Radiochemical Purity of Mo and Tc Solution Obtained after Irradiation and Dissolution of Mo-100-Enriched and Ultra-High-Purity Natural Mo Disks

Nuclear Engineering Division

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RADIOCHEMICAL PURITY OF MO AND TC SOLUTION OBTAINED AFTER IRRADIATION AND DISSOLUTION OF MO-100-ENRICHED AND ULTRA-HIGH-PURITY NATURAL MO DISKS

ABSTRACT

Four irradiations of ultra-high-purity natural Mo targets and one irradiation using 97.4% Mo-100-enriched material were performed. The purpose of these irradiations was to determine whether the presence of Sn stabilizer in the H_2O_2 used for the dissolution of sintered Mo disks can affect the radiochemical purity of the final K_2MoO_4 in 5M KOH solution. Results from radiochemical purity tests performed using thin-layer paper chromatography show that even 2–3× excess of Sn-stabilized H_2O_2 typically used for dissolution of sintered Mo disks did not affect the radiochemical purity of the final product.

1 INTRODUCTION

In 2015, a series of production irradiations were performed using enriched molybdenum-100 and natural molybdenum (Mo) [1,2]. A fine orange-brown powder was observed on the irradiated Mo disks and target housing. This powder was possibly due to corrosion of the carbon-steel shielding enclosure or the helium cooling loop. Irradiated targets were dissolved in tin (Sn)-stabilized hydrogen peroxide (H_2O_2 ; 50%), converted to ~5M potassium hydroxide (KOH), and filtered through a 0.3 μm filter. The resulting solution was light orange in color—indicating the presence of impurities, most likely iron (Fe). Radiochemical purity (RCP) tests using thin-layer chromatography (TLC) performed with the undiluted Mo solution [3,4] did not meet the specifications of $\geq 95\%$ of activity at $R_f = 0.9 \pm 0.1$ (R_f = retention factor). Therefore, in FY 2016, several irradiations were undertaken with ultra-high-purity (UHP) natural Mo and Mo-100-enriched disks without using the helium cooling system. The Mo targets were wrapped in aluminum foil and placed behind a converter. These tests were performed to investigate whether the presence of Sn introduced during the dissolution process (introduced by Sn-stabilized H_2O_2) affected the radiochemical purity of Mo and technetium (Tc) in the final product or if the inability to meet specifications was caused by another factor.

2 EXPERIMENTAL

Linac Irradiations

Argonne’s low-energy and high-power electron linac was used to irradiate the Mo targets for these experiments. This machine operates with repetition rate up to 240 Hz with ultimate beam power up to 110 J per pulse. The effective beam energy is in the range of 20–45 MeV. The

highest beam energy is about 50 MeV. A DC thermal gun produces an electron beam with amplitude up to 2.0 A and length of about 5.5 μ s. Radio frequency (RF) power is provided by two Thales TV2022A klystrons. After acceleration, the beam travels through the transport channel to the experimental hall and is delivered to the target. Steering coils and quad magnets keep the beam in proper shape and position [5].

Molybdenum irradiation runs were performed with a 40 MeV electron beam. The spectrometer ultimate range is 40 MeV, therefore the beam energy profile was optimized at a lower energy range. After that the beam energy was increased to 40 MeV based on load-line experimental data.

The typical beam energy spectrum for these experiments is shown in Figure 1. The energy spread was about ± 0.6 MeV or $\pm 1.6\%$. Since the use of convertor does not require a small beam spot size, ordinary focusing settings were used. The transverse beam size was elliptical, with a width of 5 mm and height of 3 mm. The accelerated beam pulse current was about 0.49 A. The irradiation runs were performed with repetition rate up to 60 Hz, the average beam current was up to 135 μ A, and the average beam power was 5.4 kW.

Parameters for all four runs are presented in Table 1. In the first run (04/24/16), the measured temperature reading was low because thermocouples were attached at some distance from the irradiated sample and close to the cooling water lines. In the second run (05/01/16), the physical contact of the first thermocouple was not sufficient, so the measured values were sufficiently lower than the actual temperature. In the next experiments, the applied power level was used to keep the temperature of measurements points in the range of 160–200°C.

The convertor was water cooled with a flow rate of 3 gpm. To control the temperature of the window, two 1/16 in. thermocouples were installed at the tested window holder. Various configurations for the irradiation runs are presented in Figure 2. After the beginning of the irradiation, the temperature rose to about 95% of its maximum value, which corresponds to the beam average power. After that, it asymptotically climbed to stable values in approximately one hour (Figure 3). The ultimate temperatures measured for all of the irradiation runs are presented in Table 1. A photograph of the experimental setup is shown in Figure 4. Details of the tungsten (W) converter are shown in Figure 5.

