steel line that passes across glovebox wall and ends near the solenoid vent valves manifold 88.3. Outside of recovery glovebox A. Open V-2038 for nitrogen service 88.4. **YOU WILL BE PASSING THROUGH THESE STEPS AT LEAST TWICE** A. First pass mark the black boxes ☐ B. Second pass mark the red boxes ☐ C. Third pass mark the blue boxes ☐ D. Fourth pass (if required) mark the green boxes ☐ i. NOTE – only **steps 88.6.A** through **88.6.AA** have the green boxes 88.5. At LabVIEW computer AND inside recovery glovebox 88.6. Removing residual liquid from purged sample loops A. ☐ ☐ ☐ ☐ Verify flow path through base flow meter 167/168 (valves are already open) B. ☐ ☐ ☐ ☐ Open V-0024/0025 (Base column bypass) using toggle switch at lower left corner of **[System]**.tab (using column bypass in case column still attached to system) C. ☐ ☐ ☐ ☐ Verify flow path through base column loading filter 28/29 (valves are already open) D. ☐ ☐ ☐ ☐ Open Column Stripping loop 1 on **[Sample Collection]**.tab → **[Column Stripping]**.tab by pressing the purple 66/68 button E. ☐ ☐ ☐ ☐ Open V-0134 (Base rinse) F. ☐ ☐ ☐ ☐ Open V-0156 (Effluent bottle vent) i. **V-0156 MUST BE OPEN DURING OPERATIONS – WHEN OPERATIONS ARE NOT BEING CONDUCTED CLOSE THE VALVE** G. ☐ ☐ ☐ ☐ Insert needle for Column Stripping loop 1into 60 mL square PETG bottle with septum closure H. ☐ ☐ ☐ ☐ Open Column Stripping loop 1 retrieval valves on **[Sample Collection]**.tab → **[Column Stripping]**.tab by pressing the purple 67/69 button I. ☐ ☐ ☐ ☐ Close V-0134 (Base rinse) J. ☐ ☐ ☐ ☐ Prepare 10 second timer

Step	Action
	K. Open base system purge port valve V-2033 and start timer (pre- and post-acid pump pressures will rise) L. At 10 second timer end close base system purge port valve V-2033 M. ☐ ☐ ☐ ☐ Close Column Stripping loop 1 retrieval valves on **[Sample Collection]**.tab → **[Column Stripping]**.tab by pressing the green 67/69 button N. ☐ ☐ ☐ ☐ Open V-0134 (Base rinse) O. ☐ ☐ ☐ ☐ Remove and stow needle for Column Stripping loop 1 P. ☐ ☐ ☐ ☐ Open Column Stripping loop 2 on **[Sample Collection]**.tab → **[Column Stripping]**.tab by pressing the purple 70/72 button i. The previous loop valves will automatically close when another loop is opened Q. ☐ ☐ ☐ ☐ Insert needle for Column Stripping loop 2 into 60 mL square PETG bottle with septum closure R. ☐ ☐ ☐ ☐ Open Column Stripping loop 2 retrieval valves on **[Sample Collection]**.tab → **[Column Stripping]**.tab by pressing the purple 71/73 button S. ☐ ☐ ☐ Close V-0134 (Base rinse) T. ☐ ☐ ☐ ☐ Prepare 10 second timer U. Open base system purge port valve V-2033 and start timer V. At 10 second timer end close base system purge port valve V-2033 W. ☐ ☐ ☐ ☐ Close Column Stripping loop 2 retrieval valves on **[Sample Collection]**.tab → **[Column Stripping]**.tab by pressing the green 71/73 button X. ☐ ☐ ☐ ☐ Open V-0134 (Base rinse) Y. ☐ ☐ ☐ ☐ Remove and stow needle for Column Stripping loop 2 Z. Repeat steps **88.6.P – 88.6.Y** for each Column Stripping loop, actuating the loop valves using the following purple/green buttons on **[Sample Collection]**.tab → **[Column Stripping]**.tab i. ☐ ☐ ☐ ☐ Loop 3: Valves 74/76 & 75/77 ii. ☐ ☐ ☐ ☐ Loop 4: Valves 78/80 & 79/81 iii. ☐ ☐ ☐ ☐ Loop 5: Valves 82/84 & 83/85 iv. ☐ ☐ ☐ ☐ Loop 6: Valves 86/88 & 87/89 v. ☐ ☐ ☐ ☐ Loop 7: Valves 90/92 & 91/93

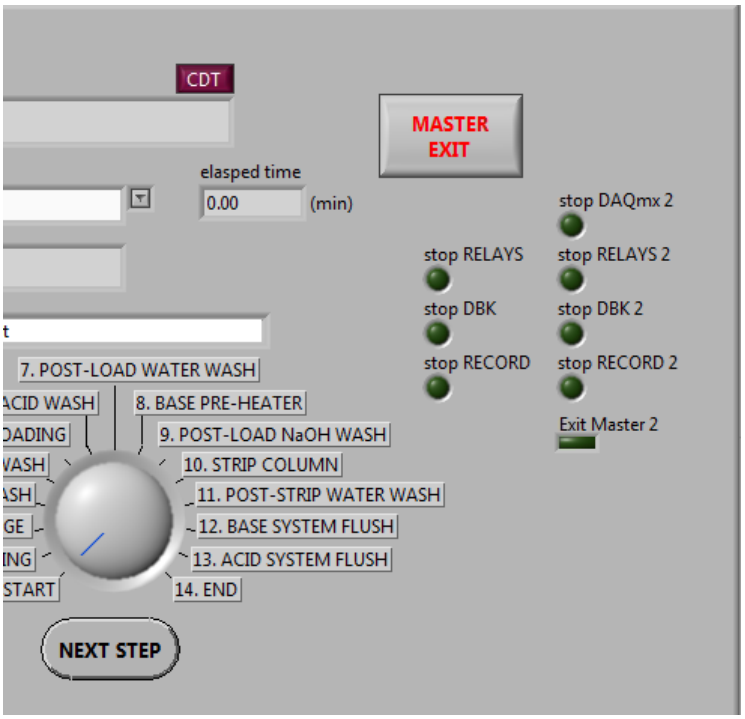
Step	Action
	vi. ☐ ☐ ☐ ☐ Loop 8: Valves 94/96 & 95/97 AA. ☐ ☐ ☐ ☐ Verify base system purge port valve V-2033 is closed BB. ***NOTE – loop 9 does not get washed as it is not used to collect a sample*** CC. Was this the third pass for this section? (blue squares ☐ checked) **PICK ONE** i. NO – go to **step 88.7** ***OR*** ii. YES – go **step 89** 88.7. Filling all sample loops with fresh H_2O A. Verify BASE Pump controller powered ON (rocker switch under front/left of BASE Pump V300 controller) B. ☐ ☐ ☐ Open V-0006 (Fresh H_2O, base manifold) C. ☐ ☐ ☐ Verify flow path through base flow meter 167/168 (valves are already open) D. ☐ ☐ ☐ Open V-0024/0025 (Base column bypass) using toggle switch at lower left corner of **[System]**.tab (using column bypass in case column still attached to system) E. ☐ ☐ ☐ Verify flow path through base column loading filter 28/29 (valves are already open) F. ☐ ☐ ☐ Open Column stripping loop 1 on **[Sample Collection]**.tab → **[Column Stripping]**.tab by pressing the purple 66/68 button G. ☐ ☐ ☐ Open V-0134 (Base rinse) H. ☐ ☐ ☐ Open V-0156 (Effluent bottle vent) i. **V-0156 MUST BE OPEN DURING OPERATIONS – WHEN OPERATIONS ARE NOT BEING CONDUCTED CLOSE THE VALVE** I. ☐ ☐ ☐ Enter flow rate **100** mL/min *Base Flow Rate Set Pt* @ **[System]**.tab J. ☐ ☐ ☐ Record calculated _____ *% Base Motor Power* @ **[System]**.tab K. ☐ ☐ ☐ Verify/Adjust BASE Pump Controller to *% Base Motor Power* for desired flow rate

Step	Action
	i. If a lower % motor power value is required due to pressure readings adjust *Base Flow Rate Set Pt* until calculated *% Base Motor Power* matches % motor power reading at controller L. ☐ ☐ ☐ Prepare 6 minute timer M. ☐ ☐ ☐ BASE Pump to **RUN** and start timer N. At 6 minute timer end BASE Pump to **STOP** O. ☐ ☐ ☐ Open Column Stripping loop 2 on **[Sample Collection]**.tab → **[Column Stripping]**.tab by pressing the purple 70/72 button i. The previous loop valves will automatically close when another loop is opened P. ☐ ☐ ☐ Prepare 75 second timer (1 min: 15 sec) Q. ☐ ☐ ☐ BASE Pump to **RUN** and start timer R. At 75 second timer end BASE Pump to **STOP** S. Repeat steps **88.7.O – 88.7.R** for each Column Stripping loop, actuating the loop valves using the following purple buttons on **[Sample Collection]**.tab → **[Column Stripping]**.tab i. ☐ ☐ ☐ Loop 3: Valves 74/76 ii. ☐ ☐ ☐ Loop 4: Valves 78/80 iii. ☐ ☐ ☐ Loop 5: Valves 82/84 iv. ☐ ☐ ☐ Loop 6: Valves 86/88 v. ☐ ☐ ☐ Loop 7: Valves 90/92 vi. ☐ ☐ ☐ Loop 8: Valves 94/96 T. ☐ ☐ ☐ Verify BASE Pump to **STOP** U. ***NOTE – loop 9 does not get washed as it is not used to collect a sample*** V. ☐ ☐ ☐ Close V-0006 (Fresh H_2O, base feed manifold) W. ☐ ☐ ☐ Close V-0134 (Base rinse) X. ☐ ☐ ☐ Close V-0156 (Effluent bottle vent) 88.8. Purging all liquid from all base paths A. Outside the glovebox i. Verify V-2038 for nitrogen service is open ii. Verify N_2 tank attached and set to 5 psig a. If not attached see step **81.1** then return to this step

Step	Action
	B. Inside of recovery glovebox i. Verify base system purge port valve V-2033 closed (valve is after base pump and before base flow meter) C. Verify ¼ in. FEP tubing nitrogen purge line attached to base system purge port valve V-2033 i. FEP tubing is attached to a stainless steel line that passes across glovebox wall and ends near the solenoid vent valves manifold D. Outside of recovery glovebox i. Verify V-2038 for nitrogen service open E. ☐ ☐ ☐ Verify flow path through base flow meter 167/168 (valves are already open) F. ☐ ☐ ☐ Open V-0024/0025 (Base column bypass) using toggle switch at lower left corner of **[System]**.tab (using column bypass in case column still attached to system) G. ☐ ☐ ☐ Verify flow path through base column loading filter 28/29 (valves are already open) H. ☐ ☐ ☐ Open Column stripping loop 1 on **[Sample Collection]**.tab → **[Column Stripping]**.tab by pressing the purple 66/68 button I. ☐ ☐ ☐ Open V-0134 (Base rinse) J. ☐ ☐ ☐ Open V-0156 (Effluent bottle vent) i. **V-0156 MUST BE OPEN DURING OPERATIONS – WHEN OPERATIONS ARE NOT BEING CONDUCTED CLOSE THE VALVE** K. ☐ ☐ ☐ Prepare 30 second timer L. Open base system purge port valve V-2033 and start timer M. At 30 second timer end close base system purge port valve V-2033 N. ☐ Open Column Stripping loop 2 on **[Sample Collection]**.tab → **[Column Stripping]**.tab by pressing the purple 70/72 button i. The previous loop valves will automatically close when another loop is opened O. ☐ ☐ ☐ Prepare 30 second timer P. Open base system purge port valve V-2033 and start timer Q. At 30 second timer end close base system purge port valve V-2033

Step	Action
	R. Repeat steps **88.8.N – 88.8.Q** for each Column Stripping loop, actuating the loop valves using the following purple buttons on **[Sample Collection]**.tab → **[Column Stripping]**.tab i. ☐ ☐ ☐ Loop 3: Valves 74/76 ii. ☐ ☐ ☐ Loop 4: Valves 78/80 iii. ☐ ☐ ☐ Loop 5: Valves 82/84 iv. ☐ ☐ ☐ Loop 6: Valves 86/88 v. ☐ ☐ ☐ Loop 7: Valves 90/92 vi. ☐ ☐ ☐ Loop 8: Valves 94/96 S. ☐ ☐ ☐ Verify base system purge port valve V-2033 is closed T. ***NOTE – loop 9 does not get washed as it is not used to collect a sample*** U. ☐ ☐ ☐ Close V-0134 (Base rinse) V. ☐ ☐ ☐ Close V-0156 (Effluent bottle vent) 88.9. Return to step **88.6**
89.	**Summary** 89.1. All loops have now been washed and purged with N_2 89.2. All loops are now ready to receive samples 89.3. If any acid flow paths are used then repeat steps **87.6** through **87.9** for the appropriate loops 89.4. If any base flow paths are used then repeat steps **88.6** through **88.9** for the appropriate loops
90.	**End of Run** \\ **Initials: _____ Date: _____ Time: _____** 90.1. ☐ Enter any final comments 90.2. ☐ Press the **MASTER EXIT** button at **[System]**.tab A. Properly stops Mo99 Remote Recovery Data Acquisition & Control Software

Step	Action



90.3. ☐ Data saved to USB stick \\ **Initials: _____ Date: _____ Time: _____**

A. Insert USB stick into computer

B. Use SyncToy 2.1 to backup files

C. Select *All Folder Pairs* (left side of window)

D. ONLY check-mark the following items (in the *Active* column)

i. C_to_USB_Labuser_docs

ii. C_to_USB_Public_docs

iii. C_to_USB_temp1

E. Press the **Run All** button (bottom right corner of window)

F. Wait for backup to complete

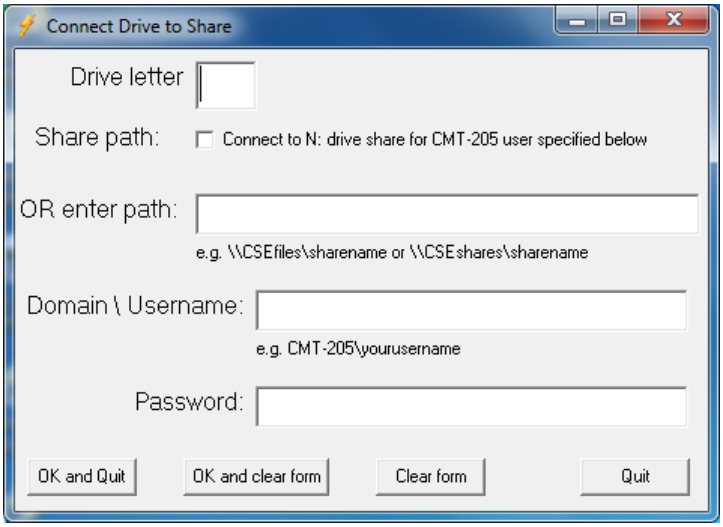
Step	Action
	G. Left click the *Safely Remove Hardware* icon and select ***Safely Remove G: drive***

H. ☐ Data saved to GTRI Mo99 Production Tests Share Drive

\\ Initials: _____ Date: _____ Time: _____

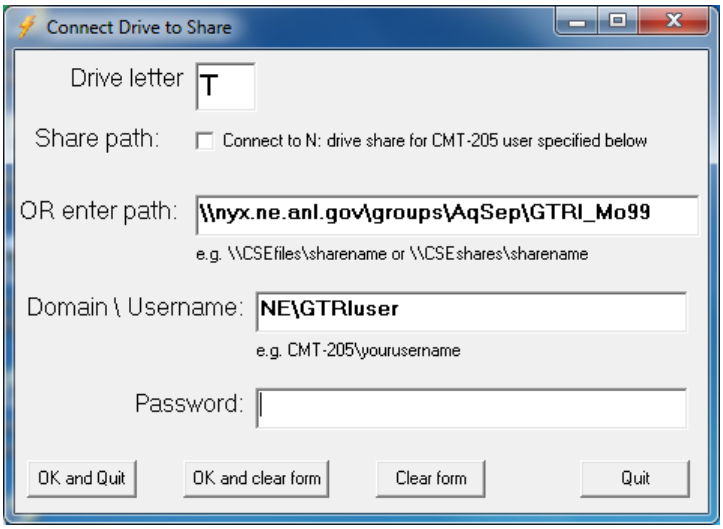
I. Open Connect Share

i. At "Drive Letter" enter T



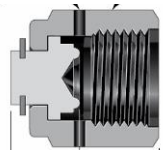
ii. Then click the "Password" field

a. The screen will change to

Step	Action
	 iii. In the "Password" field type: HY1137hy iv. Press the "OK and Quit" button v. The message "T drive is connected." should appear, click **OK** J. Use SyncToy 2.1 to backup files  i. Select *All Folder Pairs* (left side of window) ii. **ONLY** check-mark the following items (in the *Active* column) a. C_to_Tshare_labuser b. C_to_Tshare_public c. C_to_Tshare_temp1 iii. Press the **Run All** button (bottom right corner of window) iv. Wait for backup to complete K. Open Windows Explorer (folder view) L. Right click the T: drive and select ***Disconnect***
91.	**Prepare to Remove Shielded Effluent Bottle Cart** 91.1. Check most current RWP A. ☐ Sign most current RWP B. ☐ Check most current RWP for PPE requirements 91.2. ***It is recommended that two people retrieve samples***

Step	Action
	A. Person A performs the work in cabinet #3 B. Person B communicates/marks these instructions to Person A
92.	**<u>Remove Shielded Effluent Bottle Cart</u>** 92.1. At LabVIEW rack A. Power off appropriate effluent balance indicator 92.2. At recovery glovebox A. Open cabinet #3 (right side) door to full open B. Position the 4-section ramps to roll shield effluent bottle cart out of cabinet #3 C. Attach the handle to the effluent cart (handle and handle bolts should have been stored in the instrument room until effluent cart is removed) D. Remove jacks used to level shielded effluent bottle cart (stored in cabinet #3 once effluent cart is removed)  E. ☐ Verify balance lever in transport positon F. It may be necessary to pull the effluent cart out of cabinet #3 a little to break the liquid connections G. ☐ Close <u>BOTH</u> V-2012 & V-2031 for PRE-LOAD ACID WASH liquid line i. V-2012 is connected to the line to the glovebox ii. V-2031 is mounted to the manifold panel attached to the cart H. ☐ Disconnect V-2012 of PRE-LOAD ACID WASH glovebox line from effluent bottle // glovebox liquid manifold connection I. Wipe open end of V-2012 for PRE-LOAD ACID WASH liquid line from glovebox using a paper towel (may be dampened with Radiac wash) i. **FITTING MUST BE DRY** J. Install VCR cap on open end V-2012 for PRE-LOAD ACID WASH liquid line from glovebox

Step	Action



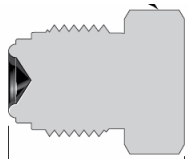
SS-4-VCR-CP, ¼ in. VCR cap

K. Wipe open end of female VCR nut for PRE-LOAD ACID WASH liquid line on effluent bottle cart using a paper towel (may be dampened with Radiac wash)

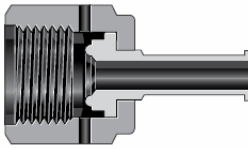
i. **FITTING MUST BE DRY**

L. Install VCR plug on open end of female VCR nut for PRE-LOAD ACID WASH liquid line on effluent bottle cart

i. VCR plugs require a VCR gasket



SS-4-VCR-P, ¼ in. VCR plug



¼ in. Female VCR nut (this end on effluent cart, VCR plug attaches to here)

M. Repeat steps **92.2.G – 92.2.L** for each liquid line valve set:

i. ☐ POST-LOAD ACID WASH valves V-2013 & V-2039

ii. ☐ POST-LOAD H_2O WASH valves V-2014 & V-2027

iii. ☐ ACID RINSE valves V-2018 & V-2019

iv. ☐ POST-LOAD NaOH WASH valves V-2015 & V-2025

v. ☐ POST-STRIP H_2O WASH valves V-2016 & V-2023

vi. ☐ BASE RINSE valves V-2017 & V-2021

92.3. Roll shielded effluent bottle cart to edge of cabinet #3

92.4. ☐ Disconnect balance cable (cables are twist lock and connectors operate smoothly when mated to one another correctly)

92.5. Stow glovebox balance cable on hook inside of cabinet #3 (prevents crushing by effluent cart)

92.6. Stow effluent balance cable on effluent cart handle (prevents crushing by effluent cart)

92.7. ☐ Disconnect liquid leak sensor cable

92.8. Stow glovebox leak sensor cable on hook inside of cabinet #3 (prevents crushing by effluent cart)

Step	Action
	92.9. Stow effluent leak sensor cable on effluent cart handle (prevents crushing by effluent cart) 92.10. ☐ Close V-2011 for glovebox vent line 92.11. ☐ Close V-2032 for PRE-LOAD ACID WASH vent line 92.12. ☐ Close V-2030 for POST-LOAD ACID WASH vent line 92.13. ☐ Close V-2028 for POST-LOAD H_2O WASH vent line 92.14. ☐ Close V-2020 for ACID RINSE vent line 92.15. ☐ Close V-2026 for POST-LOAD NaOH WASH vent line 92.16. ☐ Close V-2024 for POST-STRIP H_2O WASH vent line 92.17. ☐ Close V-2022 for BASE RINSE vent line 92.18. ☐ Disconnect V-2011 of glovebox vent line from effluent bottle // glovebox vent manifold connection 92.19. Wipe open end of V-2011 for vent line from glovebox using a paper towel (may be dampened with Radiac wash) A. **FITTING MUST BE DRY** 92.20. Install VCR cap on open end of V-2011 vent line from glovebox A. VCR caps require a VCR gasket 92.21. Wipe open end of female VCR nut for vent line on effluent bottle cart using a paper towel (may be dampened with Radiac wash) A. **FITTING MUST BE DRY** 92.22. Install VCR plug on open end of female VCR nut for vent line on effluent bottle cart A. VCR plugs require a VCR gasket 92.23. Remove shielded effluent bottle cart from cabinet #3 92.24. Remove 4-section ramps A. **DO NOT STORE 4-SECTION RAMPS IN CELL 1** Close cabinet #3 door slowly

4 Records Created by Work Process

The records listed below must be retained as indicated.

Description of Record (include form number if applicable)	Active Records Custodian	Active Records Retention	Indexing Method, Storage Medium	Federal Retention Requirements*
LEAF-PROC-024 sections 3.2.1–3.2.6, data	Facility Manager	3 years	Index by job date and name, store on	Destroy 75 years after the date of the permit

Description of Record (include form number if applicable)	Active Records Custodian	Active Records Retention	Indexing Method, Storage Medium	Federal Retention Requirements*
collection pages			paper or electronically	(DOE ADM 18.37)

*If records are maintained in a business information system that is not currently programmed to purge digital records based on age, the records may be retained in that system past the indicated destruction date.

5 Exhibit A – Hardware List

1. Swagelok VCR stainless steel gaskets: SS-4-VCR-2-GR for ¼ in
2. Verification Tank Lines
    a. Pickup line attached at bottom of tank through 1-1/2 in tri-clamp fitting
        i. ¼ in. OD x 0.21 in. ID 316SS tubing (McMaster 89785K822)
        ii. ¼ in. Swagelok x ¼ in. Swagelok union (SS-400-6)
        iii. ¼ in. OD x 3/16 in. ID FEP tubing (McMaster 2129T13)
        iv. Stainless steel ferrules can be used with FEP tubing
        v. ¼ in. Swagelok x ¼ in. VCR female connector (SS-4-WVCR-6-400)
        vi. ¼ in. double VCR male 2-way ball valve (SS-43GVCR4)
    b. Return line attached to tank cover
        i. ¼ in. OD x 0.21 in. ID 316SS tubing (McMaster 89785K512)
        ii. ¼ in. Swagelok x ¼ in. Swagelok union (SS-400-6)
        iii. ¼ in. OD x 3/16 in. ID FEP tubing (McMaster 2129T13)
        iv. ¼ in. Swagelok x ¼ in. VCR female connector (SS-4-WVCR-6-400)
        v. ¼ in. double VCR male 2-way ball valve (SS-43GVCR4)
    c. 1/8 in sample pickup & vent lines
        i. Vent line attached to tank cover
        ii. ⅛ in. OD x 0.055 in. ID 316SS tubing (McMaster 89785K511)
        iii. ⅛ in. Swagelok x ⅛ in. Swagelok union (SS-200-6)
        iv. ⅛ in. OD FEP tubing (McMaster 2129T11)
        v. ⅛ in. Swagelok fittings 2-way ball valve (SS-41GS2)
    d. Sample pickup line attached to tank cover
        i. ⅛ in. OD x 0.055 in. ID 316SS tubing (McMaster 89785K511)
        ii. ⅛ in. Swagelok x ⅛ in. Swagelok union (SS-200-6)
        iii. ⅛ in. OD FEP tubing (McMaster 2129T11)
        iv. ⅛ in. Swagelok fittings 2-way ball valve (SS-41GS2)
3. Feed connection jumper
    a. Feed valve V-3003 (SS-43GXS4)
        i. Middle arm to V-3001

1. ¼ in. OD x 3/16 in. ID FEP tubing (McMaster 2129T13)
2. ¼ in. Swagelok x ¼ in. VCR female connector (SS-4-WVCR-6-400)
    a. VCR female end faces away from 3-way valve
3. Middle arm of V-3001 to V-0003
    a. ¼ in. OD x 3/16 in. ID FEP tubing (McMaster 2129T13)
    b. ¼ in. Swagelok x ¼ in. VCR female connector (SS-4-WVCR-6-400)
        i. VCR female end faces away from 3-way valve

ii. Side arm to Verification tank pickup line V-2034
1. ¼ in. OD x 3/16 in. ID FEP tubing (McMaster 2129T13)
2. ¼ in. Swagelok x ¼ in. VCR female connector (SS-4-WVCR-6-400)
    a. VCR female end faces away from 3-way valve
3. Verification tank pickup 2-way ball valve ends in male VCR
4. VCR female end from feed jumper attaches to free male end of liquid pickup VCR 2-way ball valve

iii. Side arm from feed bottle inside glovebox
1. ¼ in. OD x 3/16 in. ID FEP tubing (McMaster 2129T13)

4. Effluent connection jumper
    a. Effluent valve V-3002 (SS-43GXS4)
        i. Middle arm from V-0011
            1. ¼ in. Swagelok x ¼ in. VCR female connector (SS-4-WVCR-6-400)
                a. VCR female end faces away from 3-way valve and attaches to V-0011
            2. ¼ in. OD x 3/16 in. ID FEP tubing (McMaster 2129T13)
        ii. Side arm to Verification tank return V-2036
            1. ¼ in. OD x 3/16 in. ID FEP tubing (McMaster 2129T13)
            2. ¼ in. Swagelok x ¼ in. VCR female connector (SS-4-WVCR-6-400)
                a. VCR female end faces away from 3-way valve
            3. Verification tank return 2-way ball valve ends in male VCR
            4. VCR female end from effluent jumper attaches to free male end of liquid pickup VCR 2-way ball valve
        iii. Side arm to effluent bottle inside glovebox

1. ¼ in. OD x 3/16 in. ID FEP tubing (McMaster 2129T13)

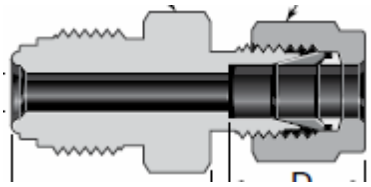
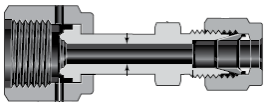
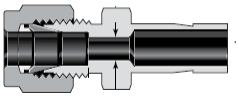


Figure 1¼ in. OD FEP tubing with SS-4-VCR-6-400 attached to each end

5. Alternative Sample Retrieval fitting assembly (p. 30)
    a. SS-4-WVCR-6-400, ¼ in. VCR x ¼ in. Swagelok nut 
    b. SS-200-R-4, 1/8 in. Swagelok nut x ¼ in. tubing stub 
    c. 1/8 in. OD FEP tubing from lower stopcock attached to the 1/8 in. Swagelok nut
6. Feed Bottle Secondary Tray
    a. McMaster 26775T23
        i. Polypropylene Plastic Pan 20-3/8” long, 19” wide, 3-1/4” high
    b. McMaster 4141T5
        i. Food-Grade Polyethylene Plastic Pan 23” long, 19” wide, 4” high
7. Effluent Bottles with silicone seals
    a. McMaster 4322T4

Semi-Clear Polypropylene Plastic Jar 64 oz./1900 ml Capacity, 5” Diameter			
Style	M	Mouth OD	2 ¾”
Capacity	64 oz./1900 mL	Graduated	No
Diameter	5”	Seal Material	Silicone Rubber
Height	8 3/8”	Includes	Lid (threaded)

    b. McMaster 4322T6

Semi-Clear Polypropylene Plastic Jar 1 Gallon/3775 ml Capacity			
Style	M	Mouth OD	2 ¾”
Capacity	1 gal./3775 ml	Graduated	No
Diameter	5 7/8”	Seal Material	Silicone Rubber
Height	11 ¼”	Includes	Lid (threaded)

    c. McMaster 4322T7

Semi-Clear Polypropylene Plastic Jar 2 Gallon/7575 ml Capacity			
Style	M	Mouth OD	2 ¾”
Capacity	2 gal./7575 ml	Graduated	No
Diameter	7 1/2”	Seal Material	Silicone Rubber
Height	13 1/8”	Includes	Lid (threaded)

8. Acid Rinse Bottle (Base Rinse Bottle is same model)
    a. McMaster 9884T14

Semi-Clear High-Density Polyethylene Plastic Jug 5 Gallon/18950 ml Capacity			
Capacity	5 gal./18950 ml	Mouth OD	3 7/8”
Width	9”	Graduation	¼ gal / 1 L
Depth	12 ¾”	Includes	Lid (threaded)
Height	15 ¾”	Handle	Stainless Steel

9. Effluent bottle connections
    a. ¼ in. Liquid connections
        i. Swagelok SS-400-1-OR
            1. ¼ in. tube x 7/16-20 straight thread O-Seal
            2. Use McMaster 94758A645 18-8 Stainless Steel Flange Nut
        ii. ¼ in. OD x 3/16 in. ID FEP tubing (McMaster 2129T13)
            1. ¼ in. Swagelok x ¼ in. VCR female connector (SS-4-WVCR-6-400)
            2. ¼ in. double VCR male 2-way ball valve (SS-43GVCR4)
                a. HANDLE POINTS TO BOTTLE (DIRECTION OF FLOW TO BOTTLE)
                b. ¼ in. VCR female cap (SS-4-VCR-CP)
                    i. Cap attached to valve when bottle not attached to effluent bottle cart liquid manifold
            3. 2-way ball valve from bottle attaches to female VCR nut on inside face of effluent bottle // glovebox liquid manifold
                    i. Effluent bottle // glovebox liquid manifold is permanently attached to an shielded effluent bottle cart
        iii. Effluent lines from glovebox
            1. There are 7 effluent lines
                a. Acid effluent lines (hexagon tags)
                    i. Pre-load acid wash
                    ii. Post-load acid wash
                    iii. Post-load water wash
                    iv. Acid rinse
                b. Base effluent lines (diamond tags)
                    i. Post-load NaOH wash
                    ii. Post-strip water wash

iii. Base rinse

2. Terminated with VCR 2-way ball valve (SS-43GVCR4)
    a. **HANDLE POINTS AWAY FROM GLOVEBOX (DIRECTION OF FLOW TO BOTTLE)**
3. Rotating VCR female union elbow (6LV-4-WVCR-9-DF)
    a. Attached to valve outlet
4. ¼ in. VCR male union (SS-4-VCR-6-DM)
    a. Attached to elbow outlet
5. Free end of VCR male union attaches to female VCR fitting at outside face of effluent bottle // glovebox liquid manifold

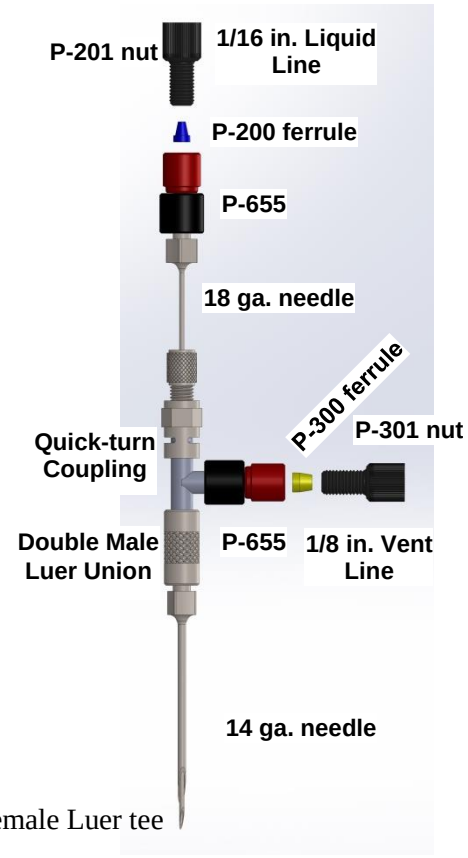
b. 1/8 in. Vent connections

i. Swagelok SS -200-1-OR

1. 1/8 in. tube x 5/16-24 straight thread O-Seal
    a. Use McMaster 93776A451 18-8 Stainless Steel Serrated Flange Locknut
2. 1/8 in. OD FEP tubing (McMaster 2129T11)
3. 1/8 in. tube x ¼ in. stub reducer (SS-200-R-4)
4. ¼ in. Swagelok x ¼ in. VCR female connector (SS-4-WVCR-6-400)
5. ¼ in. double VCR male 2-way ball valve (SS-43GVCR4)
    a. **HANDLE POINTS AWAY FROM BOTTLE (DIRECTION OF FLOW TO GAS COLLECTION SYSTEM)**
    b. ¼ in. VCR female cap (SS-4-VCR-CP) attached to valve when bottle not attached to effluent bottle cart liquid manifold
6. 2-way ball valve from bottle attaches to VCR female nut on inside face of effluent bottle // glovebox vent manifold
    a. Effluent bottle // glovebox vent manifold is permanently attached to a shielded effluent bottle cart
7. Vent line from glovebox
    a. Single vent line comes out of glovebox
        i. Line attached to solenoid valve 156 that vents to the gas collection system
    b. Terminated with double VCR male 2-way ball valve (SS-43GVCR4)
        i. HANDLE POINTS TOWARD GLOVEBOX (DIRECTION OF FLOW TO GAS COLLECTION)

c. Free end of 2-way valve attaches to female VCR fitting at outside face of effluent bottle // glovebox vent manifold

10. Pre-evacuated vials
    a. Hollister-Stier 10 mL evacuated vial (7519ZA), Fisher Scientific catalog number NC9538328
11. Sampling Retrieval Assemblies
    a. There are two (2x) needles assembled in a concentric fashion
        i. The inner 18 gauge needle handles the liquid (1/16 in. OD tubing)
        ii. The outer 14 gauge needle handles the venting (1/8 in. OD tubing)
    b. Two concentric needle assembly and bottom row 3-way Luer lock valve daisy chain assembly
        i. 14 gauge x 2 in. Luer Lock needle (vent to vacuum)
            1. Fisher 14-815-404
                a. Length (English) Needle 2 in.
                b. Diameter (Metric) Outer 2.11mm
                c. Diameter (Metric) Inner 1.6mm
                d. Length (Metric) Needle 51mm
                e. Needle Point Style Sharp, Bevel, Curved
                f. Needle Gauge 14 ga.
                g. Material (Hub) Nickel Plated Brass
                h. Material Stainless steel
                i. Hamilton 90014



        ii. Connects to double Luer lock male 316SS union
            1. McMaster 5194K31
        iii. Union connects to bottom arm of female Luer tee
            1. Polypropylene Luer adapter tees female
            2. Fisher NC0644969
        iv. Male Luer quick-turn coupling connects to upper arm of female Luer tee
            1. McMaster 5194K41
        v. 18 gauge x 6 in. Luer Lock needle (liquid) inserts through coupling
            1. 18 gauge needle inserts through 14 gauge needle
            2. 18 gauge needle tip should be slightly below 14 gauge needle tip
        vi. Fisher 01-290-25
            1. Length (English) Needle 6 in.
            2. Length (Metric) Needle 152mm
            3. Needle Point Style Deflected End
            4. Needle Gauge 18 ga.

