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ABSTRACT

A process is under development to use low-enriched uranium (LEU) metal targets for production of ^{99}Mo . The first step is to dissolve the irradiated foil. In past work, this has been done by heating a closed (sealed) vessel containing the foil and a solution of nitric and sulfuric acids. In this work, we have demonstrated that (1) the dissolver solution can contain nitric acid alone, (2) uranium dioxide is also dissolved by nitric acid alone, and (3) barrier metals of Cu, Fe, or Ni on the U foil are also dissolved by nitric acid. Changes to the dissolver design and operation needed to accommodate the uranium foil are discussed, including (1) simple operations that are easy to do in a remote-maintenance facility, (2) heat removal from the irradiated LEU foil, and (3) cold trap operation with high dissolver pressures.

INTRODUCTION

Badan Tenaga Atom Nasional (BATAN) and Argonne National Laboratory (ANL) are working on a joint R&D project to develop a LEU-metal-foil target and recover ^{99}Mo from the irradiated target by using a modified Cintichem process. The final production operation for the modified Cintichem process will be done at BATAN, which is currently producing ^{99}Mo from high-enriched uranium (HEU) UO_2 targets by the original Cintichem process [1, 2]. This process was used until 1989 at the Cintichem reactor facilities in Tuxedo, New York. These facilities are no longer operational, and the proprietary rights for the process now belong to the U.S. Department of Energy. At present, BATAN has a license to use the process in its facilities at Serpong, Indonesia.

Previous work on uranium foil dissolution using nitric-acid/sulfuric-acid mixtures was reported for both open (but covered) and closed (sealed) dissolvers [3]. In the work reported here, the operation of closed dissolvers was extended to their use with nitric acid alone, and changes were made to dissolver design and operation to facilitate its use in a remote-handled, shielded-cell facility. Our goal is to keep equipment modifications and process changes to a minimum so there can be a smooth transition from HEU as UO_2 to LEU foil. Discussed elsewhere are other aspects of the modified Cintichem process, that is, the target design and irradiation and the processing of the dissolved foil to recover ^{99}Mo [4, 5]. Successful completion of this work will enable commercial production of ^{99}Mo (for medical applications) from LEU instead of HEU.

This paper discusses progress on (1) the dissolution of LEU targets in a closed dissolver, (2) changes to dissolver design and operation, and (3) future directions.

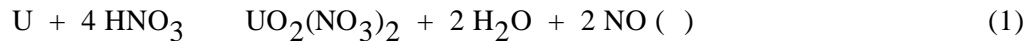
DISSOLUTION IN CLOSED DISSOLVER

Srinivasan et al. [3] demonstrated dissolution of uranium foil in a closed dissolver using a mixture of nitric acid and sulfuric acid. Based on this success, two further areas were explored. First, the use of nitric acid alone was tested in place of the mixture of nitric acid and sulfuric acid because of benefits in the waste cleanup operation. Second, the dissolution of three potential barrier metals (Cu,

Fe, and Ni) was studied, and one metal, Ni, was dissolved in a closed dissolver because development of the target is leading to the use of barrier metals on the uranium foil [4].

Uranium Foil

The overall reaction for uranium foil dissolution using nitric acid alone is



This reaction is very similar to the one for mixtures of nitric acid and sulfuric acid [3], except that the uranyl ion now forms a nitrate salt rather than a sulfate salt. In particular, the moles of NO gas generated per mole of U foil dissolved are the same. Thus, the final dissolver pressure will be the same for the same mass of uranium. Because there is no sulfuric acid in the dissolver solution, the moles of HNO₃ consumed per mole of U dissolved are doubled, from two to four. Dissolutions were carried out with nitric acid alone and the closed (sealed) stainless-steel dissolver described earlier [3]. The results are listed in Table 1. Tests 8-10, which were done earlier [3], are included here so that nitric-acid-alone results can be compared directly with those for mixed acid (3M HNO₃ and 2M H₂SO₄). Pressure-time plots for many of these tests are given below.

