

# **Full-Scale Testing of the Ambient Pressure, Acid-Dissolution Front-End Process for the Current <sup>99</sup>Mo Recovery Processes**

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**Chemical Science and Engineering Division**

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# **Full-Scale Testing of the Ambient Pressure, Acid-Dissolution Front-End Process for the Current <sup>99</sup>Mo Recovery Processes**

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by

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# **FULL-SCALE TESTING OF THE AMBIENT PRESSURE, ACID-DISSOLUTION FRONTEND PROCESS FOR THE CURRENT <sup>99</sup>MO RECOVERY PROCESSES**

## **1 INTRODUCTION**

The Global Threat Reduction Initiative (GTRI) Conversion Program is actively developing technologies for converting civilian facilities that use high enriched uranium (HEU) to low enriched uranium (LEU) fuels and targets. The conversion of conventional HEU targets to LEU for the production of <sup>99</sup>Mo production requires approximately five times the uranium in a target to maintain the <sup>99</sup>Mo yield on a per-target basis. Under GTRI, Argonne National Laboratory (Argonne) is developing two frontend options for current <sup>99</sup>Mo production processes to allow the use of LEU-foil targets. In both processes, the aim is to produce a frontend product that is compatible with current <sup>99</sup>Mo purification operations and will provide the same or a higher yield of <sup>99</sup>Mo for the same number of irradiated targets.

The two frontend processes under development as part of this project are (1) the dissolution of irradiated LEU foil (up to 250 g in a single batch) and nickel fission recoil barrier in nitric acid at ambient pressure; and (2) the electrochemical dissolution of LEU foil in series of steps that produces an alkaline (basic) solution feed for <sup>99</sup>Mo purification. This report describes results from performance tests and design optimization of the ambient pressure, nitric-acid-dissolver system. The design, fabrication, and performance test planning for this system are described in more detail in previous reports (Jerden et al. 2011a,b, 2012). Full-scale demonstrations of both of the frontend processes using irradiated uranium foils are planned to be performed at Oak Ridge National Laboratory this fiscal year.

## 2 AMBIENT PRESSURE, NITRIC-ACID-DISSOLVER SYSTEM FOR LEU FOILS

An ambient pressure, nitric-acid-dissolver system designed for the dissolution of up to 250 g of irradiated LEU foil and associated fission recoil barrier metal (e.g., Ni) has been tested using non-irradiated uranium foils. The dissolver system components were tested at full scale so that the design could be optimized in preparation for a full-scale demonstration. The key design criteria of the dissolver system that have been tested experimentally are summarized below:

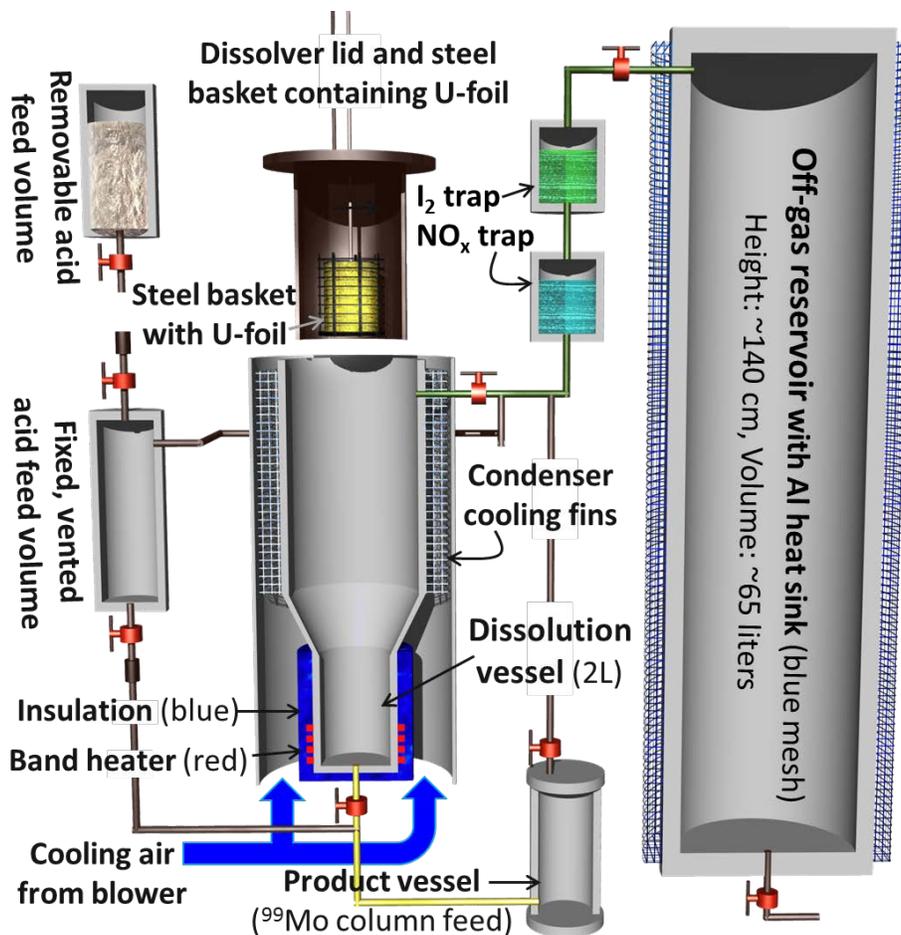
- All water vapor, reaction products, and fission gases must be contained within the dissolver system at a maximum temperature of 125°C and 2 atm of pressure (absolute) under both normal and off-normal (loss of cooling during reaction) conditions.
- The acid-feed system must be designed so that the thermally hot LEU foil (hot from decay heat) can be immersed in nitric acid without losing solution due to instantaneous boiling.
- All dissolver system components must be designed for remote operation in a hot cell facility.
- Gas-trap components must be designed to trap/neutralize all nitrogen oxide and acid gases (NO, NO<sub>2</sub>, HNO<sub>2</sub>, HNO<sub>3</sub>) and to trap iodine gas for possible extraction of economically important iodine isotopes (noble fission gases will be passively contained).

The dissolver system design basis requirements imposed by the thermal and chemical properties of the LEU-foil dissolution process and the dissolver system design and plan for performance testing are discussed in Jerden et al. (2011b). A schematic flow diagram showing the major components of the LEU-nitric acid frontend process is shown in Figure 1.

In this report, we discuss results from dissolution experiments performed with non-irradiated uranium foils and describe how the system is being optimized for the full-scale hot-cell demonstration planned for fiscal year 2013 (FY2013) (demonstration to be performed at Oak Ridge National Laboratory). Before discussing the test results, the design basis requirements for the dissolver system are summarized (for a more detailed discussion of design criteria see Jerden et al. 2011b).

### 2.1 DISSOLUTION REACTION: REACTION HEAT, DECAY HEAT, AND OFF-GAS VOLUME

The volume and concentration of nitric acid for a given experiment will depend on the mass of the metal being dissolved as well as the desired final acid concentration of the “product” solution (i.e., the solution produced by dissolution experiment). Controlling the final acid concentration is important for optimizing the <sup>99</sup>Mo extraction step that comes after dissolution.