5. Material (Hub) Micro-Mate™ Stainless Steel
6. Cadence Science 9860

vii. Male Luer lock x ¼-28 nut adapter attaches to 18 gauge needle
1. Adapter, Luer (M-lock) to 1/4-28 FB (F) PEEK 1.02 mm (0.040")
a. Fisher 14221-484
b. Upchurch (Idex) P-655
2. Upchurch Scientific™ Flangeless Nuts: Compatible with 1/4-28, Delrin
a. Upchurch Scientific™ P301X
b. Fisher 05-700-102
c. 0.25 to 28 flat bottom, Flangeless standard knurled head nut

viii. 1/16 in. OD FEP tubing attaches from male Luer lock x ¼-28 nut adapter to liquid sample solenoid valve of appropriate sampling loop

ix. 2nd male Luer lock x ¼-28 nut adapter attaches to side arm of female Luer tee

x. 1/8 in. OD FEP tubing attaches from 2nd male Luer lock x ¼-28 nut adapter to side arm of appropriate 3-way valve on bottom row of 3-way valve daisy chain assembly

c. Top row 3-way Luer lock valve daisy chain assembly
i. 1/16 in. OD FEP tubing from appropriate sample loop vent valve to female Luer lock x ¼-28 nut adapter
1. Adapter, Luer (F) to 1/4-28 FB (F), PEEK, 1.27 mm (0.050")
a. Upchurch (Idex) P-658
b. VWR 14221-486

ii. Female Luer lock x ¼-28 nut adapter to one-way Luer lock check valve
1. One-way Luer check valve
a. Fisher NC0232677

iii. One-way Luer check valve to double male Luer lock coupling
1. Cole-Parmer Polycarbonate fittings; male lock x male lock, rotating; 10/pack, Non-sterile
a. Cole-Parmer EW-30600-50
b. Fisher NC0580839

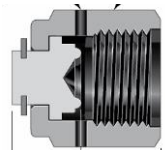
d. FEP TUBING .030 in ID x .062 in. OD x 100 ft
i. Fisher 05-7011-72
ii. This is the 1/16 in. OD tubing

e. FEP tubing 1/8 in. OD x 3/16 in. ID
i. McMaster 2129T13

f. Clean, dry Becton-Dickinson (B-D) 60 mL syringe

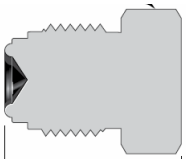
i. Used for checking operability of check valves

12. Vacuum pump for sample retrieval
    a. McMaster, 4404K29, 12VDC
13. Tweezers for retrieving samples from shields
    a. Tweezers, 12 in., McMaster, 7379A24
14. VCR Cap



SS-4-VCR-CP, ¼ in. VCR cap

15. VCR Plug



SS-4-VCR-P, ¼ in. VCR plug

16. Bottles for receiving loop washings
    a. Thermo Scientific™ Nalgene™ Square PETG Media Bottles with Septum Closure
        i. Use non-glass bottles inside the glovebox
        ii. 60 mL: Fisher 03-313-900, Thermo Scientific 3420230060, case of 200
        iii. 125 mL: Fisher 03-313-901, Thermo Scientific 3420230125, case of 96
        iv. 500 mL: Fisher 03-313-902, Thermo Scientific 3420230500, case of 40

6 Exhibit B – PFC-PCS Cheat Sheet

Acid Solution Volumes	(mL)	Water Volumes	(mL)
Leak Checking Column		Post-Load Water Wash	
Pre-Pre-Load Acid		Post-Strip Water Wash (cell)	
Pre-Load Acid		Post-Strip Water Wash (waste)	
Post Load Acid		Base System Final Rinse	
Final Acid Wash		Loop Rinsing	
Loop Rinsing		Base System Rinse	
Acid Rinse Out		Priming Lines	
Line Priming		Total Water Required	
Total Acid Required			
Base Wash Volumes	**(mL)**	**Base Strip Volumes**	**(mL)**
Base Pre-Heater Start		Column Strip	
Post-Load NaOH Wash		Priming Lines	
Priming Lines		Total NaOH Strip Required	
Total NaOH Wash Required			

Solution Properties	Density (g/mL)	Mass (g)	Volume (mL)	Conc.
Acid Solution				
Base Wash Solution				
Base Strip Solution				
Uranyl Sulfate Solution				

Flow Rate Acid Motor Powers	
Flow Rate (mL/min)	Motor %
80	
84	
100	
167	
200	
300	
Flow Rate Base Motor Powers	
Flow Rate (mL/min)	Motor %
84	

Samples Summary					
Target Mixing		Column Loading		Column Stripping	
1. □ Taken	1. □ Recovered	1. □ Taken	1. □ Recovered	1. □ Taken	1. □ Recovered
2. □ Taken	2. □ Recovered	2. □ Taken	2. □ Recovered	2. □ Taken	2. □ Recovered
3. □ Taken	3. □ Recovered	3. □ Taken	3. □ Recovered	3. □ Taken	3. □ Recovered
4. □ Taken	4. □ Recovered	4. □ Taken	4. □ Recovered	4. □ Taken	4. □ Recovered
5. □ Taken	5. □ Recovered	5. □ Taken	5. □ Recovered	5. □ Taken	5. □ Recovered
6. □ Taken	6. □ Recovered	6. □ Taken	6. □ Recovered	6. □ Taken	6. □ Recovered
7. □ Taken	7. □ Recovered	7. □ Taken	7. □ Recovered	7. □ Taken	7. □ Recovered
8. □ Taken	8. □ Recovered	8. □ Taken	8. □ Recovered	8. □ Taken	8. □ Recovered

7 Related Documents

None

8 Definitions

None

9 About this Procedure

Issuing organization:	Low Energy Accelerator Facility
Procedure owner:	S. Chemerisov
Point of contact:	S. Chemerisov
Review cycle (months):	36
Date last revised:	August 23, 2019
Date last reviewed:	August 23, 2019

10 Summary of Changes in This Version

Added Flow Rate Base Motor Powers entries and table with samples taken/retrieved to PFC-PCS Cheat Sheet

Removed all instances of recording dump tank load cell mass

Removed step 27.18 (leveling effluent cart) since this is not done

Step 39, changed 2WV-801 specification to OPEN, to match the hot cell team sheet

Added a statement to steps 42 and 43 that if steps 25 and 26 (respectively) have been performed in the last week, these steps may be skipped.

Changed units and associated calculations in steps 53.8, 54.7, 55.7, 56.7, 58.8, 59.12, 61.10, and 62.9 from minutes to seconds since the countdown timer in the software is also in seconds

Step 59.5C changed formula from 59.5A/59.5B to 59.5A x 59.5B since volume x density = mass

Removed step 61.15 since the system is already shut off from the hot cell at this point and the balance reading will not change from the beginning to the end of this step. Also changed the associated cell on p. 88 to "not applicable"

Added column to the table on p. 88 to document total acid, base wash, base strip, and water used, as well as volume remaining in acid rinse and base rinse receivers so cabinet does not need to be opened to determine if line and sample loop rinsing can commence

Made the font of the table on p. 88 smaller to make it fit on one page

Added "(if the manual dump tank valve cannot be accessed yet, proceed to **step 67**)" to step 65

Moved step 80.2.BB to 80.2.Z, where prompted to open the valve

Moved step 81.4.W to 81.4.U, where prompted to open the valve

Step 82, changed 2WV-801 specification to OPEN, to match the hot cell team sheet

Updated the link in step 85.4

Added step 44.38 to ensure flow path is through the flow meter bypass sample loop

Added step 51.2 to shut off the flow meter bypass loop and isolate the sample during the rest of processing

Added step 75.23 describing how to retrieve a sample from the flow meter bypass sample loop

Added step 77 for sampling the effluent cart bottles and updated links in all subsequent steps to be accurate

02/10/2020. Derek McLain, SSS Div.

Attachment: Mo99 Primary Recovery System – No Specific Path

Argonne Chemical and Fuel Cycle Technologies (CFC) Division drawing

Drawing Title: AMORE_MO99 RECOVERY PROCESS PHASE 2_REV23.VSD

Revision Date: 2/18/2019

Revised by John F. Krebs

AMORE_Mo99
recovery process PHA

APPENDIX 18

Resin Washing Procedure

Resin Washing Procedure

1) Weigh out approximately 200 g of dry resin into a 2 L beaker.
    a. Resin: Zirchrom TiO_2-Bulk-110(60)
2) Add 1 L of 0.5M H_2SO_4 to the beaker and place an overhead stirrer into the solution, 1-2 cm above the bottom of the beaker.
3) Turn on the stirrer and allow to mix for 20 minutes
4) Turn off the stirrer and allow the resin to settle to the bottom of the beaker for approx. 1 min.
5) Decant the used H_2SO_4 to waste.
6) Repeat steps 2-5 an additional four times, for a total of five sulfuric acid washes.
7) Add at least 0.5L of DI water to the beaker and stir for 5 minutes to rinse the resin.
8) Allow the resin to settle to the bottom of the beaker and decant the water to waste.
9) Use fresh DI water to quantitatively transfer the washed resin to a labeled plastic bottle for storage until needed.

Column Packing Procedure

1) Assemble the bottom half of the column to the specified dimensions and clamp it in a ring stand. The bottom collar should be tightened until the edge is 7/8” from the end of the threading.

Figure 1: Column with bottom frit and collar in place and connected to tubing. Top frit and collar also included

Figure 2: Distance from bottom collar to end of threading

2) Place a beaker under the column to catch water as it drains through.
3) If desired, place a mark on the inside of the column 4 ¼" above the bottom frit using a sharpie. This should be the approximate fill line of the column.
4) Slurry the previously washed titania resin (see above) with water and pour it into the column, allowing excess water to drain out the bottom while keeping a small head of fluid over the resin bed. 200 g of dry resin should be slightly more than needed.

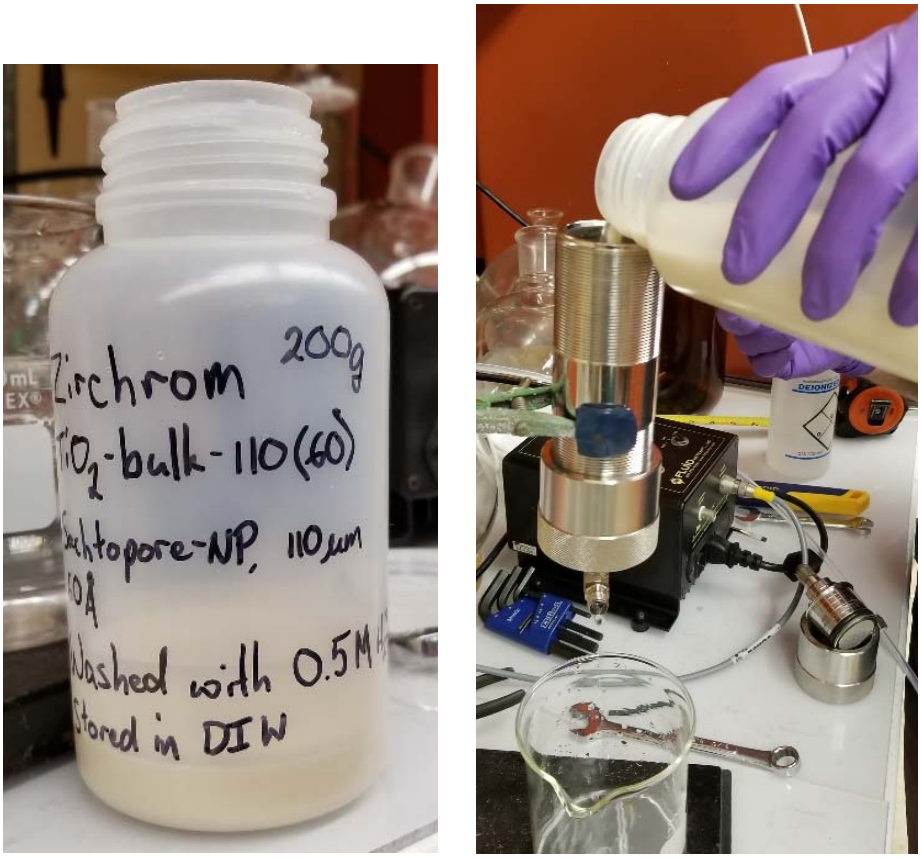


Figure 3: Left) bottle of washed resin (200g) with a small amount of water to slurry with. Right) pouring the slurried resin into the column and allowing the excess water to drip out the bottom into a beaker.

5) After filling to the top of the mark with resin, cap the bottom of the column and install the top frit and collar.
6) Hand tighten the top collar and move it to the vice, being careful not to crush the threads when securing it.
    a. **Note –** When tightening the top frit/collar assembly, water WILL be displaced. To avoid spilling it on the floor, a container should be placed under the column while tightening
7) Finish tightening the collar with a pipe-wrench, making sure the column inlet and outlet are pointing the same direction when the collar is completely tight. The overall length of the column (collar end to collar end) should be 6 ¾".

Figure 4: Tightening the top collar using a pipe-wrench and vice. Make sure inlet and outlet point the same direction.

Figure 5: Overall length of the column is 6 ¾" once tightened.

8) Prime the FMI (or other equivalent) pump and tubing with water to ensure the column is not dried out at any point during leak testing.
9) Run a line between the pump and column, column and a clean beaker with water, and the beaker and the pump. Dry the outside of the column after assembly.

Figure 6: Column/pump/beaker setup and connections

10) Begin circulating water and address any leaks. Small leaks at the swage fittings can be addressed by tightening the fitting.
11) Circulate the water for 1.5-2 hours in each direction, checking for leaks periodically. When finished, also check the beaker to see if any "fines" from the resin have made it through the frit.
    a. If fines are found, empty and rinse the beaker and then replace the water and continue circulating until no fines are present. If fines persist, the column will need to be disassembled and reassembled with new frits.
12) Set the pump to circulate water from the top of the column to the bottom and replace the bottom tube line with a pressure gauge.

Figure 7: Column with pressure gauge attached

13) Uncap the pressure gauge and fill with water by pumping it through the column.
14) Cap the pressure gauge and increase pressure to approx. 35 PSI, then shut off the pump and check for leaks. A slow pressure decrease is acceptable (0.2 PSI/min), as long as liquid leaks are not found on the column. This is sometimes caused by pressure leaking back through the pump head.
15) Allow the pressurized column to sit for 1-2 hours, checking for leaks periodically.
16) Disconnect the pump from the column to depressurize it and then cap the top column inlet.
17) Disconnect the pressure gauge from the bottom of the column and cap the bottom column outlet.
18) Store the column until the appropriate pipe can be bent and attached to assemble the complete column.
19) Once the pipe is bent and fittings/valves are attached, prime the lines with water and attach to the column.
20) Attach two thermocouples to the assembly. One to either side of the column. The overall height of the column apparatus should be 22 ½". It is better to be slightly over than slightly under.

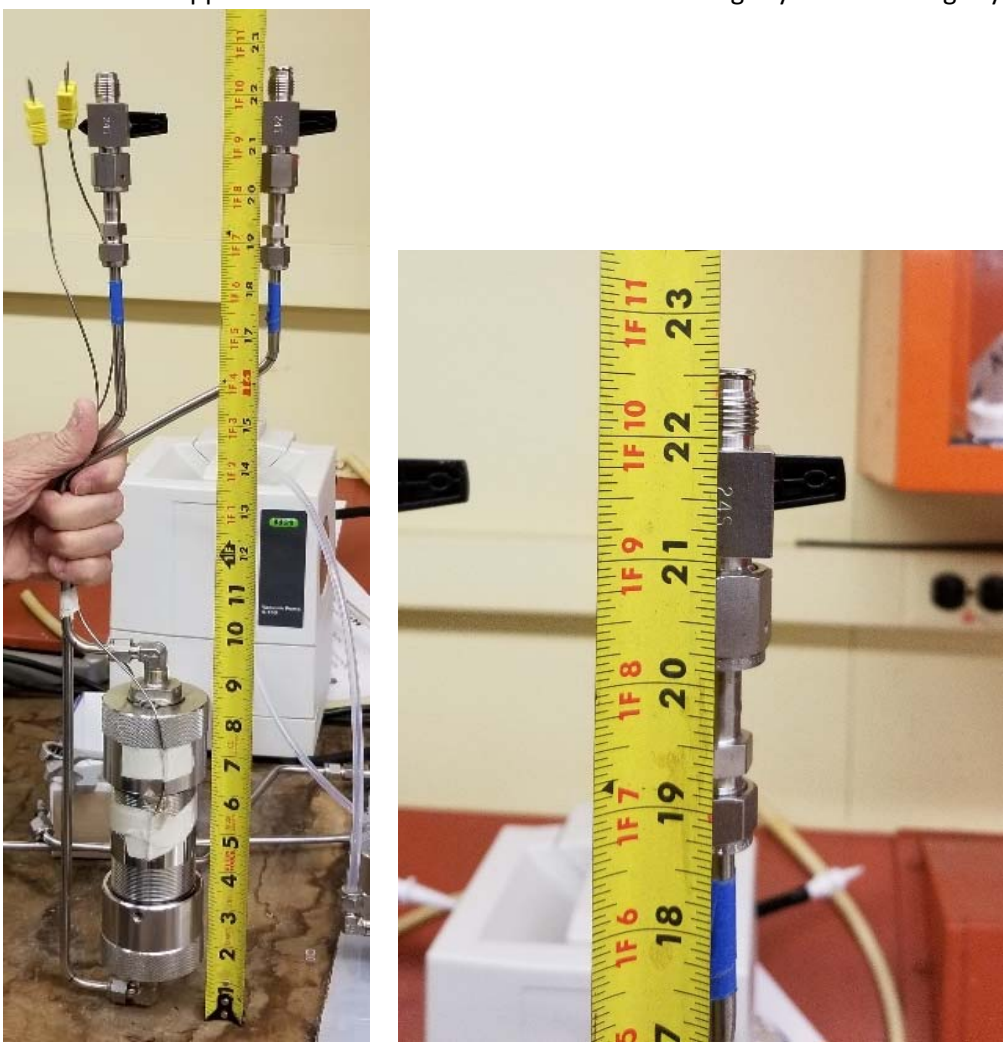


Figure 8: Overall assembly of column, pipes, fittings, and thermocouples with zoom in on overall height of 22 ½".

APPENDIX 19

LEAF-PROC-001, Rev. 1: 20L Tank Cooling System: Initial Startup, Ambient

20L Tank Cooling System: Initial Startup, Ambient

Low Energy Accelerator Facility, LEAF-PROC-001, Rev. 1

Approved:  Date: 09.12.2019

Sergey Chemerisov, Manager, IVEM/LEAF

Effective Date: 09.16.2019

1 Purpose

Establish the process for initial startup for the 20-liter (20L) tank cooling system at ambient temperature at the LEAF facility.

2 Scope

This procedure applies to the following Argonne activities and entities.

LMS core processes:	Asset Management
Organizations:	Experimental Operations and Facilities (EOF) Division
Buildings:	211
Specific locations:	LINAC
Other applicability factors:	None
Exclusions:	None
USI applicability:	Yes

3 Work Process

3.1 Introduction

During AMORE irradiation significant energy (up to 5 kW) will be deposited in the uranyl sulfate solution. To maintain desired solution temperature in 20C- 90C range solution has to be cooled. This is achieved by flowing cooling water through the reflector surrounding the solution volume and through cooling coil placed on top of the solution. Before commencing AMORE irradiation cooling system has to be turned on and operations of the system has to be verified. Procedure outlining steps necessary to perform initial startup is listed below.

3.2 Step-by-Step Procedure

The steps below are mandatory unless noted otherwise. All steps must be written into start-up check-list with logging all measurements of temperature, pressure, and flow rate. If at any step the measured value is out of compliance, or the system does not response in proper way (motor won't start, control light not switched on, etc.), immediately stop the process, inform the person in experimental duty, and initiate the troubleshooting process in accordance with proper WCD. This procedure is to be performed by Qualified Operator.

3.2.1 Actions

Step	Action
1	System in ready condition (full flow through heat exchanger and bypass valve)
2	Expansion Tank Float Switches 2.1 Fill system until both lights go on. 2.2 Start pump. 2.3 With pump running, the rest of the system will start to fill, lowering the level in the

Step	Action
	expansion tank; in turn, the bottom light will go off and pump will stop. 2.4 Refill the tank and repeat steps 1–3 until the system is full and the bottom light stays on. 2.5 Start pump and start drain water until bottom light goes off and pump stops. 2.6 With pump stopped, add 1 gallon of water to the expansion tank. 2.7 Start pump. 2.8 Drain system until top light just goes off. 2.9 Verify that approximately 1 gallon of water has been drained from the system. 2.10 Refill the tank and repeat steps 1–3 until the system is full and the bottom light stays on. 2.11 Expansion tank level is now set. 2.12 Step 2 is completed _____(Initial)
3	Expansion Tank Purge System 3.1 Purge flow rate set at 1.5±0.2 scfm. 3.2 Flow Switch interlock for purge • Reduce purge flow to 1.0±0.2 scfm. • Beam power relay should be deactivated (light on control panel is off). • Increase purge flow to 1.5±0.2 scfm. • Beam power relay is/should be activated (light on control panel is on). 3.3 Step 3is completed _____(Initial)
4	Measurements with Pump Off 4.1 Turn pump off 4.2 Pressure sensor reading should be 3.0±1.0 psi (4.5 mA) 4.3 Differential pressure sensor reading should be 0.0±0.3 psi (4.0 mA) 4.4 Step 4 is completed _____(Initial)

Step	Action
5	Measurements with pump on 5.1 Start pump 5.2 Set flow rate to 2.3±0.5 gpm using the throttle valve. 5.3 Adjust flow through DI to 0.25±0.1 gpm using upstream ball valve. 5.4 Pressure sensor reading should be 23.0±1 psi. 5.5 Differential pressure sensor reading should be 0.4±0.3 psi (4.3 mA). 5.6 Step 5 is completed. _____(Initial)
6	Flow Switch Interlock check 6.1 Reduce the flow rate from 2.3±0.3 gpm to 2.0±0.2 gpm using the throttle. 6.2 Set the flow switch to open at 2.0±0.2 gpm. 6.3 Beam power relay should be deactivated (light on control panel is off). 6.4 Increase the flow back to 2.3±0.3 gpm. 6.5 Beam power relay should be activated (light on control panel is on). 6.6 Reduce the flow to 2.0±0.2 gpm to check flow switch setting. 6.7 Increase flow to the design flow of 2.3±0.23 gpm. 6.8 Beam power relay should be activated (light on control panel is on). 6.9 Step 6 is completed. _____(Initial)
7	Chiller 7.1 Perform chiller startup steps in accordance with “Chiller Cooling System: Initial and Routine Startup”, LEAF-PROC-004. 7.2 Start and record temperatures and flow (at chiller). 7.3 Adjust flow to 2.5±0.2 gpm; outlet temperature should be 55±5°F 7.4 Step 7 is completed. _____(Initial)
8	Check out is complete
9	Date and sig _____

4 Records Created by Work Process

The records listed below must be retained as indicated.

Description of Record (include form number if applicable)	Active Records Custodian	Active Records Retention	Indexing Method, Storage Medium	Federal Retention Requirements*
Completed LEAF-PROC-001	Facility Manager	3 years	Index by job date and name, store on paper or electronically	Destroy 75 years after the date of the permit (DOE ADM 18.37)

*If records are maintained in a business information system that is not currently programmed to purge digital records based on age, the records may be retained in that system past the indicated destruction date.

5 Related Documents

This procedure implements requirements established by the following safety basis documents:

- LEAF-SAD-100, Linac Safety Assessment Document
- LEAF-ASE-100, Linac Accelerator Safety Envelope

This procedure implements requirements established by the following Argonne policies and procedures:

- LMS-PROC-188, *Accelerator Safety*
- EGS-PP-100, *Configuration Management Program Plan for Accelerators*

6 Definitions

None

7 About this Procedure

Issuing organization:	Low Energy Accelerator Facility
Procedure owner:	S. Chemerisov
Point of contact:	S. Chemerisov
Review cycle (months):	36
Date last revised:	09.05.2019
Date last reviewed:	09.12.2019

8 Summary of Changes in This Version

Rev 0: Initial release

Rev 1:

In step 3.2 changed the purge flow from 2.0 scfm to 1.5 scfm to be consistent with required purge flow.

Added additional step prior to step 4.1 to turn pump off before performing the measurements.

Added additional step prior to step 5.1 to turn pump on before performing steps in section 5.

Changed flow through DI cartridge from 0.35 to 0.25 to reflect real flow rate.

Changed expected pressure sensor reading from 24 to 23 psi to match actual pressure in the system.

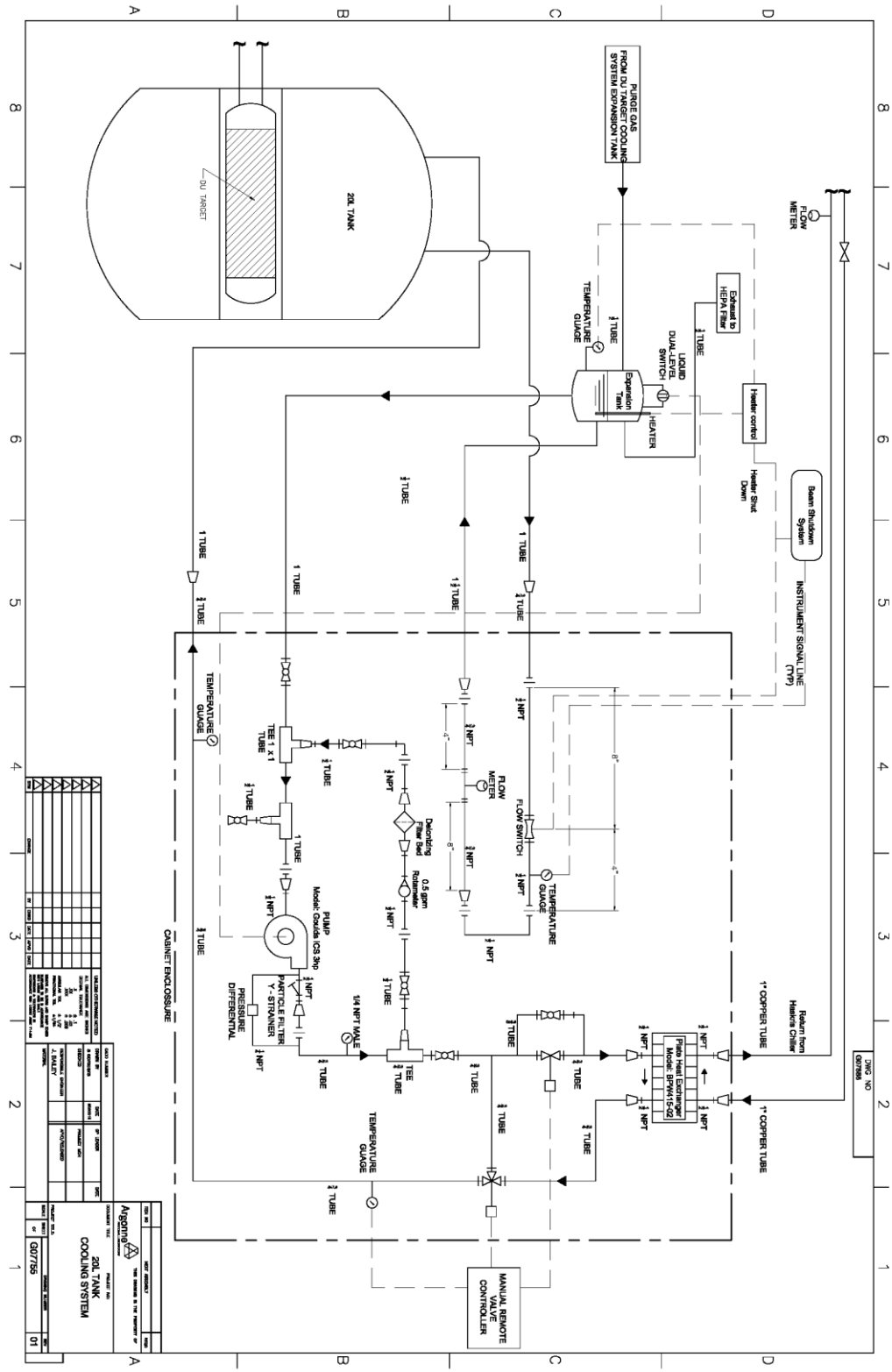
In step 6.1 changed the flow rate from 5 gpm to 2.3 gpm and from 4.5 to 2.0 gpm to reflect actual flow through the system. This flow rate is sufficient for solution cooling.

