Optimization of the Dissolution of Molybdenum Disks: FY-16 Results

Nuclear Engineering Division

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by

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CONTENTS

1	Introduction.....	1
2	Dissolution of Mo Disks to Optimize Production of Mo Targets.....	2
3	Optimization of Large-Scale Dissolution of Sintered Mo Disks	11
4	Adaptation of Benchtop Optimized Dissolution System to Hot Cell Operations.....	19
5	Acknowledgments.....	23
6	References.....	23
	Appendices.....	24

FIGURES

1	Photographs of 12×1 mm sintered and 12.4×1.5-1.9 mm 3D-printed Mo disks.	2
2	Photographs of ~29.5×2 mm sintered Mo disks.	3
3	Photograph of residual small particles that form during the dissolution of laser-melt 3D-printed Mo disks	6
4	Floating 29×0.5 mm sintered Mo disks during dissolution in 40 mL of 50% H ₂ O ₂ ; Formation of froth in 2 L reaction vessel during the dissolution of 12 g of 29×2 mm sintered Mo disks in 150 mL of 50% H ₂ O ₂	8
5	Photo of 600 g of sintered Mo disks placed inside 4 L dissolution vessel	11
6	Dissolution apparatus for processing of up to 600 g of sintered Mo disks using H ₂ O ₂	12
7	Schematic of dissolution setup with placement of thermocouples to monitor temperatures during dissolution of 600 g batch of sintered Mo disks	15
8	Temperature profiles during first 15 min for dissolution of 600 g of sintered Mo disks in 50% H ₂ O ₂	16
9	Thermal images of condensers before dissolution started; dissolution vessel during dissolution; flexible adapters between dissolution vessel and condensers; condensers during dissolution; bottom of dissolution vessel during dissolution; condensation flask containing condensed water from dissolution.....	17

10	Hot cell mockup with view from operator standpoint. No leaded glass is in place.....	20
11	Evaporation vessel with heating mantel on 10 L reaction vessel; evaporation vessel head with associated stirrer and connections to condenser system; and evaporation condenser system.	21
12	Dissolution vessel and dissolution vessel condenser system.....	22
A.1	Custom-made dissolution vessel.....	24

TABLES

1	Characteristics and dissolution results for sintered Mo disks in 40 mL of 50% H ₂ O ₂	3
2	Characteristics and dissolution results for sintered Mo disks from recycled material in 40 mL of 50% H ₂ O ₂	5
3	Characteristics and dissolution results for laser-melt 3D-printed Mo disks in 40 mL of 50% H ₂ O ₂	6
4	Characteristics and dissolution results for sintered Mo disks in 150 mL of 50% H ₂ O ₂	7
5	Characteristics and dissolution results for sintered Mo disks in 50% H ₂ O ₂ preheated to 70°C.....	9
6	Results from optimization of large-scale dissolution experiments.....	14

OPTIMIZATION OF THE DISSOLUTION OF MOLYBDENUM DISKS: FY-16 RESULTS

1 INTRODUCTION

Argonne National Laboratory, with support from the National Nuclear Security Administration's Material Management and Minimization program, is providing technical development assistance to NorthStar Medical Technologies LLC in its pursuit of two pathways for production of molybdenum-99. As a short-term answer to demands, NorthStar plans to use the $^{98}\text{Mo}(n,\gamma)^{99}\text{Mo}$ reaction (neutron capture) pathway at the University of Missouri Research Reactor. As a long-term solution, NorthStar will produce ^{99}Mo via the photonuclear reaction, $^{100}\text{Mo}(\gamma,n)^{99}\text{Mo}$, from irradiation of enriched Mo targets by an electron accelerator. Processing of irradiated targets, from either production mode, requires dissolution of the target material in H_2O_2 followed by a concentration step, addition of ferric ion to precipitate impurities, and conversion of the final solution to 5M potassium hydroxide solution of potassium molybdate.

Currently, NorthStar is using pressed and sintered Mo disks as targets. Several options are being considered for the design of Mo targets for the production of ^{99}Mo using the (γ,n) reaction. In the current design, the target holder contains a series of sintered Mo disks lined up perpendicular to two incident electron beams, one entering from each side of the target stack. In this configuration, the front-most disks absorb most of the heat from the electron beam and need to be thinner to allow for better cooling, while the middle of the target can be thicker. Distribution of the total mass of Mo allows for larger masses of Mo material and thus larger production batches of ^{99}Mo .

A limitation of the sintering approach is the production of very thin disks. On the other hand, recent advances in 3D printing allow for laser-melting on metals with high melting points, such as Mo. This approach gives more flexibility in the target design, and much thinner target components can be achieved than when the traditional press-and-sinter approach is used. Another advantage is that an entire target, including the housing, can be fabricated from Mo.

Recent progress on dissolution of sintered Mo disks is well documented [1–4]. We have demonstrated that several factors can play important roles in dissolution behavior: particle size of Mo metal used for production of targets, sintering conditions, degree of open porosity, and thickness of the sintered Mo targets.

Here we report experimental results from studies of small-scale dissolution of sintered Mo disks fabricated from various recycled and commercial Mo materials, and dissolution of 3D-printed Mo disks that were fabricated by Oak Ridge National Laboratory (ORNL). We also report on large-scale dissolution studies with 600 g batches of sintered Mo disks.

2 DISSOLUTION OF MO DISKS TO OPTIMIZE PRODUCTION OF MO TARGETS

Experimental

Sintered Mo disks and 3D-printed disks (laser-melt produced) were fabricated by ORNL. Small disks (<2 g) were dissolved in 40 mL of 50% H₂O₂ (Aqua Solutions H3050-4L) preheated to 70°C (unless stated otherwise) in a 250 mL beaker. Larger disks were dissolved in a 2 L reaction vessel. Photographs of sintered and 3D-printed disks are shown on Figures 1 and 2.

FIGURE 1 Photographs of 12×1 mm sintered and 12.4×1.5-1.9 mm 3D-printed Mo disks. U.S. penny shown for scale.

FIGURE 2 Photographs of ~29.5×2 mm sintered Mo disks. U.S. quarter shown for scale.

Results and Discussion

12×1 mm Sintered Mo Disks

Sintered Mo disks dissolved quickly with H_2O_2 that was preheated to 70°C. Dissolution of disks from the same batch in room-temperature (24°C) H_2O_2 required an activation period of 3–5 min (the time required to produce enough heat for disks to start dissolving vigorously). Characteristics of sintered Mo disks and dissolution times are listed in Table 1. Dissolution rates were in the range of ~0.76–1.05 g/min.