**FIGURE 1 Schematic Cross-Section Showing Components of the LEU Nitric-Acid-Dissolver System Being Designed at Argonne National Laboratory**

The volumes and concentrations of acid as well as the amount of nitrogen oxide gas ( $\text{NO}_x$ :  $\text{NO}$ ,  $\text{NO}_2$ ,  $\text{N}_2\text{O}_4$ ) that will be produced are determined by the following general reactions:



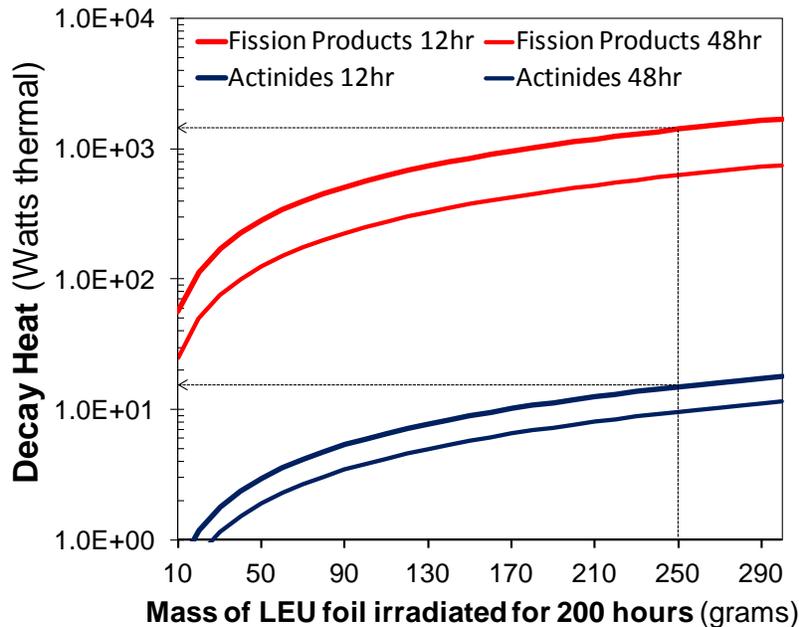
In the presence of oxygen, the  $\text{NO}(\text{g})$  produced in these dissolution reactions is rapidly converted to  $\text{NO}_2(\text{g})$ :



When water vapor and oxygen are present,  $\text{NO}_2$  is readily converted to both nitrous and nitric acid vapors [ $\text{HNO}_2(\text{g})$  and  $\text{HNO}_3(\text{g})$ ], which will dissolve in condensed water and flow back down into the dissolver.

Most of the off-gas from the dissolver will consist of the  $\text{NO}_x$  (Jerden et al. 2011b); however, ORIGEN calculations show that iodine, xenon, and krypton will also be present in the off-gas. The ORIGEN calculations, performed by Charlie Allen, University of Missouri, using ORIGEN2, Version 2.2, assume the following: irradiation of 1 g of uranium foil enriched to 19.75%  $^{235}\text{U}$ ; power =  $1.9 \times 10^{-3}$  MW; burnup =  $1.59 \times 10^{-2}$  MW days; flux =  $2.1 \times 10^{14}$  N/cm<sup>2</sup> sec; burnup is for 200 hr; foil composition is given for cooling times of 12, 24, 36, and 48 hr. The most abundant off-gas species will be iodine ( $4.2 \times 10^4$  Ci per 250 g LEU, after 12 hr cooling), xenon ( $2.6 \times 10^4$  Ci per 250 g LEU, after 12 hr cooling) and krypton ( $1.7 \times 10^3$  Ci per 250 g LEU, after 12 hr cooling). As part of this study, the feasibility of using copper metal to trap and extract iodine gas from the dissolver during the dissolution reaction has been assessed experimentally (Jerden et al. 2011b). The efficiency of NaOH to neutralize/trap the  $\text{NO}_x$  gases has also been investigated (Jerden et al. 2011b).

Thermodynamic calculations show that for the dissolution of 250 g uranium metal (~1.05 moles) the total energy released may be up to 1600 kJ, but will probably be closer to 1000 kJ. Assuming adiabatic conditions and a 30 minute reaction time, this energy would correspond to a maximum thermal power output of approximately 890 W (Jerden et al. 2011b). The ORIGEN calculations show that a total thermal output for 250 g of irradiated LEU is around 1000 W for a 12-hr cooling (Figure 2).



**FIGURE 2 Cumulative Decay Heat for Fission Products and Actinides for a Range of LEU Foil Masses after 200-hr Irradiation (Curves are for 12 and 48 hr of cooling [time out of reactor]. Calculated using ORIGEN2, assuming the following: irradiation of 1 g of uranium foil enriched to 19.75%  $^{235}\text{U}$ , power =  $1.9 \times 10^{-3}$  MW, burnup =  $1.59 \times 10^{-2}$  MW days, flux =  $2.1 \times 10^{14}$  N/cm<sup>2</sup> sec.)**

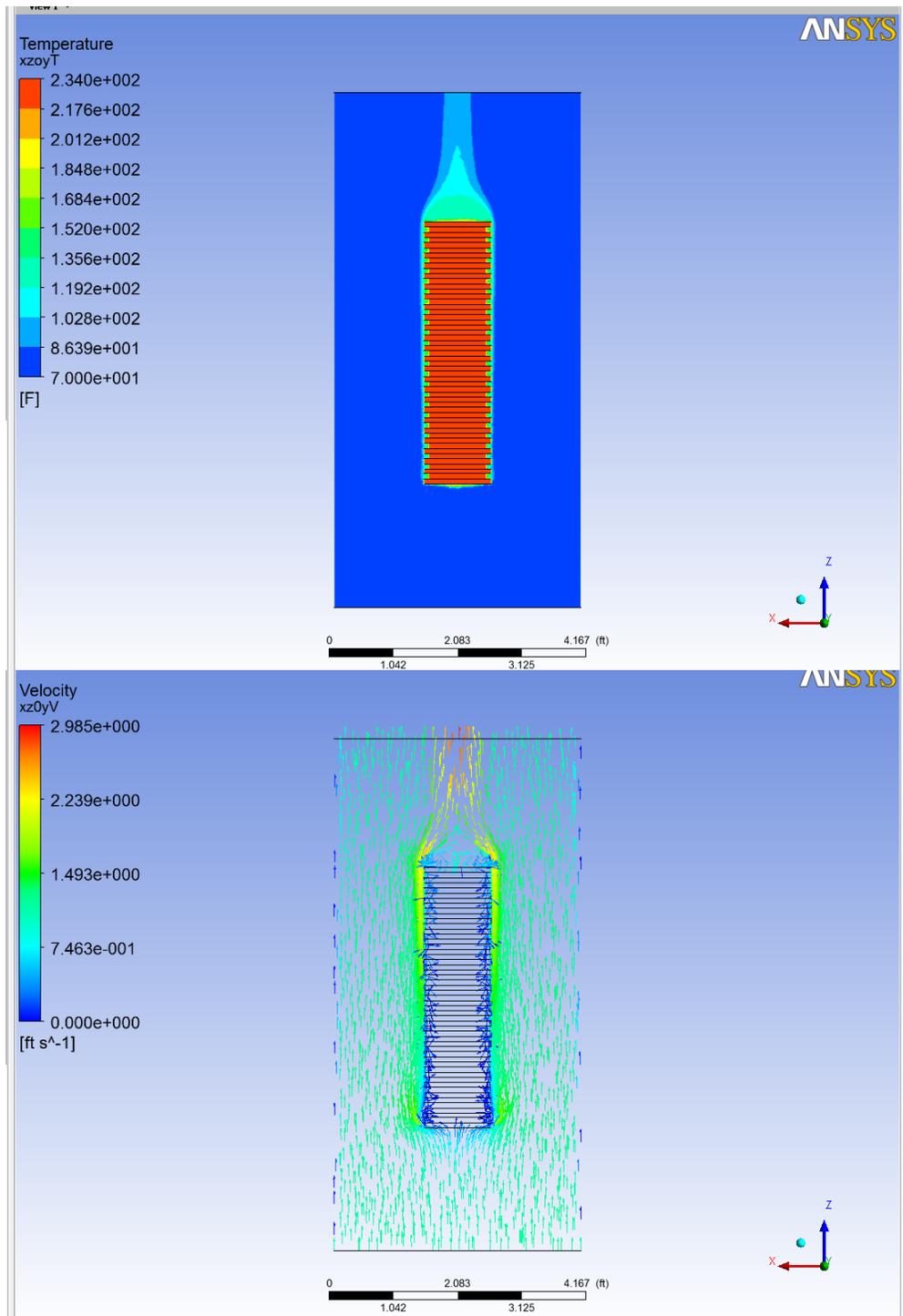
Based on the enthalpy and decay heat calculations, the cooling system for the LEU nitric acid dissolver system must be able to sink out a maximum of 2000 W (thermal). Therefore, if it is assumed that the dissolution of 250 g of irradiated LEU foil takes 30 minutes, 2000 W of thermal power will be generated and will need to be removed from the system to ensure that water vapor (and acid) is not lost during the dissolution process. Experiments in which both the reaction and decay heats are simulated using heating coils wrapped around the dissolver vessel were performed to test the performance of the dissolver cooling system.