In step 6.4 decreased the flow rate from 5 gpm to 2.3 gpm.

In step 6.6 decreased the flow rate from 4.5 gpm to 2.0 gpm.

In step 6.7 decrease the flow rate from 5 gpm to 2.3 gpm.

Exhibit A: 20L Cooling System Schematic



APPENDIX 20

LEAF-PROC-002, Rev. 1: 20L Tank Cooling System: Initial Startup, Elevated Temperature

20L Tank Cooling System: Initial Startup, Elevated Temperature

Low Energy Accelerator Facility, LEAF-PROC-002, Rev. 1

Approved:  Date: 09.05.2019

Sergey Chemerisov, Manager, IVEM/LEAF

Effective Date: 09.12.2019

1 Purpose

Establish the process for initial startup of the 20L Tank Cooling System at the LEAF/Linac facility under elevated temperature conditions.

2 Scope

This procedure applies to the following Argonne activities and entities.

LMS core processes:	Asset Management
Organizations:	Experimental Operations and Facilities (EOF) Division
Buildings:	211
Specific locations:	LINAC
Other applicability factors:	None
Exclusions:	None
USI applicability:	Yes

3 Work Process

3.1 Introduction

During AMORE irradiation significant energy (up to 5 kW) will be deposited in the uranyl sulfate solution. To maintain desired solution temperature in 20C- 90C range solution has to be cooled. This is achieved by flowing cooling water through the reflector surrounding the solution volume and through cooling coil placed on top of the solution. Before commencing AMORE irradiation cooling system has to be turned on and operations of the system has to be verified. Procedure outlining steps necessary to perform initial startup at elevated temperatures is listed below.

3.2 Step-by-Step Procedure

The steps below are mandatory unless noted otherwise. All steps must be written into start-up check-list with logging all measurements of temperature, pressure, and flow rate. If at any step the measured value is out of compliance, or the system does not response in proper way (motor won't start, control light not switched on, etc.), immediately stop the process, inform the person in experimental duty, and initiate the troubleshooting process in accordance with proper WCD. This procedure is to be performed by Qualified Operator.

3.2.1 Actions

Step	Action
1	System in ready condition (perform LEAF-PROC-001, Initial Startup Steps, Ambient)
2	Provide: • 0-20psig air supply with regulator and pressure gauge
3	Calibrate 3-Way Valve: 3.1 Pump off. 3.2 Setup the camera to observe valve movement in D-035.

Step	Action
	3.3 Put control panel in D-076 in operating mode (i.e., air supply and electric on). 3.4 Set the manual setter controller (TC108) to 0. 3.5 Increase set point until movement of valve stem actuator occurs. 3.6 Record this value (call it low). 3.7 Increase setter adjustment until movement of valve actuator stops 3.8 Record the setter setting (call it high). 3.9 The "low" to "high" setting is the range that will be used to determine the percent settings for the 3-way valve that are indicated in the table for the elevated temperature operation (Note that percent setting as determined here may be reversed from the table). 3.10 Recheck setter settings against actual valve stem positions. 3.11 Step3 is completed. _____(Initial)
4	Operation: Check 2-Way Valve 4.1 Set 2-position selector switch (SS104) to off position. 4.2 Determine and record position of actual valve stem actuator (up or down). 4.3 Set 2-position selector switch to on position. 4.4 Determine and record position of actual valve stem actuator (up or down). 4.5 Remove control air tube from the valve operator. 4.6 Determine and record position of actual valve stem actuator (up or down). This check determines the normally open or closed position of the 2-Way valve. 4.7 Step 4 is completed. _____(Initial)
5	Operation Ambient Temperature Test 5.1 Set the 2-Way valve and by pass to the full open position. 5.2 Set the 3-Way valve to full flow through the exchanger. 5.3 Pump on. 5.4 Record flow rate and pressure (should be about the same as that recorded for the LEAF-PROC-001. 5.5 Step 5 is completed. _____(Initial)

Step	Action
6	Operation Elevated Temperature Test (beam off, pump on) 6.1 Set 3-Way valve to 50% 6.2 2-Way valve and by pass valve open 6.3 Set temperature to 80°F 6.4 Reference table 6.5 Record temperature increase of coolant vs. time until steady state of 80°F is reached 6.6 Record flow rate through 20L Tank 6.7 Record chilled water temperature in and out of chiller and flow rate to 20L Tank cooling system 6.8 Repeat steps 1 through 7 for set temperatures of 110°F and 1130°F, referencing table for valve settings 6.9 Step 6 is completed. _____(Initial)
7	Over Temperature Interlock 7.1 Set over temperature beam shutdown to 130°F 7.2 Increase the water operating temperature to 135°F 7.3 Beam power relay should be deactivated (light on control panel is off) 7.4 Decrease the water operating temperature to 125°F 7.5 Beam power relay should be activated (light on control panel is on) 7.6 Reset over temperature beam shut down to 180°F 7.7 Step 7 is completed. _____(Initial)
8	Shut systems down
9	Check out is complete
10	Date and sign

4 Records Created by Work Process

The records listed below must be retained as indicated.

Description of Record (include form number if applicable)	Active Records Custodian	Active Records Retention	Indexing Method, Storage Medium	Federal Retention Requirements*
Completed LEAF-PROC-002	Facility Manager	3 years	Index by job date and name, store on paper or electronically	Destroy 75 years after the date of the permit (DOE ADM 18.37)

*If records are maintained in a business information system that is not currently programmed to purge digital records based on age, the records may be retained in that system past the indicated destruction date.

5 Related Documents

This procedure implements requirements established by the following safety basis documents:

- LEAF-SAD-100, Linac Safety Assessment Document
- LEAF-ASE-100, Linac Accelerator Safety Envelope

This procedure implements requirements established by the following Argonne policies and procedures:

- LMS-PROC-188, *Accelerator Safety*
- EGS-PP-100, *Configuration Management Program Plan for Accelerators*

6 Definitions

None

7 About this Procedure

Issuing organization:	Low Energy Accelerator Facility
Procedure owner:	S. Chemerisov
Point of contact:	S. Chemerisov
Review cycle (months):	36
Date last revised:	09.05.2019
Date last reviewed:	09.12.2019

8 Summary of Changes in This Version

Initial release

Revision 1:

Section 3 was modified to reflect use of the camera and monitor to calibrate 3-Way valve.

Temperatures settings for calibration points in section 7 reduced to 80, 110, and 130 F to reduce time necessary to verify system performance.

Reduce interlock check temperature to 130 F to coincide with highest temperature point in previous step.

Reduce beam shutdown temperature for over temperature protection to 180 F.

APPENDIX 21

LEAF-PROC-003, Rev. 0: 20L Tank Cooling System: Routine Startup, Ambient

20L Tank Cooling System: Routine Startup, Ambient

Low Energy Accelerator Facility, LEAF-PROC-003, Rev. 0

Approved:  Date: 09.05.2019

Sergey Chemerisov, Manager, IVEM/LEAF

Effective Date: 09.12.2019

1 Purpose

Establish the process for starting up the 20L tank cooling system located at the LEAF facility under routine, ambient conditions.

2 Scope

This procedure applies to the following Argonne activities and entities.

LMS core processes:	Asset Management
Organizations:	Experimental Operations and Facilities (EOF) Division
Buildings:	211
Specific locations:	LINAC
Other applicability factors:	None
Exclusions:	None
USI applicability:	Yes

3 Work Process

3.1 Introduction

During AMORE irradiation significant energy (up to 5 kW) will be deposited in the uranyl sulfate solution. To maintain desired solution temperature in 20C- 90C range solution has to be cooled. This is achieved by flowing cooling water through the reflector surrounding the solution volume and through cooling coil placed on top of the solution. Before commencing AMORE irradiation cooling system has to be turned on and operations of the system has to be verified. Procedure outlining steps necessary to perform those operations is listed below.

3.2 Step-by-Step Procedure

The steps below are mandatory unless noted otherwise. All steps must be written into start-up check-list with logging all measurements of temperature, pressure, and flow rate. If at any step the measured value is out of compliance, or the system does not response in proper way (motor won't start, control light not switched on, etc.), immediately stop the process, inform the person in experimental duty, and initiate the troubleshooting process in accordance with proper WCD. This procedure is to be performed by Qualified Operator.

3.2.1 Actions

Step	Action
1	System in ready condition (full flow through heat exchanger and by pass valve)
2	Expansion Tank Float Switches 2.1 Fill system until both lights turn on. 2.2 Start pump. 2.3 With pump running, the rest of the system will start to fill, lowering the level in the expansion tank, and in turn, the bottom light will go off and pump will stop. 2.4 Refill the tank and repeat steps 1–3 until the system is full and the bottom light stays

Step	Action
	on.
	2.5 Start pump and drain water until bottom light just goes off and pump stops.
	2.6 With pump stopped, add 1 gallon of water to the expansion tank.
	2.7 Start pump.
	2.8 Drain system until top light just goes off.
	2.9 Verify that approximately 1 gallon of water has been drained from the system.
	2.10 Refill the tank and repeat steps 1–3 until the system is full and the bottom light stays on.
	2.11 Expansion tank level is now set.
	2.12 Step 2 is completed _____(Initial)
3	Expansion Tank Purge System
	3.1 Purge flow rate set at 1.5±0.2 scfm.
	3.2 Flow Switch interlock for purge • Reduce purge flow to 1.0±0.2 scfm. • Beam power relay should be deactivated (light on control panel is off). • Increase purge flow to 1.5±0.2 scfm.
	3.3 Step 3 is completed _____(Initial) **Note:** At initial startup, set flow switch trip at 1.5 scfm.
4	Pump off measurements
	4.1 Turn pump off
	4.2 Pressure sensor reading should be 3.0±1.0 psi (4.5 mA).
	4.3 Differential pressure sensor reading should be 0.0±0.3 psi (4.0 mA).
	4.4 Step 4 is completed _____(Initial)
5	Pump on measurements
	5.1 Start pump
	5.2 Pressure sensor reading should be 23.0±1 psi.
	5.3 Differential pressure sensor reading should be 0.4±0.3 psi (4.3 mA).
	5.4 Flow through DI unit should be 0.25±0.1 gpm
	5.5 Step 5 is completed _____(Initial)

Step	Action
6	Flow switch interlock 6.1 Pump off; the beam power relay should be deactivated (light on control panel is off). 6.2 Pump on; the beam power relay should be activated (light on control panel is on). 6.3 Step 6 is completed _____(Initial)
7	Chiller 7.1 Perform chiller startup steps in accordance with "Chiller Cooling System: Initial and Routine Startup", LEAF-PROC-004. 7.2 Start and record temperatures and flow (at chiller). 7.3 Step 7 is completed _____(Initial)
8	Check out is complete _____(Initial)
9	Date and signature _____

4 Records Created by Work Process

The records listed below must be retained as indicated.

Description of Record (include form number if applicable)	Active Records Custodian	Active Records Retention	Indexing Method, Storage Medium	Federal Retention Requirements*
Completed LEAF-PROC-003	Facility Manager	3 years	Index by job date and name, store on paper or electronically	Destroy 75 years after the date of the permit (DOE ADM 18.37)

*If records are maintained in a business information system that is not currently programmed to purge digital records based on age, the records may be retained in that system past the indicated destruction date.

5 Related Documents

None

6 Definitions

None

7 About this Procedure

Issuing organization:	Low Energy Accelerator Facility
Procedure owner:	S. Chemerisov
Point of contact:	S. Chemerisov
Review cycle (months):	36
Date last revised:	09.05.2019
Date last reviewed:	09.12.2019

8 Summary of Changes in This Version

Initial release

Rev1.

In steps 3.1- 3.2 changed the purge flow from 2.0 scfm to 1.5 scfm to be consistent with required purge flow.

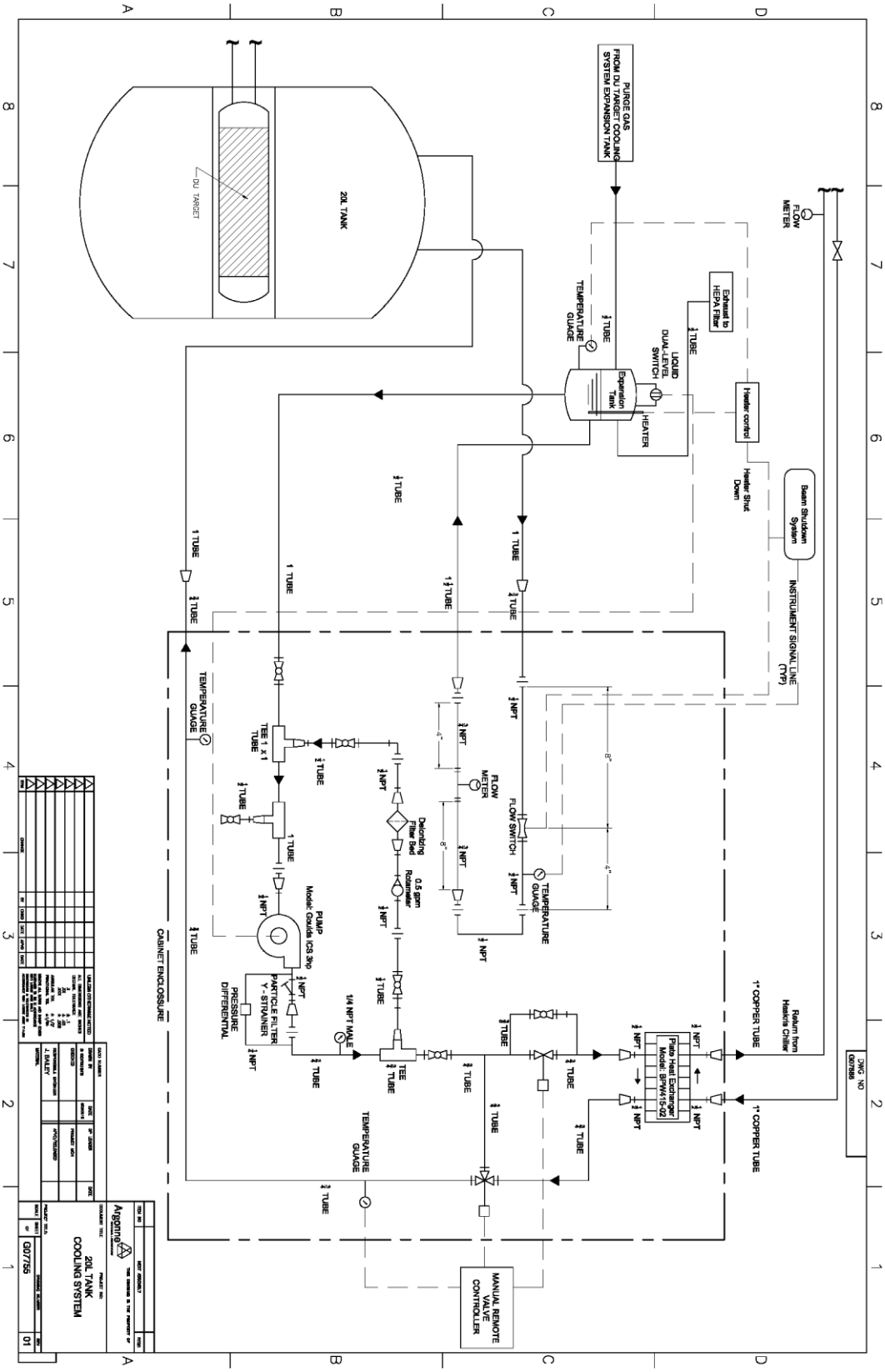
Added additional step prior to step 4.1 to turn pump off before performing the measurements.

Added additional step prior to step 5.1 to turn pump on before performing steps in section 5.

Changed flow through DI cartridge from 0.35 to 0.25 to reflect real flow rate.

Changed expected pressure sensor reading from 24 to 23 psi to match actual pressure in the system.

Exhibit A: Cooling System P&ID



APPENDIX 22

LEAF-PROC-004, Rev. 0: Chiller Cooling System: Initial and Routine Startup

Chiller Cooling System: Initial and Routine Startup

Low Energy Accelerator Facility, LEAF-PROC-004, Rev. 0

Approved:  Date: 03.25.2019

Sergey Chemerisov, Manager, IVEM/LEAF

Effective Date: 04.01.2019

1 Purpose

Establish the process for initial and routine startup of the chiller cooling system located at the LEAF facility.

2 Scope

This procedure applies to the following Argonne activities and entities.

LMS core processes:	Asset Management
Organizations:	Experimental Operations and Facilities (EOF) Division
Buildings:	211
Specific locations:	LINAC
Other applicability factors:	None
Exclusions:	None
USI applicability:	Yes

3 Work Process

3.1 Introduction

Cooling to the target cooling system and 20L solution cooling system is provided by the chiller unit located in room D-076 in building 211. Operations of the chiller and chiller water level interlock has to be verified prior to the start of AMORE irradiation. These verifications have to be completed at the day of the irradiation.

3.2 Step-by-Step Procedure

The steps below are mandatory unless noted otherwise. This procedure is to be performed by Qualified Operator.

3.2.1 Actions

Step	Action
1	System in ready condition
2	Reservoir Float Switch 2.1 Fill reservoir (to level using float level) using deionized water. 2.2 Lower level sensor to avoid tripping system 2.3 Start chiller: • Reservoir level will decrease due to filling of chilled water system • With pump running, the system will fill, lowering the level in the chiller reservoir 2.4 With the pump running, and after the reservoir has reached a steady level refill the reservoir to the full level using the float level with deionized water. 2.5 Move the sensor level up and down to assure that it is working smoothly. The water is below the trip level if the green light on the control box is off; if the water is above the

Step	Action
	trip level, the green light will be on. 2.6 Adjust the height of the level switch so that it is just in the deactivated mode (green light just goes off – starting with the green light on) 2.7 From this deactivated mode height, lower the float adjustment height down 0.6” • The switch should now be activated – green light on. 2.8 For the initial use of the system, (does not need to be done every time -- this is to confirm the limit) do the following -- With the pump running, drain system and capture the water until level switch is deactivated. Note that the chiller pump will go off when the level is tripped • The amount of water drained should be less than 1.5 gallon. If it is more than 1.5 gallon, go to step 2.9 and then repeat at step 2.5. Log the change on a tag so that the next time the level will be set at 0.3 inches. 2.9 Refill the reservoir with the water drained out.
3	Check out is complete
4	Date and Sign _____

4 Records Created by Work Process

The records listed below must be retained as indicated.

Description of Record (include form number if applicable)	Active Records Custodian	Active Records Retention	Indexing Method, Storage Medium	Federal Retention Requirements*
Completed LEAF-PROC-004	Facility Manager	3 years	Index by job date and name, store on paper or electronically	Destroy 75 years after the date of the permit (DOE ADM 18.37)

*If records are maintained in a business information system that is not currently programmed to purge digital records based on age, the records may be retained in that system past the indicated destruction date.

5 Related Documents

This procedure implements requirements established by the following safety basis documents:

- LEAF-SAD-100, Linac Safety Assessment Document
- LEAF-ASE-100, Linac Accelerator Safety Envelope

This procedure implements requirements established by the following Argonne policies and procedures:

- LMS-PROC-188, *Accelerator Safety*
- EGS-PP-100, *Configuration Management Program Plan for Accelerators*

6 Definitions

AMORE- Argonne Molybdenum Research Experiment

7 About this Procedure

Issuing organization:	Low Energy Accelerator Facility
Procedure owner:	S. Chemerisov
Point of contact:	S. Chemerisov
Review cycle (months):	36
Date last revised:	03.25.2019
Date last reviewed:	03.25.2019

8 Summary of Changes in This Version

Initial release

APPENDIX 23

LEAF-PROC-006, Rev. 2: DU Target Cooling System: Initial Startup

DU Target Cooling System: Initial Startup

Low Energy Accelerator Facility, LEAF-PROC-006, Rev. 2

Approved:  Date: 09.12.2019

Sergey Chemerisov, Manager, IVEM/LEAF

Effective Date: 09.16.2019

1 Purpose

Establish the process for initial startup of the depleted uranium (DU) target cooling system installed at the LEAF facility.

2 Scope

This procedure applies to the following Argonne activities and entities.

LMS core processes:	Asset Management
Organizations:	Experimental Operations and Facilities (EOF) Division
Buildings:	211
Specific locations:	LINAC
Other applicability factors:	None
Exclusions:	None
USI applicability:	Yes

3 Work Process

3.1 Introduction

During AMORE irradiation 20kW of electron beam from accelerator will be placed on the target. Cooling of the target is achieved by flowing cooling water through the target housing and through the spacers between target disks. **Before commencing AMORE irradiation cooling system has to be turned on, operations of the system has to be verified and interlocks preventing beam operations has to be checked [ASE2.4.1.1, 2.6.1.1]**. Steps necessary to perform those operations are listed below.

3.2 Step-by-Step Procedure

The steps below are mandatory unless noted otherwise. All steps must be written into start-up check-list with logging all measurements of temperature, pressure, and flow rate. If at any step the measured value is out of compliance, or the system does not response in proper way (motor won't start, control light not switched on, etc.), immediately stop the process, inform Facility Manager, and initiate the troubleshooting process in accordance with proper WCD. This procedure is to be performed by Qualified Linac Operator.

3.2.1 Actions

Step	Action
1	System in ready condition

Step	Action
2	Expansion Tank Float Switches 2.1 Fill expansion tank until both lights go on. 2.2 Start pump. 2.3 With pump running, the rest of the system will start to fill, lowering the level in the expansion, and in turn the bottom light will go off and pump will stop. 2.4 Refill the tank and repeat steps 1–3 until the system is full and the bottom light stays on. 2.5 Start pump and drain water until bottom light goes off and pump stops. 2.6 Stop pump and refill system to ready condition (i.e., repeat steps 1-3). 2.7 Step 2 is completed _____(Initial)
3	Expansion Tank Purge System 3.1 Set Purge flow rate to 1.5±0.2 scfm. 3.2 Flow Switch interlock for purge: • Reduce purge flow to 1.0±0.2 scfm. • Adjust flow switch to open at the 1.0±0.2 scfm. • Beam power relay should be deactivated (light on control panel is off). • Increase purge flow to 1.5±0.2 scfm. • Beam power relay is/should be activated (light on control panel is on). 3.3 Step 3 is completed _____(Initial)
4	Pump off 4.1 Pressure sensor reading should be 3.0±3.0 psi. 4.2 Differential pressure sensor reading should be 0.0±0.5 psi. 4.3 Step 4 is completed _____(Initial)
5	Start pump **5.1 Set flow rate to 42.0±1.0 gpm using the throttle valve [ASE 2.6.1.1].** 5.2 Adjust flow through DI to 0.3±0.1 gpm using upstream ball valve. 5.3 Pressure sensor reading should be 50.0±3.0 psi. 5.4 Differential pressure sensor reading across strainer should be 2.0±0.5psi. 5.5 Step 5 is completed _____(Initial)

Step	Action
6	Flow switch interlock and thermocouple interlock
	6.1 Reduce the flow rate from 42.0 gpm to 40.0±1.0 gpm using the throttle valve.
	6.2 **Set the flow switch to open at 40.0±1.0 gpm [ASE 2.6.1.1].**
	6.3 Beam power relay should be deactivated (light on control panel is off).
	6.4 **Increase the flow back to 42.0±1.0 gpm [ASE 2.6.1.1].**
	6.5 Beam power relay should be activated (light on control panel is on).
	6.6 **Reduce the flow to 40.0±1.0 gpm to check flow switch setting [ASE 2.6.1.1].**
	6.7 **Increase flow to the design flow of >42±1.0 gpm [ASE 2.6.1.1].**
	6.8 Beam power relay should be activated (light on control panel is on).
	6.9 Remove T/C at return line from DU target and insert the sensor in a 100±5°F water bath.
	6.10 **Beam power relay should be deactivated (light on control panel is off) [ASE 2.6.1.2].**
	6.11 Insert the sensor in an 80±5°F water bath.
	6.12 **Beam power relay should be activated (light on control panel is on) [ASE 2.6.1.2].**
	6.13 Reinsert T/C in return line.
	6.14 **Beam power relay should be activated (light on control panel is on) [ASE 2.6.1.2].**
	6.15 Step 6 is completed _____(Initial)
7	Chiller
	7.1 Perform chiller startup steps in accordance with "Chiller Cooling System: Initial and Routine Startup", LEAF-PROC-004.
	7.2 Start and record temperatures and flow (at chiller). _____ _____
	7.3 Adjust flow to DU target heat exchanger to 9.0±0.5 gpm, outlet temperature should be 55±5°F.
	7.4 Step 7 is completed _____(Initial)
8	Check out is complete
9	Date and sign _____

4 Records Created by Work Process

The records listed below must be retained as indicated.

Description of Record (include form number if applicable)	Active Records Custodian	Active Records Retention	Indexing Method, Storage Medium	Federal Retention Requirements*
Completed LEAF-PROC-006	Facility Manager	3 years	Index by job date and name, store on paper or electronically	Destroy 75 years after the date of the permit (DOE ADM 18.37)

*If records are maintained in a business information system that is not currently programmed to purge digital records based on age, the records may be retained in that system past the indicated destruction date.

5 Related Documents

This procedure implements requirements established by the following safety basis documents:

- LEAF-SAD-100, Linac Safety Assessment Document
- LEAF-ASE-100, Linac Accelerator Safety Envelope

This procedure implements requirements established by the following Argonne policies and procedures:

- LMS-PROC-188, *Accelerator Safety*
- EGS-PP-100, *Configuration Management Program Plan for Accelerators*

6 Definitions

None

7 About this Procedure

Issuing organization:	Low Energy Accelerator Facility
Procedure owner:	S. Chemerisov
Point of contact:	S. Chemerisov
Review cycle (months):	36
Date last revised:	09.06.2019
Date last reviewed:	09.12.2019

8 Summary of Changes in This Version

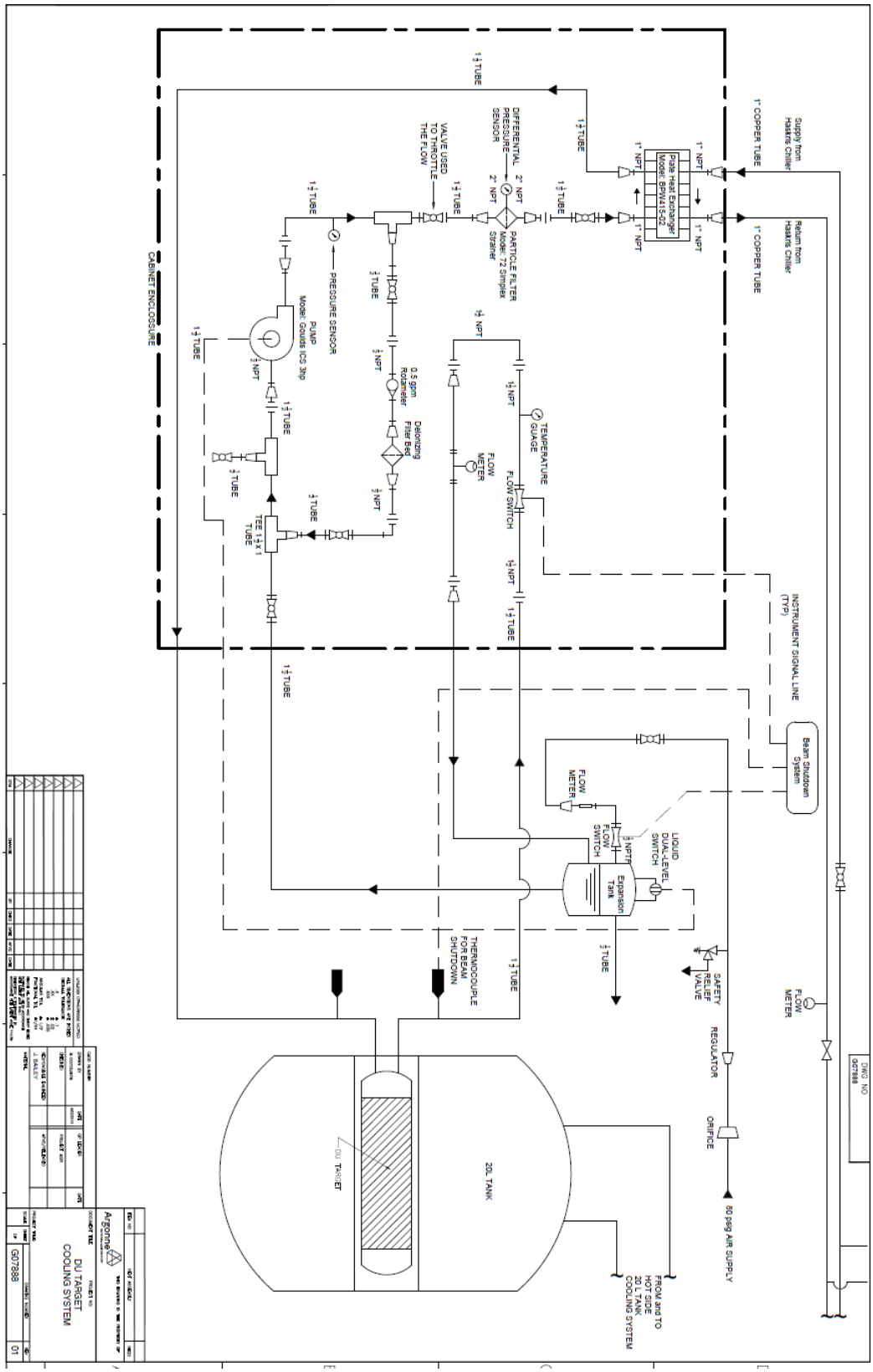
Initial release

Rev. 1. Addition of the references to the ASE controlled parameters.

Rev. 2. Change flow through the DI unit in step 5.2 from 1 gpm to 0.3 gpm.

Change the pressure readout for the pressure transducer to 50 psi to reflect real measurements in the system

Exhibit A: P&ID of the Target Cooling System



APPENDIX 24

LEAF-PROC-007, Rev. 2: DU Target Cooling System: Routine Startup Procedure

DU Target Cooling System: Routine Startup Procedure

Low Energy Accelerator Facility, LEAF-PROC-007, Rev. 2

Approved: _____ Date: 09.18.2019

Sergey Chemerisov, Manager, IVEM/LEAF

Effective Date: 09.19.2019

1 Purpose

Establish the process for starting up the depleted uranium (DU) target cooling system at the LEAF facility under routine conditions.

2 Scope

This procedure applies to the following Argonne activities and entities.

