

**PROGRESS IN DEVELOPING PROCESSES FOR CONVERTING ⁹⁹Mo PRODUCTION
FROM HIGH- TO LOW-ENRICHED URANIUM-1998**

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ABSTRACT

During 1998, the emphasis of our activities was focused mainly on target fabrication. Successful conversion requires a reliable irradiation target; the target being developed uses thin foils of uranium metal that can be removed from the target hardware for dissolution and processing. This paper describes successes in (1) improving our method for heat-treating the uranium foil to produce a random small grain structure, (2) improving electrodeposition of zinc and nickel fission fragment barriers onto the foil, and (3) showing that these fission fragment barriers should be stable during transport of the targets following irradiation. A method for quantitatively electrodepositing uranium and plutonium contaminants in the ⁹⁹Mo was also developed. Progress was also made in broadening international cooperation in our development activities.

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INTRODUCTION

To reduce nuclear-proliferation concerns, the U.S. Reduced Enrichment for Research and Test Reactors (RERTR) Program is working to reduce the use of high-enriched uranium (HEU) by substituting low-enriched uranium (LEU) fuel and targets. Low enriched uranium contains <20% ^{235}U . Currently most of the world's supply of ^{99}Mo is produced by fissioning the ^{235}U in HEU targets; generally 93% ^{235}U . Targets for the production of ^{99}Mo are generally either (1) miniature Al-clad fuel plates [1-9] or pins [10,11] containing U-Al alloy or UAl_x dispersion fuel or (2) a thin film of UO_2 coated on the inside of a stainless steel tube [12-14]. After irradiation the ^{99}Mo is separated from the uranium and fission products. First the target is either completely digested in alkaline solution for the U-Al alloy/ UAl_x dispersion targets or by dissolving the UO_2 off of the stainless steel tube using acid. Then the solution goes through a series of purification steps to achieve the final ^{99}Mo product. Each producer has its own process, and the highly competitive nature of the business and the stringent regulations governing the production of radiopharmaceuticals make the producers reluctant to change their processes.

Our program is investigating the ramifications of switching to an LEU target for the production of ^{99}Mo . To yield equivalent amounts of ^{99}Mo , an LEU targets must contain five times as much uranium as an HEU target. Substituting LEU for HEU will require changes to both target design and chemical processing. Three major challenges have been identified with substituting LEU for HEU: (1) modify the targets and purification processes as little as possible, (2) assure continued high yield and purity of the ^{99}Mo product, and (3) limit economic disadvantages.

In order to keep the target geometry the same between HEU and LEU targets requires using a denser form of uranium in order to increase the amount of uranium per target by a factor of five. It may be possible to use a highly dense LEU metal foil target as a replacement for HEU for both the acid-dissolution and basic-digestion processes. Our development activities with acid-side processing are furthest along. The irradiation and acid-side processing of the LEU metal-foil targets are being demonstrated in cooperation with researchers at the Indonesian PUSPIPTEK facility. Our recent work concerning preparation of the LEU targets for ^{99}Mo production is discussed in this paper.

One issue raised with using LEU to produce ^{99}Mo is the increased amount of ^{239}Pu generated. The ^{239}Pu is generated through neutron capture by the ^{238}U . Since the LEU targets contain significantly more ^{238}U than HEU targets about 30 times more ^{239}Pu is generated for an equivalent ^{99}Mo yield. The increased amount of ^{239}Pu increases the alpha activity in the irradiated target. However, HEU contains significantly more ^{234}U than LEU, which increases the alpha activity in HEU targets significantly. This increase in ^{234}U in HEU is a consequence of the enrichment process. Therefore, when comparing the total alpha activity of an irradiated targets the LEU is only ~20% higher than an equivalent HEU target. Nevertheless, because of this perception, we have worked on measuring the amount of alpha activity in the ^{99}Mo product to verify that product purity specifications for alpha activity can be met. This work is summarized below.

International cooperation is the key to the success of this effort. Our cooperation with international partners is discussed briefly. Conclusions and future work are discussed in the final section.