TABLE 1 Irradiation run parameters

Date	Repetition Rate (Hz)	Average Current (μ A)	Average Power (kW)	Duration (hh:mm)	T ₁ (°C)	T ₂ (°C)
04/24/16	60	133	5.4	07:50	75.0	48.9
05/01/16	60	135	5.4	10:30	67.6	164.1
05/08/16	60	135	5.4	10:20	200.8	131.0
07/31/16	44	100	4.4	05:25	183.0	118.0

FIGURE 1 Beam energy spectrum

FIGURE 2 Thermocouple placement at the sample (back view)

FIGURE 3 Thermocouple measurement history for 07/31/2016 run

Four irradiations of UHP natural Mo targets and one irradiation using 97.4% Mo-100-enriched material (Trace Science) were performed in FY 2016. The irradiation conditions and masses of the Mo targets are listed in Table 1.

FIGURE 4 Mo target wrapped in aluminum foil and placed against W converter

FIGURE 5 Exploded view of the x-ray converter. Cooling of the converter is provided by water flow between 1-mm-thick W plates

Dissolution

Irradiated Mo disks were dissolved in either 50% H₂O₂ stabilized with Sn (Aqua Solutions H3050-4L) or 30% nonstabilized H₂O₂ (Acros Organics 411885000) to investigate whether the presence of Sn as a stabilizer affects the radiochemical purity of the product by interaction of Sn with Mo and/or Tc. After complete dissolution, KOH (Alfa Aesar 44273) was added to convert the Mo to potassium molybdate (K₂MoO₄) and obtain a 5M KOH solution. Excess H₂O₂ was eliminated by evaporation, and the final volume was adjusted by adding water to make the solution 0.2 g Mo/mL solution. Impurities in the Mo-100-enriched material (540 ppm Fe, 75 ppm W, 65 ppm Cr, 39 ppm Ni, 15 ppm Cu, and 11 ppm Ge) caused an orange precipitate to form after addition of the KOH; therefore, after cooling, the solution was filtered through a 0.3 µm filter. Figure 6 is a photograph of the dissolution apparatus.

FIGURE 6 Apparatus used for dissolution of irradiated Mo-100-enriched target in hot cell

Radiochemical Purity Test

TLC was performed with ITLC-SG strips (Agilent Technologies SGI0001) and 0.1M sodium carbonate (Na₂CO₃) as a mobile phase (unless otherwise noted). A few microliters of the Mo solution were added to the paper and allowed to dry. Chromatograms were developed in 0.1M Na₂CO₃ solution. Since the solution is colorless, the front of the TLC was marked with a droplet of phenolphthalein so experimenters could observe when the mobile phase reached the front of the chromatogram. This was indicated by a purple color on the front of the chromatogram (Figure 7). After development, the TLC strips were allowed to dry, cut into 11 fractions, and gamma counted. Gamma counting was performed with a high-purity germanium (HPGe) detector.

FIGURE 7 Spotting of mobile phase (Na_2CO_3) on TLC using phenolphthalein

3 RESULTS AND DISCUSSION

The final solutions prepared from UHP disks were clear and colorless. Solution obtained after first irradiation on 4/24/16 was filtered through a 0.3 μm filter before gamma counting. Adding KOH to the solution prepared from Mo-100-enriched material led to formation of an orange precipitate (due to impurities; see Experimental section), which was removed by filtration. Clear and colorless solutions were obtained and sent to NorthStar for further analyses. Gamma counting results from solutions obtained after dissolution of irradiated Mo targets in H_2O_2 and conversion to 0.2 g Mo/mL in 5M KOH solution are presented in Tables 2–6.

TABLE 2 Gamma counting results for irradiated 1.13 g of UHP Mo disk with EOB 4/24/16 at 6 pm

Radionuclide	Total EOB (mCi)	1-Sigma Uncertainty	% of Mo-99
Mo-99	16.48	3.2%	
Mo-90	2.82	4.0%	17.1%
Nb-95m	0.79	4.0%	4.8%
Nb-95	0.11	5.5%	0.7%
Nb-96	2.34	3.2%	14.2%

EOB – end of bombardment

TABLE 3 Gamma counting results for irradiated 1.05 g of UHP Mo disk with EOB 5/01/16 at 6 pm