## 2.2 SUMMARY OF DISSOLVER SYSTEM DESIGN CALCULATIONS

A mathematical model was implemented in Mathcad for the design and design optimization of the nitric-acid LEU-foil dissolver system. This Mathcad model is supported by heat flow calculations performed using the ANSYS CFX code (see Figure 3).

The current dissolver design was based on the Mathcad/ANSYS CFX model. By comparing the model calculations with test results (discussed below) the system design, specifically the cooling system fan size and off-gas reservoir design and dimensions, has been optimized. The Mathcad design model was used for the following design aspects:

- Sizing of the cooling fins and air flow requirements from the cooling fan in order to remove both the reaction heat and decay heat from the dissolver: with a reaction heat of approximately 1600 kJ over 30 minutes and a constant decay heat of approximately 1500 W, the average required heat removal rate is 10181 kJ/hr. This value determined the original cooling system design.
- The pressure drop across the cooling fin section was calculated based on the required flow velocity as determined by the convective heat removal from the fins. The total air volume flow rate and temperature rise of the air across the fins was also calculated. These results are used to determine the cooling fan size from fan performance curves.
- The required gas volume and heat sink capacity of the off-gas reservoir was calculated. It was assumed that all the reaction heat and off gas are transferred to the reservoir (loss of cooling scenario). In addition, the steady state requirement for the decay heat removal is assumed to be dissipated by the reservoir. A thermal analysis was performed to determine an approximate value for the heat loss from the off-gas reservoir. The reservoir tank was assumed to be maintained at a uniform temperature of 100°C with an ambient air temperature of 32°C. Heat loss by radiation and heat loss by natural convection are calculated separately and then added together to determine the total heat flow from the outside of the reservoir to the air in the hot cell. Results from this calculation were used to size and design the aluminum heat sink rings that encase the off-gas reservoir tank.



**FIGURE 3** Example of Results from ANSYS CFX Model for the Off-Gas Reservoir for the Dissolver System (Calculations show that the heat sink [cooling fins] attached to the reservoir are sufficient to keep the system from becoming pressurized in the event of a complete loss of active cooling. Top: Dissolver reservoir surface temperature [red] with 1,500 W internal heat generation. Bottom: Dissolver reservoir showing natural convection velocity around the exterior.)

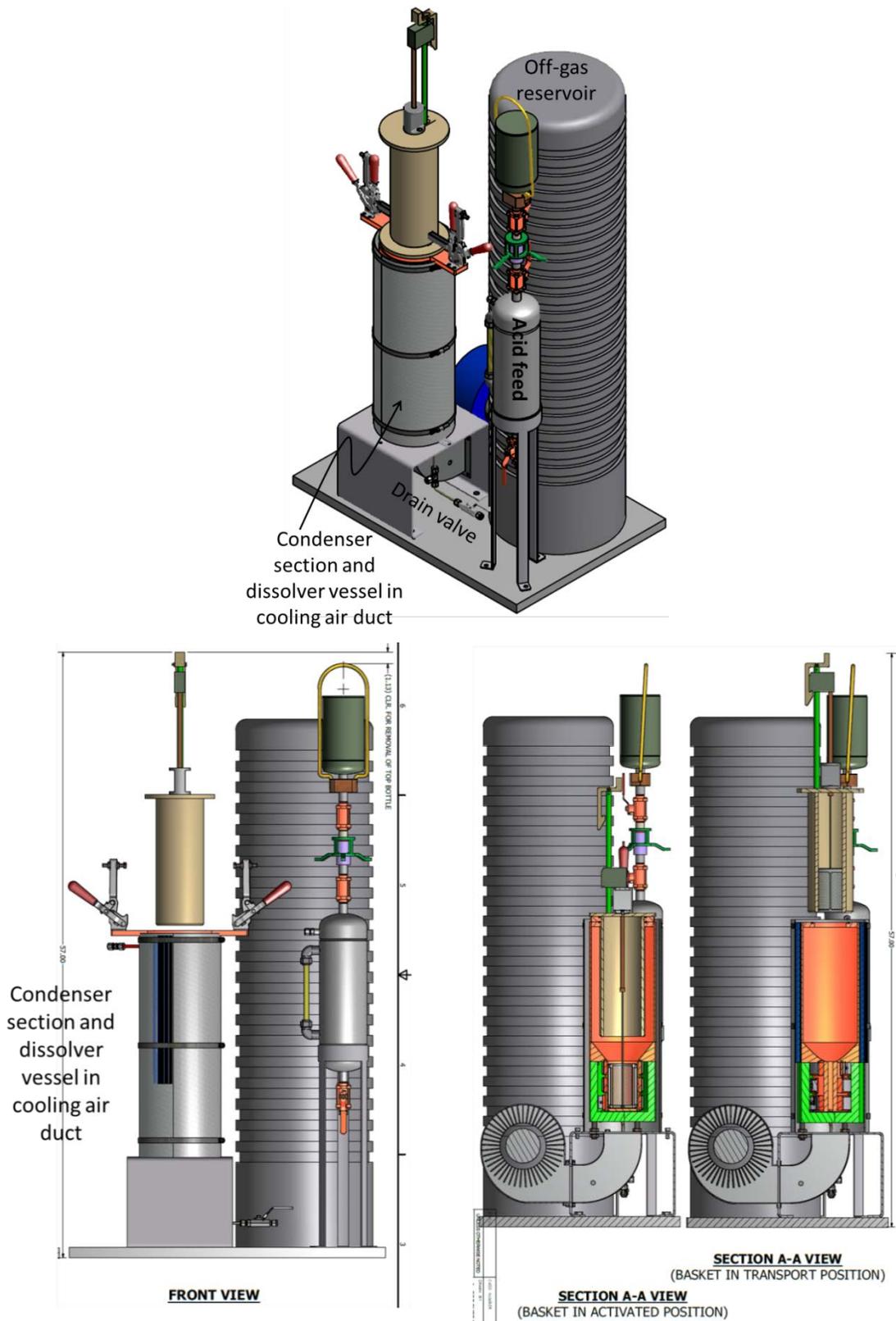
- The heat-removal capacity due to condensing of the process vapor on the inside surface of the dissolver condenser section was calculated and accounted for in the cooling system design. Boiling of the dissolver solution occurs in the vessel at the bottom of the dissolver, and the vapor moves upward to the condenser section where it condenses on the cooler walls that have been cooled by the air flow over the fins on the outer surface. The wall temperature is assumed to be below 100°C.