LEU TARGET FABRICATION

The use of an LEU metal foil target may serve as a replacement for HEU in both the acid-dissolution and basic-digestion processes. The target as currently designed is schematically shown in figure 1. The LEU target consists of a piece of uranium foil held between two concentric tubes. After assembly, end plugs are welded into the target and the center void space is back filled with helium. The target is then irradiated. After irradiation, the ends are cut-off, the inside tube pushed out, and the foil

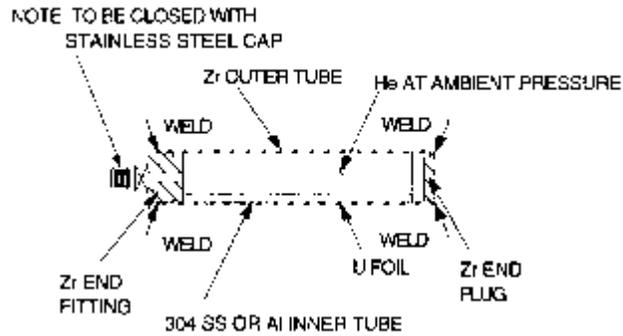


Figure 1. Schematic of LEU Foil Target

uranium recovered for processing. Several targets of this design were successfully tested at the Indonesian PUSPIPTEK facility. Preparation of the targets for this demonstration are discussed below and results from the demonstration are discussed elsewhere [15].

We have identified three issues with this target design, which were addressed for the targets fabricated for the demonstration in Indonesia. The first issue is that fission fragment recoils during the irradiation cause the uranium foil to bond to the inner and outer tubes making it impossible to slide the tubes apart and recover the uranium foil for processing. Fission fragment barriers need to be added to the uranium to prevent the uranium from bonding to the tubes [16]. The fission fragment barriers must be >7 μm thick for zinc and nickel and $>14\mu\text{m}$ thick for aluminum. We have developed a method to electrodeposit either Ni or Zn barriers onto the foil. The foil can also be wrapped in thin foils of Ni, Zn, or Al. The second issue is the anisotropic grain growth in the uranium foil. The growth can tear the fission fragment barriers during irradiation, which then allows the uranium to bond to the target tubes--preventing the ability to recover the uranium foil. We have refined a method to beta-quench these very-thin uranium foils to produce a fine, randomly oriented grain structure that prevents the tearing of the fission fragment barriers. The third issue is that the uranium foil can react with the fission fragment barriers, which may compromise their effectiveness. We have studied the reaction of the various fission fragment barriers with uranium under conditions that may occur during transport of the foils.

Heat Treatment

The last step in the fabrication of uranium foils is cold rolling to the final thickness (130 μm). This cold rolling induces preferred orientation of the crystal structure in the uranium foil. During 1998, we perfected our method for β -quenching these thin foils to produce a fine, randomly oriented grain structure. As mentioned previously a fine randomly oriented grain structure is required to prevent tearing the fission fragment barriers. After heat treatment the foils can either be electroplated or wrapped before final assembly of the targets.

The foils prepared at ANL for the demonstration in Indonesia were made of an "adjusted" uranium alloy, containing 1000 ppm aluminum and 450 ppm iron. To produce fine-grained material, the piece needs to be heated into the β region ($T>668^\circ\text{C}$) and then rapidly cooled. We used a molten-lead bath to heat-treat the foils. Molten lead is an effective means of heat-treating; it has good heat transfer characteristics and a large thermal inertia. To minimize the oxidation of the molten lead, an argon cover gas was used when the bath was hot. The uranium foils were individually sealed inside a can fabricated by beam welding two pieces of 0.015 in. (380 μm) thick stainless-steel sheet with a uranium foil sandwiched in between. The welded cans also make an ideal storage container for the heat-treated uranium foils by preventing oxidation during storage.