TABLE 1 Characteristics and dissolution results for sintered Mo disks in 40 mL of 50% H_2O_2

Disk I.D.	Mass (g)	Theor. Density (%)	Open Porosity (%)	Diameter (mm)	Thickness (mm)	50% H_2O_2 Temperature (°C)	Diss. Time (min)	Diss. Rate (g/min)
S50-12A-5	0.986	87.9	7.9	11.99	0.94	70	1.25	0.79
S50-12A-7	1.008	89.1	2.7	12.01	0.94	70	1.33	0.76
S50-12A-8	0.994	87.9	7.8	12.01	0.97	24	6.25	0.16*
S50-12A-10	0.994	86.7	8.3	12.01	0.97	70	1.00	0.99
S50-12B-2	1.052	87.4	8.6	11.99	1.05	70	1.00	1.05
S50-12B-4	1.048	87.3	8.3	11.99	1.06	24	4.00	0.26 ⁺
S50-12B-21	1.055	87.1	8.0	11.99	1.05	70	1.33	0.79

* After an activation period (5min), dissolution rate is 0.789 g/min.

⁺ After an activation period (3min), dissolution rate is 1.05 g/min.

12×1mm Disks of Recycled Mo Material

A small batch of recycled Mo material as MoO₃ obtained using a precipitation method developed at Argonne [5], and ammonium heptamolybdate (AHM) derived by solvent extraction [6], was sent to ORNL. At ORNL this material was reduced to Mo metal using a two-stage process [7], producing 12×1 mm sintered Mo disks for Argonne dissolution studies. Characteristics and dissolution rates of the sintered Mo disks are listed in Table 2.

Reduction of the AHM material led to an approximately 25–30% decrease in the sizes of obtained Mo particles. Finer AHM material with particle sizes of $131 \pm 30 \mu\text{m}$ turned into $91 \pm 25 \mu\text{m}$ Mo particles after reduction, and coarser AHM with particle sizes of $204 \pm 37 \mu\text{m}$ turned into Mo particle sizes of $158 \pm 38 \mu\text{m}$. Reduction of MoO₃ material with particle sizes of 75–150 μm led to Mo particle sizes of $125 \pm 24 \mu\text{m}$ (ANL-0815-A) and $155 \pm 45 \mu\text{m}$ (ANL-0815-B), which were agglomerates of much smaller particles, typically in the 1–10 μm range, and resulted in high-theoretical-density disks.

Disks made from recycled material did not start dissolving in room temperature H₂O₂ even after 15 min. When H₂O₂ was preheated to 70°C, the disks made from recycled AHM (ANL-1015) dissolved quickly, with dissolution rates of 0.33–0.75 g/min. There was no significant difference in dissolution rates for disks made from coarser or finer Mo material obtained after reduction of AHM.

The disks made after reduction of recycled MoO₃ (ANL-815) dissolved much slower, and continuous heat (boiling H₂O₂) was required for complete dissolution. This is due to the very high density and low open porosity of sintered Mo disks that were produced from fine Mo material.

TABLE 2 Characteristics and dissolution results for sintered Mo disks from recycled material in 40mL of 50% H₂O₂

Material	No.	Temp. /time (°C/hr)	Mass (g)	TD* (%)	Open Porosity (%)	Diameter (mm)	Thickness (mm)	Temp. (°C)	Diss. Time (min)	Diss. Rate (g/min)	Particle Size Recycled Mo/reduced Mo*
ANL-1015A	F1	1500/1	0.993	87.0	11.3	12.00	1.036	70	1.50	0.66	75–150µm AHM/91 ± 25µm Mo
ANL-1015A	F2	1500/1	1.003	86.9	11.6	12.00	1.034	70	1.67	0.60	75–150µm AHM/91 ± 25µm Mo
ANL-1015A	F3	1500/1	0.996	87.4	10.8	11.99	1.033	70	1.33	0.75	75–150µm AHM/91 ± 25µm Mo
ANL-1015A	F4	1600/4	1.002	89.9	7.2	11.79	1.021	70	3.00	0.33	75–150µm AHM/91 ± 25µm Mo
ANL-1015A	F5	1600/4	1.009	90.7	7.2	11.76	1.041	70	2.67	0.38	75–150µm AHM/91 ± 25µm Mo
ANL-1015A	F6	1600/4	1.000	91.4	7.0	11.76	1.011	70	2.33	0.43	75–150µm AHM/91 ± 25µm Mo
ANL-1015A	C1	1500/1	1.001	86.9	10.6	12.05	1.029	70	1.33	0.75	150–400µm AHM/204 ± 37µm Mo
ANL-1015A	C2	1500/1	1.004	87.5	10.3	12.05	1.021	70	1.75	0.57	150–400µm AHM/204 ± 37µm Mo
ANL-1015A	C3	1500/1	1.002	87.6	10.1	12.05	1.010	70	1.67	0.60	150–400µm AHM/204 ± 37µm Mo
ANL-1015A	C4	1600/4	1.029	87.9	9.9	11.94	1.029	70	N/A	N/A	150–400µm AHM/204 ± 37µm Mo
ANL-1015A	C5	1600/4	1.012	89.1	8.9	11.94	1.006	70	2.25	0.45	150–400µm AHM/204 ± 37µm Mo
ANL-1015A	C6	1600/4	0.995	88.3	9.8	11.94	1.006	70	2.17	0.46	150–400µm AHM/204 ± 37µm Mo
ANL-815-A	A1	1600/4	1.001	93.3	0.0	11.46	1.049	100	6.00	0.17	75–150µm MoO ₃ /125–155µm Mo ⁺
ANL-815-A	A2	1600/4	1.004	93.7	0.1	11.56	1.016	100	9.00	0.11	75–150µm MoO ₃ /125–155µm Mo ⁺
ANL-815-A	A3	1600/4	1.007	93.7	0.6	11.60	1.016	100	7.75	0.13	75–150µm MoO ₃ /125–155µm Mo ⁺
ANL-815-B	A1	1600/4	1.015	96.1	0.7	11.79	0.978	100	17.0	0.060	75–150µm MoO ₃ /125–155µm Mo ⁺
ANL-815-B	A2	1600/4	1.016	96.8	0.4	11.76	0.965	100	15.3	0.067	75–150µm MoO ₃ /125–155µm Mo ⁺
ANL-815-B	A3	1600/4	0.997	96.6	0.6	11.79	0.932	100	13.5	0.074	75–150µm MoO ₃ /125–155µm Mo ⁺

* TD = theoretical density, AHM = ammonium heptamolybdate

⁺ Most particles were agglomerates of 1–10 µm Mo particles.

Laser-Melt 3D-Printed Mo Disks

Characteristics of laser-melt 3D-printed Mo disks and dissolution times are listed in Table 3. Dissolution rates were in the range of ~0.1–0.25 g/min. In general, laser-melt 3D-printed disks dissolved more slowly than sintered Mo disks. After 1–2 min of dissolution, the disks broke down into very small pieces. Complete dissolution of the smaller particles took several additional minutes and heat provided by a hot plate. Some of the fine particles looked like tiny wires (disk Mo-45-16-9; see Figure 3).