To estimate the concentration of nitric acid required to dissolve the U foil, an analysis was completed on the dissolver heating reported for 18 g of U-foil targets dissolved in 3M HNO₃ and 2M H₂SO₄ [3]. This analysis shows that the U-foil dissolution becomes self-heating at 53°C. The correlation for the rate of U-foil dissolution as a function of temperature, nitric acid concentration, and sulfuric acid concentration [3] shows that the dissolution rate required to initiate self-heating is 1.0 mg/(min•cm²). Since the steady-state dissolver temperature is 103°C, a dissolver solution of 8M HNO₃, which reaches a dissolution rate of 1.0 mg/(min•cm²) at 68°C, should also become self-heating during uranium foil dissolution. For 3 g of U foil, both nitric acid (test 3) and mixed acid (test 8) were tested to see if they dissolved the foil at the same rate, or if the mixed-acid dissolution was slightly faster. The results, given in Fig. 1, show that test-3 dissolution is faster. The reason for this is as follows. First, only 15 mL of dissolver solution was used for test 3, 80 mL for test 8. Second, for both tests, the dissolver angle was chosen so that the liquid wets the bottom 28% of the dissolver wall. Thus, the amount of heat transferred is about the same for both tests. Thus, test 3, which has less solution, is heated more rapidly to its self-heating temperature.

Based on the success of dissolving 3 g of U foil in 8M HNO₃, similar dissolutions were done with 9 and 18 g of U foil. These curves are shown on Fig. 2 along with the corresponding mixed (3M HNO₃ and 2M H₂SO₄) acid curves. As expected, the final dissolver pressure is roughly the same for both mixed acids and nitric acid alone when the mass of U foil is the same. The 9-g tests also showed that a sharp pressure rise occurred sooner for nitric acid alone (test 4) since the volume of this solution was less. However, for the 18-g tests, the sharp pressure rise occurred sooner for the mixed acid (test 10) even though it had more solution volume. This reversal happened because the dissolver was horizontal for test 10 so that the entire dissolver surface transferred heat to the solution. Because the mixed-acid solution was spread out over the entire 46-cm (18-in.) length of the dissolver while the foil was in the bottom 10 cm (4 in.), the self-heating applied only to some of the solution, the solution near the foil. As a result, neither the rate of pressure rise nor the pressure peak was as high as for nitric acid alone (test 5). For the 9- and 18-g cases of U foil with nitric acid, the solution volume was reduced so that the final U concentration was high (1.8M), and the final HNO₃ concentration was low (0.7M). In addition, the dissolver was tilted slightly, typically 5.3°, so that all of the foil would pass through the solution during each revolution of the dissolver. As was seen earlier [3], once self-heating occurs, the increased temperatures that result give high dissolution rates even at low acid concentrations. These operating conditions are attractive because they minimize the solution volume to be processed, and the nitric acid to be neutralized.

Table 1. Dissolution of U, UO₂, or Ni in a Closed (Sealed) Dissolver Using either HNO₃ Alone or Mixed Acids