Test sets were prepared with samples that contained 30%, 40%, 50%, and 60% cold work. One set was heat-treated for 20 minutes at 700-720°C and then water quenched. A second set was heat-treated for 20 minutes at 700-720°C, water quenched, heat-treated for an additional 20 minutes at 700-720°C, and finally water quenched. These two sets of samples were then compared to samples that were not heat-treated. The samples were analyzed using X-ray diffraction (XRD) to determine how effective the heat treatment was at removing preferred orientation. After taking an XRD spectrum, the sample was rotated and another spectrum taken. Ratios of the various reflections were then compared to a reference spectrum of unoriented uranium. Variation in the ratio as the sample was rotated and deviation from the reference value indicates that preferred orientation is present.

Inspection of the XRD data showed that the ratio of the hkl reflections 111 and 113 were sensitive to preferred orientation. Table 1 gives the results of this ratio from all of the samples (Note: the reference value of unoriented uranium for 111/113 equals 6). Notice that the 111/113 ratio varies greatly from 6 in the samples that were not heat-treated. The ratio also varied as the samples were rotated. In the 50% and 60% cold-worked materials, a single 20-minute heat treatment was sufficient to remove the preferred orientation. However, in the 30% and 40% cold-worked materials, some preferred orientation was still present after the first heat-treatment but was removed by a second heat-treatment. The large amounts of strain in the 50% and 60% cold-worked pieces make it easy for the material to recrystallize, which is why one heat treatment was sufficient to remove the preferred orientation. This was not the case for the 30% and 40% cold-worked material. Overall, the double heat treatment process will remove the preferred orientation regardless of the starting amount of cold work.

Table 1. Results from XRD Analysis (Ratio of 111/113 Reflections)

| 60% CW | XRD Position | | | | | | Average |
|--------|--------------|------|------|------|------|------|----------|
| | 0° | 30° | 60° | 90° | 180° | 270° | |
| No HT | 1.52 | 1.21 | 1.10 | 0.80 | 0.67 | 0.92 | 1.04±30% |
| HT(x1) | 6.20 | 5.84 | 5.93 | 6.74 | 5.98 | 5.57 | 6.04±7% |
| HT(x2) | 5.61 | | | 5.90 | | | 5.76±4% |
| 50% CW | 0° | 30° | 60° | 90° | 180° | 270° | Average |
| No HT | 0.43 | | | 0.46 | | | 0.44±5% |
| HT(x1) | 5.52 | | | 6.15 | | | 5.84±8% |
| HT(x2) | 6.93 | | | 5.57 | | | 6.25±15% |
| 40% CW | 0° | 30° | 60° | 90° | 180° | 270° | Average |
| No HT | 1.73 | | | 1.57 | | | 1.65±7% |
| HT(x1) | 4.92 | | | 3.74 | | | 4.33±19% |
| HT(x2) | 6.44 | | | 5.48 | | | 5.96±11% |
| 30% CW | 0° | 30° | 60° | 90° | 180° | 270° | Average |
| No HT | 3.75 | | | 4.13 | | | 3.94±7% |
| HT(x1) | 4.53 | | | 4.29 | | | 4.41±4% |
| HT(x2) | 5.83 | | | 6.33 | | | 6.08±6% |

CW=Cold Work
HT=Heat Treatment

After being analyzed by XRD, the samples were etched to determine grain size by microscopy. Etching was performed by making the sample the anode in an electrolyte containing 500-g/L trichloroacetic acid at a current of ~1-2 A/cm². The sample is etched until uniform, thick, black smut

forms on the surface. After the smut is formed, it is removed by dissolution in $8M$ HNO_3 . After removing the smut, the grain structure is visible. Only the two extreme cases are shown here (30% and 60% cold work). Figure 3 shows the grain structure of the uranium foil containing 30% cold work before and after the double heat treatment. Figure 4 shows the grain structure of the uranium foil containing 60% cold work before and after the double heat treatment. It can be seen in these figures that as the amount of cold work increases, the grain structure becomes more damaged (i.e., at 60% cold work, the grains are hardly defined before heat treatment). However, after two heat treatments, the grains are well defined and small $\sim 0.1mm$. A grain size of $0.1mm$ is sufficiently small to prevent tearing the fission fragment barriers. The uranium foils placed in targets for the August irradiations in Indonesia were heat-treated using the double heat treat process.