## 2.3 DISSOLVER DESIGN OVERVIEW

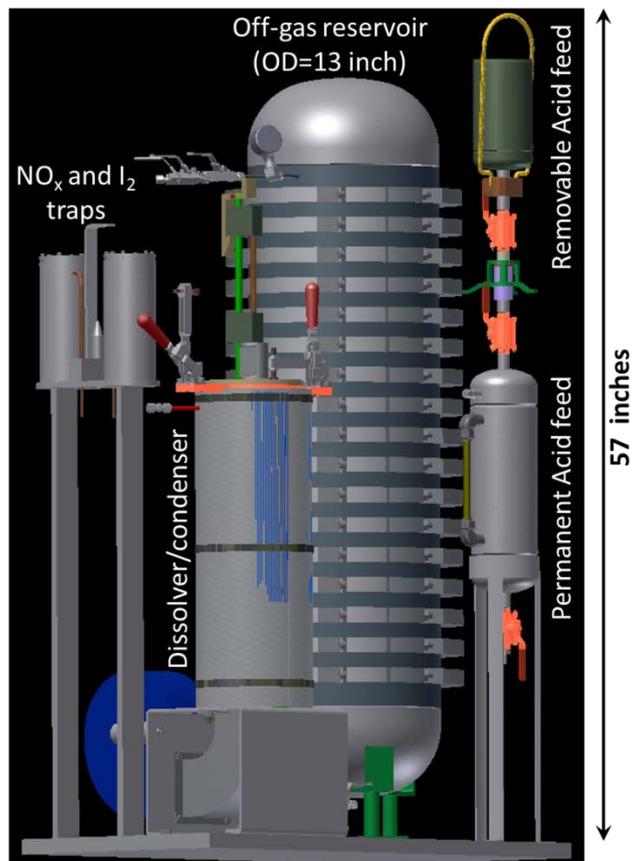
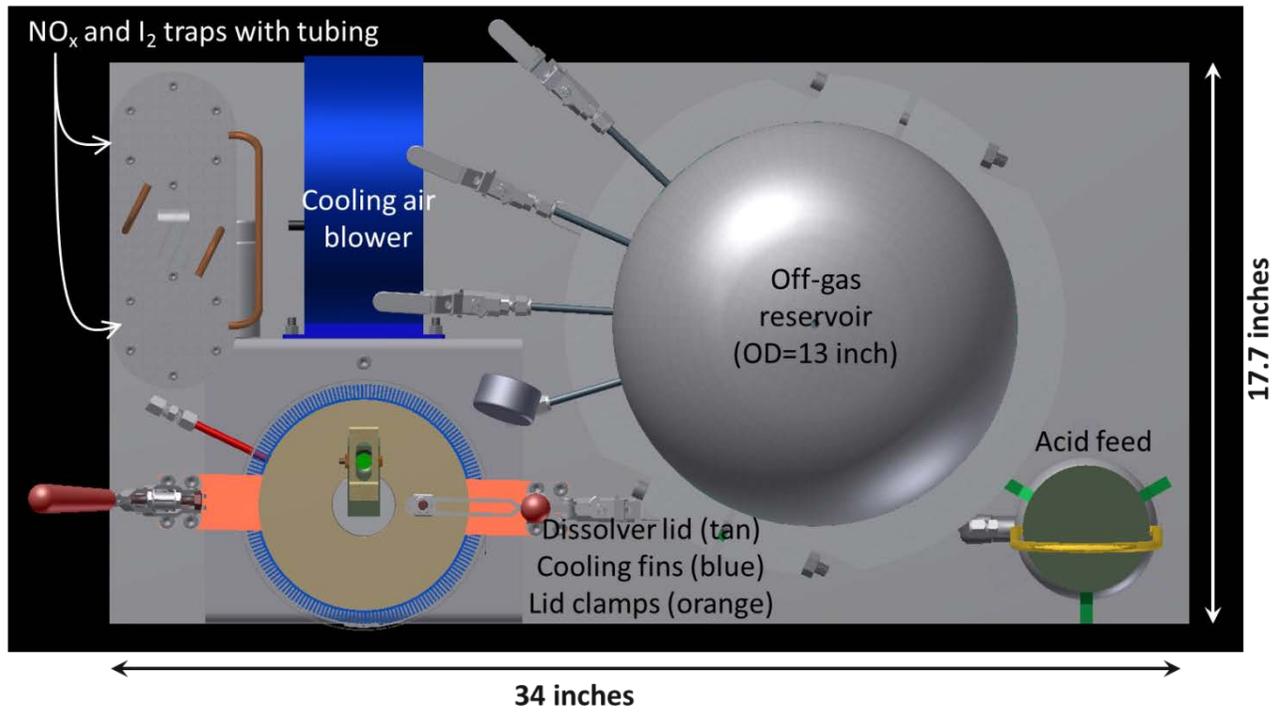
The dissolver system is designed to operate at pressures less than 2 atm (absolute) and at temperatures less than 125°C. The design and footprint of the dissolver system is shown in Figures 4 and 5. The design has been recently updated (early FY2013) to facilitate a full-scale demonstration in hot cells at Oak Ridge National Laboratory. Pictures of the dissolver system components that are currently being tested at Argonne and will be used in demonstration tests at Oak Ridge are shown in Figures 6 and 7.

The dissolver system consists of a 304 stainless steel vessel (2-L volume) connected to an approximately 65-L (30 cm × 90 cm) off-gas reservoir (Figure 7). Figure 7 shows the full-scale dissolver system setup for the non-irradiated dissolution tests discussed in this report. The dissolver vessel is open to the off-gas reservoir during the dissolution process. The volume of the reservoir and the sizing of the aluminum heat sink (fins shown in Figure 7, bottom) were chosen to provide passive containment of all water vapor and reaction products at a pressure less than 2 atm (absolute), during both normal and off-normal (loss of cooling during reaction) conditions. The role that the condenser section of the dissolver plays during a typical dissolution run is shown schematically in Figure 8.