TABLE 3 Characteristics and dissolution results for laser-melt 3D-printed Mo disks in 40 mL of 50% H₂O₂

Disk I.D.	Mass (g)	Theor. Density (%)	Open Porosity (%)	Diameter (mm)	Thickness (mm)	Temperature (°C)	Diss. Time (min)	Diss. Rate (g/min)
Mo45-16-3	1.6470	90.0	9.8	12.40	1.90	70	10.00	0.165
Mo45-16-9	1.7380	89.0	10.4	12.40	1.90	70	18.00	0.097
Mo45-23-1	1.2660	89.5	8.5	12.40	1.50	24	6.00	0.211*
Mo45-23-2	1.2840	91.0	7.5	12.40	1.50	70	+	+
Mo45-23-7	1.272	90.3	6.7	12.40	1.50	70	5.00	0.254

* Dissolution started in 24°C H₂O₂; however, fine particles had to be dissolved with additional heat using hot plate.

+ It was difficult to determine the exact time of dissolution because of profuse bubbles.

FIGURE 3 Photograph of residual small particles that form during the dissolution of laser-melt 3D-printed Mo disks

Based on a very limited number of disks, the dependency of laser-melt disk dissolution rates on open porosity was observed to be reversed compared with sintered disks. Disks with the highest degree of open porosity (10.4%) dissolved the slowest (below 0.1 g/min), while disks with the lowest open porosity (6.7%) dissolved the fastest (0.254 g/min). A possible explanation could be that the laser-melt disks with large open porosity contain fused particles with different macro- and micro-structures that require a longer time to dissolve.

29 mm Sintered Mo Disks

All disks provided by ORNL were sintered at 1,600°C for 4 hr and pressed at 138 ksi. Characteristics of the sintered Mo disks and dissolution times are listed in Table 4. To determine the best conditions for dissolution, an S100-29 disk was dissolved first by adding 110 mL of H₂O₂ preheated to 70°C. After 9.5 min, the solution turned

orange in color, indicating that most of the H₂O₂ was consumed, and the dissolution slowed down significantly. Then, 50 mL of additional H₂O₂ preheated to 70°C was added to finish the dissolution. On the basis of this initial test, further dissolutions were performed by adding 100 mL of H₂O₂ preheated to 70°C, draining the solution after 5.5–6 min, and adding 50 mL to finish the dissolution.

TABLE 4 Characteristics and dissolution results for sintered Mo disks in 150 mL of 50% H₂O₂

Disk*	Mass (g)	Theor. Density (%)	Open Porosity (%)	Diameter (mm)	Thickness (mm)	Temp. (°C)	Diss. Time (min)	Diss. Rate (g/min)
S25-36	12.1436	90.5	2.2	29.40	1.95	70	8.750	1.388
S25-37	12.1087	90.5	2.0	29.40	1.92	70	9.000	1.345
S25-38	12.0875	90.7	1.0	29.40	1.92	70	+	+
S25-39	12.1169	90.7	1.7	29.40	1.94	70	8.500	1.426
S25-40	12.0544	90.7	1.6	29.40	1.90	70	8.750	1.378
S50-41	12.1019	90.9	2.8	29.40	1.95	70	5.00	0.254
S50-42	12.1192	90.6	2.7	29.40	1.95	70	7.666	1.579
S50-43	12.0707	90.7	2.7	29.40	1.92	70	9.000	1.347
S50-44	12.0866	90.5	2.8	29.40	1.93	70	9.000	1.341
S50-45	12.0820	90.6	3.0	29.40	1.94	70	8.00	1.511
S100-29	12.0330	89.0	6.1	29.50	1.94	70	8.50	1.421
S100-30	12.0210	89.0	6.3	29.50	1.94	70	11.00	1.094
S100-31	12.0722	89.7	5.3	29.50	1.99	70	7.50	1.603
S100-32	12.1604	89.1	5.7	29.50	1.97	70	8.00	1.509
S100-33	12.0416	89.1	5.9	29.50	1.96	24	10.50 ⁺⁺	1.158 ⁺⁺
S100-34	12.0900	89.4	5.5	29.50	1.96	70	15.50	0.777
S100-35	12.0448	89.5	5.2	29.50	1.95	24	8.00 [§]	1.51 [§]

* Different amounts of ethylene bis stearamide (EBS) lubricant were used: from 0.25% to 1%. For disks marked S25, 0.25% EBS was used to make the disks; for 0.5% EBS, the disks were marked S50; and for 1% EBS, the disks were marked S100.

⁺ Most of the disk dissolved in ~13min; the remaining portion would not dissolve even after an additional 20 min.

⁺⁺ Started to dissolve after adding 100 mL H₂O₂ – activation period 8 min, dissolution rate 1.7 g/min.

[§] Started to dissolve after adding 30 mL H₂O₂ – activation period 4.5 min, dissolution rate 1.46 g/min.

Typical dissolution rates were in the range of 1.34–1.6 g/min. Disk S25-38, with 1% open porosity, didn't completely dissolve in 150 mL of H₂O₂ even after prolonged time. Although the majority of the disks (>95% of mass) dissolved in ~13 min, the remaining portion of the disks did not completely dissolve, even after an additional 20 min (without additional heat

provided). Disks that were dissolved with room-temperature H_2O_2 required an activation period before they started to dissolve quickly, usually after the H_2O_2 reached 60°C (without heating). Less H_2O_2 at the beginning (30 mL for disk S100-35) led to shorter activation periods (4.5 min), compared with an activation period of 8 min when 100 mL of H_2O_2 was used. With less mass to absorb the heat from the reaction, the solution warmed up much quicker. It appears that using 29×2 mm disks for a target should not be a problem from the dissolution point of view.

Table 5 summarizes the results from dissolution of disks with ~ 29 mm diameter and various thicknesses: 0.5, 1, 1.5, and 2 mm. All disks were manufactured from commercial Mo materials (Climax Molybdenum or H.C. Starck). All disks were dissolved in 50% H_2O_2 preheated to 70°C . Generally, ~ 13 mL of H_2O_2 was used per gram of Mo material. Based on the dissolution of a few thicker disks, it seemed the dissolution rate was slightly higher for thicker disks than thinner disks; however, this was due to the fact that thinner and lighter disks floated on top of froth during the dissolution (Figure 4a), which significantly limited their contact with solution. Thicker and heavier disks remained at the bottom of the reaction vessel for most of the dissolution despite the very vigorous reaction (Figure 4b), forming significant volume of froth ($\sim 70\%$ of 2 L vessel), and only started to float toward the end of dissolution when the mass of the disks was no longer heavy enough to keep the material from floating.