| Test ID | Initial Conc, <u>M</u> | | Soln Vol, mL | Dissol. Angle, deg. | Steady State Temp., °C | Total Time, min | Initial Metal ^a Foil or Oxide Mass, g | Calc Final Conc, <u>M</u> | |
|-----------------|------------------------|--------------------------------|--------------|---------------------|------------------------|-----------------|--|---------------------------|------------------|
| | HNO ₃ | H ₂ SO ₄ | | | | | | Metal | HNO ₃ |
| 1 | 4 | 0 | 15 | 5.3 | 102 | 44 | 2.86 | 0.80 | 0.80 |
| 2 | 4 | 0 | 15 | 5.3 | 138 | 30 | 2.74 | 0.77 | 0.93 |
| 3 | 8 | 0 | 15 | 5.3 | 102 | 30 | 2.94 | 0.82 | 4.71 |
| 4 | 8 | 0 | 21 | 5.3 | 102 | 17 | 9.14 | 1.83 | 0.69 |
| 5 | 8 | 0 | 41.2 | 5.3 | 102 | 16 | 17.86 | 1.82 | 0.71 |
| 6 ^b | 8 | 0 | 15 | 5.3 | 102 | 28 | 2.99 | 0.84 | 4.65 |
| 7 ^c | 8 | 0 | 15 | 5.3 | 102 | 12 | 2.91 | 0.63 | 5.47 |
| 8 ^d | 3 | 2 | 80 | 42 | 103 | 46 | 3.04 | 0.16 | 2.68 |
| 9 ^e | 3 | 2 | 80 | 42 | 103 | 54 | 9.98 | 0.52 | 1.95 |
| 10 ^f | 3 | 2 | 80 | 0 | 103 | 170 | 18.14 | 0.95 | 1.09 |
| 11 | 5.6 | 0 | 41 | 1.1 | 102 | 60 | 17.92 ^g | 1.62 | 1.28 |
| 12 | 8 | 0 | 55 | 5.3 | 102 | 22 | 8.29 ^h | 2.57 | 1.15 |
| 13 | 8 | 0 | 55 | 5.3 | 102 | 22 | 8.16 ^h | 2.53 | 1.26 |

^a Metal is uranium unless noted otherwise below.

^b Dissolver vent valve open for first 8.5 min during heating.

^c Dissolver quenched in ice bath after 12 min to stop dissolution reaction. As a result, the foil was not completely dissolved; 0.65 g (20%) remained.

^d Mixed acid test included from [3] for comparison with 3-g tests using nitric acid alone.

^e Mixed acid test included from [3] for comparison with 9-g test using nitric acid alone.

^f Mixed acid test included from [3] for comparison with 18-g test using nitric acid alone.

^g Test used UO₂ particles were used in place of U foil.

^h Test used Ni foil was used in place of U foil. The foil size was 76 x 102 x 0.127 mm (3 x 4 x 0.005 in.).