LMS core processes:	Asset Management
Organizations:	Experimental Operations and Facilities (EOF) Division
Buildings:	211
Specific locations:	LINAC
Other applicability factors:	None
Exclusions:	None
USI applicability:	Yes

3 Work Process

3.1 Introduction

During AMORE irradiation 20kW of electron beam from accelerator will be placed on the target. Cooling of the target is achieved by flowing cooling water through the target housing and through the spacers between target disks. Before commencing AMORE irradiation cooling system has to be turned on, operations of the system has to be verified and interlocks preventing beam operations has to be checked. Steps necessary to perform those operations are listed below. This procedure has to be performed on the day of irradiation.

3.2 Step-by-Step Procedure

The steps below are mandatory unless noted otherwise. This procedure is to be performed by Qualified Operator (QLO). QLO should indicate initial each step in this procedure indicating that all required conditions are satisfied.

3.2.1 Actions

Step	Action
1	System in ready condition

Step	Action
2	Expansion Tank Purge System 2.1 Purge flow rate set at 1.5±0.2 scfh _____ 2.2 Flow Switch interlock for purge • Reduce purge flow to 1.0±0.2 scfm _____ • Beam power relay should be deactivated (light on control panel is off) _____ • Increase purge flow to 1.5±0.2 scfm_____ • Beam power relay is should be activated (light on control panel is on) _____ **Note:** At initial startup, set flow switch trip at 1.5±0.2 scfm
3	Pump off 3.1 Pressure sensor reading should be 4.5 ma (3±3psi) _____ 3.2 Differential pressure sensor reading should be 4.0 ma (0±0.5psi) _____
4	Start pump 4.1 Pressure sensor reading should be 12.4 ma (50.0±3.0 psi) _____ 4.2 Differential pressure sensor reading should be 6.4 ma (2.0±0.5psi) _____ 4.3 Flow through DI unit should be 0.3±0.1 gpm_____ 4.4 Flow though the target is > 42 gpm _____ **[ASE 2.6.1.1]**
5	Flow switch interlock and thermocouple interlock 5.1 Pump off; the beam power relay should be deactivated (light on control panel is off) _____ **[ASE 2.6.1.2]** 5.2 Pump on; the beam power relay should be activated (light on control panel is on) _____ **[ASE 2.6.1.2]** 5.3 Remove thermocouple (T/C) at return line from DU target and insert the sensor in a 32C water bath _____ **[ASE 2.6.1.2]** 5.4 Beam power relay should be deactivated (light on control panel is off) _____ **[ASE 2.6.1.2]** 5.5 Reinsert T/C in return line _____ 5.6 Beam power relay should be activated (light on control panel is on) _____ **[ASE 2.6.1.2]** 5.7 Verify the temperature of the water at the exit of the target is <20C_____ **[ASE 2.6.1.1]**

Step	Action
6	Chiller 6.1 Perform chiller startup steps _____ 6.2 Start and record temperatures and flow (at chiller) Temperature _____ Flow _____
7	Check out is complete _____
8	Date and Sign off _____

4 Records Created by Work Process

The records listed below must be retained as indicated.

Description of Record (include form number if applicable)	Active Records Custodian	Active Records Retention	Indexing Method, Storage Medium	Federal Retention Requirements*
Completed LEAF-PROC-007	Facility Manager	3 years	Index by job date and name, store on paper or electronically	Destroy 75 years after the date of the permit (DOE ADM 18.37)

*If records are maintained in a business information system that is not currently programmed to purge digital records based on age, the records may be retained in that system past the indicated destruction date.

5 Related Documents

This procedure implements requirements established by the following safety basis documents:

- LEAF-SAD-100, Linac Safety Assessment Document
- LEAF-ASE-100, Linac Accelerator Safety Envelope

This procedure implements requirements established by the following Argonne policies and procedures:

- LMS-PROC-188, Accelerator Safety
- EGS-PP-100, Configuration Management Program Plan for Accelerators

6 Definitions

None

7 About this Procedure

Issuing organization:	Low Energy Accelerator Facility
Procedure owner:	S. Chemerisov
Point of contact:	S. Chemerisov
Review cycle (months):	36
Date last revised:	09.17.2019
Date last reviewed:	09.18.2019

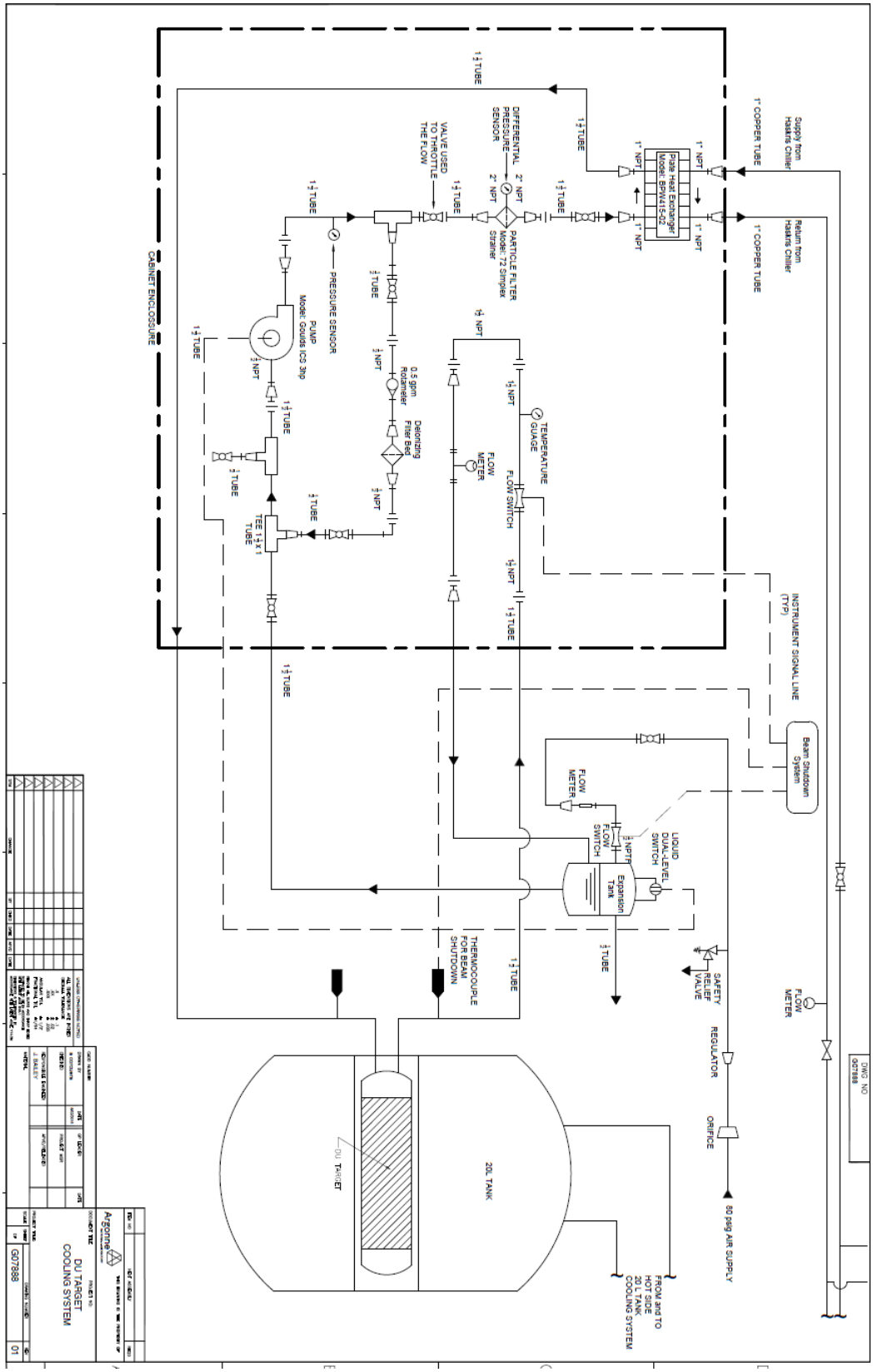
8 Summary of Changes in This Version

Initial release

Rev. 1. Addition of the references to the ASE controlled parameters.

Rev. 2. Changed purge flow rate in step 2.1 and 2.2 from 2.0 schf to 1.5 schf to reflect actual purge flow rate in the system. Changed expected value for pressure transducer from 53.2 psi to 5050±3 psi to reflect actual pressure generated by the pump and to be consistent with LEAF-PROC-006

Exhibit A: P&ID of the Target Cooling System

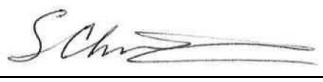


APPENDIX 25

LEAF-PROC-027, Rev. 0: LEAF Linac General Operating Procedure

LEAF Linac General Operating Procedure

Low Energy Accelerator Facility, LEAF-PROC-027, Rev. 0

Approved:  Date: 03.25.2019

Sergey Chemerisov, Manager, IVEM/LEAF

Effective Date: 04.01.2019

1 Purpose

Establish the process for operation of the Linac Facility in building 211.

2 Scope

This procedure applies to the following Argonne activities and entities.

LMS core processes:	Asset Management
Organizations:	Experimental Operations and Facilities (EOF) Division
Buildings:	211
Specific locations:	LINAC
Other applicability factors:	None
Exclusions:	None
USI applicability:	Yes

3 Work Process

3.1 Introduction

This document contains the procedures for general operation of the Linac, including start up, normal operation, stand by and shut down.

If any malfunctions occur, which make it impossible to satisfactorily complete any of the procedures; such malfunctions must be corrected before continuing. Due to the complexity of the equipment and the multitude of malfunctions which may occur, no attempt is made to specify repair procedures. All repairs must be made in compliance with applicable safety standards.

3.2 Step-by-Step Procedure

The steps below are mandatory unless noted otherwise. This procedure is to be performed by Qualified Linac Operator (QLO).

3.2.1 Actions

Step	Action
1	**Set Up Procedure for Linac Operator** Follow access procedures in LEAF-PROC-010, Linac Shielding Procedure, Section .2 when entering potential beam areas. 1.1 Determine which beam line the experimenter will use for his run. 1.2 AMORE experimentation shall be limited to five** full irradiation runs (full run is any run where more than 175 kW*hrs delivered to the target). 1.3 AMORE irradiation run shall be limited to an integrated energy deposition of ≤700kw-hrs** without prior approval from DOE-Argonne Site Office (ASO).

Step	Action
	1.4 Maximum average beam current in the port located in D-017 (Pit) room is 200 µA**. 1.5 Maximum average beam current for beam port located in D-035 (Cell 1) is 200 µA** for the ports equipped with aluminum window and 1.5 mA** for water cooled beryllium window. 1.6 Maximum beam energy for any operations o fthe accelerator is limited to 60MeV.** 1.7 Put quartz over window (if needed) and set up water-cooled beam stopper (if required). 1.8 Hook signal cable to beam stopper and note cable used. 1.9 Set up TV camera with monitor or OTR-camera, if required. 1.10 Position Beam Port switch(es) (B.P.) the appropriate position(s). If a B.P. has to be moved, assure that the Linac exit valve is closed prior to moving B.P.
2	**Start Up Procedure for Linac Operator** 2.1 In Room D-101 (Control Room): 2.1.1 Log vacuum (<1 x 10-7 Torr) (If any vacuum readings are higher than 1 X 10-7 Torr, check the reason.) 2.1.2 Turn on control power using both the switch and the key. 2.1.3 Run up injector filament voltage (filament needs to be higher than what is mentioned below in A and B) A. 65% for nsec or picosec work B. Approximately 55% for 1.5 A gun. (Try to run gun close to emission limit, this will give flattest pulse.) 2.1.4 If running nsec or picosec pulse, run short-pulse pulser amplitude control to zero, so pulser tubes will be in conditioning mode. 2.1.5 Reset trigger generator 2.2 In Room D-117 (Modulator Room): 2.2.1 At sub-station, turn on 480 V disconnect. 2.2.2 Turn on klystron cooling water pump (check water level in make-up tank is approx. one-half full). 2.2.3 Turn on modulator cabinet cooling fan. 2.2.4 Turn on modulator room cooling fans (if weather is warm outside). 2.2.5 Clear core bias interlocks, if not already clear. 2.2.6 Check thyratron control panel (reservoir, filament voltage). If not at marked meter value, adjust to correct setting. 2.2.7 If pico-sec run, turn on sub-harmonic buncher (SHB) power supply control voltage and SHB radio frequency (RF) preamplifier.

Step	Action
	2.2.8 Turn on Modulator high voltage power supply breakers #1 and #2 2.3 In Basement 2.3.1 Turn on one of the two main cooling water pumps 2.3.2 If booster pump is needed turn on booster pump per posted instructions 2.4 In Room D-017 (Pit): 2.4.1 Charge main buncher load with dry nitrogen to 15 pounds per square inch gauge (PSIG). 2.4.2 Turn on auxiliary cooling water pump. 2.4.3 Turn on buncher/injector cooling water pump. (note that the following 3 pumps should be on at least 30 minutes prior to putting RF into the waveguides as the waveguides must come up to temperature) 2.4.4 Turn on accelerating waveguide (W.G.) #1 cooling water pump 2.4.5 Turn on W.G. #2 cooling water pump. 2.4.6 Turn on Circulator (formerly W.G. #3) cooling water pump. 2.4.7 Charge W.G. #1, and W.G. #2 transmission waveguides and loads with SF6 to 12÷15 PSIG. 2.4.8 Check SF6 pressure in injector tank (5 PSIG). Fill if needed. 2.4.9 Valve off SF6 tank and secure. 2.4.10 Check local accelerator interlock panel to see if all water and gas interlock lights are out. 2.4.11 Turn on 270° magnet water pump if magnet is required for target setup. 2.4.13 Check that optical tube shields are properly positioned. A. Both ends of Instrument room tube. B. Both ends of Spectrograph room tube. Linac is now ready to turn on for operation.
3	**General Operation for Linac Operator** 3.1 Secure the Facility Secure all areas of the facility that will be exposed to the beam, following the procedures in LEAF-PROC-10, Linac-Shielding Procedure, Section 3.2. 3.2 Turn on the Linac In Modulator Room: 3.2.1 Check klystron filament current and set to correct value as indicated on front of power supplies.

Step	Action
	3.2.2 Set up quadrupole patch panels for desired quadrupole arrangement using log sheet for correct placement. 3.2.3. Set up steering patch panel for desired steering arrangement using log sheet for correct placement. 3.2.4 Set water and air interlocks on 270° magnet system (if used for this run). 3.2.5 Turn M.G. regulator "ON" (if used for this run). 3.2.6 Turn amplidyne regulator "ON" (if used for this run). 3.2.7 Turn on sub-harmonic buncher amplifier high voltage (HV) power supply and adjust to voltage noted on front of power supply (if used for this run). 3.2.8 Turn on Bi-polar Steering power supplies (if used for this run). Linac is now ON and ready for TUNE UP. 3.3 Linac Tune Up (General) for Linac Operator 3.3.1 Check radiation detectors by pressing the calibrate switches and determining that the meters read properly and that the system alarms properly. 3.3.2 Recheck vacuum pressures, if any pressures are above the listed value, linac cannot be operated until conditions are corrected. A. Injector (<1 X 10^{-7} Torr) B. Accelerator (<1 X 10^{-7} Torr) C. Table #2 (IG^{-7}) (<5 X 10^{-7} Torr) D. Table #3 (IG^{-8}) (<5 X 10^{-7} Torr) 3.3.3 If pressures are within operating range, open all required beam valves. 3.3.4 Turn on modulator high voltage. 3.3.5 Watching klystron #1 and #2 current waveforms on console oscilloscope, slowly raise the "voltage adjust" on the modulator control panel for each modulator until the current reaches the desired value. 3.3.6 Watching W.G. #1 and W.G. #2 load signals (R.F. signals), increase R.F. drive levels to klystrons #1 and #2 until waveforms stop increasing. At this point klystron drive is saturated. 3.3.7 Using log sheet for the last known run for experimenter of the day, insert phase and power numbers from that run. 3.3.8. Using the same log sheet, set up transport system (quadrupoles, steering and bending magnets, etc.). 3.3.9 Set injector selector to desired pulse length, turn off injector pulses and turn on injector high voltage. (Refer to Appendix A for current and repetition rate limitations). 3.3.10 Turn on injector pulses.

Step	Action
	3.3.11 A. For µsec pulses, watching beam amplitude and shape on scope, raise and lower RF phases until maximum beam current is obtained for the best pulse shape. B. For nano-sec and picosec pulses, watching beam current meter, raise and lower RF phases until maximum beam current is obtained. 3.3.12 Adjust lens #1 and #2 for maximum beam current. 3.3.13 If a defined energy distribution of the electrons is necessary, an energy spectrum can be plotted by bending the beam at Table #2 - 90° port. Use the EPICS program to measure and set the energy. 3.3.14 Log the settings of the Linac parameters. (Print out and retain as a record the Daily Log (Appendix B) generated by the Linac software.) Linac is now ON and ready for use. 3.4 Linac Operation for Linac Operator Return the beam to the experimenter's port, shape beam spot to desired shape with Quadrupoles and steering. **Linac operator must be present in the control room all the time during beam-on operations. If one operator have to leave control room for any reason while beam is on, other operator has to take his place in the control room or Linac has to be placed in stand-by mode.**
4	**Stand-by Operation for Linac Operator** For any long delay in use of the Linac, it should be placed in stand-by as follows: 4.1 Turn off modulator high voltage key switch and remove. 4.2 Turn off injector high voltage. 4.3 Turn off Helmholtz power supply. 4.4 Place short pulser in pulsed diode mode (if used). 4.5 Close exit valve from Linac accelerator section NOTE: Potential beam area may be entered during Stand-By Operation. Vault must be secured before leaving the Stand-By Operation mode.
5	**Machine Shut Down Instructions for Linac Operator** 5.1 If sub-harmonic buncher is on, run down and turn off high voltage power supply and RF preamplifier (in modulator room). 5.2 Run gun filament voltage to minimum (at control console). Place short pulser in pulsed diode mode (if used). Turn off Aux. Power switch 5.3 If bending magnet is in use, turn off silicon controlled rectifier power supply (SCR), set generator current to zero and degauss magnet twice, then turn off generator (at control console).

Step	Action
	5.4 Turn off Helmholtz coils and close B.V. 2 and B.V. 5.
	5.5 Turn off the control power (fire switch at control console).
	5.6 Turn off the 480 disconnect connector – circuit breaker 2B (in modulator room):
	5.7 Turn off the contactors for the EMI power supplies (cabinets on east wall of modulator room. Check to make sure that the power is off to the capacitor charging supplies in modulators 1 and 2 (lights should be off on power supply.)
	5.8 Open any of the following which are closed if they are not required to be left closed by the experimental review documentation: A. Pit door. B. Pit-cell gate C. Cell #1 door. D. Cell #2 gate.
	5.9 Turn off all unnecessary lighting.

** Denotes the ASE controlled parmeters.

4 Records Created by Work Process

The records listed below must be retained as indicated.

Description of Record (include form number if applicable)	Active Records Custodian	Active Records Retention	Indexing Method, Storage Medium	Federal Retention Requirements*
Linac Daily Log Sheet	Facility Manager	3 years	Index by job date and name, store on paper or electronically	Destroy 75 years after the date of the permit (DOE ADM 18.37)

*If records are maintained in a business information system that is not currently programmed to purge digital records based on age, the records may be retained in that system past the indicated destruction date.

5 Related Documents

This procedure implements requirements established by the following safety basis documents:

- LEAF-SAD-100, Linac Safety Assessment Document
- LEAF-ASE-100, Linac Accelerator Safety Envelope

This procedure implements requirements established by the following Argonne policies and procedures:

- LMS-PROC-188, *Accelerator Safety*
- EGS-PP-100, *Configuration Management Program Plan for Accelerators*

6 Definitions

None

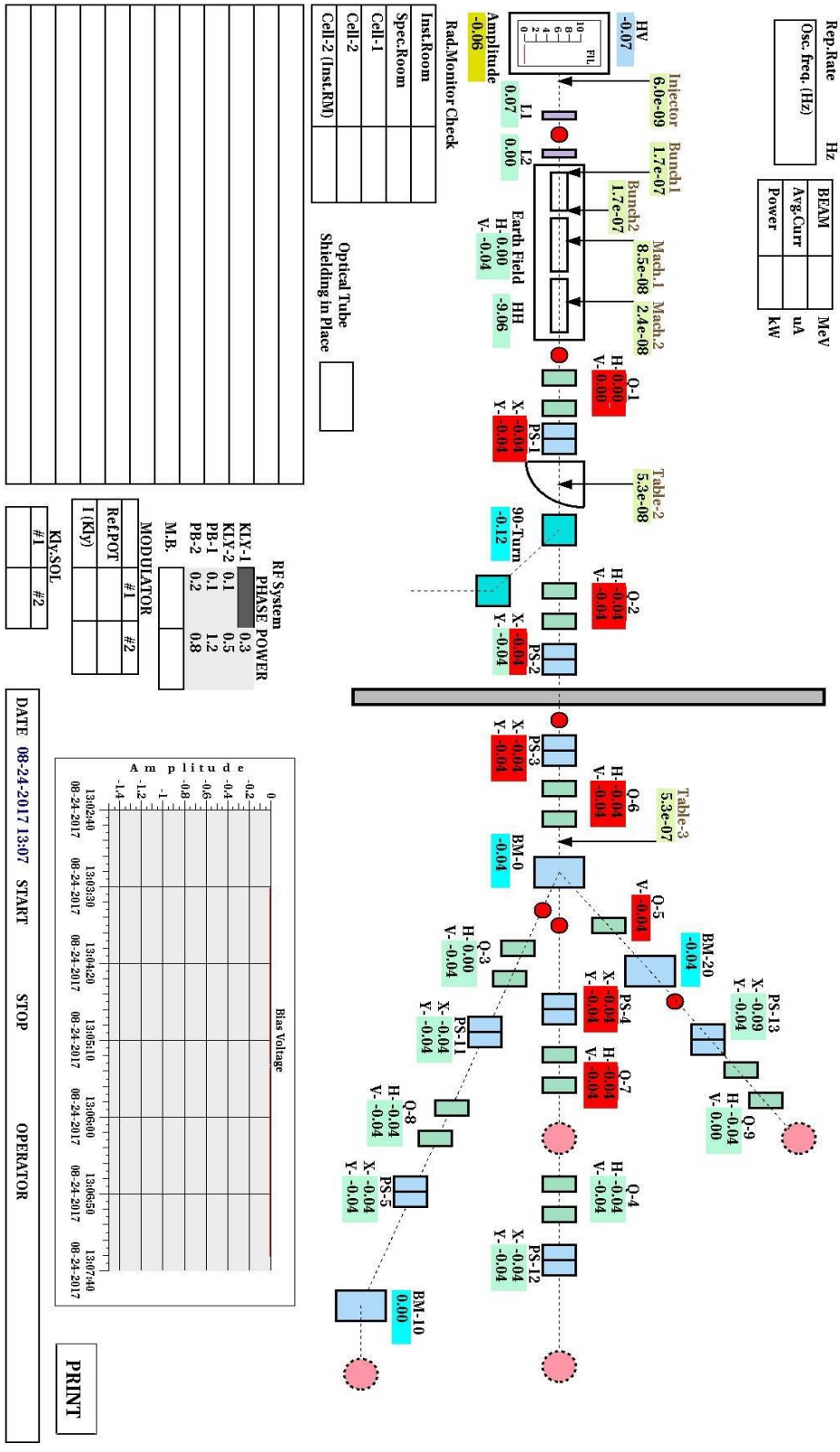
7 About this Procedure

Issuing organization:	Low Energy Accelerator Facility
Procedure owner:	S. Chemerisov
Point of contact:	S. Chemerisov
Review cycle (months):	36
Date last revised:	03.25.2019
Date last reviewed:	03.25.2019

8 Summary of Changes in This Version

Initial release.

Appendix A: Example Linac Daily Log Sheet



APPENDIX 26

LEAF-PROC-012, Rev. 0: AMORE Startup Checklist for Beam on Target

AMORE Startup Checklist for Beam on Target

Low Energy Accelerator Facility, LEAF-PROC-012, Rev. 0

Approved:  Date: 03.26.2019

Sergey Chemerisov, Manager, IVEM/LEAF

Effective Date: 04.01.2019

The current version of this document resides at https://leaf-docdb.ne.anl.gov/cgi-bin/DocumentDatabase. Printed or electronically downloaded copies may be obsolete. Before using such a copy for work direction, employees must verify that it is current by comparing its revision number to that shown in the on-line version.

1 Purpose

Establish the process for verifying all conditions are met to start irradiation for AMORE experiment at the Linac facility.

2 Scope

This procedure applies to the following Argonne activities and entities.

LMS core processes:	Asset Management
Organizations:	Experimental Operations and Facilities (EOF) Division
Buildings:	211
Specific locations:	LINAC
Other applicability factors:	None
Exclusions:	None
USI applicability:	Yes

3 Work Process

3.1 Introduction

In order to put beam on target for AMORE experiments multiple system has to be operational and in proper configuration to perform irradiation. This procedure identifies the step to verify readiness for the beginning of the experiment.

3.2 Step-by-Step Procedure

The steps below are mandatory unless noted otherwise. This procedure is to be performed by Linac operator or properly trained facility personnel.

3.2.1 Actions

Step	Action (Initial)
1	DU Target Cooling System 1.1 Perform routine startup procedure _____ 1.2 Coolant flow to the target is on _____ 1.3 Flowmeter calibration is current _____ 1.4 Thermocouple calibration is current _____ 1.5 Air purge calibration is current _____ 1.6 Verify that stop of the purge gas flow will interrupt interlock chain _____

Step	Action (Initial)
2	20L Tank Cooling System 2.1 Perform routine startup procedure _____ 2.2 Verify coolant flow is on _____ 2.3 Verify stop of the pump will interrupt interlock chain _____
3	Chiller 3.1 Perform Initial and Routine startup procedure for the chiller _____ 3.2 Verify coolant flow through the heat exchanger _____
4	20L tank 4.1 Verify 20L tank is ready _____ 4.2 Verify thermocouples temperatures are recorded _____ 4.3 Verify temperatures are in required range _____ 4.4 Verify stop of the pump will interrupt interlock chain _____
5	Separation glovebox 5.1 Verify glovebox is in ready condition _____
6	Gas collection and analysis system 6.1 Gas collection system is in ready condition _____ 6.2 Gas analysis system is in ready condition _____ 6.3
7	Fire department 7.1 Notify fire department about start of AMORE irradiation by calling 2-6131 _____

4 Records Created by Work Process

The records listed below must be retained as indicated.

Description of Record (include form number if applicable)	Active Records Custodian	Active Records Retention	Indexing Method, Storage Medium	Federal Retention Requirements*
This completed procedure	Facility Manager	3 years	Index by job date and name, store on paper or electronically	Destroy 75 years after the date of the permit (DOE ADM 18.37)

*If records are maintained in a business information system that is not currently programmed to purge digital records based on age, the records may be retained in that system past the indicated destruction date.

5 Related Documents

This procedure implements requirements established by the following safety basis documents:

- LEAF-SAD-100, Linac Safety Assessment Document
- LEAF-ASE-100, Linac Accelerator Safety Envelope

This procedure implements requirements established by the following Argonne policies and procedures:

- LMS-PROC-188, *Accelerator Safety*
- EGS-PP-100, *Configuration Management Program Plan for Accelerators*

6 Definitions

None

7 About this Procedure

Issuing organization:	Low Energy Accelerator Facility
Procedure owner:	S. Chemerisov
Point of contact:	S. Chemerisov
Review cycle (months):	36
Date last revised:	03.20.2019
Date last reviewed:	

8 Summary of Changes in This Version

Initial release.

APPENDIX 27

LEAF-PROC-011, Rev. 3: LEAF D-024 Hot Cell. 211/D-024 Hot Cell Operations AMORE

LEAF D-024 Hot Cell. 211/D-024 Hot Cell Operations AMORE

Low Energy Accelerator Facility, LEAF-PROC-011, Rev. 3

Approved:  Date: 12.23.2020

Sergey Chemerisov, Manager, IVEM/LEAF

Effective Date: 01.04.2021

1 Purpose

This procedure provides instructions for performing Argonne Molybdenum Research Experiment (AMORE) Phase II Tests at Building 211 Low Energy Accelerator Facility (LEAF) 211-D024 Hot Cell. The document also includes a Work Aid for operations involving the Concentration Column and LEU Modified Cintichem.

2 Scope

This procedure applies to the following Argonne activities and entities.

LMS core processes:	Asset Management
Organizations:	Experimental Operations and Facilities (EOF) Division
Buildings:	211
Specific locations:	LINAC
Other applicability factors:	None
Exclusions:	None
USI applicability:	Yes

3 Work Process

3.1 Introduction

This document provides instructions for AMORE ^{99}Mo Phase II Tests –211-D024 Hot Cell Operations, including a Work Aid for the Concentration Column and LEU Modified Cintichem operations. The flow diagram of the concentration column has been attached to this document and is posted at the job-site.

3.2 Step-by-Step Procedure

Sections 3.2.1 through 3.2.2 are mandatory and must be performed exactly as written. Sections 3.2.3 through 3.2.8 are considered guidance and are not required to be performed exactly as written. This procedure is to be performed by trained personnel.

3.2.1 Concentration Column

Step	Action
	Warning: Steps in this section and the next (3.2.1 and 3.2.2) must be performed exactly as written.
1	Follow Sections 3.2.3 through 3.2.6 of this procedure. These sections are meant to provide a step-by-step operation of the experiment. If deviations from the steps are made, they must be documented in the associated laboratory notebook and referenced in pen on the printout of this procedure.

Step	Action
2	The operator/worker may not deviate from items in Section 3.2.1.
3	All workers must have all required training for the work they are performing up to date.
4	All workers must have been read in and briefed for all RWPs required for the work they are performing.
5	The hot cell must be smeared under HPT guidance prior to entry.
6	Effluent bottles may not be reused, replace as necessary with HPT support.
7	The 3L 5-neck vessel must be sealed prior to receiving any solution from the recovery glove box. 7.1 pH probe is installed for Mo-99 processing solution from Recovery Glove Box. 7.2 24/40 plug in place of pH probe for solutions other than Mo-99 processing or wash/rinse solutions being received after AMORE tests. Other solutions may include water, acids, or bases used during commissioning or general testing of AMORE systems. 7.3 All ports are stoppered with Teflon adapter or septum.
8	**PRIOR** to transfer of the solution from recovery glove box, the Primary Recovery Team must be informed that D-024 hot cell is prepared to receive solution. Step-by-step operation of the experiment are provided in Section 3.2.4 and 3.2.5.
9	Hot cell systems are connected to gas collection by opening the gas collection valve (tri-dent valve, 2WV-803). The valve is located inside the hot cell just to the left of center of the hot cell near the floor (left of the concentration column valve board). An image of this valve is posted at the worksite. This valve should be closed during non-operational periods unless used to vent effluent bottles. 9.1 This valve must be open to connect pressure equalization lines (VNT-13 to gas collection system, image and flow diagram posted at work site). 9.2 This valve must be open to connect the LMC vacuum exhaust to gas collection 9.3 This valve must be open to vent effluent bottles in secondary under the hot cell
10	The 3L 5-neck vessel must be connected to gas collection system at all times when solution received from the recovery glove box is present, including during receiving. 10.1 This may be accomplished by one of or both methods described in 10.1.1 and 10.1.2. 10.1.1 Recovery Glovebox team opens solenoid valve NCSV-802 (image and flow diagram posted at work site). 10.1.2 Hot cell team opens vale 2WV-801 (image and flow diagram posted at work site).