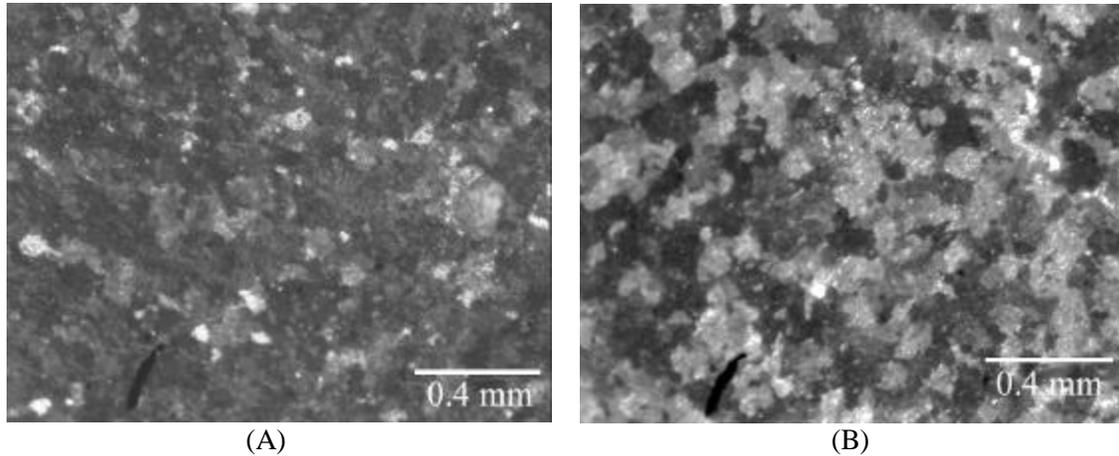


Figure 2. Uranium Foil Cold Worked 30%; (A) No Heat Treatment; (B) Double Heat Treated

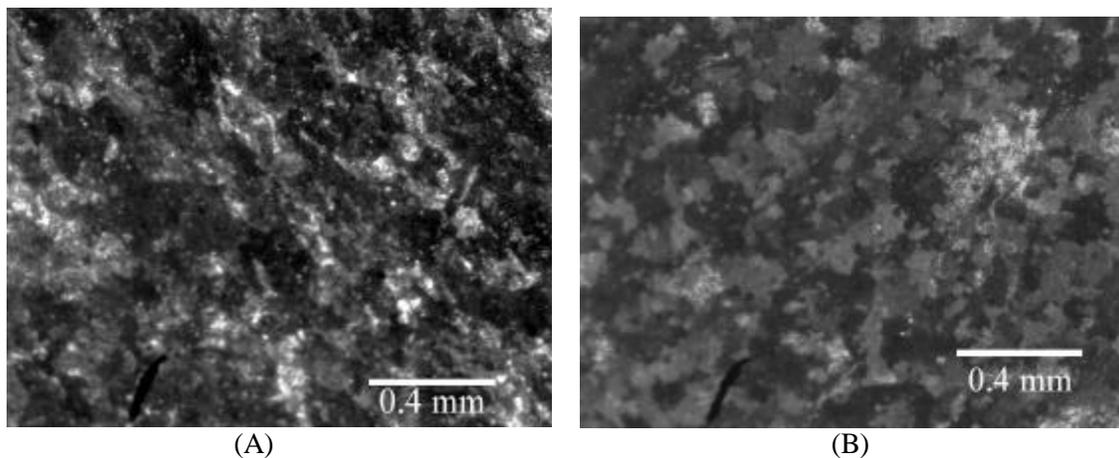


Figure 3. Uranium Foil Cold Worked 60%; (A) No Heat Treatment; (B) Double Heat Treated

Electroplating

The foil surface preparation and electroplating techniques are described elsewhere[17]. In general though, the foils were degreased in trichloroethylene in an ultrasonic cleaner for three minutes, then rinsed successively in methyl alcohol and water to remove any organic material. The foil was then immersed in $8M$ HNO_3 to remove the oxide coating. After the oxide layer was removed, the foil was rinsed with water and then etched in a $5.33M$ $FeCl_3$ solution at $40^\circ C$. The foil was then rinsed with water

in an ultrasonic cleaner. After etching the foil was weighed, its thickness was measured, and then it was immersed in 8M HNO₃ for 15 seconds to activate the surface before being electroplated.