FIGURE 4 (a) Floating 29×0.5 mm sintered Mo disks during dissolution in 40 mL of 50% H_2O_2 ; (b) Formation of froth in 2 L reaction vessel during the dissolution of 12 g of 29×2 mm sintered Mo disks in 150 mL of 50% H_2O_2

TABLE 5 Characteristics and dissolution results for sintered Mo disks in 50% H₂O₂ preheated to 70°C

Disk I.D.	Material	Press (ksi)	Sinter (°C/hr)	Mass (g)	Density (g/cm ³)	TD Sintered (%)	Open Porosity (%)	Diameter (mm)	Thickness (mm)	Diss. Time (min)	Diss. Rate (g/min)	50% H ₂ O ₂ (mL)	H ₂ O ₂ / Mo (mL/g)
PMS-1	Climax PM + 0.5%SA	100	1600/4	3.0797	9.14	89.6	7.3	28.8	0.53	1.5	2.05	40	13.0
PMS-2	Climax PM + 0.5%SA	100	1600/4	3.0923	9.08	89	9.1	28.8	0.54	1.75	1.77	40	12.9
PMS-3	Climax PM + 0.5%SA	100	1600/4	3.0725	8.95	87.7	8.5	28.8	0.53	1.633	1.88	40	13.0
PMA5-3	Climax PM + 0.5%EBS	100	1600/2	3.0996	9.04	88.6	9.3	29	0.53	1.5	2.07	40	12.9
PMA5-24	Climax PM + 0.5%EBS	100	1600/4	6.0576	8.99	88.1	6.7	29.3	1	2.75	2.20	60	9.9
PMA5-26	Climax PM + 0.5%EBS	100	1600/4	6.0533	9.00	88.2	6.9	29.3	1	2.25	2.69	80	13.2
PMA5-27	Climax PM + 0.5%EBS	100	1600/4	9.0418	8.92	87.5	7.7	29.3	1.49	4	2.26	100	11.1
PMA5-28	Climax PM + 0.5%EBS	100	1600/4	9.0453	8.92	87.5	7.8	29.3	1.5	3.33	2.72	100	11.1
PM5A-31	Climax PM + 0.5%EBS	100	1600/4	12.0738	8.90	87.3	7.9	29.4	2	5.5	2.20	150	12.4
PMA5-32	Climax PM + 0.5%EBS	100	1600/4	12.0781	8.91	87.4	7.9	29.4	1.99	4.17	2.90	150	12.4
PMA-13	Climax PM + 0.25%EBS	100	1600/4	3.0899	9.23	90.5	2.4	28.8	0.52	2	1.54	40	12.9
PMA-27	Climax PM + 0.25%EBS	100	1400/2	3.1001	8.71	85.4	---	29.2	0.53	1.33	2.33	40	12.9
PMA-40	Climax PM + 0.25%EBS	100	1600/4	3.0946	9.1	89.2	6.2	28.8	0.53	1.75	1.77	40	12.9
PMA-41	Climax PM + 0.25%EBS	100	1600/4	3.0952	9.06	88.8	6.3	28.8	0.54	1.75	1.77	40	12.9
PMA-43	Climax PM + 0.25%EBS	100	1600/4	3.0975	9.09	89.1	6.4	28.8	0.53	1.72	1.80	40	12.9
PMA-44	Climax PM + 0.25%EBS	100	1600/4	3.0893	9.05	88.7	6.4	28.8	0.53	1.78	1.74	40	12.9
PMA-46	Climax PM + 0.25%EBS	100	1600/4	3.0732	9.07	88.9	6.6	28.8	0.53	1.75	1.76	40	13.0
PM-2	Climax PM	100	1500/16	3.0804	7.86	77.1	20.9	27.3	0.73	1.33	2.32	40	13.0
PM-3	Climax PM	100	1500/1	3.0818	7.10	69.6	28.9	28.3	0.7	1.17	2.63	40	13.0
PM-4	Climax PM	100	1500/1	3.1083	8.68	85.1	12.7	29.3	0.55	1.33	2.34	40	12.9
PM-5	Climax PM	100	1500/1	3.0806	8.54	83.7	14.3	29.3	0.55	1.33	2.32	40	13.0
PM-7	Climax PM	100	1600/4	3.1087	9.08	89	6.6	28.8	0.54	1.75	1.78	40	12.9
PM-9	Climax PM	100	1600/1	3.0834	8.87	87	9.9	29.1	0.54	1.33	2.32	40	13.0
PM-10	Climax PM	100	1600/1	3.1099	8.88	87.1	9.6	29.1	0.55	1.417	2.19	40	12.9
PM-11	Climax PM	100	1500/1	3.0844	8.76	85.9	12.2	29.3	0.54	1.417	2.18	40	13.0
PM-12	Climax PM	100	1600/4	3.0989	9.03	88.5	8.6	28.7	0.54	1.58	1.96	40	12.9
PM-14	Climax PM	145	1600/2	3.0966	9.36	91.8	3.4	29.3	0.5	1.75	1.77	40	12.9
PM-15	Climax PM	145	1600/4	3.1242	9.47	92.8	1	29.1	0.52	2	1.56	40	12.8
PM-16	Climax PM	145	1600/2	3.0198	9.40	92.2	3.9	29.3	0.55	2	1.51	40	13.2

TABLE 5 (Cont.)