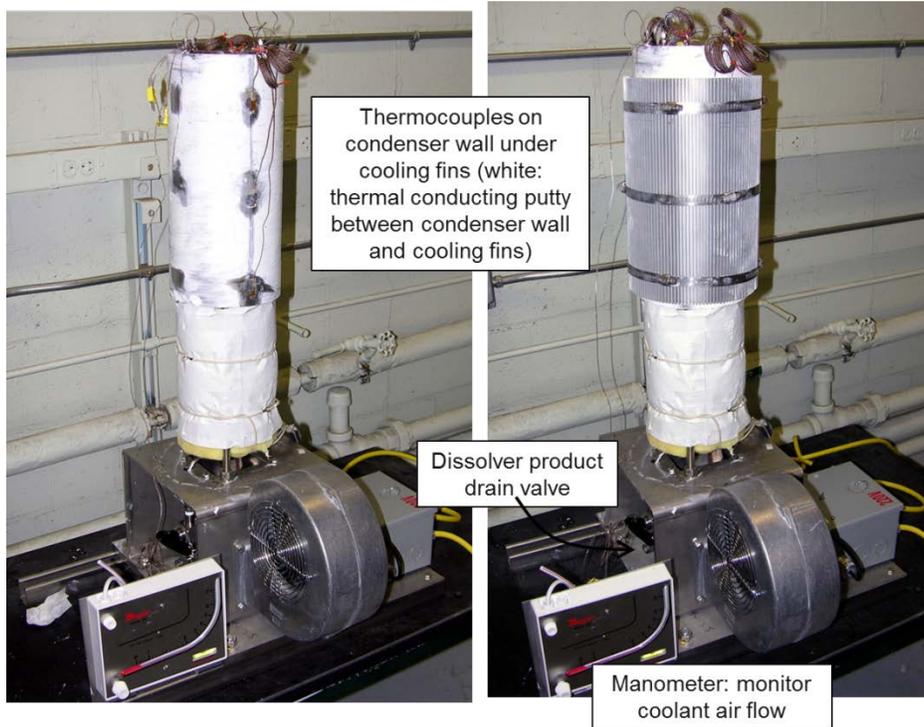
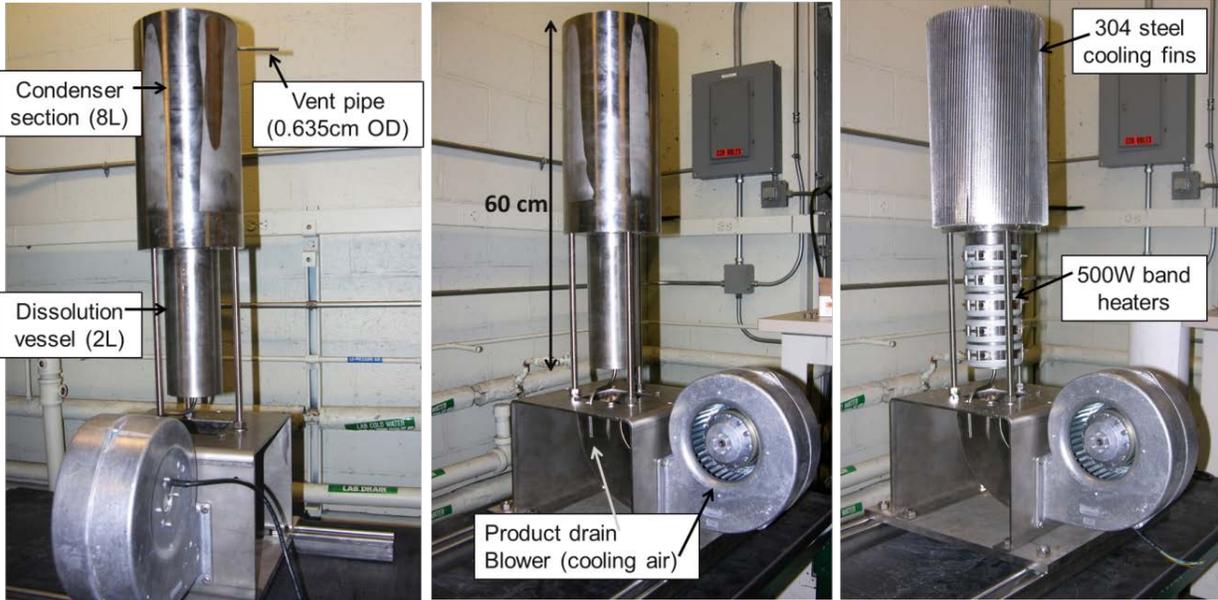
The dissolution process is started by first lowering the uranium foil (contained within a steel mesh basket) into the dissolver vessel and then sealing the vessel with a metal cap. Nitric acid is then added to the vessel using a two-chamber acid feed system that is designed to avoid pressurization of the acid bottle in the event that the dissolution reaction begins instantaneously when the acid is added. The dissolver vessel is cooled by forced air blown from the base of the unit. The temperature of the dissolver solution is monitored by a thermocouple. The dissolver vessel is insulated so that the top of the vessel is cooled continuously during the reaction. Heat loss from the top of the vessel is optimized by the presence of steel cooling fins attached to the condenser part of the dissolver system. This design causes the water and acid vapors to condense along the walls at the top of the vessel during the dissolution reaction (as acid is boiling). This process is shown schematically in Figure 8.



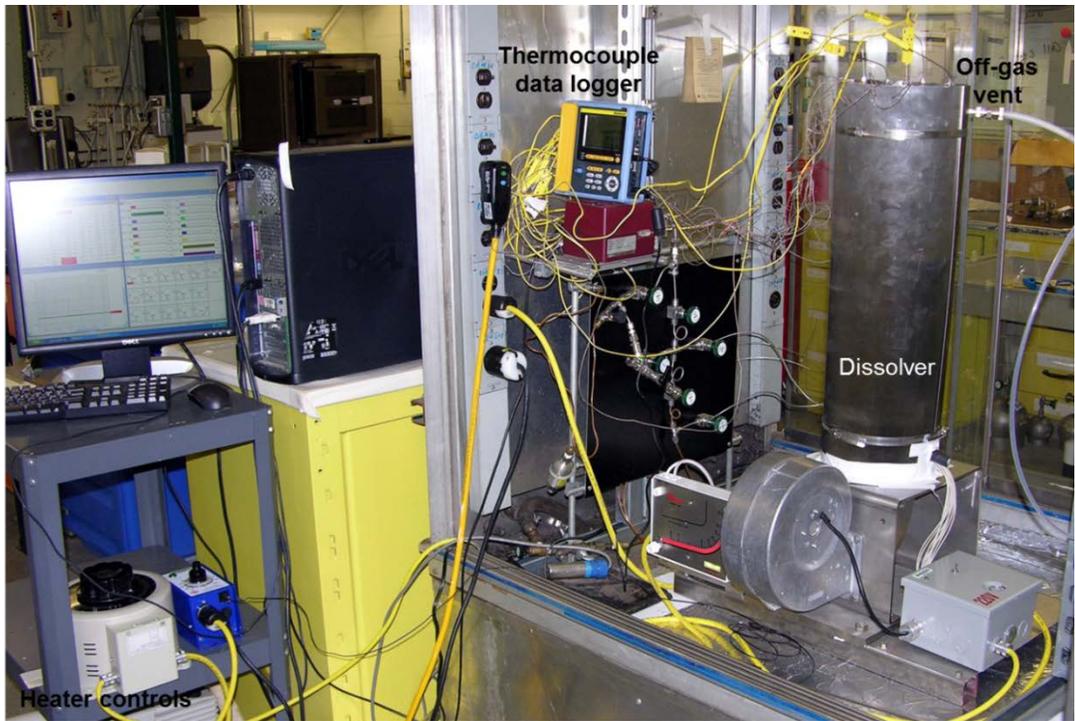
**FIGURE 4** Portions of the Ambient-Pressure, Nitric-Acid-Dissolver System Fabrication Drawings