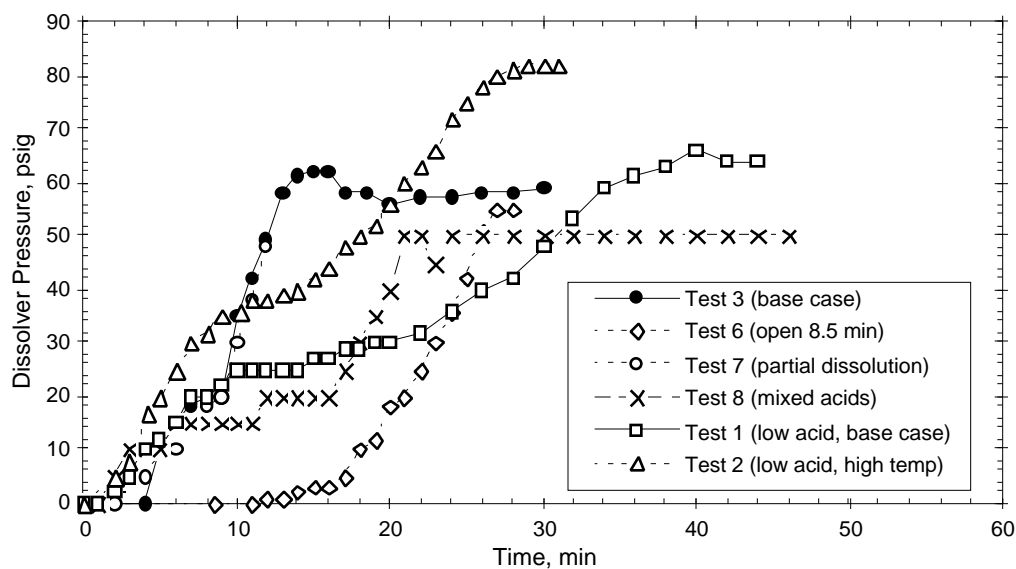


Figure 1. Pressure-Time Curves for Dissolution of 3 g of U Foil

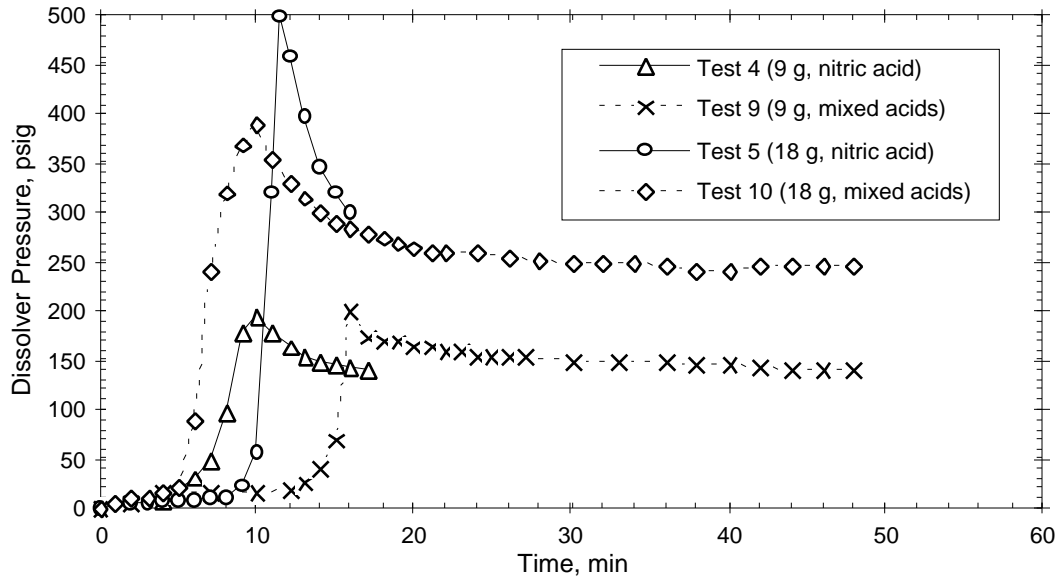


Figure 2. Pressure-Time Curves for Dissolution of 9 and 18 g of U Foil

Additional tests with 3 g of U foil, also plotted in Fig. 1, show (1) the need for the closed dissolver to be sealed, (2) the time at which complete U-foil dissolution occurs, (3) the effect of lower acid concentrations, and (4) the effect of higher steady-state dissolver temperatures. The need to have the dissolver closed and sealed is shown by test 6. This test was like the base case (test 3) except that the vent valve was accidentally left open for the first 8.5 min. After the valve was closed, the dissolver pressure started to increase, but at a lower rate. When the pressure leveled off at 28 min, the dissolver was cooled quickly and opened. The U foil was found to have been completely dissolved.

The time at which the foil completely dissolves was explored in test 7. This test was like test 3 (base case) except that the dissolver heating was stopped, and the dissolver was plunged into an ice bath after 12 min. The dissolver from test 3 had been heated for 30 min before it was cooled and opened. At 12 min, the dissolver pressure was 80% of the way to its peak pressure. When the dissolver from test 7 was opened, it was found that the 0.13-mm-thick (5-mil-thick) foil was 80% dissolved. As can be seen in Fig. 3, the original shape of the foil was pretty much intact. However, there were many fine holes in the foil, and portions of the foil on the right in Fig. 3, which were at the very bottom of the dissolver, have completely dissolved away. Thus, from analysis of tests 6 and 7, complete dissolution of U foil in a closed, sealed dissolver can be identified by either (1) the occurrence of a pressure peak or (2), if there is no maximum pressure, the point at which the pressure becomes steady. Note that, if the dissolver is not sealed, self-heating effects are minimized (since steam that is formed can escape and carry off the excess heat), and the dissolution time increases sharply (by a factor of two or more).