Disk I.D.	Material	Press (ksi)	Sinter (°C/hr)	Mass (g)	Density (g/cm ³)	TD Sintered (%)	Open Porosity (%)	Diameter (mm)	Thickness (mm)	Diss. Time (min)	Diss. Rate (g/min)	50% H ₂ O ₂ (mL)	H ₂ O ₂ / Mo (mL/g)
S5-1	Starck SD + 0.5%EBS	100	1600/4	3.1545	8.82	86.5	9	28.9	0.55	1.5	2.10	40	12.7
S5-2	Starck SD + 0.5%EBS	100	1600/4	3.1481	8.8	86.3	9.3	28.9	0.56	1.5	2.10	40	12.7
S5-4	Starck SD + 0.5%EBS	100	1600/4	3.12	8.81	86.4	9.4	28.9	0.55	1.42	2.20	40	12.8
S5-5	Starck SD + 0.5%EBS	100	1600/4	3.1447	8.89	87.2	8.9	28.9	0.55	1.5	2.10	40	12.7
S5-6	Starck SD + 0.5%EBS	131	1600/4	3.0303	9.08	89	6.5	29.3	0.5	1.33	2.28	40	13.2
S5-8	Starck SD + 0.5%EBS	131	1600/4	3.0393	9.03	88.5	6.8	29.3	0.51	1.42	2.14	40	13.2
S5-9	Starck SD + 0.5%EBS	148	1600/4	3.0263	9.20	90.2	4.7	29.4	0.5	1.5	2.02	40	13.2
S5-10	Starck SD + 0.5%EBS	148	1600/4	3.0511	9.22	90.4	4.4	29.4	0.5	1.58	1.93	40	13.1
S5-11	Starck SD + 0.5%EBS	148	1600/4	3.0234	9.21	90.3	4.4	29.5	0.49	1.75	1.73	40	13.2
S5-12	Starck SD + 0.5%EBS	123	1600/4	3.0262	8.97	87.9	7.6	29.3	0.51	1.5	2.02	40	13.2
S5-13	Starck SD + 0.5%EBS	123	1600/4	3.0208	8.95	87.7	7.6	29.3	0.51	1.5	2.01	40	13.2
S5-15	Starck SD + 0.5%EBS	123	1600/4	3.0228	8.93	87.5	8	29.3	0.51	1.42	2.13	40	13.2
S5-16	Starck SD + 0.5%EBS	123	1600/4	3.0302	8.89	87.2	8.4	29.3	0.51	1.5	2.02	40	13.2
S5-17	Starck SD + 0.5%EBS	131	1600/4	3.0069	9.07	89	–	29.4	0.49	1.5	2.00	40	13.3
S5-18	Starck SD + 0.5%EBS	131	1600/4	3.028	9.15	89.7	–	29.4	0.49	1.33	2.28	40	13.2
S5-22	Starck SD + 0.5%EBS	137	1600/4	3.0078	8.80	86.3	–	29.5	0.5	1.42	2.12	40	13.3
S5-23	Starck SD + 0.5%EBS	137	1600/4	3.0053	8.84	86.7	–	29.5	0.5	1.42	2.12	40	13.3
S5-24	Starck SD + 0.5%EBS	137	1600/4	2.984	8.96	87.8	–	29.5	0.49	1.58	1.89	40	13.4
S5-26	Starck SD + 0.5%EBS	137	1600/4	3.0036	8.97	87.9	–	29.5	0.49	1.42	2.12	40	13.3
S5-27	Starck SD + 0.5%EBS	145	1600/4	3.0225	8.97	87.9	–	29.5	0.49	1.58	1.91	40	13.2
S5-28	Starck SD + 0.5%EBS	145	1600/4	3.0027	9.00	88.2	–	29.5	0.49	1.5	2.00	40	13.3
S5-30	Starck SD + 0.5%EBS	145	1600/4	3.0096	9.09	89.1	–	29.5	0.48	1.5	2.01	40	13.3
S5-31	Starck SD + 0.5%EBS	145	1600/4	3.0106	9.28	91	–	29.5	0.47	1.42	2.12	40	13.3
RTP5-9	Starck SD + 0.25%EBS	87.5	1600/4	3.0084	8.97	87.9	–	28.7	0.52	1.83	1.64	40	13.3
RTP5-10	Starck SD + 0.25%EBS	87.5	1600/4	2.9906	9.23	90.5	–	28.7	0.5	1.75	1.71	40	13.4
S2-3	Starck SD + 0.25%EBS	100	1600/4	3.1297	8.60	84.3	7.8	28.9	0.55	1.5	2.09	40	12.8
S2-4	Starck SD + 0.25%EBS	100	1600/4	3.0964	8.73	85.6	8.4	28.9	0.54	1.42	2.18	40	12.9

3 OPTIMIZATION OF LARGE-SCALE DISSOLUTION OF SINTERED MO DISKS

Experimental

Sintered Mo disks received from NorthStar (26×1 mm) and ORNL (29×0.5 mm) were dissolved using 50% H₂O₂ in 600 g batches in a glass reaction vessel. H₂O₂ (50%) stabilized with Sn (Aqua Solutions, Inc., Technical grade, H30504L) was used for these dissolutions. Disks were placed into a 4 L dissolution vessel with a glass frit on the bottom (Figure 5).

The dissolution apparatus is shown in Figure 6. All parts of dissolution apparatus are identified and referenced in the Appendix A. The apparatus consists of a 4 L dissolution vessel with glass-fritted filter and valved-drain (stopcock) at the bottom to allow for draining dissolved portions of Mo material into an evaporation vessel by vacuum. A Julabo FL 1701 with cooling capacities of 1.7 kW at 20°C, 1.1 kW at 0°C, 0.85 kW at -10°C, and 0.4 kW at -20°C and flow rate capacity of 23 L/min was used. However, it should be noted that due to small tubing connections (11 mm at the largest serration) and small internal diameter of the condenser coils, the actual coolant (50% ethylene glycol) flowrate through the system was 0.32 L/min.

FIGURE 5 Photo of 600 g of sintered Mo disks placed inside 4 L dissolution vessel

Hydrogen peroxide was slowly added from the top of the vessel using a peristaltic pump. Shortly after addition of H₂O₂, vigorous dissolution of the Mo disks began, producing yellow-colored froth and solution and significant bubble formation due to heat produced from the reaction. Water vapor produced from the reaction was condensed using three high-efficiency condensers (Chemglass CG-1215-C) and collected in collection flask. The condensers were used to replace the lower-efficiency condensers (used in the previous system [1]) in order to trap the majority of vapors evolved during the dissolution process. Here the evolved gases flowed up and out of the dissolution vessel to the high-efficiency condensers in two parallel flow paths, down

the water-cooled jackets, and up a single high-efficiency condenser exhausted to the atmosphere (Figure 6, center). The parallel flow paths were established to lower the internal pressure by raising the volume capacity of the flow path. A 3 L round bottom was put in place to collect the condensation of the evolved dissolution vapors. A lower drain spout allowed for easy removal of the volume collected. During the increased mass dissolution experiments (600 g instead of 300 g), this vessel needed to be drained at least once during processing. The new chiller proved to be more efficient than the previous one, but was also unable to keep up with the exothermic process. On several occasions, the dissolution needed to be halted in order for the chiller to reestablish an acceptable chilled water supply.

FIGURE 6 Dissolution apparatus for processing of up to 600 g of sintered Mo disks using H_2O_2 . Evaporation vessel (*left*), is heated with an oversized heating mantle and heat tape. The associated condensation vessel is visible behind the evaporation vessel. The dissolution vessel (*right*) is connected to the evaporation vessel through the lower drain. The associated condensation vessel (*center*) is connected to the dissolution vessel by two flexible adapters and can be drained at any point during the experiment through a lower drain.