Step	Action
11	The 3L 5-neck vessel is never opened when processed solution from the recovery glove box is present. 11.1 Never remove the pH probe when processing solutions are present. 11.2 Never remove the septum when processing solutions are present. 11.3 Never remove the Teflon ports when processing solutions are present.
12	The 3L 5-neck vessel may only be opened when removing the wash solution sent from the recovery glove box. This solution is only sent after full processing of the irradiated solution has been completed (after the LEU Modified Cintichem Process).
13	The wash solution must be removed prior to performing the next AMORE process.

END OF SEGMENT

3.2.2 LEU Modified Cintichem Operations

Step	Action
	Warning: Steps in this Section (3.2.2) must be performed exactly as written.
1	Follow Sections 3.2.7 through 3.2.8 of this procedure. These sections are meant to provide a step-by-step operation of the experiment. If deviations from the steps are made, they must be documented in the associated laboratory notebook and referenced in pen on the printout of the procedure.
2	The operator/worker may not deviate from items in section 3.2.2.
3	All workers must have all required training for the work they are performing up to date.
4	All workers must have been read in and briefed for all RWPs required for the work they are performing.
5	The D-024 hot cell anti-chamber must be smeared under HPT guidance prior to use of the D-024 hot cell anti-chamber.
6	Reagent bottles, sampling syringes, and vials may not be reused. Replace as necessary with HPT support.
7	Hot cell systems are connected to gas collection by opening the gas collection valve (tri-dent valve, 2WV-803). The valve is located inside the hot cell just to the left of center of the hot cell near the floor (left of the concentration column valve board). An image of this valve is posted at the worksite. This valve should be closed during non-operational periods unless used to vent effluent bottles. 7.1 This valve must be open to connect pressure equalization lines (VNT-13 to gas collection system. Image and flow diagram is posted at work site.) 7.2 This valve must be open to connect the LMC vacuum exhaust to gas collection 7.3 This valve must be open to vent effluent bottles in secondary under the hot cell 7.4 Verify the gas collection valve (tri-dent valve, 2WV-803) is OPEN prior performing LMC process in hot cell. Image and flow diagram is posted at work site. Date: _____ Time: _____
8	Make sure that for any operation when solutions are added to LMC bottles, columns, or sample vials, that they are pressure equilibrated. The gas collection line can be used for pressure equalization. This prevents pressurization of LMC bottles, columns, and sample vials.

Step	Action
9	When LMC processing in hot cell has concluded (including sample collection), close the gas collection valve (tri-dent valve, 2WV-803). The valve is located inside the hot cell just to the left of center of the hot cell near the floor (left of the concentration column valve board). An image of this valve is posted at the worksite. This valve should be closed during non-operational periods unless used to vent effluent bottles or any LMC bottles. Verify the gas collection valve (tri-dent valve, 2WV-803) is CLOSED after performing LMC process in hot cell. Image and flow diagram is posted at work site. Date: _____ Time: _____
10	If gas collection valve needs to be left open, provide explanation and notify gas collection system custodian, Mike Kalensky, at x2-4168. Explanation: _____ _____ _____ _____

END OF SEGMENT

Note: Sections 3.2.3 through 3.2.8 are considered guidance and are not required to be performed exactly as written.

3.2.3 Concentration Column

Step	Action
1	Prepare D-024 Hot Cell for AMORE operations 1.1 Number of workers suggested for these Work Aides: a. Minimum for Steps 1.2 and 1.3, dependent on D-024 hot cell radiological posting and discussions with Health Physics (HP). i. Worker 1: _____ ii. Worker 2 (optional): _____ iii. HPT: _____ b. If respirator is required, a minimum of two personnel and one Health Physics Tech (HPT) are required. 1.2 Confirm ≤500,000 dpm removable contamination within D-024 hot cell a. D-024 Hot Cell manipulator operator b. HP Tech 1.3 Setup of AMORE operations inside D-024 Hot Cell a. Entry worker – worker entering D-024 Hot Cell b. Watch worker – worker standing watch outside of D-024 Hot Cell i. Hands items to entry worker as needed ii. Responsible for these work aides and that all steps are checked off iii. Documents preparations in lab notebook 1.4 Inside-D024 HP Tech monitors activities inside 211-D024 1.5 Outside-D024 HP Tech assists Inside-D024 HP Tech with getting smears counted
2	Verify RWPs are current and workers read-in 2.1 Use https://apps.inside.anl.gov/rwp/permits 2.2 RWP suffix -211-004 a. Title: Work in a contamination area without engineering controls (e.g., benchtop, room) b. Verify RWP active c. Verify personnel identified for assignment in step 1.2.a is read in d. Verify personnel identified for assignment in step 1.2.b is read in 2.3 RWP suffix -211-024

Step	Action
	a. Title: Transfers in and out of the D-024 Hot cell and shielded glovebox antechambers (transfer ports) b. Verify RWP active c. Verify personnel identified for assignment in step 1.2.a is read in d. Verify personnel identified for assignment in step 1.2.b is read in 2.4 RWP suffix -211-030 a. Title: Transfer Equipment / Material in or out of D-024 hot cell b. Verify RWP active c. Verify personnel identified for assignment in step 1.3.a is read in d. Verify personnel identified for assignment in step 1.3.b is read in e. Verify personnel identified for assignment in step 1.4 is read in f. Verify personnel identified for assignment in step 1.5 is read in
3	Verify Permit Required Confined Space (PRCS) requirements 3.1 Is this permit needed? Verify personnel identified for assignment in step 1.3.a has completed and is up-to-date on PRCS entry training, ESH113A. a. D-024 hot cell is classified as a PRCS (211-0D-008) that requires 2-persons for single person entry b. Fill out permit for confined space entry (used only for that job and then permit is terminated)
4	Confirm supply of PPE for setup of AMORE Operations inside D-024 Hot Cell 4.1 Entry Worker PPE (worker identified in 1.3.a) a. Training. Verify personnel identified for assignments is up-to-date on the following training if respirator required (confirm with HP). • MEDCERT 114 Respirator Medical Certification • ESH 118 Resp. Protection – Air-purifying Respirator • ESH 118PR Resp. Protection – Air-purifying Respirator Practical Exercise b. Full respirator or PPE as required by the RWP. c. Double Tyvek coveralls or PPE as required by the RWP. • Use one to two sizes larger to allow for reaching/stretching d. First pair of gloves – Nitrile, long cuff e. Second pair of over-gloves – Nitrile, latex, or other chemical resistant gloves f. Shoe covers – Orange, rubber

Step	Action
	g. Tape – Vinyl tape h. Non-permeable gauntlets – Required for working with solutions 4.2 Watch worker PPE and Inside-D024 HP Tech a. Training. Verify personnel identified for assignments has following training up-to-date • MEDCERT 114 Respirator Medical Certification • ESH 118 Resp. Protection – Air-purifying Respirator • ESH 118PR Resp. Protection – Air-purifying Respirator Practical Exercise b. Full respirator or PPE as required by the RWP. c. Single Tyvek coveralls or PPE as required by the RWP. d. First pair of gloves – Nitrile, long-cuff e. Second pair of over-gloves – Nitrile, latex, or other chemical resistant gloves f. Shoe covers – Orange, rubber 4.3 Outside-D024 HP Tech a. Lab coat b. First pair of gloves – Nitrile, long-cuff c. Second pair of over-gloves – Nitrile, latex or other chemical resistant gloves d. Shoe covers – Orange, rubber
5	
6	Use checklist for pre-job brief • Verify all workers have initialed pre-job brief at conclusion of briefing
7	Confirm ≤500,000 dpm removable contamination within D-024 hot cell • Work under RWP 211-030 or RWP specified by HP (e.g., RWP 211-004) • While waiting for results proceed to next step
8	Outside the D-024 Hot Cell 8.1 Stage lab notebook and pen a. ANL notebook serial number: _____ b. Notebook page number(s): _____ 8.2 Stage equipment and chemicals for AMORE operations 1. Concentration column (see Figure 1). Keep column ends plugged with solid plastic screw plugs

Step	Action
	2. Four (4x) 60 mL septa vials: • Receiving vials for concentration column processing steps • Each vial is labeled, dated and placed in D-024 hot cell. • Verify vessels labeled: ☐ Acid wash ☐ And date ☐ Water wash ☐ And date ☐ Waste #1 ☐ And date ☐ Waste #2 ☐ And date 3. One (1x) Cintichem double ended bottle, "Mo-99 product vial" ☐ Sealed at both ends with rubber septa and aluminum crimp cap ☐ Upper crimp cap flap removed ☐ Lower crimp cap flap removed ☐ Labeled RF-1 ☐ Labeled with date placed in D-024 hot cell

Figure 1 Omnifit Benchmark column 15 mm O.D. x 100 mm L

Step	Action
8 (cont.)	4. Five (5x) 20 mL sampling septa vials • Each vial is labeled, dated and placed in D-024 hot cell • Verify vials labeled ☐ Mo-99 product ☐ And date Mass:_____ ☐ Acid wash ☐ And date Mass:_____ ☐ Water wash ☐ And date Mass:_____ ☐ Feed initial ☐ And date Mass:_____ ☐ Waste #2 ☐ And date Mass:_____ 5. Five (5x) Luer-Lock tip syringes Concentration column processing solutions: 1. Water reservoir fill • Syringe size x2 = 60 mL • Volume = 120 mL • Verify syringe labeled 2. 0.01 M HNO_3 reservoir fill • Syringe size = 60 mL • Volume = 60 mL • Verify syringe labeled 3. 1 M NaOH reservoir fill • Syringe size = 60 mL • Volume = 60 mL • Verify syringe labeled 4. 10 M NaOH • Syringe size = 10 mL • Volume = 10 mL • Verify syringe labeled ☐ And Date 5. Syringe – 30 mL x 1 in needle • 30 mL with Luer-Lock tip syringe • Load with 20 mL 8 M HNO_3 • Verify syringe labeled ☐ And Date 6. Bottle of 8 M HNO_3 • Bottle #1 – 140 mL 8 M HNO_3 • Verify bottle labeled 7. Six (6x) sampling syringes

Step	Action
	1. Syringes do not need to be labeled • Syringes are single use • Liquid sample not stored in syringe 2. Syringe #1 – 1 mL x 6 in. needle • 1 mL with Luer-Lock tip syringe • Fisher Scientific p/n 14-823-30 • Becton Dickinson p/n BD 309628 • 20 gauge x 8 in. Luer-Lock stainless steel needle • Fisher Scientific p/n 14-825-15AA • Cadence Science p/n 4187 3. Syringes #2-#5 – 1 mL x 5 in. needle • 1 mL with Luer-Lock tip syringe • Fisher Scientific p/n 14-823-30 • Becton Dickinson p/n BD 309628 • 18 gauge x 5 in. Luer-Lock stainless steel needle • Fisher Scientific p/n 14-817-105 • Air-Tite p/n N165 4. Syringe #6 – 3 mL x 8 in. or longer needle • 3 mL with Luer-lock tip • Fisher Scientific p/n 14-823-435 • Becton Dickinson p/n BD 309657 • 20 gauge x 8 in. Luer-Lock stainless steel needle • Fisher Scientific p/n 14-825-15AA • Cadence Science p/n 4187 8. Two (2x) 24/40 septa • Off-white • Part number 9. Herculite for D-024 Hot Cell door threshold • Verify extra pre-cut sheet(s) are available, stored under D-024 Hot Cell access door • If no extra sheets are available, cut 2–5 new sheets, 2 ft W by 4 ft L 10. Stage low level waste container • With liner 11. Stage paper towels 12. Stage zip lock bag • For previous concentration column

Step	Action
	13. One (1x) 1 L bottle with cap • Receiving vessel for final rinse contents of 3 L 5-neck flask
9	Clear area below D-024 Hot Cell access door 9.1 Remove/re-locate any unnecessary equipment 9.2 Ensure step stool is available
10	Verify step stool is in good working order
11	If interior smears come back ≤500000 dpm proceed to step #13
12	Open D-024 Hot Cell access door **NOTE:** Door is heavy • Use slow motions when opening/closing • Use latch on left side of door between wall and door to open • Use two hands to open door slowly until door rests on bumper – **DO NOT** bounce door off of bumper • Door will maintain open position when full open
13	At open D-024 Hot Cell access door 13.1 HP performs dose rate surveys 13.2 HP performs smears 1. Inner side of door 2. Door frame 3. Herculite sheet rolled up inside of D-024 hot cell ledge 13.3 BEFORE entry into the hot cell, the sheet is surveyed by the HP Tech 1. Verify Herculite sheet survey completed 2. Survey comes back **with no loose contamination** found: • Proceed with entry into D-024 Hot Cell 3. Survey comes back **contaminated**: • Replace outer gloves frequently, especially if torn or liquid found on gloves • Roll-up Herculite with containment of contamination inside of roll and remove from access door threshold and place in low-level waste receptacle • Survey area under Herculite just removed 3.a. Survey comes back with no loose contamination found – proceed to 13.4 3.b. Survey comes back with loose contamination found

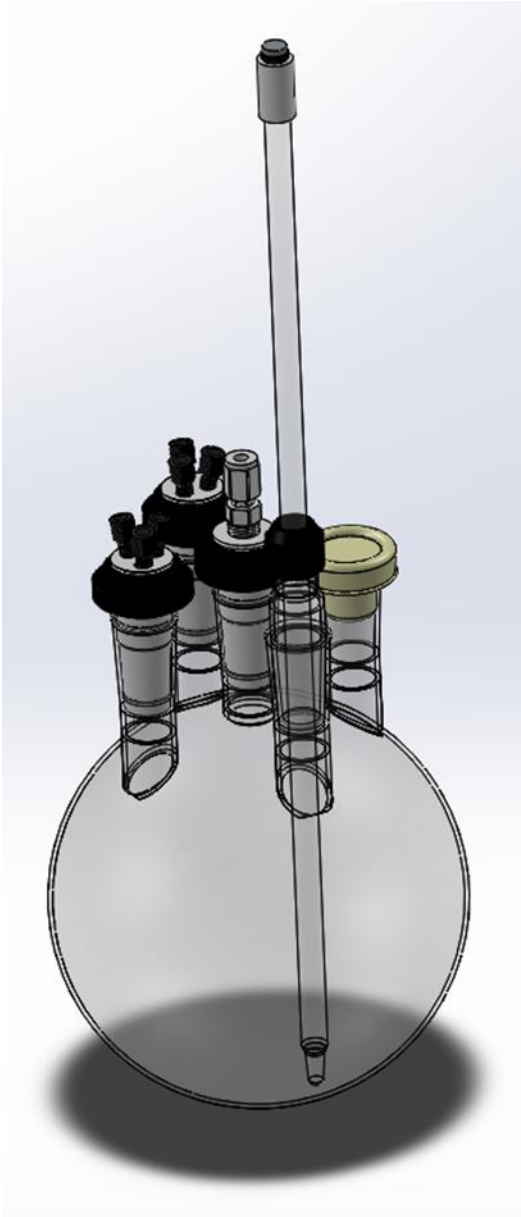
Step	Action
	• If within RWP limits, proceed with non-wet cleaning methods as described in WCD 55632 Task 2 • Survey and repeat as necessary to remove loose contamination and then proceed to 13.4. • If above RWP limits stop job 4. Retrieve new piece of Herculite 5. Install Herculite over D-024 Hot Cell access door threshold 6. Proceed with entry into D-024 Hot Cell 13.4 Remove any waste not previously removed from within hot cell • Place in low level waste container
14	Inside D-024 Hot Cell 14.1 Place four (4x) 60 mL septa vessels in white rack 14.2 Place five (5x) 20 mL sampling septa vials in white rack 14.3 Load NaOH solution reservoir with 1 M NaOH from 60 mL syringe 14.3.1 Pull syringe plunger to help relieve any build-up of pressure within reservoir while needle engaged with bottle 14.4 Load HNO_3 solution reservoir with 0.01 M HNO_3 from 60 mL syringe 14.4.1 Pull syringe plunger to help relieve any build-up of pressure within reservoir while needle engaged with bottle 14.5 Load Water solution reservoir with water from 60 mL syringe 14.5.1 Pull syringe plunger to help relieve any build-up of pressure within reservoir while needle engaged with bottle 14.5.2 Ensure water reservoir is full. If not full, add water as necessary to fill reservoir 14.6 Place sample syringes inside hot cell 14.6.1 Preferred position: center of hot cell, in front of valve board 14.7 Inspect solution plastic transfer lines. The solution transfer lines MUST be replaced at least every two years. Check the date on the log sheet on the front of the D-024 Hot Cell and verify the last time the lines were replaced. Date of replacement:_____ Today's date:__ _____ If this date is >2 years from today's date, replace the lines. If this date is < 2 years from today's date, proceed with the next step. 14.7.1 Defects are tubing crimped, brittle, flattened 14.7.1.1 From 5-way valve to Waste #1 60 mL vial (liquid effluent line)

Step	Action
	• Line in good working order ☐ date and time of inspection: Date:_____ Time:_____If signs of defects replace • Date of replacement: _____ 14.7.1.2 From 5-way valve to Acid Wash 60 mL vial (liquid effluent line) • Line in good working order ☐ date and time of inspection: Date:_____ Time:_____If signs of defects replace • Date of replacement: _____ 14.7.1.3 From 5-way valve to Water Wash 60 mL vial (liquid effluent line) • Line in good working order ☐ date and time of inspection: Date:_____ Time:_____If signs of defects replace • Date of replacement: _____ 14.7.1.4 From 5-way valve to RF-1 Cintichem 1-A bottle (liquid effluent line) • Line in good working order ☐ date and time of inspection: Date:_____ Time:_____If signs of defects replace • Date of replacement: _____ 14.7.1.5 From 3-way valve at FMI pump outlet to 3 L receiving vessel (liquid bypass line) • Line in good working order ☐ date and time of inspection: Date:_____ Time:_____If signs of defects replace • Date of replacement: _____ 14.7.1.6 From 3-way valve feed source selector to FMI pump inlet • Line in good working order ☐ date and time of inspection: Date:_____ Time:_____PEEK tubing can become brittle • If signs of defects replace • Date of replacement: _____ 14.7.1.7 From FMI pump outlet to 3-way valve destination selector • Line in good working order ☐ date and time of inspection: Date:_____ Time:_____PEEK tubing can become brittle • If signs of defects replace • Date of replacement: _____

Step	Action
	14.7.1.8 From 3-way valve at FMI pump outlet to 3 L receiving vessel (liquid bypass line) • Line in good working order ☐ date and time of inspection: Date:_____ Time:_____If signs of defects replace • Date of replacement: _____ 14.7.1.9 From 4-way valve at FMI pump outlet to 3 L receiving vessel (liquid feed line) • Line in good working order ☐ date and time of inspection: Date:_____ Time:_____If signs of defects replace • Date of replacement: _____ 14.7.1.10 From 2-way valve at From Recovery Glovebox line to 3 L receiving vessel (liquid feed line) • Line in good working order ☐ date and time of inspection: Date:_____ Time:_____If signs of defects replace • Date of replacement: _____ 14.7.1.11 From 0.1 M NaOH feed bottle to 4-way valve (liquid feed line) • Line in good working order ☐ date and time of inspection: Date:_____ Time:_____If signs of defects replace • Date of replacement: _____ 14.7.1.12 From Water feed bottle to 4-way valve (liquid feed line) • Line in good working order ☐ date and time of inspection: Date:_____ Time:_____If signs of defects replace • Date of replacement: _____ 14.7.1.13 From 1 M NaOH feed bottle to 4-way valve (liquid feed line) • Line in good working order ☐ date and time of inspection: Date:_____ Time:_____If signs of defects replace • Date of replacement: _____ 14.7.1.14 From valved quick-disconnect fitting to needle for concentration column ops (VQD-24, vent line) • Line in good working order ☐ date and time of inspection: Date:_____ Time:_____If signs of defects replace

Step	Action
	• Date of replacement: _____ 14.7.1.15 From vacuum pump base trap to needle for Cintichem ops (vent line) • Line in good working order ☐ date and time of inspection: Date:_____ Time:_____If signs of defects replace • Date of replacement: _____ 14.7.1.16 From tee below 2-way valve 2WV-19 to 3 L receiving vessel (vent line) • Line in good working order ☐ date and time of inspection: Date:_____ Time:_____If signs of defects replace • Date of replacement: _____ 14.8 Inspect SS Quick Disconnect valves (QDV806, 805, and 804) 14.8.1 Defects are embrittlement, cracks, or inability to connect/disconnect 14.8.1.1 From Effluent bottle enclosure QDV804 • Line in good working order ☐ date and time of inspection: Date:_____ Time:_____If signs of defects replace • Date of replacement: _____ 14.8.1.2 From ¼" butyl rubber vent line QDV805 • Line in good working order ☐ date and time of inspection: Date:_____ Time:_____If signs of defects replace • Date of replacement: _____ 14.8.1.3 From Cintichem vacuum QDV-806 • Line in good working order ☐ date and time of inspection: Date:_____ Time:_____If signs of defects replace • Date of replacement: _____ 14.9 Inspect PV Quick Disconnect valves (QDPV810, and 807) 14.9.1 Defects are embrittlement, cracks, or inability to connect/disconnect 14.9.1.1 From Cintichem needle QDPV807 • Line in good working order ☐ date and time of inspection: Date:_____ Time:_____If signs of defects replace • Date of replacement: _____

Step	Action
	14.9.1.2 From ¼" butyl rubber vent line QDVP810 • Line in good working order ☐ date and time of inspection: Date:_____ Time:_____If signs of defects replace • Date of replacement: _____ 14.10 Inspect Solenoid valve NCSV802 14.10.1 Energize valve and place magnet on string next to valve body, if magnetized valve assumed to be working • Line in good working order ☐ date and time of inspection: Date:_____ Time:_____If signs of defects replace • Date of replacement: _____ 14.11 Inspect SS valve 2WV801 14.11.1 Rotate valve, if valve rotates with slight friction, valve assumed to be working. If valve rotates freely, no friction, valve must be replaced • In good working order ☐ date and time of inspection: Date:_____ Time:_____If signs of defects replace • Date of replacement: _____ 14.12 Transfer final rinse contents from 3 L 5-neck vessel into 1 L bottle 14.12.1 Disconnect clamp(s) holding 3 L vessel 14.12.2 Remove rubber septum (pour from this neck) 14.12.2.1 Do not pour the stir bar into the 1 L bottle. 14.12.3 Remove 24/40 Telfon tubing adapters as necessary DO NOT remove lines from Teflon adaptor 14.12.3.1 Ensure no "sproing" of solution as lines removed from 3 L 5-neck vessel 14.12.3.2 Use paper towel to dab or grasp lines as they are removed from vessel 14.12.4 Prepare to use magnet to retain stir bar in 3 L vessel 14.12.5 Pour contents of vessel into 1 L bottle 14.12.5.1 Stir bar is still within vessel after solution removed 14.12.6 At completion of transfer wipe 24/40 ground glass joint clean 14.12.6.1 Inside and outside 14.12.7 Remove 1 L bottle 14.12.7.1 Bottle is placed in a zip lock bag • Place 1 L bottle in bag held by outside worker

Step	Action
	• Outside worker seals bag and places on herculite or paper towel on floor for HPT to survey • Replace outer gloves 14.12.8 Replace 24/40 rubber septum 14.12.8.1 Rubber sides of septum rolled over 24/40 joint exterior 14.12.9 Verify stir bar in reaction vessel. See Figure 2.  **Figure 2**

Step	Action
	14.13 Check and verify operation of balance
	14.14 Calibrate pH probe
	14.15 Wipe down any surfaces as needed
	14.16 Remove any waste and un-needed items
	14.17 Replace outer gloves
	14.18 Exit from interior hot cell
	14.19 Roll-up Herculite and place inside hot cell on access door threshold
	14.19.1 Final diameter of roll should fit on interior ledge of hot cell without spilling over either edge
	14.20 Replace outer gloves
	14.21 Entry Worker slowly close door with both hands
	14.21.1 KEEP HANDS ON OUTER MOST EDGES OF DOOR
	14.21.2 DO NOT LET DOOR SLAM SHUT
	14.22 Verify handle is in closed position and is holding door closed
15	HP Tech surveys workers and workers exit CA per training
16	HP Tech down post area
	16.1 Smears are taken of the floor, access door and horizontal surfaces in immediate vicinity
	16.2 Workers wait in vicinity
	16.2.1 HP releases respirator
	16.2.2 HP releases room
	16.2.3 HP tags waste bags

END OF SEGMENT

3.2.4 Prepare for Receipt of Primary Recovery Column Strip Solution from Primary Recovery Glovebox

Step	Action
1	Preparation. The following steps require two personnel: 1.1 Primary manipulator operator 1.1.1 Operates manipulators 1.1.1.1. Name:_____ 1.1.1.2. Date and time:_____ 1.2 Recorder/secondary manipulator operator 1.2.1 Tracks progress using these work aides – primary function 1.2.1.1. Name:_____ 1.2.1.2. Date and time:_____ 1.2.1.3. Name:_____ 1.2.1.4. Date and time:_____ 1.2.2 Records all values to lab notebook – primary function 1.2.3 Operates manipulators – secondary function as needed
2	Weigh all bottles and vials (Use this data in the summary table at the end of 3.2.6 (Page35) 2.1 Record model number of balance used: Model No.: _____ Date of last balance calibration check: _____ 2.2 Waste #1: _____ g (60 mL vial) 2.3 Waste #2: _____ g (60 mL vial) 2.4 Water wash: _____ g (60 mL vial) 2.5 Nitric acid wash: _____ g (60 mL vial) 2.6 RF-1: _____ g (Cintichem bottle)
3	Check pH probe MUST BE PERFORMED BEFORE SOLUTION TRANSFERRED FROM RECOVERY GLOVE BOX 3.1 pH meter on 3.2 Remove pH probe from storage holder 3.3 Insert pH probe into 3 L 5-neck vessel 3.4 End of segment 1.4.1 Date and Time:_____

4	Select appropriate storage vessel on solution storage container (below D-024) 4.1 Record <u>current</u> position of 6-way valve: ❶ ❷ ❸ ❹ ❺ ❻ (circle one) 4.2 Calculate next position: (value from step 4.1) + 1 = _____ 4.3 Rotate handle of 6-way valve to next position for current experiment: ❶ ❷ ❸ ❹ ❺ ❻ (circle one) 4.4 If bottle position other than the value calculated in 2.4.2 is used, give reason: _____ _____ _____ 4.5 Sign and date log sheet attached to D-024 Hot Cell 4.6 Record value of 10kg load cell holding 3 L 5-neck flask: _____ g • All tubing is attached • pH probe is inserted • Stir plate in place 4.7 Verify gas collection needle from VNT-13 inserted into Waste #1 60 mL vial 4.8 Open manual 2-way valve solution valve, 2WV-701 (image and flow diagram posted at work site), from Primary Recovery Glovebox line 4.8.1 Rotate valve handle counter-clockwise so that handle points down and is now in vertical position **<u>Checking D024 Hot Cell 3-L/5-Neck Flask Installation for Receipt of Mo-99 solution</u>** \\ **Recovery Member Name: _____PRINT Date: _____** **Time: _____** **D024 Hot Cell Ops Member Name: _____PRINT Date: _____** **Time: _____** ### SYSTEMS INTERFACE STEP ### 4.8.2 Contact a Recovery Glove Box Operation team member 4.8.3 Appropriate team member **<u>INITIALIZES</u>** every step in this section **4.8.4 Inside D024 Hot Cell** 4.8.4.1 ☐ Recovery ☐ HotCellOps Verify 3-L/5-neck flask in place 4.8.4.2 ☐ Recovery ☐ HotCellOps Verify pH probe inserted and not broken

Step	Action
	4.8.4.3 ☐ Recovery ☐ HotCellOps Verify plastic feed line attached to center neck 4.8.4.4 ☐ Recovery ☐ HotCellOps Verify septum secured in 24/40 port 4.8.4.5 ☐ Recovery ☐ HotCellOps Verify multi-port Teflon neck adapters inserted and attached to other two ports 4.8.4.6 ☐ Recovery ☐ HotCellOps Verify flask on balance 4.8.4.7 ☐ Recovery ☐ HotCellOps Verify balance is ON 4.8.4.8 Record balance reading: _____ grams 4.8.4.9 ☐ Recovery ☐ HotCellOps Verify 2WV-701 liquid valve OPEN (image and flow diagram posted at work site) • Handle parallel to the long axis of valve body 4.8.4.10 ☐ Recovery ☐ HotCellOps Verify plastic line attached between 2WV-701 and center neck of flask (image and flow diagram posted at work site) 4.8.4.11 ☐ Recovery ☐ HotCellOps Verify 2WV-801 vent valve OPEN (image and flow diagram posted at work site). • Handle parallel to the long axis of valve body 4.9 Inform Primary Recovery Glovebox team D-024 Hot Cell is prepared to receive 4.10 Primary Recovery Glovebox team open valve NCSV-802 or SWV-801 to vent 3L-5-neck vessel (image and flow diagram posted at work site) 4.11 Valve is open _____ (date) _____ (time)
5	Record beam parameters 5.1 Start of irradiation: _____ (date) _____ (time) 5.2 Energy: _____ MeV 5.3 Power: _____ kW 5.4 End of irradiation: _____ (date) _____ (time)

END OF SEGMENT

3.2.5 Receive Primary Recovery Column Strip Solution from Primary Recovery Glovebox

Step	Action
1	Preparation. The following steps require two personnel: 1.1 Transfer began: _____(date) _____(time) 1.2 Records dose rates measured by HP Tech into lab notebook 1.1.1 One of the following personnel 1.1.2 Primary manipulator operator • Operates manipulators 1.1.3 Recorder/secondary manipulator operator • Tracks progress using these work aides • Records all values to lab notebook 1.3 HP Tech Conducts dose measurements within 211-D024
2	Dose rates BEFORE initiation of transfer 2.1 Average dose rate at 211-D024 doorway: _____ 2.2 Average dose rate at 30 cm from transfer line: _____ 2.3 Average dose rate of lines on tact with lead bricks: _____
3	3.1 Transfer end: _____(date) _____(time) 3.2 Once solution transfer complete, record value of 10kg load cell holding 3 L 5-neck flask: _____ g

END OF SEGMENT

3.2.6 Process Primary Recovery Strip Product Through Concentration Column

Step	Action
1	Time of operation: _____ (date) _____ (time)
2	The following steps require two personnel 2.1 Primary manipulator operator • Operates manipulators • Name:_____ 2.2 Recorder/Secondary manipulator operator • Tracks progress using these work aides • Records all values to lab notebook • Name:_____ • Name:_____
3	Close manual 2-way valve, 2WV-701 from Primary Recovery Glovebox line (image and flow diagram posted at work site) • Turn valve clockwise so that valve handle is perpendicular to the floor
4	If not already open, open 2WV-801 to gas collection system 4.1 Vent line From 3 L 5-neck flask to gas collection system 4.2 Inform Primary Recovery Glovebox team that the 3 L 5-neck flask is now manually open to gas collection system and solenoid valve NCSV-802 can be closed (image and flow diagram posted at work site)
5	Fine adjustment of the pH of the primary recovery strip product 5.1 Turn on stir plate 5.2 Set stir rate to _____ 5.3 Adjust pH of recovery column primary strip product to pH 2 with appropriate solution: 1.3.1.8 M HNO_3: 1.3.1.1. Obtain mass of full syringe: _____g 1.3.2.10 M NaOH: 1.3.2.1. Obtain mass of full syringe: _____g 5.4 Insert needle of 30 mL syringe loaded with solution from 5.3 into septum of 3 L 5-neck vessel 5.4.1 If pH >10 observed, add 5 mL 8 M HNO_3 and observe change in pH (If pH <1 observed, add 5 mL 10 M NaOH and observe change in pH) 5.4.2 Allow pH to settle 5.4.3 Continue until pH <10 (or pH > 2 if using NaOH) observed and then proceed if not add another 5 mL of 8 M HNO_3 (or 10 M NaOH)