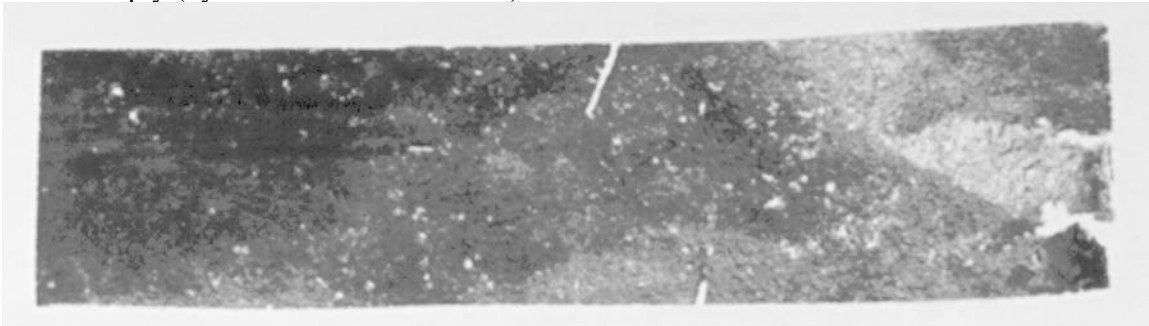


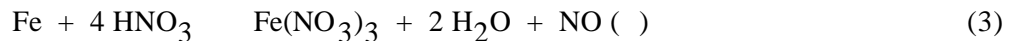
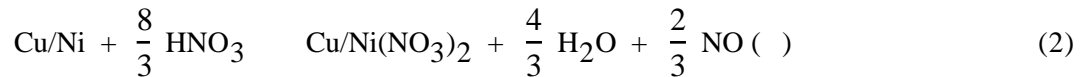
Figure 3. Photo of Partially (80%) Dissolved U Foil

The effect of lower acid concentrations and higher steady-state dissolver temperatures was explored in tests 1 and 2. For test 1, the acid concentration was lowered from 8M HNO₃ (base case) to 4M HNO₃ so that the self-heating dissolution rate of 1 mg/(min•cm²) occurred at 100°C, very close to the steady-state dissolver temperature of 102°C. At these conditions, the foil dissolved more slowly, as shown in Fig. 1. When test 1 was repeated at a higher steady-state dissolver temperature of 138°C (test 2), the foil dissolved more rapidly, as expected. The higher final pressure was a result of the higher dissolver temperature.

To increase the uranyl nitrate concentration and decrease the nitric acid concentration so that they were close to those for tests 4 and 5 using LEU, we dissolved an appropriate mass of UO₂ particles in the stainless-steel dissolver containing HNO₃ with an initial concentration of 5.6M (test 11). The pressure-time curve (not shown here) indicated that dissolution was complete after 33 ± 11 min. As with the low-acid case for U foil, there was no pressure peak that would indicate a rapid self-heating reaction.

Barrier Metals

The use of 10-µm metal barriers between the U foil and the container walls is being considered because the U foil bonds to the container walls due to fission recoil during irradiation [4]. The barrier metal will be bonded to the U foil and will have to be dissolved. The overall dissolution reactions for metal barriers of Cu and Ni, and Fe using nitric acid alone are



Dissolution rates for these three metals were measured over a variety of conditions in a covered, but unsealed, centrifuge tube in a constant-temperature bath. The results, listed in Table 2, indicate that all three metals dissolve faster than U; Ni is five times faster, Cu is 190 times faster, and Fe is 560 times faster. Equations 2 and 3 and the ideal gas law were used in calculating the dissolution pressure for a two-sided barrier on a typical 18-g U-foil target with barrier dimensions of 76 x 102 x 0.010 mm (3 x 4 x 0.00039 in.), a dissolver temperature of 103°C, and a gas volume of 282 mL. The results indicate an increase in the final dissolver pressure of 23, 35, and 25 psig for Cu, Fe, and Ni, respectively, over that for U alone. Thus, the dissolver pressure will be increased about 10% by the presence of a two-sided barrier. This pressure increase is within the design limits of the stainless-steel dissolver, so that any of these three barrier materials can be used without affecting the operation of the U-foil dissolver.