As the level of foam reached above half the height of the dissolution vessel, addition of H_2O_2 was slowed, and as the solution turned to a dark yellow-orange color, the dissolved portion of Mo was drained into the evaporation vessel. The dissolution was continued by adding fresh

H₂O₂. This process was repeated throughout the dissolution of the Mo disks. In this manner, the dissolution and evaporation steps were performed in parallel, with partial solutions. Gentle heat (50°C) was applied under vacuum to the initial volumes received in the evaporation/concentration vessel to destroy excess H₂O₂ and begin the evaporation process. Because excessive heating causes rapid expulsion of gases and foam formation upon subsequent additions of dissolved Mo disks to the evaporation vessel, a relief valve was attached to relieve pressure and decrease the foam layer as needed.

After complete dissolution, the final portion of dissolved Mo was transferred to the evaporation vessel. The dissolution vessel was rinsed with KOH (Sigma Aldrich) (900 g of 85% KOH dissolved in 740 mL of H₂O) in portions and then transferred to the evaporation vessel. Reaction of KOH with H₂O₂ is vigorous and this step was completed with extreme care. During evaporation, the solution was continuously stirred with an overhead stirrer (Arrow electric stirrer, CG-2021) using a glass shaft (CG-2074) with a Teflon paddle that was connected to the stirrer motor using a flexible drive shaft (CG-2047). The combined solutions were heated to destroy H₂O₂ and to bring the solution to the desired volume as required.

When the evaporation step was completed, the final solution was filtered by vacuum through a high-capacity filter (Polycap 36 TC, 0.6/45µm). Filtration was performed to remove impurities that precipitated under the alkaline conditions, such as iron. The filtration step usually required ~15 min. After filtration, 900 g of solid 85% KOH was added. To make Mo ~0.2 g/mL as K₂MoO₄ in 5M KOH solution, water was added as necessary to bring the density of the solution to 1.55 g/mL.

Results and Discussion

No significant changes were made to the evaporation vessel. The heating mantle, heat tape, and condensers performed as expected. Gently heating the solution that was transferred from the dissolution vessel accelerated the decomposition of H₂O₂. The solution, in general, was not heated to evaporation temperatures until after the complete dissolution of Mo and transfer into the dissolution vessel. If evaporation was required, the vessel was heated with both the heat tape and the heating mantle. Vacuum was applied at all times.

Dissolution of 600 g of 26×1 mm Disks

The dissolution of 600 g of 26×1 mm disks was not significantly different from the dissolution of 300 g disks [1]. The summary of dissolution tests with 600 g of sintered Mo disks is listed in Table 6. Dissolution rates were approximately the same from the optimized 300 g dissolution rates. With twice the mass of disks, approximately twice the amount of H₂O₂ and twice the amount of time was required to fully dissolve 600 g of disks. The slight increase in H₂O₂ can be attributed to the self-destruction of H₂O₂. Hydrogen peroxide decomposes with heat, and this reaction is vigorous and exothermic [2]. Consumption of H₂O₂ can be controlled by regulating the flow rate of H₂O₂ addition. The transfer of dissolved Mo to the evaporation vessel was determined by the color of the solution. As the color of the solution changed to dark yellow-orange, the solubility limit of the Mo-peroxo species was being reached; as a result, the

flow of H_2O_2 was decreased or stopped and Mo-peroxo solution was drained to the evaporation vessel. This led to a more economical consumption of H_2O_2 , which can shorten total processing times.

Dissolution of 29.5×0.5 mm Disks

The dissolution rate of the larger-diameter and thinner disks was significantly faster than the 26×1 mm disks (Table 6). The faster dissolution rate could partially be attributed to the larger surface area per mass of the disks. Assuming the porosities of the disks are equal, the surface area of the 29.5×0.5 mm disks is 1,413.24 mm², while the surface area of the 26×1 mm disks is 1143.54 mm². The increased surface area allows for greater contact with the H_2O_2 and thus more surface upon which the reaction may occur. Due to significantly better efficiency of dissolution, no additional time for evaporation was required, which led to significantly shorter total processing time.

TABLE 6 Results from optimization of large-scale dissolution experiments

Disks (mm)	Mass (g)	H_2O_2 (L)	Water Condensed from Diss. (L)	Diss. Time (hr)	Evaporation Time (hr)	Total Time (hr)	Total Diss. Rate (g/min)
26×1	603.7	9.5	5.4	2.5	1.5	4.5	2.24
26×1	600.5	9.0	5.9	2.75	1	4.0	2.5
26×1	602.4	6.5	4.3	2.0	1.5	3.75	2.68
29.5×0.5	596.8	6.8	4.7	1.8	0	2.25	4.42

Reaction Heat and Cooling Capacity

During the last dissolution of 26×1 mm disks, thermocouples were installed to monitor changes in the temperature at four different locations (Figure 7). Temperatures were monitored at:

- T₁ – inlet of coolant before entering the first condenser
- T₂ – outlet of coolant after third condenser (directly connected to dissolution vessel)
- T₃ – water vapor on top of the dissolution vessel
- T₄ – water vapor exiting the last condenser (open to atmosphere)

FIGURE 7 Schematic of dissolution setup with placement of thermocouples to monitor temperatures during dissolution of 600 g batch of sintered Mo disks

The temperature profiles of the four points monitored are shown in Figure 8. The headspace temperature of the dissolution vessel rose from 24°C to 99°C within 30 sec. Due to the heat of reaction, the temperature of coolant leaving the condensers rose from -0.3°C to 85°C in ~15 min of processing time. As dissolution began, gas was observed exhausting from the last condenser (open to atmosphere). However, the temperature of those gases decreased from 6°C to -2°C in first 30 sec of dissolution. This suggests that at this point of dissolution, exhausted gas is oxygen and not water vapor. However, as the dissolution progressed the temperature began to increase, reaching ~23°C after ~14 min. During the 2 hr of dissolution, it was observed that the cooling capacity of the chiller was not sufficient and the temperature at the exhaust point of the condenser open to atmosphere reached 60°C (not shown in Figure 8), indicating that some portion of water vapor may have been exhausted. However, the amount of water vapor that escaped was minimal, as there were only short periods of time with elevated exhaust temperatures.

FIGURE 8 Temperature profiles during first 15 min for dissolution of 600 g of sintered Mo disks in 50% H₂O₂

Thermal images of the system before and during dissolution are shown in Figure 9. Figures 9a and 9b show the thermal images of dissolution setup before dissolution, while Figures 9c–9h are thermal images from during the dissolution. The volume of water condensed in the first 15 min of dissolution was used to determine the heat of dissolution:

$$\Delta H = mc\Delta T \quad (1)$$

where ΔH is the heat absorbed by water, m is mass, and c is specific heat ($c = 4.18 \text{ J/g/}^\circ\text{C}$). The mass of water that condensed during the first 15 min of the dissolution was $\sim 900 \text{ g}$; the difference between room temperature and the boiling temperature of water was calculated as $\Delta T = 76^\circ\text{C}$.