Radionuclide	Total EOB (mCi)	1-Sigma Uncertainty	% of Mo-99
Mo-99	15.32	3.1%	
Mo-90	1.83	3.2%	11.9%
Mo-93m	0.01	16.8%	0.1%
Nb-95m	0.79	3.3%	5.2%
Nb-95	0.07	1.9%	0.5%
Nb-96	1.98	3.1%	12.9%

EOB – end of bombardment

TABLE 4 Gamma counting results for irradiated 1.06 g of UHP Mo disk with EOB 5/08/16 at 6 pm

Radionuclide	Total EOB (mCi)	1-Sigma Uncertainty	% of Mo-99
Mo-99	13.54	3.1%	
Mo-90	2.16	3.5%	16.0%
Nb-95m	0.72	3.4%	5.3%
Nb-95	0.06	3.5%	0.4%
Nb-96	1.89	3.2%	14.0%

EOB – end of bombardment

TABLE 5 Gamma counting results for irradiated 1.03 g of UHP Mo disk with EOB 5/08/16 at 6 pm

Radionuclide	Total EOB (mCi)	1-Sigma Uncertainty	% of Mo-99
Mo-99	16.11	3.1%	
Mo-90	2.49	3.5%	15.5%
Nb-95m	0.83	3.4%	5.2%
Nb-95	0.07	2.6%	0.4%
Nb-96	2.22	3.1%	13.8%

EOB – end of bombardment

TABLE 6 Gamma counting results for irradiated 2.09 g of 97.4% Mo-100-enriched disks with EOB 7/31/16 at 3:45 pm

Radionuclide	Total EOB (mCi)	1-Sigma Uncertainty	% of Mo-99
Mo-99	123.2	0.10%	
Mo-93m	0.04	58.4%	0.03%
Nb-95m	0.05	21.8%	0.04%
Zr-95	0.01	16.8%	0.01%

EOB – end of bombardment

When comparing gamma counting results for filtered samples (Tables 2 and 6), it is obvious that Nb was not removed when Mo in 5M KOH solution was filtered with no resultant precipitation of Fe—very similar proportions of Nb isotopes to Mo-99 were observed as in the unfiltered samples (Tables 3–5). When Fe precipitate formed during processing of an Mo-100-enriched target and was filtered, a significant portion of Nb was removed, which is indicated by a much lower relative percent of Mo-99 activity (compare Tables 2–5, where Nb-95m activity was ~5% of Mo-99, with Table 6, where Nb-95m activity was only 0.04% of Mo-99 when Fe precipitate was filtered).

Paper chromatograms obtained from solution of processed UHP disks irradiated on 04/24/16 are shown in Figures 8 and 9. It is evident that when 0.2M HCl in acetone is used as a mobile phase, the retention factor of Mo (present in macro concentration) is significantly affected. This effect is due to conversion of MoO_4^{2-} species, which are present in highly alkaline solution, to molybdenyl species MoO_2^{2+} , which predominate under acidic conditions. For subsequent tests, therefore, Na_2CO_3 solution was used as the mobile phase.

Figures 10 and 11 show TLC for a sample irradiated on 05/06/16. Much cleaner chromatograms were obtained when Mo solution in KOH was diluted $3.5\times$ with water (Figure 11). Due to the relatively low Mo-99 activity produced and very small aliquots taken for TLC, $3.5\times$ dilution was chosen so that enough activity could still be detected on fractions of the chromatograms. However, for high activities of Mo-99, higher dilution could be chosen.

FIGURE 8 Retention factors for Mo, Tc, and Nb after spotting 20 µL undiluted solution of Mo in 5M KOH on TLC and eluting in 0.1M Na₂CO₃. UHP Mo dissolved in 30% H₂O₂ nonstabilized with EOB (04/24/16).

FIGURE 9 Retention factors for Mo, Tc, and Nb after spotting 20 µL of undiluted solution of Mo in 5M KOH on TLC and eluting in 0.2M HCl in acetone. UHP Mo dissolved in 30% H₂O₂ nonstabilized with EOB (04/24/16).

FIGURE 10 Retention factors for Mo, Tc, and Nb after spotting 20 μL of undiluted solution of Mo in 5M KOH on TLC and eluting in 0.1M Na_2CO_3 . UHP Mo dissolved in 50% H_2O_2 Sn-stabilized with EOB (05/01/16).

FIGURE 11 Retention factors for Mo, Tc, and Nb after spotting 20 μL of 3.5 $\times$ diluted solution of Mo in 5M KOH with water on TLC and eluting in 0.1M Na_2CO_3 . UHP Mo dissolved in 50% H_2O_2 Sn-stabilized with EOB (05/01/16).