In two tests, Ni foil was used in the closed (sealed) stainless-steel dissolver. The results of these tests were very similar. Their pressure-time plots (not shown here) indicated that the Ni dissolved easily, as expected. The final gas pressure for this piece of Ni foil was expected to be 159 psia. Instead, a much lower pressure, about 40 psia, was actually realized. The difference in these two pressures may be due to NO gas solubility in the dissolver solution.

CHANGES TO DISSOLVER DESIGN AND OPERATION

As the design of the Cintichem dissolver and the dissolver procedure are modified, there are three process constraints. First, the dissolution of the LEU-foil target must be rapid since the product ⁹⁹Mo has a short half-life (t_{1/2} = 66 h). The goal is to dissolve the target in less than one hour, while producing ⁹⁹Mo having a yield and purity comparable to that of the HEU product. Second, the dissolution must be done in a sealed vessel capable of withstanding the pressure generated by the release of gaseous reaction products. A sealed dissolver keeps the dissolution rate high and contains volatile fission products, so that they can subsequently be released in a controlled fashion.

Third, the LEU dissolver should be similar to the HEU dissolver so that existing dissolution facilities for the Cintichem process can be used with only minor modifications.

Table 2. Dissolution Rates for Metal Foil or Wire in Closed (Unsealed) Dissolver using either Nitric Acid Alone or Mixed Acids

| Mass of Metal Dissol'd, g | Nitric Acid Conc, <u>M</u> | Sulfuric Acid Conc, <u>M</u> | Soln Vol, mL | Dissol. Temp., °C | Time to Dissol., min | Avg. Dissol. Area, cm ² | Metal Dissol. Rate, mg/(min•cm ²) | Metal Dissol. Rate rel. to U Model |
|---------------------------|----------------------------|------------------------------|--------------|-------------------|----------------------|------------------------------------|---|------------------------------------|
| Copper | | | | | | | | |
| 0.115 | 8 | 0 | 4 | 70 | 0.23 | 2.00 | 250 | 230 |
| 0.11 | 8 | 0 | 5 | 70 | 0.31 | 2.00 | 177 | 163 |
| | | | | | | | | 194 ^a |
| Iron | | | | | | | | |
| 0.2572 | 8 | 0 | 3 | 53 | 2 | 0.46 ^b | 280 | 565 |
| Nickel | | | | | | | | |
| 0.011 | 0.8 | 0 | 3 | 70 | 12.5 | 2.54 | 0.35 | 18.8 |
| 0.014 | 1.2 | 0 | 3 | 70 | 8 | 3.16 | 0.55 | 15.7 |
| 0.014 | 1.6 | 0 | 3 | 70 | 7 | 3.12 | 0.64 | 11.3 |
| 0.011 | 2 | 0.5 | 3 | 70 | 4.3 | 2.45 | 1.04 | 8.2 |
| 0.012 | 3 | 2 | 3 | 70 | 1.5 | 2.78 | 2.88 | 1.4 |
| 0.012 | 4 | 0 | 3 | 70 | 3.3 | 2.62 | 1.39 | 4.7 |
| 0.011 | 4 | 0.5 | 3 | 70 | 2.58 | 2.52 | 1.69 | 4.1 |
| 0.011 | 4 | 2 | 3 | 70 | 1.3 | 2.54 | 3.33 | 0.8 |
| 0.012 | 6 | 0 | 3 | 70 | 1.6 | 2.68 | 2.80 | 4.5 |
| 0.014 | 7 | 0 | 3 | 70 | 1.25 | 3.08 | 3.64 | 4.3 |
| 0.011 | 8 | 0 | 3 | 70 | 0.78 | 2.54 | 5.55 | 5.1 |
| 0.0115 | 8 | 0 | 3 | 70 | 0.78 | 2.58 | 5.71 | 5.3 |
| | | | | | | | | 5.1 ^a |
| Uranium | | | | | | | | |
| 0.13 | 8 | 0 | 4 | 70 | 73 | 1.27 | 1.40 | 1.3 |