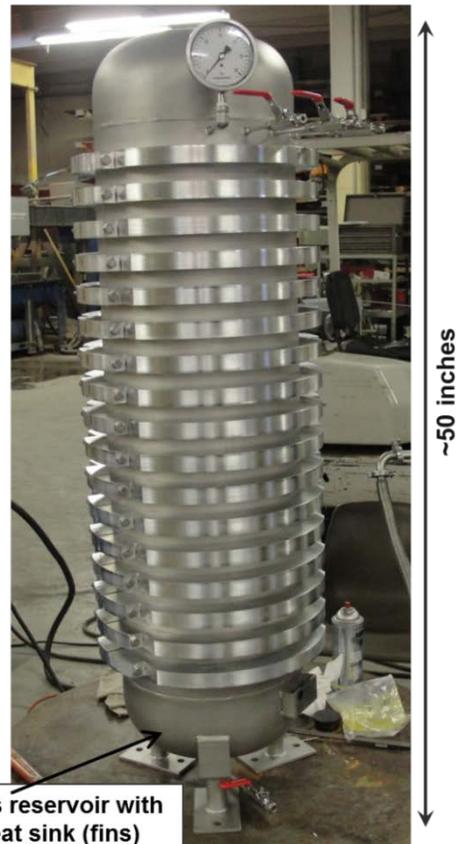
**FIGURE 5 Design Drawings of Ambient-Pressure, Nitric-Acid-Dissolver System Showing Layout and Key Dimensions**



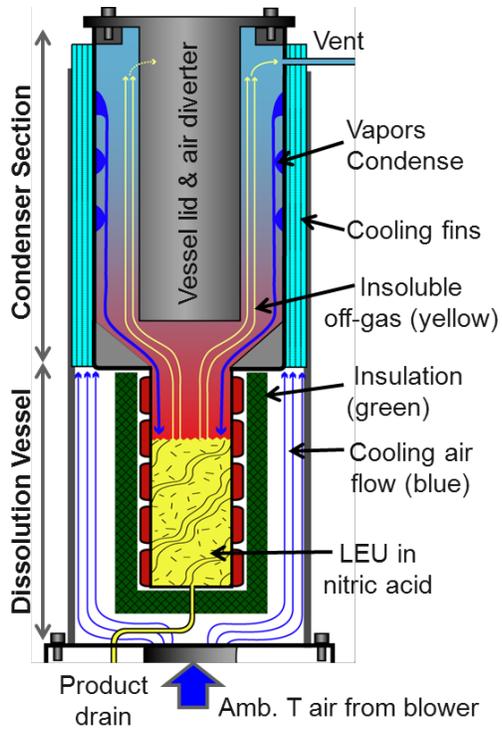
**FIGURE 6 Dissolver/Condenser and Cooling Fan Portion of Full-Scale Dissolver Prototype Used in Performance Tests**



Dissolver test set-up in radiological hood



**FIGURE 7 Full-Scale Dissolver Prototype Used in Non-irradiated Uranium Foil Performance Tests**



**FIGURE 8 Schematic Cross-Section through the Condenser and Dissolution Vessel Sections of the Dissolver System**

### 3 DISSOLVER SYSTEM PERFORMANCE TESTS

Two types of experiments were performed: (1) Condensation performance tests were run using boiling water to confirm and quantify the performance of the condenser section of the dissolver system. An array of thermocouples was used in the initial testing to measure all relevant thermal gradients during the dissolution reaction process (inside and outside the dissolver vessel and condenser section). (2) Non-irradiated uranium dissolution tests were also performed to measure the loss of  $\text{NO}_x$  from the dissolver and to test the performance of the NaOH-based  $\text{NO}_x$  scrubber. The general procedures used for the experiments are summarized as follows.

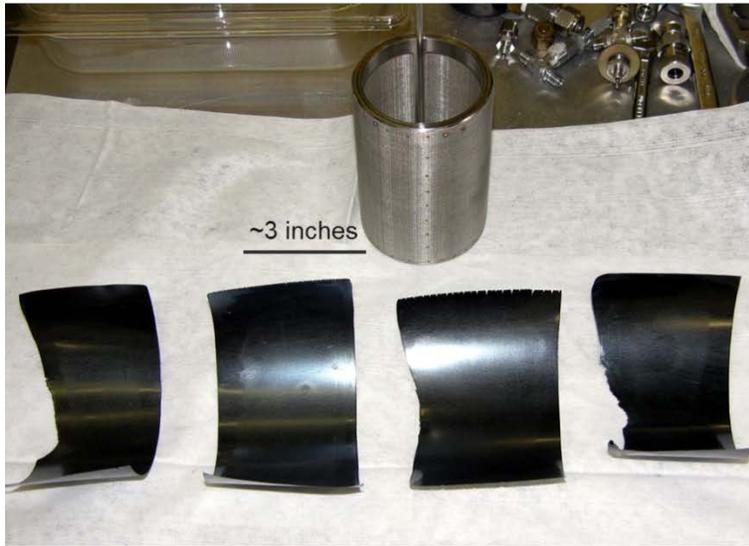
*Condenser section performance tests:* The purpose of these experiments was to determine the heat-exchange capacity between the coolant air flow on the outside of the condenser section and the condensing vapor to liquid on the inside of the vessel. Band heaters attached to the base of the dissolver vessel (Figures 5) were used to simulate the exothermic heat from the LEU dissolution reaction, as well as the decay heat from the irradiated foil. The condenser section performance tests were performed as follows:

- 2 L of deionized water was fed into the dissolver vessel. The dissolver was sealed using a bolted lid on the top of the condenser section. At this point, the heaters were off, and the vessel was at ambient temperature ( $\sim 24^\circ\text{C}$ ).
- The blower was turned on, and the monitoring and recording of temperatures and cooling air flow velocity were initiated. The temperatures were allowed to stabilize before starting the next step.
- The heaters were turned on at low power, and the transient temperature increase was monitored and recorded.
- Once the system reached steady state (water boiling), temperatures within and outside the dissolver were continuously monitored using a computer data logger. The cooling-air flow velocity was measured, and the electrical power to the heaters was measured.
- The experiments were run at this steady-state condition for approximately 1 hr. The vent tube was constantly monitored to see if vapor was escaping from the condenser section.
- After 1 hr of running the experiment at steady state, the heaters were turned off, and the blower was kept on until the vessel returned to ambient temperature.
- When the vessel was at ambient temperature, the water was drained out (using the product drain valve), and its mass/volume was measured to determine how much vapor was lost from the condenser section during the experiment.

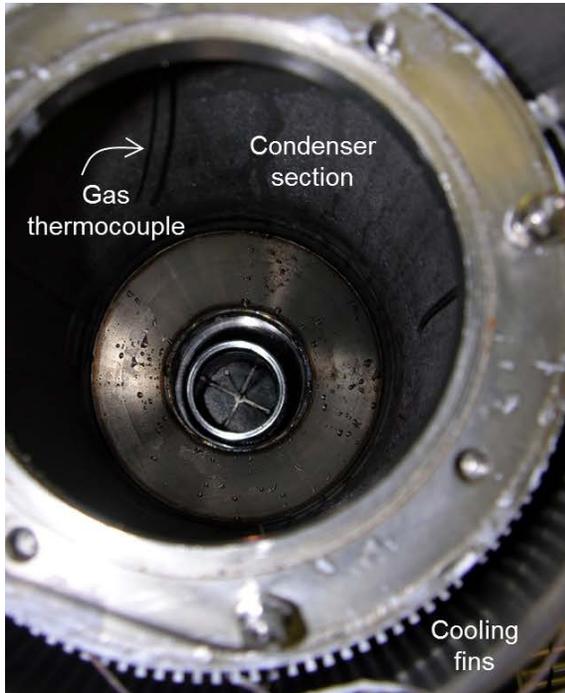
*Depleted uranium metal dissolution tests:* Similar to the “water” tests described above, the purpose of these experiments was to determine the heat-exchange capacity between the coolant-air flow on the outside of the condenser section and the condensing vapor to liquid on the inside of the vessel. Band heaters attached to the base of the dissolver vessel (Figures 6) were used to simulate the exothermic heat from the LEU dissolution reaction and the decay heat from the irradiated foil. The difference between these tests and the “water” tests was that the uranium metal was dissolved in nitric acid during the experiment so that the behavior of the NO<sub>x</sub> and acid gases could be observed and the performance of a simple NaOH NO<sub>x</sub> trap tested. Pictures from the loading of uranium foils into the dissolver for these dissolution tests are shown in Figure 9. The dissolution tests were performed as follows:

- 133 g of depleted uranium metal foil (surrogate for LEU foil) was lowered into the base of the dissolver vessel using a steel mesh basket (Figure 9).
- 500 mL of 7 molar nitric acid was fed into the bottom of the vessel. In some experiments, nitric acid was fed into the dissolver prior to the metal foil being added, while in other tests, the acid was added after the metal foil had been lowered into the vessel. The dissolver vessel was then sealed. At this point the heaters were off and the vessel was at ambient temperature.
- The blower was then turned on, and the monitoring and recording of the temperatures and air flow velocity was initiated. The temperatures were allowed to stabilize before initiating the next step.
- The heaters were turned on at low power, and the transient temperature increase was measured.
- The temperature of the acid was monitored and recorded continuously during the exothermic dissolution reaction.
- Once the acid was boiling, the heaters were turned off, and the blower was kept on. The temperature within the dissolver vessel was monitored and recorded continuously until it reached ambient temperature.
- The dissolver solution was removed using the product drain valve, and its mass was measured to determine the loss of acid from the dissolver during the experiment.
- The dissolver lid was removed and the interior of the dissolution vessel was examined using a borescope (1-m-long flexible fiber optic camera) to determine whether any undissolved solids remained in the dissolver.

Dissolver basket and DU foils used in dissolution tests



Basket containing DU foils lowered into dissolver vessel



**FIGURE 9** Loading of Basket with Depleted Uranium Foils (133.1 g) and Lowering of Basket into Dissolver in Preparation for Non-irradiated Uranium Dissolution Tests

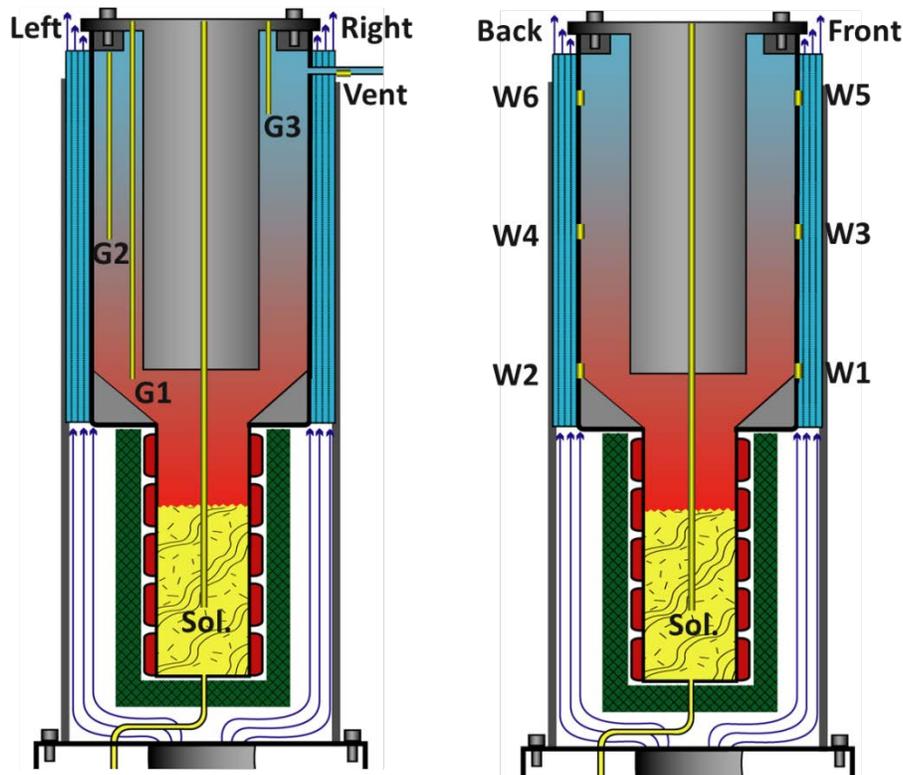
### 3.1 DISCUSSION OF DISSOLVER PERFORMANCE TEST RESULTS

The approximate locations of thermocouples for the dissolver performance tests are shown in Figure 10. Results from the tests are shown in Figures 11 and 12.

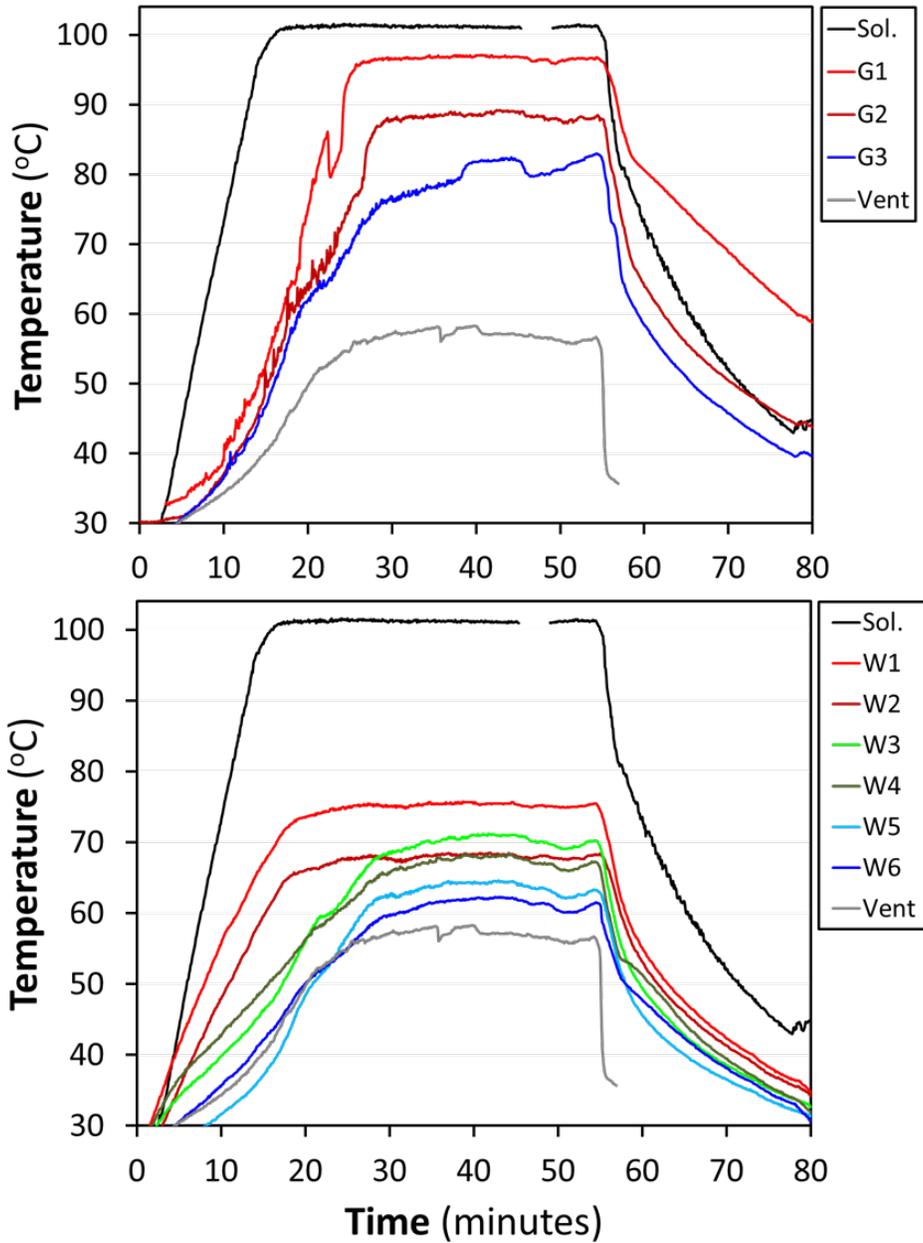
The linear flow velocity of the cooling fan was measured throughout each experiment. The cooling air flow remained constant at 26.1 m/s where it entered the cooling fin section at the base of the dissolver. The cooling air flow measured at the top of the dissolver, where it exits through the cooling fins was consistently 19.8 m/s. The loss of flow velocity is due to turbulence as the cooling air travels up through the dissolver duct work and is channeled into the cooling fins, and perhaps by small leaks in the steel sheath that contains the fins.