Step	Action
	5.5 Slowly add (dropwise) 8 M HNO_3 (or 10 M NaOH) until pH 2 is reached 5.6 Obtain mass of spent syringe: _____g
6	Collect sample of primary strip product after acidification 6.1 Stir primary strip product for at least 5 minutes after acidification complete 6.2 Weigh empty 20 mL vial labeled "Feed Init": _____ g _____ (date) _____ (time) 1.2.1.Use this data in the summary table at the end of 3.2.6 (Page35) 6.3 Use 3 mL syringe with 8 in. needle 6.4 Insert needle of 3 mL syringe into septum port of 3 L 5-neck flask 6.5 Draw 1–1.5 mL of solution into syringe 6.6 Remove tip of needle from solution 6.7 Draw 0.5 mL of flask head space into syringe 6.8 Prepare sample vial to receive 3 mL syringe needle 6.9 Draw ~75% of the needle out of the setpum 6.10 Grasp needle near base to avoid "sproing" and remove needle and 3 mL syringe from 3 L 5-neck flask and immediately insert into septum of 20 mL sample vial 6.11 Deliver syringe contents into sample vial 6.12 With needle still in the sample vial plunge the plunger of the 3 mL syringe several times to void all volume 6.13 Remove 3 mL syringe and set aside 6.14 Weigh 20 mL vial labeled "Feed Init" with sample: _____ g _____ (date) _____ (time) 1.14.1. Use this data in the summary table at the end of 3.2.6 (Page35) 6.15 Set syringe and needle aside for later disposal
7	Turn on temperature controllers marked column heater and pre-heater and pre-equilibrate column 7.1 Verify heater settings 7.1.1 Verify Pre-Heater set to 110 ºC 7.1.2 Verify Column Heater set to 90 oC 7.1.3 Turn on Pre-Heater controller by hitting the "reset" button 7.1.4 Turn on Column Heater controller by hitting the "reset" button 7.2 Water to column in UP flow direction

Step	Action
	7.2.1 Verify liquid needle from 7-way "Output" valve (7WV-709) inserted into Waste #1 60 mL vial
	7.2.2 Verify gas collection needle from VNT-13 inserted into Waste #1 60 mL vial
	7.2.3 Turn 7-way "Output" (7WV-709) valve to Waste
	7.2.4 Turn bottom "Column Control" 3-way valve (3WV-706) to upflow
	7.2.5 Turn upper "Column Control" 3-way valve (3WV-707) to upflow
	7.2.6 Turn left "Column Control" 3-way valve (3WV-708) to upflow
	7.2.7 Turn right "Column Control" 3-way valve (3WV-705) to upflow
	7.2.8 Turn upper "Pump Control" (inlet) 3-way valve (3WV-703) to COLUMN
	7.2.9 Turn lower "Pump Control" (outlet) 3-way valve (3WV-704) to COLUMN
	7.2.10 Turn feed source "Input" 5-way valve (5WV-702) to water feed bottle "Water"
	7.2.11 Open 2WV-803 to gas collection
	7.2.12 Verify pump pitch set to _____ micrometer reading
	7.2.13 Calculate pump power setting
	7.2.13.1 Desired flow rate = 50 mL/min
	7.2.13.2 Pitch value = _____ in.
	7.2.13.3 (576 mL/min) x (% motor power) x (pitch value) = output flow rate (mL/min)
	7.2.14 Verify V300 pump controller powered ON
	7.2.14.1 Rocker switch at bottom of controller housing
	7.2.15 Verify V300 pump controller to STOP
	7.2.15.1 Display alternates between % motor power setting and the word OFF
	7.2.16 Verify V300 pump controller set to _____ % motor power
	7.2.16.1 Recommended 67.2%
	7.2.17 Verify V300 pump controller set to FWD (forward)
	7.2.18 Verify V300 pump controller set to MANUAL
	7.2.19 Set timer to 26 seconds (22mL)
	7.2.20 Press RUN on V300 pump controller and start timer
	7.2.21 At timer end press STOP on V300 pump controller
	7.3 Water to column in DOWN flow direction
	7.3.1 Turn bottom "Column Control" 3-way valve (3WV-706) to downflow
	7.3.2 Turn upper "Column Control" 3-way valve (3WV-707) to downflow

Step	Action
	7.3.3 Turn left “Column Control” 3-way valve (3WV-708) to downflow 7.3.4 Turn right “Column Control” 3-way valve (3WV-705) to downflow 7.3.5 Verify V300 pump controller set to _____ % motor power 7.3.5.1 Flow rate = 50 mL/min 7.3.5.2 Recommended 67.2% 7.3.6 Set timer to 26 seconds (22mL) 7.3.7 Press RUN on V300 pump controller and start timer 7.3.8 At timer end press STOP on V300 pump controller 7.4 Acid (0.01 M HNO_3) to column in UP flow direction 7.4.1 Turn bottom “Column Control” 3-way valve (3WV-706) to upflow 7.4.2 Turn upper “Column Control” 3-way valve (3WV-707) to upflow 7.4.3 Turn left “Column Control” 3-way (3WV-708) valve to upflow 7.4.4 Turn right “Column Control” 3-way valve (3WV-705) to upflow 7.4.5 Turn feed source “Input Valve” 5-way valve (5WV-702) to acid feed bottle “HNO_3” 7.4.6 Verify V300 pump controller set to _____ % motor power 7.4.6.1 Flow rate = 50 mL/min 7.4.6.2 Recommended 67.2% 7.4.7 Set timer to 26 seconds (22mL) 7.4.8 Press RUN on V300 pump controller and start timer 7.4.9 At timer end press STOP on V300 pump controller
8	Prepare Mo99 transfer lines for column loading 8.1 Turn upper “Pump Control” valve (pump outlet) 3-way valve (3WV-703) to BYPASS (return to 3 L 5-neck flask) 8.2 Verify lower “Pump Control” valve (pump inlet) 3-way valve (3WV-704) is positioned to column 8.3 Turn “Input” feed source 5-way valve (5WV-702) to 3 L 5-neck flask “Feed” 8.4 Verify V300 pump controller set to _____ % motor power 8.4.1 Flow rate = 50 mL/min 8.4.2 Recommended 67.2% 8.5 Set timer to 20 seconds (16.7 mL)

Step	Action
	8.6 Press RUN on V300 pump controller and start timer
	8.7 At timer end press STOP on V300 pump controller
9	Load Mo99 product on to concentration column in UP flow direction
	9.1 Verify "Input" 4-way valve turned to 3 L 5-neck flask "Feed"
	9.2 Turn "Output" 7-way valve (7WV-709) to 6-way eluent bottle directing valve "Phase I Effluent"
	9.2.1. Ensure ball valve of solution line connected to effluent bottle is OPEN
	9.2.2. Ensure that the black luer-lock valve connected to the effluent bottle is OPEN
	9.2.3. Ensure that black luer-lock valve for sampling the effluent bottle is CLOSED
	9.2.4. Ensure that gas collection ball valve connected to the effluent bottle is OPEN
	9.2.5. Ensure that the black luer-lock valve connected to the gas collection needle is CLOSED or is inserted in to one of the 60 mL septum collection vials
	9.3 Verify bottom "Column Control" 3-way valve (3WV-706) to upflow
	9.4 Verify upper "Column Control" 3-way valve (3WV-707) to upflow
	9.5 Verify left "Column Control" 3-way valve (3WV-708) to upflow
	9.6 Verify right "Column Control" 3-way valve (3WV-705) to upflow
	9.7 Verify upper "Pump Control" (pump outlet) 3-way valve (3WV-703) to "Column"
	9.8 Verify lower "Pump Control" (pump inlet) 3-way valve (3WV-704) to feed source 4-way valve "Column"
	9.9 Turn "Input" (feed source) 5-way valve (5WV-702) to 3 L 5-neck flask "Feed"
	9.10 Turn off stirring
	9.11 Verify V300 pump controller set to _____ % motor power
	9.11.1 Flow rate = 50 mL/min (67.2%)
	9.12 Set timer to 50 minutes
	9.13 Press RUN on V300 pump controller and start timer
	9.14 Record time pump on: _____ (date) _____ (time)
	9.15 At timer end prepare to tilt vessel to process all of solution
	9.16 At air bubbles in flask pickup line press STOP on V300 controller
	9.17 Record time pump off: _____ (date) _____ (time)
10	Post-load acid wash
	10.1 Verify liquid needle from "Output" 7-way valve (7WV-709) inserted into Acid Wash 60 mL vial

Step	Action
	10.2 Turn “Output” 7-way valve (7WV-709) to Acid Wash 60 mL vial “acid wash”
	10.3 Verify bottom “Column Control” 3-way valve (3WV-706) to upflow
	10.4 Verify upper “Column Control” 3-way valve (3WV-707) to upflow
	10.5 Verify left “Column Control” 3-way valve (3WV-708) to upflow
	10.6 Verify right “Column Control” 3-way valve (3WV-705) to upflow
	10.7 Verify upper “Pump Control” (pump outlet) 3-way valve (3WV-703) to “Column”
	10.8 Verify lower “Pump Control” (pump inlet) 3-way valve (3WV-704) to feed source 4-way valve “Column”
	10.9 Turn “Input” (feed source) 5-way valve (5WV-702) to acid bottle “HNO_3”
	10.10 Insert “Gas collection” vent needle from VNT-13 into Acid Wash 60 mL vial
	10.11 Verify V300 pump controller set to _____ % motor power
	10.11.1 Flow rate = 50 mL/min (67.2%)
	10.12 Set timer to 51 seconds
	10.13 Press RUN on V300 pump controller and start timer
	10.14 At timer end press STOP on V300 pump controller
11	Post-load water wash
	11.1 Verify liquid needle from “Output” 7-way valve (7WV-709) inserted into Water Wash 60 mL vial
	11.2 Turn “Output” 7-way valve (7WV-709) to Water Wash 60 mL vial
	11.3 Verify bottom “Column Control” 3-way valve (3WV-706) to upflow
	11.4 Verify upper “Column Control” 3-way valve (3WV-707) to upflow
	11.5 Verify left “Column Control” 3-way valve (3WV-708) to upflow
	11.6 Verify right “Column Control” 3-way valve (3WV-705) to upflow
	11.7 Verify upper “Pump Control” (pump outlet) 3-way valve (3WV-703) to “Column”
	11.8 Verify lower “Pump Control” (pump inlet) 3-way valve to (3WV-704) feed source 4-way valve “Column”
	11.9 Turn “Input” (feed source) 5-way valve (5WV-702) to water bottle
	11.10 Insert “Gas Collection” vent needle from VNT-13 into Water wash 60 mL vial
	11.11 Verify V300 pump controller set to _____ % motor power
	11.11.1 Flow rate = 50 mL/min (67.2%)
	11.12 Set timer to 53 seconds
	11.13 Press RUN on V300 pump controller and start timer

Step	Action
	11.14 At timer end press STOP on V300 pump controller
12	Product strip 12.1 Verify Water needle from "Output" 7-way valve (7WV-709) inserted into Waste #2 60 mL vial 12.2 Verify Mo-99 product bottle, RF-1 Mo99 product (Cintichem style bottle, see Figure 3) is prepared and present  **Figure 3** Cintichem style bottle, 60 mm O.D. (bare glass) x 172 mm L, Safety coated 12.3 Verify "Input" 5-way valve to Water 12.4 Turn bottom "Column Control" 3-way valve (3WV-706) to downflow 12.5 Turn upper "Column Control" 3-way valve (3WV-707) to downflow 12.6 Turn left "Column Control" 3-way valve (3WV-708) to downflow 12.7 Turn right "Column Control" 3-way valve (3WV-705) to downflow 12.8 Verify upper "Pump Control" (pump outlet) 3-way valve (3WV-703) to "Column" 12.9 Verify lower "Pump Control" (pump inlet) 3-way valve to (3WV-704) feed source 4-way valve "Column" 12.10 Turn "Input" (feed source) 5-way valve (5WV-702) to NH_4OH bottle "NH_4OH" 12.10.1 Feed bottle is filled with 1 M NaOH 12.11 Verify "Gas Collection" vent needle from VNT-13 inserted into Waste #2 60 mL vial 12.12 Verify V300 pump controller set to _____ % motor power

Step	Action
	12.12.1 Flow rate = 11 mL/min (14.8%) 12.13 Set timer to 2 minutes (120 seconds) 12.14 Press RUN on V300 pump controller and start timer 12.15 At timer end press STOP on V300 pump controller 12.16 Turn "Output" 7-way valve (7WV-709) to RF-1 Mo99 product bottle "Product" 12.17 Insert "Gas Collection" needle from VNT-13 into RF-1 Mo99 product bottle 12.18 Verify V300 pump controller set to _____ % motor power 12.18.1 Flow rate = 11 mL/min (14.8%) 12.19 Set timer to 6 minutes (360 seconds) 12.20 Press RUN on V300 pump controller and start timer 12.21 At timer 1.36 minutes left 12.21.1 Verify feed in NaOH feed bottle 12.21.2 If feed bottle liquid level is too low to reach end of timer press STOP on V300 controller 12.22 At timer end press STOP on V300 pump controller
13	Post-strip water wash 13.1 Insert Mo-99 needle from "Output" 7-way valve (7WV-709) Waste #1 valve port into Waste #2 60 mL vial 13.2 Turn "Output" 7-way valve (7WV-709) to Waste #2 60 mL vial 13.3 Turn bottom "Column Control" 3-way valve (3WV-706) to downflow 13.4 Turn upper "Column Control" 3-way valve (3WV-707) to downflow 13.5 Turn left "Column Control" 3-way valve (3WV-708) to downflow 13.6 Turn right "Column Control" 3-way valve (3WV-705) to downflow 13.7 Verify upper "Pump Control" (pump outlet) 3-way valve (3WV-703) to "Column" 13.8 Verify lower "Pump Control" (pump inlet) 3-way valve (3WV-704) to feed source 4-way valve "Column" 13.9 Turn "Input" (feed source) 5-way valve (5WV-702) to Water bottle 13.10 Insert "Gas Collection" vent needle from VNT-13 into Waste #2 60 mL vial 13.11 Adjust V300 pump controller to _____ % motor power 13.11.1 Flow rate = 50 mL/min (67.2%) 13.12 Set timer to 1 minute (60 seconds) 13.13 Press RUN on V300 pump controller and start timer

Step	Action
	13.14 At timer end press STOP on V300 pump controller
14	Turn off J-Kern controllers
15	Weigh RF-1 Mo99 product bottle: _____ g _____ (date) _____ (time) Use this data in the summary table at the end of 3.2.6 (Page35)
16	Return RF-1 Mo99 product bottle to vial rack
17	Verify that 2WV-019 to gas collection system is still open 17.1 Vent line From 3 L 5-neck flask to gas collection system will remain open until system washed from Primary Recovery Column
18	Leave pH probe in vessel until next entry into cell
19	Turn off pump controller
20	Place gas collection line into RF1 “Mo-99 product bottle”
21	Sample solutions 21.1. Shake all vials with manipulators and obtain mass of each vessel 21.1.1. Waste #1: _____ g (60 mL vial) 21.1.2. Waste #2: _____ g (60 mL vial) 21.1.3. Water Wash: _____ g (60 mL vial) 21.1.4. Nitric Acid Wash: _____ g (60 mL vial) 21.1.5. Mo-99 Product (Cintichem Vessel): _____ g (60 mL vial) Use this data in the summary table at the end of 3.2.6 (Page35) 21.2. Use 1 mL syringes with 6” needles to sample vials. Pull the plunger to ~50% of the syringe shaft. Remove needle from solution while keeping needle within vessel being samples. Pull plunger to ~80% of the syringe shaft. Remove needle from vessel and inject sample into appropriate sampling vessel. Record mass of sample 21.2.1 Waste #2 vessel with sample: _____ g (20 mL vial) 21.2.2 Water wash vessel with sample: _____ g (20 mL vial) 21.2.3 Nitric acid wash vessel with sample: _____ g (20 mL vial) Use this data in the summary table at the end of 3.2.6 (Page35) 21.3. Sample Effluent Bottle 21.3.1. Ensure effluent bottle is connected to gas collection system 21.3.1.1. Ensure VQD-014 is connected

Step	Action
	21.3.1.2. Ensure gas collection needle for small bottles is in a septum bottle or closed 21.3.1.3. Ensure effluent bottle black luer lock valve is OPEN 21.3.1.4. Ensure effluent bottle ball valve is OPEN 21.3.2. Connect effluent bottle solution line to syringe 21.3.2.1. Ensure that 7WV-709 "OUTPUT" 7-way valve is directed towards any other output than "Phase I Effluent" – "OUTPUT" valve is NOT connected to effluent bottle being sampled. 21.3.2.2. Ensure effluent bottle solution ball valve is OPEN 21.3.2.3. Connect syringe (suggested 20-mL syringe with valve and plunger fully extended) to black luer lock at "t" connection to effluent bottle. 21.3.3. Mix and collect sample 21.3.3.1. Open black luer lock connecters 21.3.3.2. Depress syringe plunger to force air into the effluent bottle to mix system **(LEAVE ~5 ML OF AIR WITHIN THE SYRINGE)** 21.3.3.3. Pull syringe plunger up and down 3X to further mix the system **(LEAVE ~5 ML OF AIR WITHIN THE SYRINGE)** 21.3.3.4. Pull plunger to take sample (suggested ~1-3 mL) 21.3.3.5. Invert syringe so that solution is on plunger side and remaining 21.3.3.6. Depress syringe until bubbles are noticed in effluent bottle – OR – until sample is near top of syringe barrel (the point of this step is to void the lines and ensure no solution is in disconnect points) 21.3.3.7. CLOSE all solution black luer lock valves (2x) 21.3.3.8. Disconnect the syringe from the effluent bottle system while maintaining ONE black luer lock valve to the syringe. 21.3.3.9. Affix a needle to the black luer lock valve still connected to the syringe. 21.3.3.10. Inject solution into appropriately marked 20 mL septum vessel and record mass. 21.2.3.3.10.1 Effluent sample: _____ g (20 mL vial) Use this data in the summary table at the end of 3.2.6 (Page35)
22	Notify LMC team of completion Date:_____ Time:_____
23	Within one week of the AMORE experiment being completed the plastic lines that radioactive material transferred through them should be rinsed with water.

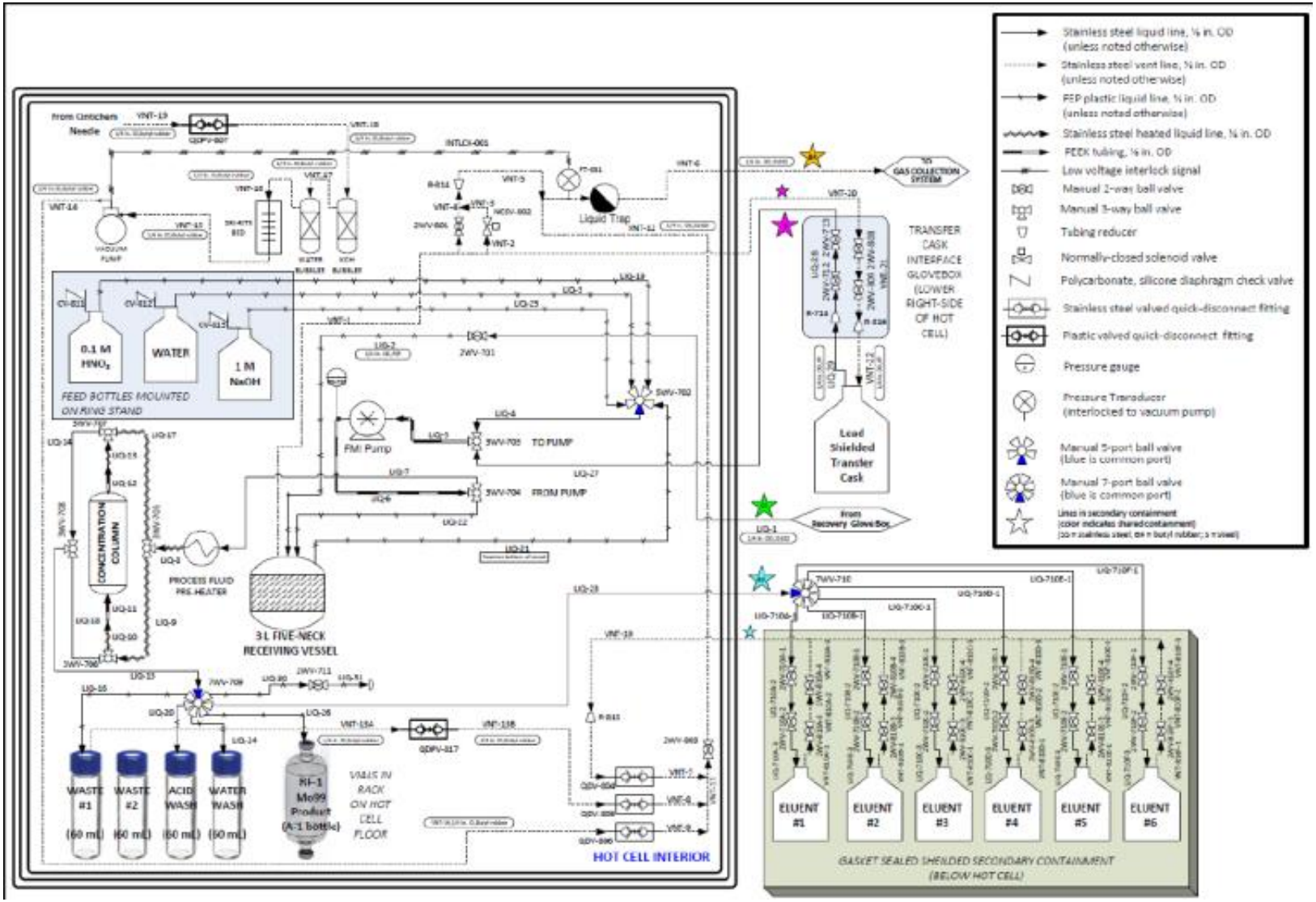
Step	Action
	Water wash pathway of column and acid wash line 23.1 Verify acid wash needle from "Output" 7-way valve (7WV-709) inserted into an empty 60 mL vial or collection bottle 23.2 Turn "Output" 7-way valve (7WV-709) to Acid Wash 23.3 Verify bottom "Column Control" 3-way valve (3WV-706) to upflow 23.4 Verify upper "Column Control" 3-way valve (3WV-707) to upflow 23.5 Verify left "Column Control" 3-way valve (3WV-708) to upflow 23.6 Verify right "Column Control" 3-way valve (3WV-705) to upflow 23.7 Verify upper "Pump Control" (pump outlet) 3-way valve (3WV-703) to "Column" 23.8 Verify lower "Pump Control" (pump inlet) 3-way valve to (3WV-704) feed source 4-way valve "Column" 23.9 Turn "Input" (feed source) 5-way valve (5WV-702) to water bottle 23.10 Insert "Gas Collection" vent needle from VNT-13 into collection bottle 23.11 Verify the luer-lock valve on the line is open 23.12 Verify V300 pump controller is ON and set to _____ % motor power Flow rate = 50 mL/min (67.2%) 23.13 Press RUN on V300 pump controller and start timer 23.14 Observe vial for liquid and rinse line for at least 10 seconds 23.15 Press STOP on V300 pump controller once rinse is complete Acid wash line rinse completed: ☐ Date:_____ Time:_____ Water wash pathway of column and water wash line 23.16 Verify water wash needle from "Output" 7-way valve (7WV-709) inserted into an empty 60 mL vial or collection bottle 23.17 Turn "Output" 7-way valve (7WV-709) to Water Wash 23.18 Verify bottom "Column Control" 3-way valve (3WV-706) to upflow 23.19 Verify upper "Column Control" 3-way valve (3WV-707) to upflow 23.20 Verify left "Column Control" 3-way valve (3WV-708) to upflow 23.21 Verify right "Column Control" 3-way valve (3WV-705) to upflow 23.22 Verify upper "Pump Control" (pump outlet) 3-way valve (3WV-703) to "Column" 23.23 Verify lower "Pump Control" (pump inlet) 3-way valve to (3WV-704) feed source 4-way valve "Column" 23.24 Turn "Input" (feed source) 5-way valve (5WV-702) to water bottle

Step	Action
	23.25 Insert “Gas Collection” vent needle from VNT-13 into collection bottle 23.26 Verify the luer-lock valve on the line is open 23.27 Verify V300 pump controller is ON and set to _____ % motor power Flow rate = 50 mL/min (67.2%) 23.28 Press RUN on V300 pump controller and start timer 23.29 Observe vial for liquid and rinse line for at least 10 seconds 23.30 Press STOP on V300 pump controller once rinse is complete Water wash line rinse completed: ☐ Date:_____ Time:_____ Water wash pathway of column and Mo99 Product line 23.31 Verify Mo99 Product needle from “Output” 7-way valve (7WV-709) inserted into an empty 60 mL vial or collection bottle 23.32 Turn “Output” 7-way valve (7WV-709) to Mo99 Product 23.33 Verify bottom “Column Control” 3-way valve (3WV-706) to upflow 23.34 Verify upper “Column Control” 3-way valve (3WV-707) to upflow 23.35 Verify left “Column Control” 3-way valve (3WV-708) to upflow 23.36 Verify right “Column Control” 3-way valve (3WV-705) to upflow 23.37 Verify upper “Pump Control” (pump outlet) 3-way valve (3WV-703) to “Column” 23.38 Verify lower “Pump Control” (pump inlet) 3-way valve to (3WV-704) feed source 4-way valve “Column” 23.39 Turn “Input” (feed source) 5-way valve (5WV-702) to water bottle 23.40 Insert “Gas Collection” vent needle from VNT-13 into collection bottle 23.41 Verify the luer-lock valve on the line is open 23.42 Verify V300 pump controller is ON and set to _____ % motor power Flow rate = 50 mL/min (67.2%) 23.43 Press RUN on V300 pump controller and start timer 23.44 Observe vial for liquid and rinse line for at least 10 seconds 23.45 Press STOP on V300 pump controller once rinse is complete Mo99 Product wash line rinse completed: ☐ Date:_____ Time:_____ Water wash pathway of column and waste wash line

Step	Action
	23.46 Verify waste needle from "Output" 7-way valve (7WV-709) inserted into an empty 60 mL vial or collection bottle 23.47 Turn "Output" 7-way valve (7WV-709) to Waste 23.48 Verify bottom "Column Control" 3-way valve (3WV-706) to upflow 23.49 Verify upper "Column Control" 3-way valve (3WV-707) to upflow 23.50 Verify left "Column Control" 3-way valve (3WV-708) to upflow 23.51 Verify right "Column Control" 3-way valve (3WV-705) to upflow 23.52 Verify upper "Pump Control" (pump outlet) 3-way valve (3WV-703) to "Column" 23.53 Verify lower "Pump Control" (pump inlet) 3-way valve to (3WV-704) feed source 4-way valve "Column" 23.54 Turn "Input" (feed source) 5-way valve (5WV-702) to water bottle 23.55 Insert "Gas Collection" vent needle from VNT-13 into collection bottle 23.56 Verify the luer-lock valve on the line is open 23.57 Verify V300 pump controller is ON and set to _____ % motor power Flow rate = 50 mL/min (67.2%) 23.58 Press RUN on V300 pump controller and start timer 23.59 Observe vial for liquid and rinse line for at least 10 seconds 23.60 Press STOP on V300 pump controller once rinse is complete Waste wash line rinse completed: ☐ Date:_____ Time:_____
24	Rinse Effluent Transfer Line with Water 24.1 Turn "Output" 7-way valve (7WV-709) to "Phase 1 Effluent" 24.2 Turn bottom "Column Control" 3-way valve (3WV-706) to downflow 24.3 Turn upper "Column Control" 3-way valve (3WV-707) to downflow 24.4 Turn left "Column Control" 3-way valve (3WV-708) to downflow 24.5 Turn right "Column Control" 3-way valve (3WV-705) to downflow 24.6 Verify upper "Pump Control" (pump outlet) 3-way valve (3WV-703) to "Column" 24.7 Verify lower "Pump Control" (pump inlet) 3-way valve (3WV-704) to feed source 4-way valve "Column" 24.8 Turn "Input" (feed source) 5-way valve (5WV-702) to Water bottle 24.9 Adjust V300 pump controller to _____ % motor power Flow rate = 50 mL/min (67.2%)

Step	Action
	24.10 Set timer to 30 seconds 24.11 Press RUN on V300 pump controller and start timer Stop V300 pump controller after time has elapsed Effluent line rinse completed: ☐ Date:_____ Time:_____
25	Effluent line water rinse back into 3-L 5-neck flask 25.1 Verify "Input" 4-way valve turned to 3 L 5-neck flask "Feed" 25.2 On V300 pump control change direction of pump from forward to reverse 25.3 Turn "Output" 7-way valve (7WV-709) to 6-way eluent bottle directing valve "Phase I Effluent" 25.3.1 Ensure ball valve of solution line connected to effluent bottle is OPEN 25.3.2 Ensure that the black luer-lock valve connected to the effluent bottle is OPEN 25.3.3 Ensure that black luer-lock valve for sampling the effluent bottle is CLOSED 25.3.4 Ensure that gas collection ball valve connected to the effluent bottle is OPEN 25.3.5 Ensure that the black luer-lock valve connected to the gas collection needle is CLOSED or is inserted in to one of the 60 mL septum collection vials 25.4 Verify bottom "Column Control" 3-way valve (3WV-706) to upflow 25.5 Verify upper "Column Control" 3-way valve (3WV-707) to upflow 25.6 Verify left "Column Control" 3-way valve (3WV-708) to upflow 25.7 Verify right "Column Control" 3-way valve (3WV-705) to upflow 25.8 Verify upper "Pump Control" (pump outlet) 3-way valve (3WV-703) to "Column" 25.9 Verify lower "Pump Control" (pump inlet) 3-way valve (3WV-704) to feed source 4-way valve "Column" 25.10 Turn "Input" (feed source) 5-way valve (5WV-702) to 3 L 5-neck flask "Feed" 25.11 Verify V300 pump controller set to _____ % motor power 25.11.1 Flow rate = 50 mL/min (67.2%) 25.12 Observe solution transfer during this step 25.13 Press RUN on V300 pump controller and start timer 25.14 As transfer nears completion prepare to tilt vessel to transfer all of the rinse solution 25.15 At air bubbles in flask pickup line press STOP on V300 controller Effluent line rinse completed: ☐ Date:_____ Time:_____
26	Transfer Water Rinse solution to Effluent storage below hot cell 26.1 Verify "Input" 4-way valve turned to 3 L 5-neck flask "Feed"

Step	Action
	26.2 Change V300 pump controller direction to forward 26.3 Turn “Output” 7-way valve (7WV-709) to 6-way eluent bottle directing valve “Phase II Effluent” 26.4 Verify bottom “Column Control” 3-way valve (3WV-706) to upflow 26.5 Verify upper “Column Control” 3-way valve (3WV-707) to upflow 26.6 Verify left “Column Control” 3-way valve (3WV-708) to upflow 26.7 Verify right “Column Control” 3-way valve (3WV-705) to upflow 26.8 Verify upper “Pump Control” (pump outlet) 3-way valve (3WV-703) to “Column” 26.9 Verify lower “Pump Control” (pump inlet) 3-way valve (3WV-704) to feed source 4-way valve “Column” 26.10 Turn “Input” (feed source) 5-way valve (5WV-702) to 3 L 5-neck flask “Feed” 26.11 Verify gas collection is open to effluent storage bottles and feed is directed to an un-used bottle 26.12 Verify V300 pump controller set to _____ % motor power Flow rate = 50 mL/min (67.2%) 26.13 Observe solution transfer until completion 26.14 Press RUN on V300 pump controller and start timer 26.15 At transfer completion prepare to tilt vessel to process all of solution 26.16 At air bubbles in flask pickup line press STOP on V300 controller Effluent water rinse completed: ☐ Date:_____ Time:_____