^aGeometric mean for metal dissolution ratios given above.

^bThis metal sample was a long, thin wire. Thus, the average area for this sample, which was used to calculate the dissolution rate, was one-half of the initial area. All other samples were thin pieces of metal foil so that the initial area (both sides) was the average and final area as well.

Design

Dissolver Opening - In the past, opening (and closing) the flanged stainless-steel dissolver has been done with four socket-head cap screws that go through the lid flange and into the threads in the dissolver-body flange [3]. Since the hex-bar wrench used to turn these cap screws must come down through the lid assembly, opening (and closing) is difficult to do with remote manipulators. An improved design was developed. In this design, a new dissolver body will be built that has four eye bolts attached to the underside of the dissolver-body flange. Both this flange and the lid flange will be slotted so that the bolts can swing into place with wing nuts or bar knobs already on the screw. In only two or three turns, each bolt can be brought up tight. This bar-knob design is preferred because any available bar (for example, the shaft of a screwdriver or the handle of a wrench) less than 11 mm (7/16 in.) thick can be used to turn the knob.

Cold Finger Pressurization - After dissolution, the gas generated must be removed before ⁹⁹Mo can be recovered from the solution. This is done by connecting the dissolver to a copper cold finger (cold trap). When 18 g of uranium is used as metal rather than UO₂, this creates a potential design

problem for the cold finger since the metal generates three times as much NO gas as the oxide. To estimate this pressure, the final dissolver pressure at 103°C and at 25°C is plotted in Fig. 4 for all the U and UO₂ dissolver tests reported above, as well as all of the earlier tests [3]. The model assumes that the dissolver (1) initially has air at 14.7 psia (760 mm Hg), (2) has the appropriate water/steam pressure for its temperature, (3) has 50 mL of solution, and (4) contains the amount of NO that would be released if the dissolution reaction is that given by Eq. 1. For the UO₂ test, the effective amount of U is taken to be one-third of the actual amount of U. The model works quite well at 103°C. However, at 25°C, the model pressure is above the observed pressures. This deviation is attributed to the solubility of NO in the dissolver solution. This solubility increases as temperature decreases.

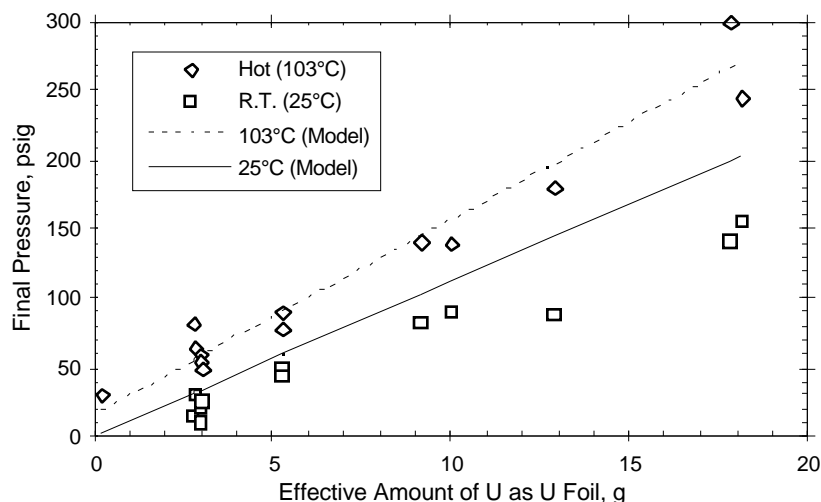


Figure 4. Effect of Uranium Mass on Final Dissolver Pressure at Room Temperature (R.T.) and 103°C