For both tests, 1700 W of thermal power was supplied to the dissolver cup via band heaters (heater configuration shown in Figure 6). Key observations from these experiments are summarized below:

- No measurable water loss was detected from the deionized water condenser tests.
- Dissolution of 133 g of depleted uranium foil in 7 molar nitric acid (initial) was not complete after 1 hr under boiling conditions.

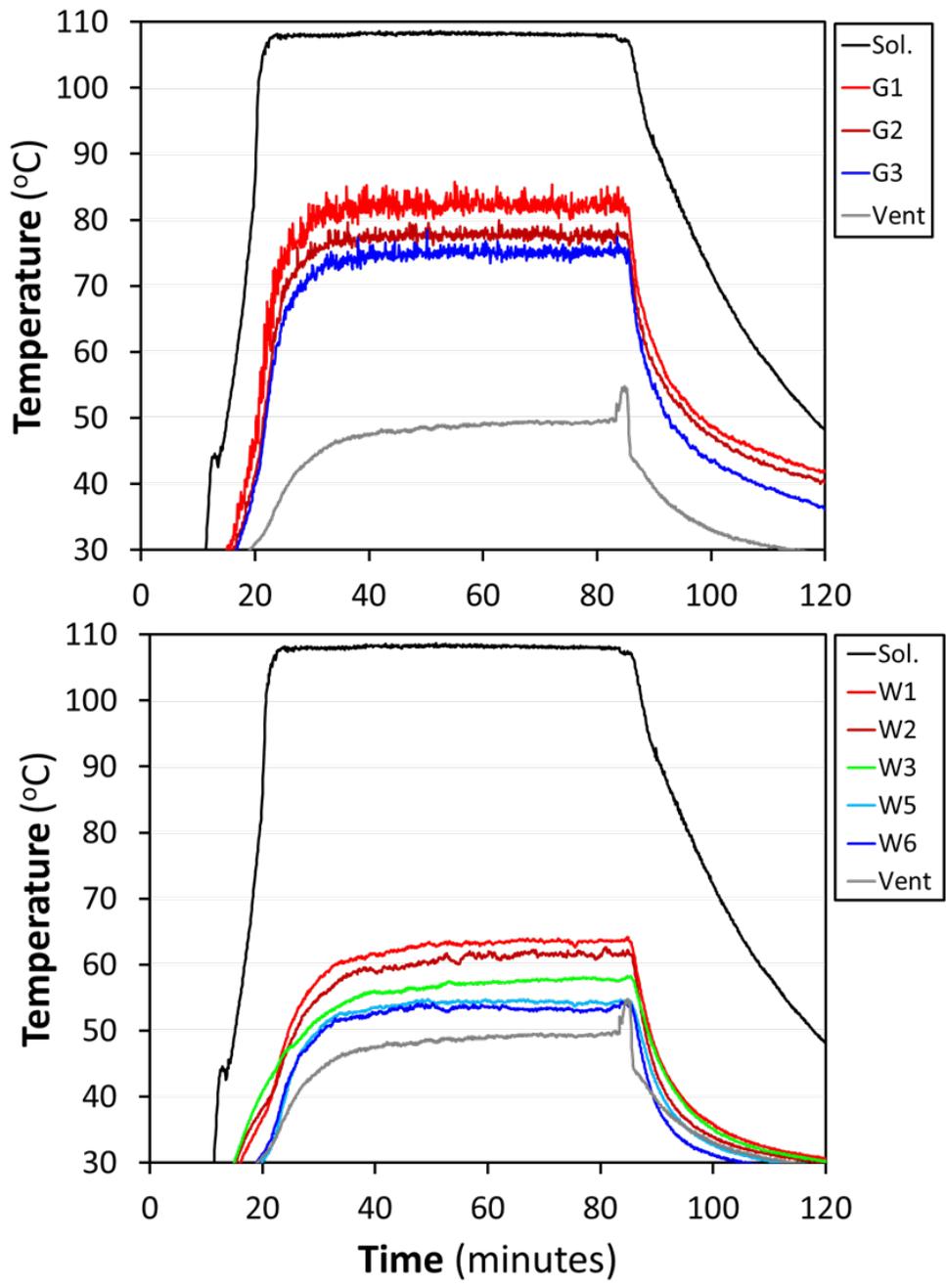


**FIGURE 10** Approximate Location of Thermocouples for Dissolver Performance Tests



**FIGURE 11 Thermal Profiles within the Dissolver and Condenser Sections during Water Tests**

- Dissolution of 133 g of DU foil in 7 molar nitric acid (initial) was complete after 2 hr under boiling conditions.
- No measurable solution loss was detected during DU dissolution tests; however,  $\text{NO}_x$  gas was observed escaping intermittently during the dissolution process. The amount of  $\text{NO}_x$  lost from the dissolver and its effect of the dissolution rate and efficiency are being further investigated in ongoing experiments.



**FIGURE 12 Thermal Profiles within the Dissolver and Condenser Sections during Dissolution of 133 g of Depleted Uranium Foil**

#### 4 SUMMARY AND FUTURE/ONGOING WORK

- A full-scale prototype of the ambient-pressure, nitric acid LEU-foil dissolver system (capable of dissolving 250 g of irradiated LEU for  $^{99}\text{Mo}$  extraction) was designed and tested.
- Tests results show that the cooling system of the dissolver (reflux condenser) is sufficient to remove all heat produced by the exothermic dissolution reaction and continuous decay heat of the irradiated foil.
- The dissolution of 133 g of depleted uranium foil was completed successfully in approximately 2 hr. The dissolution time can be minimized by optimizing the starting acid concentration to account for the effects of the loss of  $\text{NO}_x$  and acid gases from the dissolver during dissolution. These optimization dissolution experiments, using test setup described in this report, are ongoing.
- The performance tests of the off-gas traps for  $\text{NO}_x$  (NaOH trap) and iodine (copper metal or silver loaded zeolite) will be completed and these gas traps will be added to the dissolver system design.
- The complete dissolver system (including gas traps and off-gas reservoir) will be tested in a manipulator mock-up facility to ensure that the dissolver system can be easily operated in a production scale in a hot-cell facility.

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