FIGURE 9 Thermal images of: (a, b) condensers before dissolution started; (c, d) dissolution vessel during dissolution; (e) flexible adapters between dissolution vessel and condensers; (f) condensers during dissolution; (g) bottom of dissolution vessel during dissolution; (h) condensation flask containing condensed water from dissolution

ΔH determined based on Eq. 1 was 286.3 kJ. $\Delta H_{\text{vap}} = 2,257$ kJ/kg of water; therefore, total heat from reaction was:

$$\Delta H_T = \Delta H + 0.9\Delta H_{\text{vap}} = 2,317.6 \text{ kJ} \quad (2)$$

where 0.9 represents 0.9 kg of water (900 mL). This was converted to power with Eq. 3:

$$P = \Delta H_T/t = 2,317.6/900 = 2.58 \text{ kW} \quad (3)$$

where t is time. Based on these calculations, the capacity of the chiller used in this experiment (see Experimental section) was far exceeded. A chiller with higher cooling capacity should be used in the future if release of water is of concern. In our experimental setup, water vapor was exhausted inside of a chemical fume hood and did not cause concern; however, release of water vapor in hot cell can cause significant fogging and pooling or free-standing water. An example of a chiller with sufficient cooling capacity is the Julabo FL4003 recirculating cooler, which has 4 kW cooling capacity at 20°C, and 2.4 kW at 0°C.

Summary and Conclusions

Dissolution studies were performed on 600 g of Mo disks with an optimized system, developed from multiple 300 g dissolution studies. The average dissolution time from the three experiments performed with 26×1 mm disks was ~4.1 hr, giving average dissolution rates of 2.3 g/min. These large-batch rates were faster than those observed for single 26×1 mm disks. Shorter total processing time was observed during the processing of 29.5×0.5 mm disks because the additional time for evaporation was not required. When the optimized system was used, the dissolution rate for 600 g of thinner disks was 4.44 g/min, which was significantly faster than for the thicker 26×1 mm disks. The increased condenser capacity helped to minimize the amount of vapor to escape the system, conservatively estimated to be <5%. To eliminate all vapor escape, a recirculating chiller with >3 kW cooling capacity is required.

4 ADAPTATION OF BENCHTOP OPTIMIZED DISSOLUTION SYSTEM TO HOT CELL OPERATIONS

Processing production-level quantities—thousands of curies—of irradiated Mo will be performed in a hot cell. NorthStar is currently designing its hot cell for this work. One of the primary concerns is that the system described in Section 2 of this report is composed entirely of glass. Glass could break within a hot cell, where metallic manipulators perform the operations needed for processing hundreds of grams of irradiated Mo. Furthermore, the system requires cleaning between processing runs, and periodic maintenance. Therefore, there is a need to demonstrate that the equipment can be set up, maintained, and operated in a hot cell environment. Argonne has configured a hot cell mockup to determine the required dimensions for the optimized system and ensure operations can be performed within the confined space of a hot cell.

Figure 10 shows an image of the hot cell mockup (sketches of modules can be found in Appendix B). The system fits within the dimensions of 140×112×160 cm (w×d×h). This layout allows for additional ~70 cm of working space in front of the setup. These dimensions allow ample room for an antechamber tray pass-through, additional valves, and other required attachments (e.g., condenser water lines, liquid disposal lines). There is also room for retrieving disks from irradiation holders, preparing disks to add to the dissolution vessel, performing standard operations, and storing the manipulators when not in use. The manipulators must be able to reach from floor to the height of the tallest vessel (0–127 cm). The system components are modular so that individual components can be removed and replaced when maintenance or replacement is needed.

Because they are too heavy and cumbersome to remove with manipulators alone, the modules are equipped with rings on the top to allow hooks from cranes to lift them. The manifolds are made of lightweight aluminum and can be bolted to the floor to avoid inadvertently knocking them over. Alternatively, the bases could be made to fit into notches in the hot cell floor or a lock-and-key mechanism could be devised. A large exterior hot cell door would be required to bring the modules in and out of the hot cell, or a long antechamber in which the modules could lay on their sides could be devised.

Flexible adapters are used to connect the evaporation vessel to its condenser system and the dissolution vessel to its condenser system. These flexible adapters are required unless fixed positioning is used for the system components. Even then, the system may prove difficult to wield with manipulators. Every connection must be gas tight, to provide adequate vacuum and no loss of material or vapor. It is unclear at this point whether flexible or rigid connections better suit the system.

FIGURE 10 Hot cell mockup with view from operator standpoint. No leaded glass is in place. *Left to right:* evaporation vessel, evaporation vessel condenser and collection vessel, dissolution vessel condensers and collection vessel, and dissolution vessel.

Evaporation System

The evaporation vessel (Figures 11A and 11B) is currently ~127 cm in height, with a diameter of ~34 cm at its widest point. The associated collection system (Figure 11C) has a height of ~97 cm and a diameter of ~24 cm at its widest point. A glass evaporation vessel is needed to view the interior liquid level, as the vessel is too cumbersome to weigh. Graduations can be marked on the exterior of the vessel to indicate the solution volume. The vessel alone is ~85 cm tall. The vessel's head, shown in Figure 11B, adds another ~35 cm from the combined ~18 cm head and the ~17 cm adapter to the stirring mechanism. The condenser system is attached with flexible adapters with glass 24/40 (standard taper) adapters.

The condenser system connects to the evaporation vessel by two pathways, to maximize the flow path volume. Two high-efficiency condensers and a gas inlet are affixed to a 3 L round-bottom vessel with a bottom drain. The bottom drain is currently controlled with a twist stopcock that can be replaced with a T-valve (for use with manipulators) or with a remotely operable valve. Condensate must be drained after every process.

FIGURE 11 (A) Evaporation vessel with heating mantle on 10L reaction vessel; (B) Evaporation vessel head with associated stirrer and connections to condenser system; and (C) Evaporation condenser system.

Dissolution System

The dissolution vessel (Figures 12A and 12B) has a height of ~97 cm and a diameter of ~31 cm at its widest point. Figure 12C shows the associated condenser system; it has a height of ~102 cm and a diameter of ~28 cm at its widest point. The glass vessel is needed to view the dissolution process. The operator must be able to observe the disks, the color of the solution, and the height of the reaction foam. The vessel itself is ~66 cm tall. The vessel's head is ~16.5 cm tall and the flexible adapters add another ~5 cm. The condenser system is attached with flexible adapters with glass 24/40 (standard taper) adapters.

The condenser system connects to the dissolution vessel by two pathways, to maximize the flow path volume. Three high-efficiency condensers are affixed to a 5 L round-bottom vessel with a bottom drain. The bottom drain is currently controlled with a twist stopcock that can be replaced with a T-valve (for use with manipulators) or with a remotely operable valve. Condensate must be drained after every process. The central condenser is open to the atmosphere to avoid pressurization of the system.