When the cold finger is in liquid nitrogen, it condenses the NO and fission-product gases so that there is a vacuum in the system consisting of the cold finger and the dissolver. However, when the cold finger is disconnected from the dissolver and allowed to come to room temperature, the maximum cold finger pressure will be 290 psig. This value is based on the relative volumes of the dissolver and the cold finger and a final dissolver pressure of 200 psig or less, which is taken from Fig. 4 for 18 g of U foil. To be sure the cold finger can take this pressure, it was subjected to pneumatic testing at 550 psig. This pressure is the allowable pressure for the cold finger and was calculated using an allowable stress of 6000 psi for annealed copper [6]. Based on the calculation, the existing cold finger can be used with 18 g of U foil. We have also shown that enough NO gas is removed by the cold finger to allow normal processing of the dissolver solution by the Cintichem process.

Operation

Twelve hours after the LEU is removed from the reactor, it is dissolved. At that time, the heat (thermal power) from this 5-day irradiated foil will be 4.0 W/g. With this thermal power, a total of 72 W for 18 g of LEU, the foil, which is 76 x 102 x 0.127 mm (3 x 4 x 0.005 in.), will have a temperature between 230 and 330°C in air. Inside the dissolver, the foil will be somewhat hotter, 252 to 440°C. When solution is added to the dissolver, it will be heated up by the foil at the rate of 5.1°C/min if there is 41 mL of solution, 2.8°C/min if there is 80 mL of solution. To prevent uranium foil dissolution from starting prematurely, that is, while solution is still being added to the dissolver, the dissolver will be put into liquid nitrogen before solution is added. After the solution is added, the valve to the septum through which the dissolver solution was added will be closed. The dissolver will then be removed from the liquid nitrogen and put into the rotation jig for normal heating. By following this procedure, it is expected that premature dissolution can be avoided.

Detailed analysis of this process will be done to ensure that each step will go as outlined here. The procedure will then be tested with 18 g of irradiated LEU foil at BATAN.

FUTURE DIRECTIONS

Future work includes (1) designing, building, and testing the improved design to open and close the dissolver, (2) completing an analysis of the cooling procedure for the irradiated foil, and, (3) testing the modified dissolver with 18 g of LEU foil. If the dissolver works as expected, the amount of LEU foil required for production runs will be determined, and a dissolver will be designed for that mass of LEU foil.

SUMMARY

Previous work that involved dissolving up to 18 g of uranium foil in a modified Cintichem dissolver with $3M$ HNO_3 and $2M$ H_2SO_4 has been extended to the use of $8M$ HNO_3 alone. Additional work showed how (1) decreasing HNO_3 concentration decreases the U-foil dissolution rate, (2) increasing the steady-state dissolver temperature increases the dissolution rate, (3) foil dissolution is complete when the dissolver pressure passes through a maximum or reaches a steady value, (4) HNO_3 alone can also be used to dissolve UO_2 particles, (5) solution volume and dissolver orientation can affect both the onset of self-heating and the peak pressure, and (6) barrier metals of Cu, Fe, or Ni can be used on the surface of the U foil. Work on the dissolver design and operation showed how (1) the dissolver can be opened and closed more quickly, (2) the present cold finger can be used for tests with 18 g of irradiated LEU foil, (3) evacuation of the dissolver before heating and NO gas removal by an evacuated cold finger after dissolution are required if the normal Cintichem process is to be used, and (4) liquid nitrogen will be needed to cool the dissolver before solution is added to the irradiated foil. When the analysis of this last operation is completed, we will be ready to demonstrate the modified Cintichem process with 18 g of irradiated LEU foil.

ACKNOWLEDGMENTS

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