After heat treatment, two foils were plated for the demonstration in Indonesia. One was plated with nickel and the other with zinc; the plated thickness targets were 10 μm and 15 μm respectively. Plating was done at a current density of 20 mA/cm² and the electroplating time was adjusted to obtain the desired plated thickness. The thicknesses of the electroplated foils were measured by using a micrometer and by weighing before and after etching and after plating. The micrometer-caliper measurements are made by measuring at 10 – 12 locations around the foil and vary by ± 8% at all stages of plating. The uranium mass losses from etching were 4.9 and 5.8% for the two foils. The final thicknesses of the fission fragment barrier foils used for the demonstration in Indonesia are shown in Table 2.

| Plate | Expected Thickness, μm | Thickness by Micrometer, μm | Thickness by Mass, μm |
|-------|------------------------|-----------------------------|-----------------------|
| Zn | 15 | 21 | 17.1 |
| Ni | 10 | 9 | 10.9 |

Fission Fragment Barrier Reaction with Uranium

Rough calculations showed that it may be possible for the target to reach a temperature of 375°C during transport. We were concerned that this may cause problems in target disassembly because of reaction of the barrier with the uranium. Samples used to investigate the reaction of fission fragment barrier materials were prepared by compressing metal powder (either aluminum or zinc containing less than one weight percent oxide) around a piece of DU foil which was wrapped with nichrom wire to mark the interface. The samples were compressed at ~60,000 psi for thirty seconds in a die. Figure 5 (A) shows a sample compact. Interaction with nickel fission fragment barriers was studied using samples that had been electroplated with nickel. The samples were then sealed under rough vacuum in aluminum tubing. Reaction of the barriers with the uranium was then simulated by heating the sample.

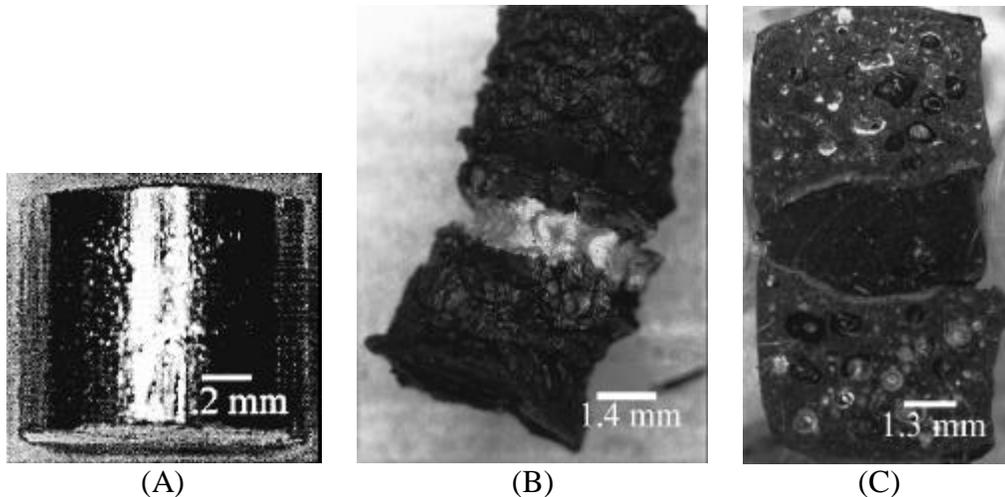


Figure 4. Sample Zn/U Compact with (A) No Heat-Treatment and Following Reaction of Zinc with Uranium at 450°C for (B) 16 and (C) 24 Hours