FIGURE 12 Dissolution vessel (A and B) and dissolution vessel condenser system (C)

Conversion to Stainless Steel

Stainless steel provides a chemically resistant material with durability that far exceeds glass. As previously mentioned, the dissolution and evaporation vessels must remain glass. A shield or shroud can be used to protect the vessels from inadvertent bumps with manipulators or other tools within the hot cell. Components eligible for conversion to stainless steel are the condensation systems (dissolution and evaporation), including the evaporation and dissolution vessel heads/lids. The vessel heads can be replaced with stainless steel flat caps to eliminate some of the headspace needed.

Introducing the disks to process through the flat cap of the dissolution vessel can be accomplished by several methods. One is to include a slide mechanism, where the disks are loaded one at a time. Another, more efficient, method is to use an insert that is loaded with the disks (presumably the irradiation target holder), introduce the disks into the dissolution vessel through a pass-through in the stainless steel cap, and have a mechanism that releases the disks into the vessel. The pass-through can be sealed with a plug once the target holder releases its payload. This requires a modest redesign of the dissolution holder. Flexible or rigid connections can be used to connect the modular systems together. Aside from the durability, an added benefit to condensation systems of stainless steel is that remotely operated or electric valves can be used to drain the system, minimizing remote operator manipulations.

5 ACKNOWLEDGMENTS

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APPENDIX A: LIST OF PART NUMBERS FOR DISSOLUTION APPARATUS

FIGURE A.1 Custom-made dissolution vessel (modified from 4 L reaction vessel, ChemGlass CG-1920-05)

Dissolution vessel lid 4-neck, 24/40 joint – ChemGlass CG-1941-03
www.chemglass.com/product_view.asp?pnr=CG-1941

Flexible adapters male to female 24/40 – SigmaAldrich ST/NS 24/40 – Z530891
www.sigmaaldrich.com/catalog/product/aldrich/z530891?lang=en®ion=US

4L evaporation vessel with drain valve – ChemGlass CG-1924-05
www.chemglass.com/product_view.asp?pnr=CG-1924

High Efficiency condensers for diss. vessel. (3pcs) – ChemGlass CG-1215-C-01
www.chemglass.com/product_view.asp?pnr=CG-1215-C

3L condensing flask 3-neck with drain valve – ChemGlass CG-1523-V-04
www.chemglass.com/product_view.asp?pnr=CG-1523-V

Evap. Vessel lid – 5 neck, 24/40 – ChemGlass CG-1946-01

www.chemglass.com/product_view.asp?pnr=CG-1946

Stirrer bearing – ChemGlass CG-2077-U-01

www.chemglass.com/product_view.asp?pnr=CG-2077-U

Stirrer shaft – ChemGlass CG-2074-01

www.chemglass.com/product_view.asp?pnr=CG-2074

Arrow overhead stirrer – ChemGlass CG-2021-01

www.chemglass.com/product_view.asp?pnr=CG-2021

Flexible drive shaft – ChemGlass CG-2047-01

www.chemglass.com/product_view.asp?pnr=CG-2047

Clamp for reaction flanges (2pcs) – ChemGlass CG-141-T-14

www.chemglass.com/product_view.asp?pnr=CG-141-T

Heating mantle for 4L vessel with drain – ChemGlass CG-10009-18

www.chemglass.com/product_view.asp?pnr=CG-10009

Condenser for evaporation vessel (2pcs) – ChemGlass CG-1215-A-10

www.chemglass.com/product_view.asp?pnr=CG-1215-A

1L condensing flask 3-neck with drain valve – ChemGlass CG-1523-V-02

www.chemglass.com/product_view.asp?pnr=CG-1523-V

Adapters 75° angle (2pcs) – 24/40 joint ChemGlass CG-1022-01

www.chemglass.com/product_view.asp?pnr=CG-1022

Adapter straight (2pcs) – 24/40 joint ChemGlass CG-1003-01

www.chemglass.com/product_view.asp?pnr=CG-1003

Inlet adapter 24/40joint (3pcs) ChemGlass CG-1014-E

www.chemglass.com/product_view.asp?pnr=CG-1014-E

Flow control adapter – ChemGlass CG-1029-G-01

www.chemglass.com/product_view.asp?pnr=CG-1029-G

JKEM controller MODEL 270

www.jkem.com/high-safety-controller-model-270

Recirculating cooler – Julabo FL 1701 (recommend one with higher cooling capacity; this one had highest capacity with 110V)

<https://punchout.fishersci.com/shop/products/fl1701-cooler-20-40c/507533950>

www.julabo.com/us/products/recirculating-coolers/fl1701-recirculating-cooler

Vacuum pump – plenty of options, should be corrosion resistant

APPENDIX B: HAND SKETCHES FOR HOT CELL MOCKUP HOLDERS

PETER TRAC
GLASSWARE SUPPORT
COLUMN # 1

ASSY

ASSY W/O NEW TOP PLATE
WEIGHS 13 1/2 LBS

1/3

PROGRAMS 1085
108113 OUTSIDE CONTOUR
1081131 CENTER HOLE
1081132 $\frac{1}{2}$ -13 HOLES

PETER TRAC
NORTH STAR
GLASS WARE SUPPORT
COLUMN #1

BASE PLATE $\frac{1}{2}$ " ALUMINUM STR

2/3

PETER TKAC
NORTH STAR
GLASS WARE SUPPORT
COLUMN #1

FIRST PLATE UP
FROM BASE $\frac{1}{8}$ ALUMINUM
SUPPORTS ROUND FLASK

HOLES
105 H33

2 ND PLATE UP, TOP
OF ROUND FLASK $\frac{1}{16}$ ALUMINUM

3/3

PETER TRAC
NORTH STAR
GLASSWARE SUPPORT
COLUMN #1

1081134 O.D.

COLUMN # 1

LIFT PLATE $\frac{1}{4}$ ALUMINUM

5/5

COLUMN # 2
ASSY.

COLUMN #2

COLUMN # 2

UPPER BEAKER SUPPORT PLATE

1/16 ALUMINUM

3/3

COLUMN # 3
ASSY

1/4

2/4

COLUMN #3

TOP OF FLASK PLATE
 $\frac{1}{16}$ ALUMINUM

CONDENSER SUPPORT PLATE

COLUMN # 3

4/4

ASSY COLUMN # 4
 WEIGHT 36 #

COLUMN # 4
TOTAL WEIGHT 36 #

COLUMN #4

BEAKER SUPPORT PLATE $\frac{1}{8}$ ALUMINUM

PLATE TOP OF BEAKER
 $\frac{1}{16}$ ALUMINUM

COLUMN #4

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