Figures 12 and 13 show TLC results from irradiation of two UHP Mo disks performed on 05/08/16. No significant difference in TLC profile is observed between samples dissolved with 50% H_2O_2 Sn-stabilized (Figure 12) or 30% H_2O_2 nonstabilized (Figure 13), indicating that the presence of Sn (~55 ppm of Sn) does not affect the radiochemical purity of Mo or Tc. Figure 14 shows TLC results for an Mo-100-enriched disk where Fe precipitate was observed and filtered. Due to removal of Nb by filtration, no Nb was observed on TLC.

FIGURE 12 Retention factors for Mo, Tc, and Nb after spotting 20 μ L of 3.5 $\times$ diluted solution of Mo in 5M KOH with water on TLC and eluting in 0.1M Na₂CO₃. UHP Mo dissolved in 50% H₂O₂ Sn-stabilized with EOB (05/08/16).

FIGURE 13 Retention factors for Mo, Tc, and Nb after spotting 20 μ L of 3.5 $\times$ diluted solution of Mo in 5M KOH with water on TLC and eluting in 0.1M Na₂CO₃. UHP Mo dissolved in 30% H₂O₂ nonstabilized with EOB (05/08/16).

FIGURE 14 Retention factors for Mo and Tc after spotting 20 μ L of 5 $\times$ diluted solution of Mo in 5M KOH with water on TLC and eluting in 0.1M Na₂CO₃. Mo-100-enriched disk dissolved in 50% H₂O₂ Sn-stabilized with EOB (07/31/16).

Results of RCP tests with 0.1M Na₂CO₃ as the mobile phase are summarized in Table 7, and indicate that no radiochemical impurities occurred in the final solutions, and that even elevated amounts of Sn-stabilized H₂O₂ (up to 31 mL/g-Mo) did not affect the RCP test. The usual consumption of 50% H₂O₂ for large-scale dissolution of up to 600 g of sintered Mo disks is 11–16 mL/g-Mo [6]. Based on the chromatograms obtained (Figures 4–10), it is recommended that Mo solutions in 5M KOH be diluted with water before the RCP test is performed. This recommendation is made because the interaction between high salt content and the mobile phase causes the behavior of both Tc and Mo on TLC to be misleading. It should be noted that 0.1M Na₂CO₃ can also be used as a mobile phase for RCP for both Tc and Mo. In some cases, a small portion of Tc was found at R_f = 0.1. It is unclear, however, whether any interaction between Tc and Nb (or other impurities) occurs.

These experiments conclude that the radiochemical purity of the product is not affected by the introduction of Sn with Sn-stabilized H₂O₂, or the presence of up to 500 ppm Fe and other impurities (see Experimental section) in the enriched Mo-100 target. It is likely that during the FY 2015 production runs, impurities affecting RCP were introduced during irradiation (corrosion found on the target). For all future irradiations using the helium cooling system, precautions should be taken to eliminate the source of impurities during the irradiation.

TABLE 7 Summary of RCP results for irradiated four UHP Mo targets and one Mo-100-enriched target with 0.1M Na₂CO₃ as a mobile phase

EOB	Mo (g)	$R_f = 0.9 \pm 0.1$		H ₂ O ₂ Used	H ₂ O ₂ mL/g-Mo
		Mo-99	Tc-99m		
04/24/16	UHP, 1.13	91.3%*	89.5%*	30/NS	13.3
05/01/16	UHP, 1.05	99.7%	96.5%	50/Sn-stab	9.5
05/08/16	UHP, 1.06	99.5%	95.8%	50/Sn-stab	18.9
05/08/16	UHP, 1.03	99.3%	94.6%	30/NS	9.7
07/31/16	Mo-100, 2.09	98.4%	98.3%	50/Sn-stab	31.0

* Undiluted solution of Mo in 5M KOH

EOB – end of bombardment; NS – nonstabilized; Sn-stab – tin stabilized

4 CONCLUSION

Four UHP natural Mo, and one Mo-100-enriched target were irradiated using Argonne's electron linac to produce Mo-99, and to determine whether radiochemical purity specifications can be met when Sn-stabilized H₂O₂ is used during dissolution. It was determined that even 2–3× excess of Sn-stabilized H₂O₂ typically used for dissolution of sintered Mo disks did not affect the radiochemical purity of the final product. In the case of Mo-100-enriched targets, impurities such as 540 ppm Fe, 75 ppm W, 65 ppm Cr, 39 ppm Ni, 15 ppm Cu, and 11 ppm Ge in enriched material also did not affect the RCP. Highly alkaline Mo solution in 5M KOH required dilution with water to obtain good chromatographic behavior.

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