Nickel and aluminum did not react with uranium foil at temperatures up to 450°C. Zinc-uranium reactions at temperatures below 375°C are minimal. At a temperature of 400°C, the zinc and uranium begin to react. The uranium in these samples is completely consumed at temperatures near or above the melting point of zinc (419.6°C). Figure 5 (B) shows the extensive reaction of zinc with uranium; this sample was heated at 450°C for 16 hours. Figure 5 (C) shows a section of the reaction product of zinc with uranium; this sample was heated at 450°C for 24 hours. Samples of the reaction product were analyzed using ICP-AES. The product typically contained 8 - 10 w% with the remainder zinc. The zinc-uranium-reaction products were analyzed by Electron Dispersive Spectroscopy (EDS), which indicates that some previously unpublished phases may have been formed. However, reaction of the fission fragment barriers with uranium should not be a problem under normal irradiation and transport conditions.

MEASUREMENT OF ALPHA ACTIVITY CONTAMINATION IN ⁹⁹MO PRODUCT

The product purity standards for alpha contamination in ⁹⁹Mo are very stringent. Since LEU targets contain significantly more ²³⁸U than HEU targets, they generate about 30 times more ²³⁹Pu for an equivalent yield of ⁹⁹Mo. The increased amount of ²³⁹Pu increases the alpha activity in the irradiated target; thus, meeting the product purity for alpha contamination may be more difficult. However, HEU contains significantly more ²³⁴U than LEU, which increases the alpha activity in HEU targets significantly. Therefore, the total alpha activity of irradiated LEU targets is only ~20% higher than for an equivalent HEU target. Although the total alpha activity in an irradiated LEU sample is not significantly different from that in a comparable HEU target, the great concern over plutonium has made the measurement of alpha contamination an essential component of our efforts to convert ⁹⁹Mo production to LEU. We are investigating an electrodeposition technique that will allow us to measure very low levels of alpha activity from the ⁹⁹Mo product solutions using alpha pulse height analysis.

In this method a sample of the ⁹⁹Mo product solution is added to an electrolyte containing ammonium bioxalate and ammonium chloride. The uranium and plutonium are then electrodeposited at ~0.15 A/cm² onto a polished 1 in. (2.5cm) diameter stainless steel planchet. After electrodeposition, the planchet is counted in an alpha spectrometer to determine the alpha activity in the sample of the ⁹⁹Mo product solution. We studied the yield of uranium and plutonium on the planchet as a function of electrodeposition time. We also investigated the effects of additional mass and beta activity from ⁹⁹Mo on the analysis of the sample.

Figure 6 shows the yield of uranium and plutonium as a function of electrodeposition time from electrolyte containing 0.08M ammonium bioxalate / 1M ammonium chloride. As seen in figure 6, the yield of uranium and plutonium are greater than 80% for electrodeposition times longer than 90 minutes. It is also shown that the yield of plutonium and uranium are not affected by the addition of molybdenum (as both stable molybdate and ⁹⁹Mo). However ⁹⁹Mo does show up in the alpha spectrum of the sample. Figure 7 shows the alpha spectrum of a sample containing ⁹⁹Mo and a sample that does not contain ⁹⁹Mo. As seen in figure 7 beta activity from ⁹⁹Mo can be detected using the alpha spectrometer. In fact, the shapes of the uranium and plutonium alpha peaks are different in figures 7A and 7B. This change is due to coincidence summing of an alpha and beta particle in the detector. While coincidence summing will not affect the measured alpha activity, it will distort the shape of the peaks and tend to broaden them. However coincidence summing of multiple beta particles from the ⁹⁹Mo may cause a general increase in the background activity of the instrument in the alpha sensitive region. This may cause a decrease in sensitivity of the analysis. Further investigation into the coincidence summing of multiple beta particles needs to be completed. However in general this method looks very promising for the measurement of very-low levels of alpha activity in the ⁹⁹Mo product.