Stainless steel liquid line, ¼ in. OD (unless noted otherwise)
Stainless steel vent line, ¼ in. OD (unless noted otherwise)
PFP plastic liquid line, ¼ in. OD (unless noted otherwise)
Stainless steel heated liquid line, ¼ in. OD
PEEK tubing, ¼ in. OD
Low voltage interlock signal
Manual 2-way ball valve
Manual 3-way ball valve
Tubing reducer
Normally-closed solenoid valve
Polycarbonate, silicone diaphragm check valve
Stainless steel valved quick-disconnect fitting
Plastic valved quick-disconnect fitting
Pressure gauge
Pressure Transducer (interlocked to vacuum pump)
Manual 5-port ball valve (blue is common port)
Manual 7-port ball valve (blue is common port)
Lines in secondary containment (color indicates shared containment) (SS = stainless steel, BR = butyl rubber, S = vinyl)
From Cintichem Needle
VNT-19
QDPV-807
VNT-18
INTLCH-005
VNT-17
VNT-16
VACUUM PUMP
VNT-15
SKID KITS BID
ULTRA BUBBLER
VNT-14
VNT-13
VNT-12
VNT-11
Liquid Trap
VNT-10
VNT-9
VNT-8
VNT-7
VNT-6
VNT-5
VNT-4
VNT-3
VNT-2
VNT-1
NCDV-802
2WV-806
TO GAS COLLECTION SYSTEM
TRANSFER CASK INTERFACE GLOVEBOX (LOWER RIGHT-SIDE OF HOT CELL)
Lead Shielded Transfer Cask
from Recovery Glove Box
0.1 M HNO3
WATER
1 M NaOH
FEED BOTTLES MOUNTED ON RING STAND
CV-811
CV-812
CV-813
2WV-701
FMI Pump
5WV-702
5WV-705 TO PUMP
5WV-704 FROM PUMP
CONCENTRATION COLUMN
PROCESS FLUID PRE-HEATER
3 L FIVE-NECK RECEIVING VESSEL
7WV-708
7WV-709
7WV-710
7WV-711
WASTE #1 (60 mL)
WASTE #2 (60 mL)
ACID WASH (60 mL)
WATER WASH (60 mL)
RI-1 Mo99 Product (A-1 bottle)
VIALS IN RACK ON HOT CELL FLOOR
QDPV-817
QDV-804
QDV-808
QDV-896
HOT CELL INTERIOR
LIQ-710F-1
LIQ-710E-1
LIQ-710D-1
LIQ-710C-1
LIQ-710B-1
LIQ-710A-1
ELUENT #1
ELUENT #2
ELUENT #3
ELUENT #4
ELUENT #5
ELUENT #6
GASKET SEALED SHIELDED SECONDARY CONTAINMENT (BELOW HOT CELL)

Concentration Column Summary Table

	Mass of Empty Vessel	**Mass of Vessel with Solution**	**Mass of Solution**
Feed (3L vessel)			
Waste #1			
Waste #2			
Water Wash			
Nitric Acid Wash			
Mo-99 Product (RF-1 Cintichem bottle)			

Concentration Column Sample Summary Table

	Mass of Empty Sampling vessel	**Mass of Sampling Vessel with Sample**	**Mass of sample**
Feed Initial			
Waste #2			
Water Wash			
Nitric Acid Wash			
Effluent			

END OF SEGMENT

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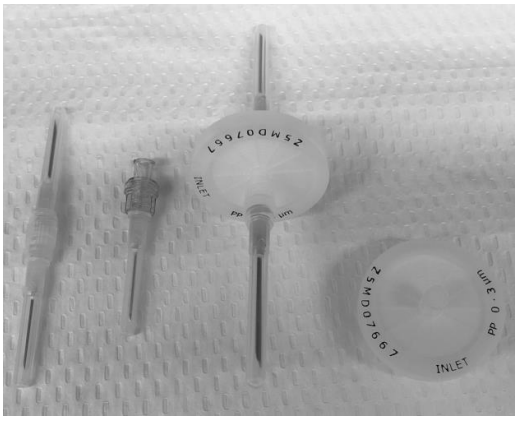
3.2.7 Prepare for Cintichem Operations

Step	Action
1	1.1 Verify that 2% alpha-benzoin oxime in 0.4M NaOH has been freshly prepared within 24 hours as operation is taking place. Date: _____ Time: _____ (solution must be <24 hours old) 1.2 Verify that molybdenum carrier solution (10 mg Mo/mL) has been freshly prepared. Date: _____ Time: _____ (solution must be <7 days old) 1.3 OPTIONAL STEP Verify that Ag/C column has been washed with 0.2M NaOH prior to using in hot cell. Washed Date: _____ Time: _____ 1.4 Verify that combination column (HZO/Ag/C) has been washed with 0.2M NaOH and pH of effluent was checked and was alkaline prior to using in hot cell. Washed Date: _____ Time: _____ 1.4.1 Verify pH of eluent was alkaline ☐
2	Stage the following solutions and glassware: 2.1 Sampling syringes 2.1.1 1 mL syringe x 5" needle with luer lock two-way valve ☐ 2.1.2 1 mL syringe x 5" needle with luer lock two-way valve ☐ 2.1.3 10 mL syringe x 5" needle with luer lock two-way valve ☐ 2.2 Sampling vials 2.2.1 20mL LSC vial with septa for RF1 bottle ☐ 2.2.2 20mL LSC vial with septa for RFW bottle ☐ 2.2.3 20mL LSC vial with septa for 1-B bottle ☐ 2.3 Cintichem bottles 2.3.1 Double-sided bottle labeled RF-2 (plastic coated) ☐ see Fig. 4 2.3.2 Flat bottom bottle labeled RFW (plastic coated) ☐ see Fig. 4 2.3.3 51-mm fritted glass column with ~20mL of glass beads – pre wet ☐ see Fig. 4 2.3.4 Double-sided bottle labeled 1-A (plastic coated) ☐ see Fig. 4 2.3.5 Flat bottom bottle labeled 1-B Mo99 Product (plastic coated) ☐ see Fig. 4

Step	Action
	2.3.6 Spare double-sided bottle (plastic coated) ☐ see Fig. 4 2.3.7 AgC/ZrO/AC column ☐ See Fig. 4 2.3.8 Charcoal filter ☐ See Fig. 4 2.4 Double-sided needles 2.4.1 Three 18 gauge double-sided needles ☐ To make one pair: connect two 18 gauge needles with male to male luer - see Fig. 5 2.4.2 Four 18 gauge double-sided needles with luer lock valve ☐ To make one pair: connect two 18 gauge needles with male to male luer and two-way valve 2.4.3 Three 16 gauge double-sided needles with luer lock valve ☐ To make one pair: connect two 16 gauge needles with male to male luer and two-way valve 2.4.4 Three one-way luer vent valve with 21 gauge needle ☐ see Fig. 5 2.4.5 Two 0.3µm 45mm filter with two 16 gauge needles connected via male to male luer ☐ - see Fig. 5 2.4.6 Three 21 gauge vent needles ☐ 2.5 All solutions, glassware and needles enter through D-024 Hot Cell transfer port 2.6 All syringes use a luer-lock 18 gauge 1-1.5 in. disposable needle 2.7 When solution is prepared, enter a ✓ in column I 2.8 When solution is staged in 211-D024, enter a ✓ in column II 2.9 When solution is placed into the transfer port, enter a ✓ in column VIII

Step	Action
	 **Figure 4** LMC glassware. From left to right: flat bottom bottle, double-sided bottle, 51-mm fritted glass column containing glass beads (~20mL of glass beads), AgC/ZrO/AC column, charcoal filter column (empty shown). All glassware uses crimps to hold septa in place  **Figure 5** From left to right: double-sided needle with male to male luer connector in the middle, luer one-way luer check valve needle, 40mm 0.3um filter with needles, 40mm 0.3um filter
3	Transfer Cintichem equipment and solutions into D-024 Hot Cell 2.1 The following steps require two personnel and an HPT 2.1.1 Open D-024 hot cell antechamber door according to RWP • HP Tech to perform survey and release area for work

Step	Action
	2.1.2 Personnel 1 2.1.2.1 Puts items into transfer port 2.1.3 Personnel 2 Recorder 2.1.3.1 Tracks items as they are place in transfer port 2.1.3.2 Mark column VIII in Table 1 (page 37) 2.1.4 HP Tech • Conducts dose measurements and smears by D-024 Hot Cell transfer port 2.2 Work under RWP suffix -211-024 **Title:** Transfers in and out of the D-024 and shielded glovebox antechambers (transfer ports) 2.3 Place Cintichem process items into transfer port: 2.3.1 Place large items on the antechamber sliding tray 2.3.2 Place syringe needles into Styrofoam tray in back of antechamber sliding tray • Ensure stopcock in closed position for solutions with H_2O_2 2.3.2.1 0.4M NaOH with ~1% H_2O_2 2.3.2.2 0.2M NaOH with ~1% H_2O_2 See Figures 6 and 7 for examples of aluminum needle guards.

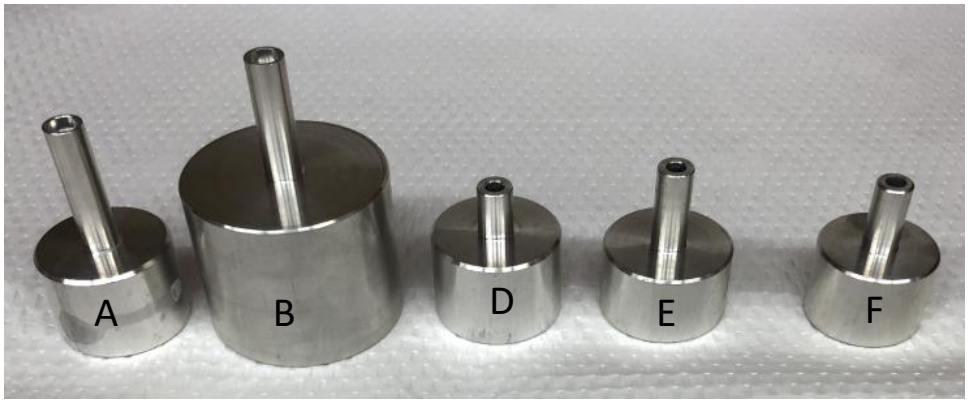
Step	Action
	 **Figure 6** Aluminum Needle Guards (Different Models), Model C Not Shown  **Figure 7** D-Type Aluminum Needle Guard With Side Port for Vacuum Line

Table 1 Solutions for LEU Modified Cintichem: To Be Passed Into D-024 Hot Cell Via Transfer Port (process steps are reference to Section 3.2.8)

Prepared I	Staged in 211-D024 II	Syringe Size (cc) III	Solution Volume (mL) IV	Solution V	Purpose VI	Process Step # VII	Transfer Port VIII
		25	15	10M HNO_3	Acidification of RF-1 bottle	6	
		5	4.0	NaI carrier 4.0 mL at 1 mg/mL	AgI ppt	11	
		1	0.5	10% $AgNO_3$ in 0.1 M HNO_3	AgI ppt	13	
		1	1.0	1 M HCl	Aid in ppt. of NaI/Cl	18	
		25	11	4 M HNO_3	Rinse the filter	33	
		1	0.5	Mo carrier (10 mg/mL)	Mo carrier	43	
		25	25	2.5% $KMnO_4$	Mo oxidation	44	
		2	1.5	Rh carrier (8 mg/mL)	Rh carrier	47	
		2	2.0	Ru carrier (5 mg/mL)	Ru carrier	50	
		25	20.0	Fresh 2% alpha-benzoin-oxime in 0.4 M NaOH Date: _____ Time: _____ (solution must be <24 hours old)	Mo ppt	54	
		25	20.0	0.1M HNO_3	Rinse	75	
		25	20.0	0.1M HNO_3	Rinse-second	81	
		25	20.0	0.1M HNO_3	Rinse-third	87	
		10	10.0	0.1M HNO_3	Rinse	101	
		10	10.0	0.1M HNO_3	Rinse-second	114	
		10	10.0	0.1M HNO_3	Rinse-third	127	
		10	10.0	0.1M HNO_3	Rinse-fourth	147	
		10	10.0	0.1M HNO_3	Rinse-fifth	167	
		10	10.0	0.4M NaOH with ~1% H_2O_2 Use polycarbonate 2-way valve between needle and syringe	Dissolve ppt	198	
		10	10.0	0.2M NaOH with ~1% H_2O_2 Use polycarbonate 2-way valve between needle and syringe	Dissolve ppt	221	
		10	10.0	0.2M NaOH.	Fritted column rinse	243	
		5	4.0	1 mg/mL NaI	AgI ppt	256	
		1	0.5	10% $AgNO_3$ in 0.1 M HNO_3	AgI ppt	257	
		10	10.0	0.2 M NaOH	Ag/C/HZO/AC column rinse	274	

END OF SEGMENT

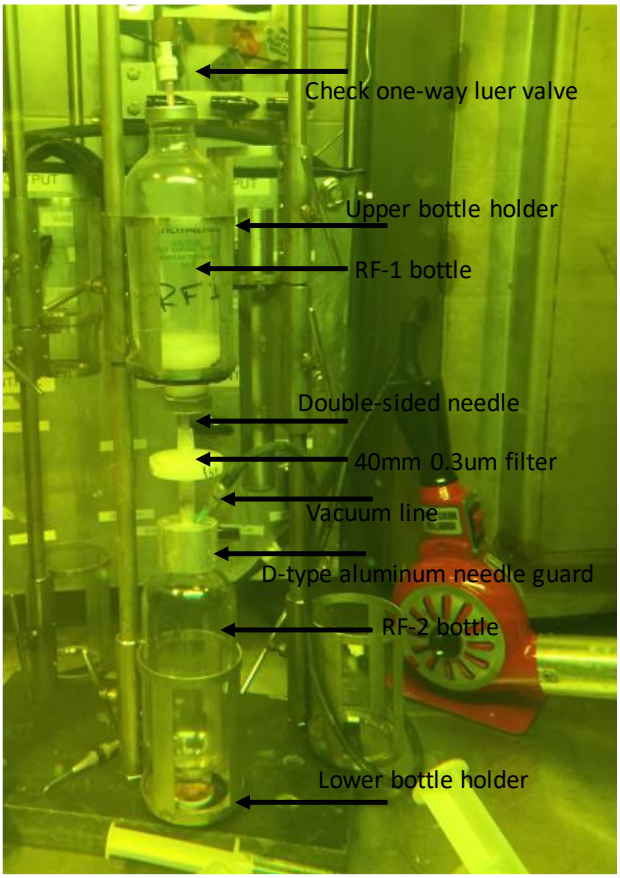
3.2.8 Conduct LEU Modified Cintichem Process

Step	Action
1	The following steps require two personnel 1.1 Primary manipulator operator: NAME: _____ • Operates manipulators 1.2 Recorder/Secondary manipulator operator: NAME:_____ 1.2.1 Tracks progress using these work aides 1.2.2 Records all values to lab notebook 1.2.3 Operates manipulators as needed START OF LMC OPERATIONS: DATE:_____ TIME: _____
2	Weigh the following Cintichem glassware components: 2.1 Use the 4000 g Ohaus balance for weighing 2.2 RF-1 bottle: _____ g // _____ (date) _____ (time) (double-sided bottle) 2.2.1 With Mo99 product from concentration column 2.2.2 Raw Fission #1 2.3 RFW bottle: _____ g // _____ (date) _____ (time) (flat bottom bottle) 2.3.1 This bottle is empty 2.3.2 Raw Fission Waste 2.4 1-B bottle: _____ g // _____ (date) _____ (time) (flat bottom bottle) 2.4.1 This bottle is empty 2.4.2 1-B Mo-99 product 2.5 RF-1 sample vial: _____ g // _____ (date) _____ (time) (20mL LSC vial with septum) 2.5.1 Solution sample after acidification 2.5.2 Raw Fission #1 after acidification 2.6 RFW sample vial: _____ g // _____ (date) _____ (time) (20mL LSC vial with septum) 2.6.1 Solution sample of RFW 2.6.2 Raw Fission waste sample

Step	Action
	2.7 1-B sample vial: _____ g // _____ (date) _____ (time) (20mL LSC vial with septum) 2.7.1 Mo-99 product solution sample after LMC process 2.7.2 1-B Mo-99 product
3	**ALL USED NEEDLES ARE PLACED IN HDPE BOTTLE HALF FILLED WITH CHARCOAL IMMEDIATELY AFTER USE** **NOTE:** In some instances, iodine precipitation step may be performed before acidification. This could lead to more efficient removal of iodine
4	Take sample of RF-1 Mo-99 product into 20mL LSC sample vial labeled RF-1 4.1 Verify valve 2WV-803 to gas collection is OPEN. If not open, open it (image and flow diagram posted at work site) _____ (date) _____ (time) 4.2 Insert gas collection vent needle (VNT-19) into 20mL sample vial labeled RF-1 4.3 Insert a 1-mL syringe with needle into the RF-1 bottle • To remove a ~0.5 mL sample 4.4 After the solution is drawn into the syringe lift the needle out of the solution – DO NOT REMOVE THE SYRINGE + NEEDLE AT THIS TIME 4.5 Draw a small amount of head space from RF-1 • About 0.2-0.3 mL 4.6 Remove the 1 mL syringe + needle and immediately insert it into the weighed sampling vial 4.7 DO NOT PUSH THE PLUNGER ON THE SYRINGE YET 4.8 Add the ~0.5 mL sample to the vial 4.9 With needle still in the sample vial plunge the plunger of the syringe several times to void all volume 4.10 Remove the 1 mL syringe from the vial 4.11 Remove the vent needle from the sampling vial 4.12 Weigh the sample vial + sample: _____ g // _____ (date) _____ (time)
5	Weigh RF-1 Mo99 product bottle after sample was taken: _____ g // _____ (date) _____ (time)
6	Determine volume of 10 M HNO3 required to produce ~1.2 M HNO_3 solution in RF-1 6.1 Mass RF-1 bottle after sample was taken _____ g from step 3.2.8. Step 2.2

Step	Action
	6.2 Mass RF-1 bottle, empty _____ g obtained during Concentration Column procedure, 3.2.6. summary table (Page35) 6.3 Difference _____ g 6.4 0.25 mL of 10 M HNO_3 per 1 mL of product, 3.2.8 step 6.3 x 0.25 = _____ mL of 10 M HNO_3
7	Set 2 minute timer
8	Insert gas collection vacuum needle from KOH bubbler into RF-1 bottle
9	Turn on vacuum
10	Turn off vacuum
11	Add 4.0 mg of NaI carrier (4.0 mL at 1 mg/mL) into the RF-1 bottle // _____ (date) _____ (time) 11.1 Remove syringe after addition 11.2 Contains Mo solution in ~1.2M HNO_3 11.3 Approximate volume = ~50 mL **NOTE:** In some instances iodine precipitation step may be performed before acidification. This could lead to more efficient removal of iodine
12	Shake RF-1 bottle
13	Add 0.5 mL of 10% $AgNO_3$ in 0.1 M HNO_3 // _____ (date) _____ (time) • Remove the syringe after addition
14	Shake RF-1 bottle • White precipitate should form
15	Start timer (2 minutes)
16	At timer end, proceed to next step
17	Set 2 minute timer
18	Add 1 mL of 1.0 M HCl // _____ (date) _____ (time) 18.1 Additional Precipitate should form 18.2 Remove the syringe after addition is complete
19	Start timer
20	At timer end, proceed to next step

Step	Action
21	Remove gas collection vacuum needle (VNT-19) from KOH bubbler from RF-1
22	Mount RF-2 bottle into bottom holder
23	Attach aluminum needle guide type D to RF-2
24	Insert 16 gauge needle with 0.3 µm filter and 16 gauge needle assembly into aluminum needle guide on RF-2 and push through the RF-2 septum
25	Place aluminum needle guide type E (optional) on filter/needle assembly
26	Adjust upper holder to hold RF-1 bottle
27	Insert one-way luer check valve needle to RF-1 top septum
28	Insert RF-1 bottle onto top needle of filter assembly and press down to puncture the septum of RF-1
29	Insert gas collection vacuum needle (VNT-19) from KOH bubbler into RF-2 bottle through aluminum needle guide port (see Figure 4 for reference) 29.1 Turn on vacuum // _____ (date) _____ (time) 29.2 Liquid will be drawn through the filter into RF-2 (example of iodine filtration setup is shown in Figure 8)
30	Wait until all of solution has passed from RF-1 into RF-2

Step	Action
31	Turn off vacuum // _____ (date) _____ (time)  **Figure 8** Example of AgI Filtration Setup
32	Remove RF-1 bottle
33	Add 11 mL of 4 M HNO_3 to RF-1 bottle // _____ (date) _____ (time) 33.1 Remove the syringe after addition is complete 33.2 Rinses the precipitate in RF-1
34	Insert RF-1 bottle onto top needle and press down to puncture the septum of RF-1
35	Gas collection vacuum needle (VNT-19) from KOH bubbler is still connected in RF-2 bottle through aluminum needle guide point 35.1 Turn on vacuum // _____ (date) _____ (time) 35.2 Liquid will be drawn through the filter into RF-2
36	Wait until all of solution has passed from RF-1 through 0.3µm filter into RF-2
37	Turn off vacuum // _____ (date) _____ (time)

Step	Action
38	Remove one-way luer check valve needle from top septum of RF-1 and place into Styrofoam tray in back of antechamber sliding tray
39	Remove RF-1 from the 0.3 µm filter assembly 39.1 Set aside for waste
40	Remove the 0.3 µm filter assembly from RF-2 40.1 Place inside of wide-mouth HDPE bottle and seal 40.2 Set aside for waste
41	Remove gas collection vacuum line (VNT-19)
42	Remove aluminum needle guide from RF-2
43	Add 0.5 mL of Mo carrier (10 mg/mL) to RF-2 // _____ (date) _____ (time) 43.1 Remove the syringe after addition is complete
44	Insert a a gas collection vent needle into RF-2 bottle and slowly add 2.5 % $KMNO_4$ solution dropwise to RF-2 until a deep pink color persists for ~30 seconds // _____ (date) _____ (time) 44.1 This may require up to 25 mL of solution 44.2 Remove the syringe after addition is complete
45	Add 1.5 mL of Rh carrier (8 mg/mL) to RF-2 // _____ (date) _____ (time) 45.1 Remove the syringe after addition is complete
46	Remove RF-2 from holder and shake RF-2
47	Return RF-2 to holder
48	Add 2.0 mL of Ru carrier (5 mg/mL) to RF-2 // _____ (date) _____ (time)
49	Remove RF-2 from holder and shake RF-2
50	Return RF-2 to holder
51	Set timer for 1 minute
52	Add 20 mL of fresh 2% alpha-benzoin-oxime (in 0.4M NaOH) to RF-2 // _____ (date) _____ (time) 52.1 Remove the syringe after addition is complete
53	Remove gas collection vacuum needle (VNT-19) from KOH bubbler from RF-2
54	Remove RF-2 from holder and shake RF-2

Step	Action
55	Return RF-2 to holder and start timer for 1 minute
56	Place RFW in bottom holder of second ring stand
57	Insert aluminum needle guide type D into RFW bottle
58	Insert 16 gauge double-sided needle assembly into RFW
59	Insert aluminum needle guide type C on top of needle assembly (optional)
60	Position holder for 51mm fritted glass column above double-sided needle
61	Place 51-mm fritted glass column into upper needle
62	Place aluminum needle guide type B on top of 51mm fritted glass column (optional)
63	Insert 16 gauge double-sided needle assembly into upper septum port of 51-mm fritted glass column
64	Place aluminum needle guide type C on top of 51mm fritted glass column (optional)
65	Position top bottle holder above 16 gauge double sided needle assembly
66	Insert Gas Collection vacuum needle (VNT-19) from KOH bubbler into RFW bottle through vacuum line port in D type aluminum needle guide
67	Insert one-way luer check valve needle into RF-2 upper septum port
68	Place RF-2 bottle into top holder and press down to puncture the septum of RF-2
69	Turn on vacuum // _____ (date) _____ (time) 69.1 Solution will flow from RF-2 into 51-mm fritted glass column then into RFW

Step	Action
	69.2 Mo-ABO precipitate will collect on a frit of 51 mm fritted glass column (see example of Mo-ABO precipitate filtration in Figure 9  **Figure 9** Example of Mo-ABO Filtration Step
70	Once all solution has passed through the system, remove one-way luer check valve needle from RF-2.
71	Turn off vacuum // _____ (date) _____ (time)
72	Remove RF-2 bottle from holder
73	Add 20 mL of 0.1 M HNO_3 to RF-2 bottle (Rinse #1) // _____ (date) _____ (time) 73.1 Remove the syringe after addition is complete 73.2 Shake the RF-2 bottle 73.3 Rinses precipitate from RF-2 bottle
74	Insert one-way luer check valve needle into RF-2 upper septum port
75	Mount RF-2 bottle back on top of assembly
76	Turn on vacuum // _____ (date) _____ (time) • Solution will flow from RF-2 into 51-mm fritted glass column then into RFW

Step	Action
77	Once all solution has passed through the system, remove one-way luer check valve needle from RF-2
78	Turn off vacuum // _____ (date) _____ (time)
79	Add 20 mL of 0.1 M HNO_3 to RF-2 bottle (Rinse #2) // _____ (date) _____ (time) 79.1 Rinses precipitate from RF-2 bottle 79.2 Remove the syringe after addition is complete
80	Insert one-way luer check valve needle into RF-2 upper septum port
81	Mount RF-2 bottle back on top of assembly
82	Turn on vacuum // _____ (date) _____ (time) • Solution will flow from RF-2 into 51-mm fritted glass column then into RFW
83	Once all solution has passed through the system, remove one-way luer check valve needle from RF-2
84	Turn off vacuum // _____ (date) _____ (time)
85	Add 20 mL of 0.1 M HNO_3 to RF-2 bottle (Rinse #3) // _____ (date) _____ (time) 85.1 Rinses precipitate from RF-2 bottle 85.2 Remove the syringe after addition is complete
86	Insert one-way luer check valve needle into RF-2 upper septum port
87	Mount RF-2 bottle back on top of assembly
88	Turn on vacuum // _____ (date) _____ (time) • Solution will flow from RF-2 into 51-mm fritted glass column then into RFW
89	Once all solution has passed through the system, turn off vacuum
90	Inspect the RF-2 bottle for remnants of the precipitate before removing from the holder 90.1 If any of the alpha-benzoin-oxime precipitate remains in the bottle, repeat Section 3.2.8, Step 75, above, until the RF-2 bottle doesn't contain any removable precipitate **NOTE:** Some portion of Mo-ABO precipitate may stick to the wall of bottle and cannot be easily removed 90.2 If the RF-2 bottle is satisfactorily clean, proceed to next step
91	Remove one-way luer check valve needle from RF-2 and store in Styrofoam 91.1 Remove RF-2 from the top holder and set aside for waste

Step	Action
92	Remove the 16 gauge double-sided needle assembly from the top of the 51-mm fritted glass column and dispose in waste
93	Remove the 51-mm fritted glass column from the holder and place into designated holder • This column contains Mo-99
94	Connect the gas collection vacuum line (VNT-19) into top septum of 51mm fritted glass column
95	Turn on vacuum for a few seconds
96	Turn off vacuum
97	Remove the gas collection vacuum line from the top septum
98	Connect the gas collection vacuum line (VNT-19) into top septum of RFW
99	Add 10 mL of 0.1 M HNO_3 to the 51-mm fritted glass column into the chamber with glass beads (chamber with vacuum) (Rinse #1) // _____ (date) _____ (time) 99.1 Remove the syringe after addition is complete 99.2 Rinse the precipitate and glass beads
100	Shake the 51-mm fritted glass column
101	Return the 51-mm fritted glass column to the holder and allow the wash solution to be drawn down to the RFW bottle
102	Connect one-way luer check valve needle on top of the 51-mm fritted glass column
103	Turn on vacuum // _____ (date) _____ (time) • Solution will flow from 51-mm fritted glass column into RFW
104	Once all solution has passed through the system, turn off vacuum // _____ (date) _____ (time)
105	Remove the 51-mm fritted glass column from the holder
106	Remove the one-way luer check valve needle from top of the 51-mm fritted glass column
107	Connect the gas collection vacuum line (VNT-19) into top septum of 51mm fritted glass column
108	Turn on vacuum for a few seconds
109	Turn off vacuum // _____ (date) _____ (time)
110	Remove the gas collection vacuum line (VNT-19) from the top septum
111	Connect the gas collection vacuum line (VNT-19) into top septum of RFW

Step	Action
112	Add 10 mL of 0.1 M HNO_3 to the 51-mm fritted glass column into the chamber with glass beads (chamber with vacuum) (Rinse #2) // _____ (date) _____ (time) 112.1 Remove the syringe after addition is complete 112.2 Rinses the precipitate and glass beads
113	Shake the 51-mm fritted glass column
114	Return the 51-mm fritted glass column to the holder and allow the wash solution to be drawn down to the RFW bottle
115	Connect one-way luer check valve needle on top of the 51-mm fritted glass column
116	Turn on vacuum // _____ (date) _____ (time) • Solution will flow from 51-mm fritted glass column into RFW
117	Once all solution has passed through the system, turn off vacuum // _____ (date) _____ (time)
118	Remove the gas collection vacuum line from the RFW
119	Remove the 51-mm fritted glass column from the holder
120	Equilibrate both chambers of fritted column by connecting one-way luer check valve needle 120.1 No vacuum in both chambers
121	Flip the bottle upside down and connect the vacuum line above the frit of the 51-mm fritted glass column – this is the chamber not containing glass beads
122	Turn on vacuum
123	Turn off vacuum
124	Remove vacuum line
125	Add 10 mL of 0.1 M HNO_3 to the chamber not containing glass beads – chamber with vacuum (Rinse #3) // _____ (date) _____ (time) 125.1 Remove the syringe after addition is complete
126	Insert one-way luer check valve needle to the same chamber to equilibrate pressure (no vacuum in any of the chambers of the fritted column)
127	Remove one-way luer check valve needle
128	Flip the bottle, so the 10 mL of 0.1 M HNO_3 is under the frit (normal orientation)
129	Connect vacuum line above the frit (chamber with glass beads)
130	Turn on vacuum // _____ (date) _____ (time)