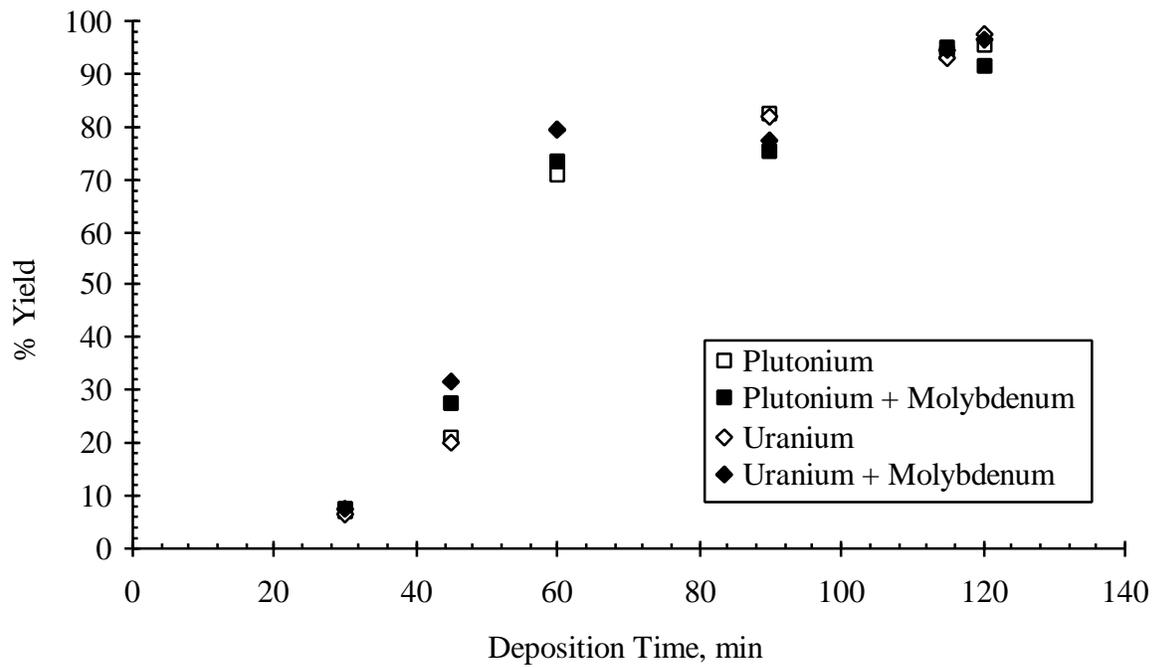


Figure 5. Uranium/Plutonium Yield as a Function of Electrodeposition Time

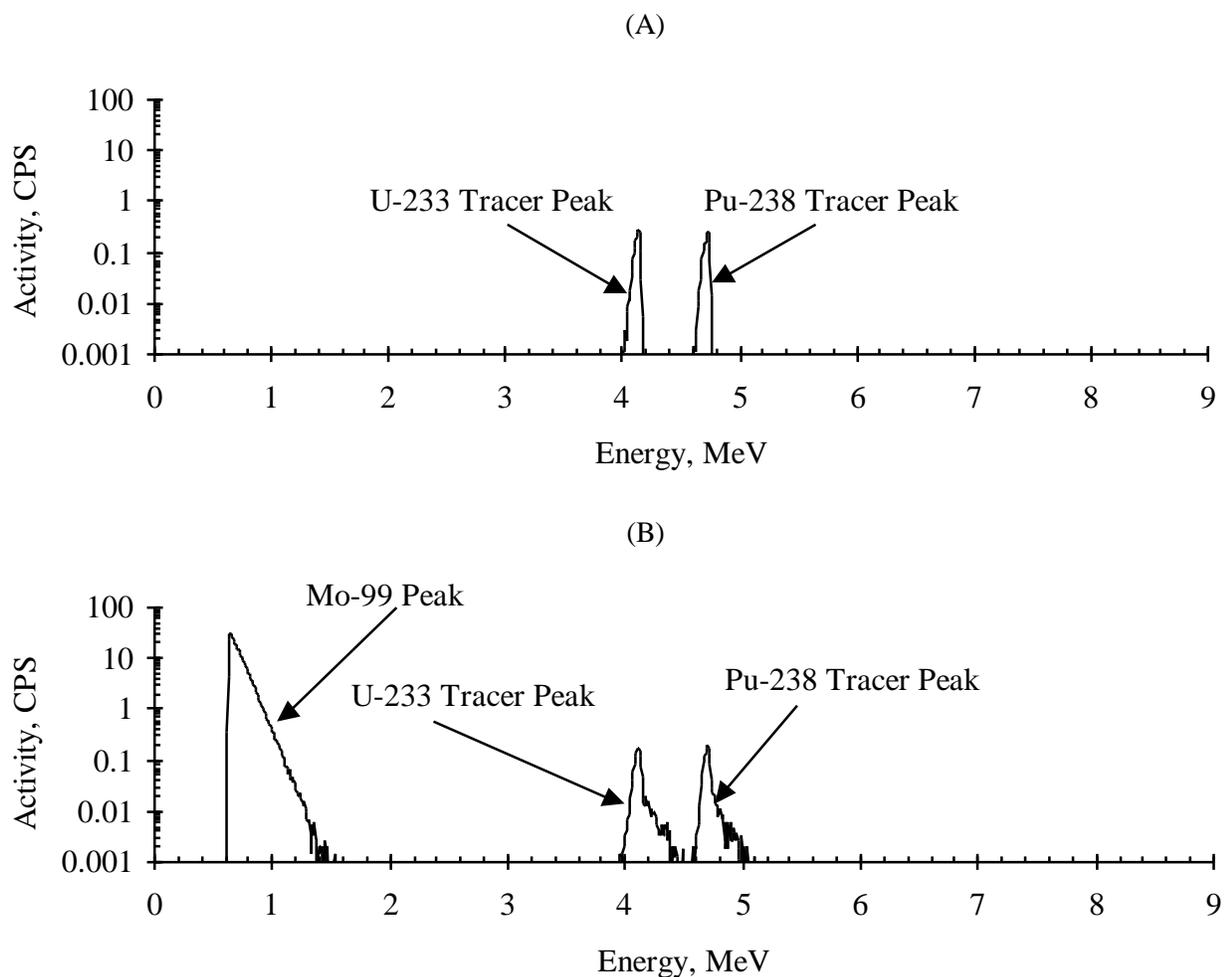


Figure 6. Spectrum of Electrodeposited Sample (A) with out ^{99}Mo (B) with ^{99}Mo

INTERNATIONAL COOPERATION

International cooperation is the key to the success of this effort. We continue to work on several fronts in that area. Our cooperation with the Indonesian Badan Tenaga Atom Nasional (BATAN) is moving forward. The first two of a series of full-scale demonstrations of the target and process were completed in August 1998 [15]. A cooperation agreement with the Argentine Comisión Nacional de Energía Atómica (CNEA) and Argonne National Laboratory is near completion. The technical areas of the agreement were easily settled; intellectual property concerns have taken more time. We expect the full agreement to be in-place by the end of 1998. We are also in the planning stage of a cooperation with the Australian National Science and Technology Organization (ANSTO) which will provide the opportunity to develop optimized target designs. Both organizations anticipate full cooperation to begin in the next few months. The Institut National des Radioéléments (IRE) in Belgium has volunteered to cooperate with Argonne National Laboratory in future activities to test the irradiation and processing of an LEU metal target. We are looking for this cooperation to follow development of a base-side target and processing with the Argentines.

CONCLUSIONS AND FUTURE WORK

Testing and development activities are continuing on using LEU targets for the production of ^{99}Mo . The three issues we identified with current target design have been addressed. The success of the target in the demonstration at the Indonesian BATAN facility showed the LEU target using metal foil is a viable target for the production of ^{99}Mo .

We are also developing a method that looks promising for the determination of the alpha activity contamination in the ^{99}Mo product. The method should be more reliable and more accurate than the standard Cintichem method.

Securing the Argentine CNEA as a commercial partner for base-side processing will greatly assist in (1) refining our design of the dissolution system, (2) focusing our development activities and (3) demonstrating processing of irradiated full-scale targets. The RERTR Program wishes to work with all current and future producers of ^{99}Mo to assure that, ultimately, no HEU is needed for ^{99}Mo production.

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