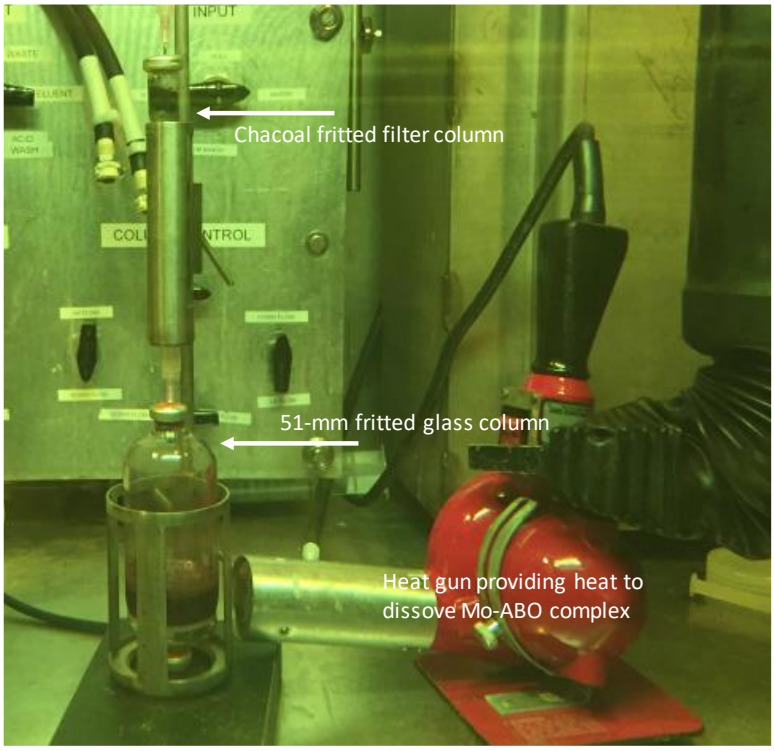
Step	Action
131	Turn off vacuum // _____ (date) _____ (time)
132	Remove vacuum line
133	Flip the column again, so the 10mL 0.1M HNO_3 solution passes through the frit into the chamber containing glass beads and Mo-ABO precipitate • This rinses the frit that may contain small particles of Mo-ABO precipitate
134	Shake the 51-mm fritted glass column
135	Return the 51-mm fritted glass column to the holder and allow the wash solution to be drawn down to the RFW bottle
136	Connect one-way luer check valve needle on top of the 51-mm fritted glass column
137	Turn on vacuum // _____ (date) _____ (time) • Solution will flow from 51-mm fritted glass column into RFW
138	Once all solution has passed through the system, turn off vacuum // _____ (date) _____ (time)
139	Remove the 51-mm fritted glass column from the holder
140	Equilibrate both chambers of fritted column by connecting one-way luer check valve needle (no vacuum in both chambers)
141	Flip the bottle upside down and connect the vacuum line above the frit of the 51-mm fritted glass column – this is the chamber not containing glass beads
142	Turn on vacuum
143	Turn off vacuum // _____ (date) _____ (time)
144	Remove the vacuum line
145	Add 10 mL of 0.1 M HNO_3 to the chamber not containing glass beads – chamber with vacuum (Rinse #4) 145.1 Remove the syringe after addition is complete
146	Insert one-way luer check valve needle to the same chamber to equilibrate pressure (no vacuum in any of the chambers of the fritted column)
147	Remove one-way luer check valve needle
148	Flip the bottle, so the 10 mL of 0.1 M HNO_3 is under the frit (normal orientation)
149	Connect vacuum line above the frit (chamber with glass beads)
150	Turn on vacuum

Step	Action
151	Turn off vacuum // _____ (date) _____ (time)
152	Remove the vacuum line
153	Flip the column again, so the 10mL 0.1M HNO_3 solution passes through the frit into the chamber containing glass beads and Mo-ABO precipitate • This rinses the frit that may contain small particles of Mo-ABO precipitate
154	Shake the 51-mm fritted glass column
155	Return the 51-mm fritted glass column to the holder and allow the wash solution to be drawn down to the RFW bottle
156	Connect one-way luer check valve needle on top of the 51-mm fritted glass column
157	Turn on vacuum // _____ (date) _____ (time) • Solution will flow from 51-mm fritted glass column into RFW
158	Once all solution has passed through the system, turn off vacuum // _____ (date) _____ (time)
159	Remove the 51-mm fritted glass column from the holder
160	Equilibrate both chambers of fritted column by connecting one-way luer check valve needle (no vacuum in both chambers)
161	Flip the bottle upside down and connect the vacuum line above the frit of the 51-mm fritted glass column – this is the chamber not containing glass beads
162	Turn on vacuum
163	Turn off vacuum // _____ (date) _____ (time)
164	Remove vacuum line
165	Add 10 mL of 0.1 M HNO_3 to the chamber not containing glass beads – chamber with vacuum (Rinse #5) // _____ (date) _____ (time) 165.1 Remove the syringe after addition is complete
166	Insert one-way check luer valve needle to the same chamber to equilibrate pressure (no vacuum in any of the chambers of the fritted column)
167	Remove one-way luer check valve needle
168	Flip the bottle, so the 10 mL of 0.1 M HNO_3 is under the frit (normal orientation)
169	Connect vacuum line (VNT-19) above the frit (chamber with glass beads)
170	Turn on vacuum

Step	Action
171	Turn off vacuum // _____ (date) _____ (time)
172	Remove the vacuum line
173	Flip the column again, so the 10mL 0.1M HNO_3 solution passes through the frit into the chamber containing glass beads and Mo-ABO precipitate • This rinses the frit that may contain small particles of Mo-ABO precipitate
174	Shake the 51-mm fritted glass column
175	Return the 51-mm fritted glass column to the holder and allow the wash solution to be drawn down to the RFW bottle
176	Connect one-way luer check valve needle on top of the 51-mm fritted glass column
177	Turn on vacuum // _____ (date) _____ (time) • Solution will flow from 51-mm fritted glass column into RFW
178	Once all solution has passed through the system, turn off vacuum // _____ (date) _____ (time)
179	Verify that the acid wash appears clear **NOTE:** Over-time, some precipitate may form in the RFW bottle. This is due to precipitation of excess ABO.
180	Remove the 51-mm fritted glass column from the holder
181	Save the 51-mm fritted glass column with precipitate • This contains Mo-99
182	Remove the double-sided needle from RFW and dispose the needle assembly
183	Remove the aluminum needle guide from RFW
184	Weight the RFW bottle 186.1 Weigh the RFW bottle: _____ g // _____ (date) _____ (time)
185	Place the RFW into a bottle holder

Step	Action
186	Sample the RFW bottle solution 186.1 Insert a vent needle into 20mL sample vial labeled RFW sample 186.2 Insert a 10-mL syringe into the RFW bottle 188.2.1 To remove a ~5 mL sample 186.3 After the solution is drawn into the syringe lift the needle out of the solution – DO NOT REMOVE THE SYRINGE + NEEDLE AT THIS TIME 186.4 Draw a small amount of head space from RFW 188.4.1 About 0.5 mL 186.5 Remove the 10 mL syringe + needle and immediately insert it into the weighed sampling vial 186.6 DO NOT PUSH THE PLUNGER ON THE SYRINGE YET 186.7 Add the ~5 mL sample to the vial 186.8 With needle still in the sample vial plunge the plunger of the syringe several times to void all volume 186.9 Remove the 10 mL syringe from the vial 186.10 Remove the vent needle from the sampling vial 186.11 Weigh the sample vial + sample: _____ g // _____ (date) _____ (time)
187	Connect vacuum line (VNT-19) into RFW bottle
188	Turn on vacuum for a few seconds
189	Turn off vacuum // _____ (date) _____ (time)
190	Remove vacuum line (VNT-19) from RFW bottle
191	Store the RFW bottle under vacuum
192	Insert a new 1-A bottle into the bottom holder
193	Place aluminum needle guide type D on top of 1-A bottle
194	Insert a 18 gauge double-sided needle assembly with luer valve into the aluminum needle guide and pierce the top septum port of the 1-A bottle 194.1 Leave the valve closed
195	Insert the 51-mm fritted glass column with precipitate into a holder for dissolution

Step	Action
196	Inject 10 mL of 0.4 M NaOH with ~1% H_2O_2 into the 51-mm fritted glass column from underside of frit// _____ (date) _____ (time) 196.1 Remove the syringe after addition is complete
197	Set an aluminum needle guide type B (optional) on top of the 51-mm fritted glass column
198	Insert a 18 gauge double-sided needle assembly with luer valve into the top septum of the 51-mm fritted glass column 198.1 Leave the valve closed
199	Position short column holder above the needle assembly
200	Place the charcoal column into the holder and pierce the septum by pressing down on charcoal column
201	Insert the Gas Collection vent needle (VNT-13) into a charcoal filter
202	Open the valve on double-sided needle assembly above 51mm fritted column
203	☐ Verify the valve on double-sided needle assembly above 51mm fritted column is OPEN
204	Heat the 51-mm fritted glass column that contains the solution with forced hot air until the solution begins to boil // _____ (date) _____ (time) • See Figure 10 as an example of Mo-ABO dissolution setup.
205	Remove heat from the 51-mm fritted glass column by turning off heat gun and moving away into a safe location. 205.1 Record date and time: // _____ (date) _____ (time)

Step	Action
206	Start timer and wait 5 minutes  **Figure 10** Example of Setup for Dissolution of Mo-ABO Precipitate
207	At timer end close the luer lock valve on double sided needle assembly.
208	Slide the charcoal vent higher, so it disconnects from the double sided needle
209	Remove the 51-mm fritted glass column from the dissolution holder and place onto the top needle of the double-sided needle assembly inserted in the 1-A bottle
210	Insert the gas collection vacuum needle (VNT-19) from KOH bubbler into the 1-A bottle // _____ (date) _____ (time)
211	Connect one-way luer check valve needle on top of the 51-mm fritted glass column
212	Turn on vacuum // _____ (date) _____ (time) 214.1 The solution will be drawn from 51mm fritted glass column into the 1-A bottle
213	Once all solution has passed through the system, turn off vacuum // _____ (date) _____ (time)
214	When all solution has been transferred close the valve on double-sided needle assembly above 1-A bottle
215	Verify the valve on double-sided needle assembly above 51mm fritted column is CLOSED

Step	Action
216	Remove the one-way luer check valve needle from the 51-mm fritted glass column
217	Remove the 51-mm fritted glass column from the double needle union
218	Insert the 51-mm fritted glass column with precipitate into a holder for dissolution
219	Inject 10 mL of 0.2 M NaOH with ~1% H_2O_2 into the 51-mm fritted glass column from underside of frit// _____ (date) _____ (time) 219.1 Remove the syringe after addition is complete
220	Set an aluminum needle guide type B (optional) on top of the 51-mm fritted glass column
221	Insert a 18 gauge double-sided needle assembly with luer valve into the top septum of the 51-mm fritted glass column 221.1 Leave the valve closed
222	Position short column holder above the needle assembly
223	Place the charcoal column into the holder and pierce the septum by pressing down on charcoal column
224	Insert the Gas Collection vent needle (VNT-19) into a charcoal filter
225	Open the valve on double-sided needle assembly above 51mm fritted column
226	Verify the valve on double-sided needle assembly above 51mm fritted column is OPEN
227	Heat the 51-mm fritted glass column that contains the solution with forced hot air until the solution begins to boil // _____ (date) _____ (time)
228	Remove heat from the 51-mm fritted glass column by turning off heat gun and moving away into a safe location // _____ (date) _____ (time)
229	Start timer and wait 5 minutes
230	At timer end close the luer lock valve on double sided needle assembly.
231	Slide the charcoal vent higher, so it disconnects from the double sided needle
232	Remove the 51-mm fritted glass column from the dissolution holder and place onto the top needle of the double-sided needle assembly inserted in the 1-A bottle
233	Insert the gas collection vacuum needle (VNT-19) from KOH bubbler into the 1-A bottle
234	Connect one-way luer check valve needle on top of the 51-mm fritted glass column
235	Turn on vacuum // _____ (date) _____ (time) 235.1 The solution will be drawn from 51mm fritted glass column into the 1-A bottle

Step	Action
236	Once all solution has passed through the system, turn off vacuum // _____ (date) _____ (time)
237	When all solution has been transferred close the valve on double-sided needle assembly above 1-A bottle
238	Verify the valve on double-sided needle assembly above 51mm fritted column is CLOSED
239	Remove the one-way luer check valve needle from the 51-mm fritted glass column
240	Remove the 51-mm fritted glass column from the double-sided needle assembly
241	Inject 10 mL of 0.2 M NaOH into the 51-mm fritted glass column from underside of frit // _____ (date) _____ (time) 241.1 Rinsing the 51 mm fritted glass column 241.2 Remove the syringe after addition is complete
242	Place the 51mm fritted glass column on top of double-sided needle assembly above 1-A bottle
243	Connect one-way luer check valve needle on top of the 51-mm fritted glass column
244	Open the valve on double-sided needle assembly above 1A bottle
245	Verify the valve on double-sided needle assembly above 1-A bottle is OPEN
246	Turn on vacuum // _____ (date) _____ (time) 246.1 The rinse solution will be pulled into the 1-Abottle
247	Once all the solution has passed through the system, turn off vacuum // _____ (date) _____ (time)
248	Remove the 51-mm fritted glass column from the double needle union assembly
249	Remove the double-sided needle assembly from the 1-A bottle
250	Pull vacuum into 1-A bottle
251	Turn off vacuum // _____ (date) _____ (time)
252	Remove aluminum needle guide from 1-A bottle
253	Set timer for 5 minutes
254	Inject 4.0 mg NaI solution into 1-A bottle (4.0 mL of 1 mg/mL NaI) // _____ (date) _____ (time) 254.1 Shake bottle

Step	Action
255	Inject 0.5 mL 10 10 % $AgNO_3$ in 0.1 M HNO3 solution // _____ (date) _____ (time) 255.1 Shake bottle 255.2 Precipitate will form
256	Start timer and wait 5 minutes
257	Place 1-B bottle at the bottom of stand
258	Place aluminum needle guide type D on top of 1-B bottle
259	Insert the 18 gauge double-sided needle assembly into aluminum needle guide on top of 1-B bottle
260	Place aluminum needle guide type F on top of double sided needle assembly
261	Position long column holder above double-sided needle assembly
262	Place 3-phase column (AgC/ZrO/AC) column on top of assembly, push down to pierce all septa
263	Place aluminum needle guide type C on top of 3-phase column
264	Insert the 16 gauge double-sided needle assembly into needle guide
265	Place aluminum needle guide type E on top of double sided needle assembly
266	Position top bottle holder above needle
267	Place one-way luer check valve needle in top septum of 1-A bottle
268	Place 1-A bottle on top of assembly and push down to pierce all septa
269	Insert the Gas Collection vacuum needle (VNT-19) from KOH bubbler into 1-B bottle
270	Allow gravity flow to move solution through 3-phase column and into 1-B bottle 270.1 Toggle vacuum on/off as needed to initiate flow
271	After all solution has left 1-A bottle, remove 1-A bottle from assembly
272	Inject 10 mL of 0.2 M NaOH into 1-A bottle // _____ (date) _____ (time)
273	Shake and replace on top of assembly
274	Allow gravity flow to move solution through 3-phase column and into 1-B bottle 274.1 Toggle vacuum on/off as needed to initiate flow
275	After gravity flow completed use vacuum to remove all solution from 3-phase column and collect solution in 1-B

Step	Action
276	Disassemble the system and dispose 1-A bottle and 3-phase column for waste 276.1 Keep the 1-B bottle containing purified Mo-99 product
277	Weigh the 1-B bottle: _____ g // _____ (date) _____ (time)
278	Sample the 1-B bottle solution 278.1 Insert the one-way luer check valve needle into the 1-B bottle 278.2 Insert a 1-mL syringe into the 1-B bottle 278.2.1 To remove a ~0.5 mL sample 278.3 After the solution is drawn into the syringe lift the needle out of the solution – **NOTE:** DO NOT REMOVE THE SYRINGE + NEEDLE AT THIS TIME 278.4 Draw a small amount of head space from 1-B bottle 278.4.1 About 0.2-0.3 mL 278.5 Remove the 1 mL syringe + needle and immediately insert it into the weighed sampling vial 278.6 DO NOT PUSH THE PLUNGER ON THE SYRINGE YET 278.7 Insert the vent needle into sampling vial 278.7.1 Ensure the tip of the needle is in the top 25% of the vial 278.8 Add the ~0.5 mL sample to the vial 278.9 With needle still in the sample vial plunge the plunger of the 3 mL syringe several times to void all volum 278.10 Remove the 1 mL syringe from the vial 278.11 Remove the vent needle from the sampling vial 278.12 Weigh the sample vial + sample: 278.12.1 _____ g // _____ (date) _____ (time)

Table 2. LMC Summary table with bottle masses

Bottle	Empty mass, g	Full mass, g	Mass of sample, g
RF-1	_____,g (3.2.4 step 2.6)	_____,g (3.2.8 step 5)	
RFW	_____,g (3.2.8 step 2.3)	_____,g (3.2.8 step 184)	
1-B	_____,g (3.2.8 step 2.4)	_____,g (3.2.8 step 277)	

Table 3. LMC Sample Summary Table

Sample	Empty mass, g	Full mass, g	Mass of sample, g
RF-1	_____,g (3.2.8 step 2.5)	_____,g (3.2.8 step 4.12)	
RFW	_____,g (3.2.8 step 2.6)	_____,g (3.2.8 step 186.11)	
1-B	_____,g (3.2.8 step 2.7)	_____,g (3.2.8 step 278.12)	

END OF SEGMENT

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3.2.9 Receiving WASH/Rinse Solutions from Recovery Glove Box

Follow the steps below to receive wash/rinse solutions from Recovery Glove Box.

Step	Action
1	Documentation for wash/rinse solutions from Recovery Glove Box \\ **Recovery Member Name: _____**PRINT **Date: _____ Time: _____** **D024 Hot Cell Ops Member Name: _____**PRINT **Date: _____ Time: _____** ### SYSTEMS INTERFACE STEP ###
2	Appropriate team member **INITIALIZES** every step in this section
3	Inside D024 Hot Cell 3.1 ☐ Recovery ☐ HotCellOps Verify 3-L/5-neck flask in place 3.2 ☐ Recovery ☐ HotCellOps Verify pH probe inserted and not broken or sealed with 24/40 stopper 3.3 ☐ Recovery ☐ HotCellOps Verify plastic feed line attached to center port/neck 3.4 ☐ Recovery ☐ HotCellOps Verify septum is secured 3.5 ☐ Recovery ☐ HotCellOps Verify multi-port neck adapters inserted and attached to other two ports/necks 3.6 ☐ Recovery ☐ HotCellOps Verify adequate free volume in 3-L/5-neck flask 3.6.1 3-L/5-neck flask will receive ~1000 mL of washout solution 3.7 ☐ Recovery ☐ HotCellOps Verify balance is ON 3.8 Record balance reading: _____ grams 3.9 ☐ Recovery ☐ HotCellOps Verify 2WV-701 liquid valve OPEN (image and flow diagram posted at work site) 3.9.1 Handle parallel to the long axis of valve body 3.10 ☐ Recovery ☐ HotCellOps Verify plastic line attached between 2WV-701 and center neck of flask (image and flow diagram posted at work site)

Step	Action
	3.11 ☐ Recovery ☐ HotCellOps Verify 2WV-801 vent valve CLOSED (image and flow diagram posted at work site) 3.11.1 Handle perpendicular to the long axis of valve body 3.12 Verify liquid trap to Gas Collection System is: 3.12.1 ☐ Recovery ☐ HotCellOps Attached 3.12.2 ☐ Recovery ☐ HotCellOps Good condition (not cracked/broken), lines attached 3.12.3 ☐ Recovery ☐ HotCellOps Verify empty 3.13 Proceed with instructions from Recovery Glove Box to transfer solution.
4	The wash solution must be removed prior to performing the next AMORE process.

4 Records Created by Work Process

The records listed below must be retained as indicated.

Description of Record (include form number if applicable)	Active Records Custodian	Active Records Retention	Indexing Method, Storage Medium	Federal Retention Requirements*
Completed procedure	Facility Manager	3 years	Index by job date and name, store on paper or electronically	Destroy 75 years after the date of the permit (DOE ADM 18.37)

*If records are maintained in a business information system that is not currently programmed to purge digital records based on age, the records may be retained in that system past the indicated destruction date.

5 Related Documents

This procedure implements requirements established by the following safety basis documents:

- LEAF-SAD-100, Linac Safety Assessment Document
- LEAF-ASE-100, Linac Accelerator Safety Envelope

This procedure implements requirements established by the following Argonne policies and procedures:

- LMS-PROC-188, *Accelerator Safety*
- EGS-PP-100, *Configuration Management Program Plan for Accelerators*

6 Definitions

None

7 About this Procedure

Issuing organization:	Low Energy Accelerator Facility
Procedure owner:	D. Rotsch
Point of contact:	D. Rotsch
Review cycle (months):	36
Date last revised:	03.25.2019
Date last reviewed:	03.25.2019

8 Summary of Changes in This Version

Initial release

LEAF-PROC-011 CHANGES LOG DATE: 8/28/2019

3.2.3. step 1. Action 1.1.a – added

i. Worker 1:_____

ii. Worker 2 (optional):_____

iii. HPT:_____

3.2.3. step 5 – removed

3.2.3. step 8. Action 5.1 – added “Water reservoir fill”

3.2.3. step 8. Action 5.2 – added “HNO_3 reservoir fill”

3.2.3. step 8. Action 5.3 – added “1M NaOH reservoir fill”

3.2.3. step 8. Action 5.5.4 – added “ And Date”

3.2.3. step 8. Action 5.5.5 – removed:

- Fisher Scientific p/n #
- Becton Dickinson p/n BD #

3.2.3. step 8. Action 5.7.1 – changed “8 in.” to “6 in.”

3.2.3. step 8. Action 5.7.4 – changed “8 in.” to “8 in. or longer”

3.2.4 step 1. Action 1.1.1 – added

1.2.3.1. Name:_____

1.2.3.2. Date and time:_____

3.2.4 step 1. Action 1.1.2 – added

1.1.1.1. Name:_____

1.1.1.2. Date and time:_____

1.1.1.3. Name:_____

1.1.1.4. Date and time:_____

3.2.4 step 2. – added “(Use this data in the summary table at the end of 3.2.6”

3.2.4 step 2. Action 2.6 – removed “1-A bottle”

3.2.4 step 3. – added step:

3.6 End of segment

Date and Time:_____

3.2.4 step 3. Removed:

3.3 Insert probe into pH 2 standard

3.4 pH value = _____ time: _____ date: _____

3.2.4 step 4. Action 4.10 – added “or manual valve SWV-801”

3.2.5. step 1 – added

1.1 Transfer began: _____(date) _____(time)

3.2.5. step 3 – added

3.1 Transfer end: _____(date) _____(time)

3.2.6 step 2 action 2.1 – added

Name:_____

3.2.6 step 2 action 2.2 – added

Name:_____

3.2.6 step 5 Action 5.3 – changed section to

5.3 Adjust pH of recovery column primary strip product to pH 2 with appropriate solution:

8 M HNO_3:

Obtain mass of full syringe: _____g

10 M NaOH:

Obtain mass of full syringe: _____g

3.2.6 step 5 Action 5.4 – added (If pH <1 observed, add 5 mL 10 M NaOH and observe change in pH)

3.2.6 step 5 Action 5.4.3 – adjusted text "Continue until pH <10 (or pH > 2 if using NaOH) observed and then proceed if not add another 5 mL of 8 M HNO_3 (or 10 M NaOH)"

3.2.6 step 5 Action 5.5 – adjusted text "Slowly add (dropwise) 8 M HNO_3 (or 10 M NaOH) until pH 2 is reached"

3.2.6 step 5 – added Action – "Obtain mass of spent syringe: _____g"

3.2.6 step 6 action 6.2 – added "(Use this data in the summary table at the end of 3.2.6"

3.2.6 step 6 action 6.14 – added "(Use this data in the summary table at the end of 3.2.6"

Upper "Pump Control" (pump outlet) 3-way valve (3WV-703) to "Column", VALVE 3WV-704 changed to 3WV-703 throughout procedure

Lower "Pump Control" (pump inlet) 3-way valve (3WV-704) to feed source 4-way valve "Column", VALVE 3WV-705 changed to 3WV-704 throughout procedure

3.2.6 Step 12 Action 12.1 – changed text to "Verify Water needle from "Output" 7-way valve (7WV-709) inserted into Waste #2 60 mL vial"

3.2.6 Step 12 Action 12.2 – changed text to "Verify Mo-99 product bottle, RF-1 Mo99 product (Cintichem style bottle, see Figure 3) is prepared and present."

3.2.6 Step 12 Action 12.2 – changed text to "Verify "Input" 5-way valve to Water"

3.2.6 Step 12 Action 12.11 – changed text to "Verify "Gas Collection" vent needle from VNT-13 inserted into Waste #2 60 mL vial"

3.2.6 Step 12 Action 12.11 – changed time from 5 minutes to 6 minutes (360 sec)

3.2.6 Step 13 Action 13.1 – changed text to "Insert Mo-99 needle from "Output" 7-way valve (7WV-709) Waste #1 valve port into Waste #2 60 mL vial"

3.2.6 Step 13 Action 13.1 – changed vial from Waste #1 to Waste #2

3.2.6 Step 15 – added "Use this data in the summary table at the end of 3.2.6"

Added 3.2.6 step 19 – Turn off pump controller

Added 3.2.6 step 20 – Place gas collection line into RF1

Added 3.2.6 step 21 – Sample vials

21.1. Shake all vials with manipulators and obtain mass of each vessel

21.1.1. Waste #1: _____ g (60 mL vial)

21.1.2. Waste #2: _____ g (60 mL vial)

21.1.3. Water Wash: _____ g (60 mL vial)

21.1.4. Nitric Acid Wash: _____ g (60 mL vial)

21.1.5. Mo-99 Product (Cintichem Vessel): _____ g (60 mL vial)

Use this data in the summary table at the end of 3.2.6

21.2. Use 1 mL syringes with 6" needles to sample vials. Pull the plunger to ~50% of the syringe shaft. Remove needle from solution while keeping needle within vessel being samples. Pull plunger to ~80% of the syringe shaft. Remove needle from vessel and inject sample into appropriate sampling vessel. Record mass of sample

21.2.1 Waste #2 vessel with sample: _____ g (20 mL vial)

21.2.2 Water wash vessel with sample: _____ g (20 mL vial)

21.2.3 Nitric acid wash vessel with sample: _____ g (20 mL vial)

Use this data in the summary table at the end of 3.2.6

Added 3.2.6 step 22 – Notify LMC team of completion

Date:_____ Time:_____

Added Concentration Column Summary Tables

Concentration Column Summary Table

	Mass of Empty Vessel	**Mass of Vessel with Solution**	**Mass of Solution**
Feed (3L vessel)			
Waste #1			
Waste #2			
Water Wash			
Nitric Acid Wash			
Mo-99 Product (Cintichem bottle)			

Concentration Column Sample Summary Table

	Mass of Empty Sampling vessel	**Mass of Sampling Vessel with Sample**	**Mass of sample**

Feed Initial			
Waste #2			
Water Wash			
Nitric Acid Wash			

3.2.7. Step 1.3 – "OPTIONAL STEP" was added

3.2.7. Step 1.4.1–2.4.6 - check box added

3.2.7 steps 2.5-2.12 renumbered

3.2.7. Steps 2.10 –2.11 are now 2.7-2.9 check symbol added

3.2.7. Steps 3.2.2 Work under appropriate currently approved RWP (replaced RWP #)

Table 1 – added line on top of the table to include syringe with 10M HNO3

3.2.8 step 1.1 added NAME: Primary manipulator operator: NAME: _____

3.2.8 step 1.2 added NAME: Recorder/Secondary manipulator operator: NAME:_____

After step 3.2.8 step 1.3 added: START OF LMC OPERATIONS: DATE:_____ TIME: _____

3.2.8 steps 2.2-2.7 added date and time fields

3.2.8 step 4.1 added date and time field

3.2.8 step 4.5 corrected RFW to RF-1

3.2.8 step 6.2 added reference to empty RF-1 bottle weight

3.2.8 step 6.4 corrected step reference to 3.2.8 step 6.3

3.2.8 step 18 added date and time fields

3.2.8 step 24 changed the description of step to: Insert 16 gauge needle with 0.3 µm filter and 16 gauge needle assembly into aluminum needle guide on RF-2 and push through the RF-2 septum

3.2.8 step 29 added date and time fields

3.2.8 step 31 added date and time fields

3.2.8 step 35.1 and 37 added date and time fields

3.2.8 step 44 added: Insert a gas collection vent needle into RF-2 bottle and slowly…

3.2.8 steps 45-46 removed, old step 47 becomes step 45

3.2.8 step 71 becomes 69 added date and time fields

3.2.8 step 73 becomes 71 added date and time fields

Same edits in old steps added date and time fields – steps: 75, 78, 80, 81, 82, 84, 86, 87, 90, 101, 105, 106, 111, 113, 118, 119, 127, 132, 133, 139, 140, 145, 153, 159, 160, 165, 167, 173, 179, 180, 191, 198, 207, 214, 215, 237, 238, 248, 249, 253, 256, 257, 274

Table 2 added with summary masses of RF-1, RFW and 1-B bottles

Table 3 added with summary masses of RF-1, RFW and 1-B samples

UPDATES as of 2/20/2020

Section 3.2.6 step 9.2: Changed "Phase II Effluent" to "Phase I Effluent" see text below

Section 3.2.6

9.2 Turn "Output" 7-way valve (7WV-709) to 6-way eluent bottle directing valve "Phase I Effluent"

Section 3.2.6 added the following:

9.2.1. Ensure ball valve of solution line connected to effluent bottle is OPEN

9.2.2. Ensure that the black luer-lock valve connected to the effluent bottle is OPEN

9.2.3. Ensure that black luer-lock valve for sampling the effluent bottle is CLOSED

9.2.4. Ensure that gas collection ball valve connected to the effluent bottle is OPEN

9.2.5. Ensure that the black luer-lock valve connected to the gas collection needle is CLOSED or is inserted in to one of the 60 mL septum collection vials

21.3. Sample Effluent Bottle

21.3.1. Ensure effluent bottle is connected to gas collection system

21.3.1.1. Ensure VQD-014 is connected

21.3.1.2. Ensure gas collection needle for small bottles is in a septum bottle or closed

21.3.1.3. Ensure effluent bottle black luer lock valve is OPEN

21.3.1.4. Ensure effluent bottle ball valve is OPEN

21.3.2. Connect effluent bottle solution line to syringe

21.3.2.1. Ensure that 7WV-709 "OUTPUT" 7-way valve is directed towards any other output than "Phase I Effluent" – "OUTPUT" valve is NOT connected to effluent bottle being sampled.

21.3.2.2. Ensure effluent bottle solution ball valve is OPEN

21.3.2.3. Connect syringe (suggested 20-mL syringe with valve and plunger fully extended) to black luer lock at "t" connection to effluent bottle.

21.3.3. Mix and collect sample

21.3.3.1. Open black luer lock connecters

21.3.3.2. Depress syringe plunger to force air into the effluent bottle to mix system (LEAVE ~5 ML OF AIR WITHIN THE SYRINGE)

21.3.3.3. Pull syringe plunger up and down 3X to further mix the system (LEAVE ~5 ML OF AIR WITHIN THE SYRINGE)

21.3.3.4. Pull plunger to take sample (suggested ~1-3 mL)

21.3.3.5. Invert syringe so that solution is on plunger side and remaining

21.3.3.6. Depress syringe until bubbles are noticed in effluent bottle – OR – until sample is near top of syringe barrel (the point of this step is to void the lines and ensure no solution is in disconnect points)

21.3.3.7. CLOSE all solution black luer lock valves (2x)

21.3.3.8. Disconnect the syringe from the effluent bottle system while maintaining ONE black luer lock valve to the syringe.

21.3.3.9. Affix a needle to the black luer lock valve still connected to the syringe.

21.3.3.10. Inject solution into appropriately marked 20 mL septum vessel and record mass.

21.2.3.3.10.1 Effluent sample: _____ g (20 mL vial)

Updated Table to include effluent sample:

Concentration Column Sample Summary Table

	Mass of Empty Sampling vessel	**Mass of Sampling Vessel with Sample**	**Mass of sample**
Feed Initial			
Waste #2			
Water Wash			
Nitric Acid Wash			
Effluent			

Section 3.2.3.8 Step 8.2.4 updated to include mass of each 20 mL vessel

4. Five (5x) 20 mL sampling septa vials

- Each vial is labeled, dated and placed in D-024 hot cell
- Verify vials labeled
  - ☐ Mo-99 product ☐ And date Mass:_____
  - ☐ Acid wash ☐ And date Mass:_____
  - ☐ Water wash ☐ And date Mass:_____
  - ☐ Feed initial ☐ And date Mass:_____
  - ☐ Waste #2 ☐ And date Mass:_____

Updates on 11/2020 to Rev. 3

Added comment in section 3.2.3 step 14 that the lines must be replaced at least every 2 years.

Section 3.2.6 Step 23-26 were added to include a water rinse of the lines after the AMORE experiment has been completed.

Schematic of the concentration column system added to PROC.

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