



Pacific Northwest
NATIONAL LABORATORY

Proudly Operated by Battelle Since 1965

Procedure for Uranium-Molybdenum Density Measurements and Porosity Determination

August 2016

Ramprashad Prabhakaran
Arun Devaraj
Vineet Joshi
Curt Lavender

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor Battelle Memorial Institute, nor any of their employees, makes **any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights.** Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or Battelle Memorial Institute. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

PACIFIC NORTHWEST NATIONAL LABORATORY
operated by
BATTELLE
for the
UNITED STATES DEPARTMENT OF ENERGY
under Contract DE-AC05-76RL01830

Printed in the United States of America

Available to DOE and DOE contractors from the
Office of Scientific and Technical Information,
P.O. Box 62, Oak Ridge, TN 37831-0062;
ph: (865) 576-8401
fax: (865) 576-5728
email: reports@adonis.osti.gov

Available to the public from the National Technical Information Service
5301 Shawnee Rd., Alexandria, VA 22312
ph: (800) 553-NTIS (6847)
email: orders@ntis.gov <<http://www.ntis.gov/about/form.aspx>>
Online ordering: <http://www.ntis.gov>



This document was printed on recycled paper.

(8/2010)

Procedure for Uranium-Molybdenum Density Measurements and Porosity Determination

Ramprashad Prabhakaran
Arun Devaraj
Vineet Joshi
Curt Lavender

August 2016

Prepared for
the U.S. Department of Energy
under Contract DE-AC05-76RL01830

Pacific Northwest National Laboratory
Richland, Washington 99352

Purpose/Scope

The purpose of this document is to provide guidelines for preparing uranium–molybdenum (U-Mo) specimens, performing density measurements, and computing sample porosity. Typical specimens (solids) will be sheared to small rectangular foils, disks, or pieces of metal. A mass balance, solid density determination kit, and a liquid of known density will be used to determine the density of U-Mo specimens using the Archimedes principle. A standard test weight of known density would be used to verify proper operation of the system. By measuring the density of a U-Mo sample, it is possible to determine its porosity.

Prerequisites and Precautions

Staff members who perform work in accordance with this procedure must be qualified. Assigned staff shall read and comply with applicable work control procedures and other applicable operating procedures.

Instructions for operating the equipment can be found in the manufacturers' instruction manuals. Do not operate the sectioning saw in the presence of flammable liquids, gases, or dust.

Handling radioactive materials/sources can result in exposure of workers to doses of radiation (internal and external). Care should be taken to identify these materials. Avoid direct handling of radioactive materials, and use remote handling tools.

Samples must be kept clean and properly stored for testing. Perform density measurements immediately after cleaning specimens.

Care must be taken to ensure that liquids are stored in properly labeled leak-proof closed containers. Good housekeeping practices should be followed during all operations in the fume hood, glove box, and on bench tops.

Contents

Purpose/Scope.....	iii
Prerequisites and Precautions	v
1.0 Introduction	1
2.0 Sample Receipt and Identification.....	1
3.0 Sample Sectioning for Uranium–Molybdenum Density Measurement.....	2
3.1 Equipment/Supplies	2
3.2 Preparation of the Cutting Equipment.....	2
3.3 Retrieving Materials for Sectioning	3
3.4 Sectioning.....	3
3.5 Job Completion and Cleanup	3
4.0 Sample Acid Cleaning Prior to Density Measurements	4
4.1 Equipment/Supplies	4
4.2 Acid Cleaning Uranium-Molybdenum Samples	4
5.0 Definition of Density and Archimedes’ Principle	5
6.0 Density Measurement Setup.....	5
6.1 Temperature	6
6.2 Choosing a Liquid of Known Density.....	6
6.3 Air Buoyancy	7
6.4 Air Bubbles	7
6.5 Immersion Depth of the Gem Holder.....	7
6.6 Surface Tension of the Liquid.....	8
6.7 Porous Bodies.....	8
7.0 Calibration of Analytical Mass Balance	8
7.1 Equipment	8
7.2 Procedure.....	8
8.0 Example – Analytical Mass Balance Verification.....	9
9.0 Density Measurement Setup.....	9
10.0 Density Measurement Operating Procedure	10
10.1 Equipment	10
10.2 Operating Procedure.....	10
10.3 Example with Buoyancy Indication	11
11.0 Example – Density Measurement with a Density Standard	11
12.0 Example – Density Measurement of U-Mo Specimen	12
13.0 Predicting Porosity from Uranium-Molybdenum Density	13
14.0 Upon Completion of Density Measurement	13
15.0 Waste Management	14
16.0 References	14

Appendix A Test Weight Calibration Certificate from the Vendor	A.1
Appendix B Mass Balance Verification.....	B.1
Appendix C Density Standard – Report from the Vendor	C.1
Appendix D Density Measurement Setup Verification	D.1
Appendix E Uranium–Molybdenum Density Measurement	E.1

1.0 Introduction

The U.S. Department of Energy's, National Nuclear Security Administration, Office of Material Management and Minimization, requires the use of metallic fuel to meet the objectives of the Reactor Conversion Program for the fleet of U.S. high-performance research reactors. The metallic fuel selected to replace the current fuel is low-enriched uranium 10-wt% molybdenum alloy (U-10Mo) in the form of a thin sheet or foil encapsulated in AA6061 aluminum alloy. The Fuel Fabrication Capability Pillar of Reactor Conversion has undertaken a series of tasks to meet fuel performance and schedule requirements, and a series of tasks have been undertaken that increase the understanding of the impact of processing conditions on the microstructure of the final fuel.

The chemistry of raw materials and processing methods can directly impact the final U-10Mo alloy quality, which is partially determined by the amount of undesirable phases such as carbides, MoSi_2 (molybdenum di-silicide), or oxide phases in the final U-10Mo alloy product and the effective ^{235}U enrichment in the U-10Mo matrix, devoid of second phases. At present, a clear correlation between the final U-10Mo microstructure and raw material chemistry or processing methods is lacking.

To establish quality benchmarks for fabricated U-10Mo fuel, mass balance calculations were developed to estimate the carbide volume fraction in final U-10Mo alloy as a function of carbon concentration. We also developed a mathematical model for estimating the final ^{235}U effective enrichment in the uranium–molybdenum (U-Mo) matrix of U-10Mo alloy after accounting for the loss of ^{235}U in the carbide phase. This model was validated by comparing its predictions with results from detailed microstructural characterizations and image processing undertaken at Pacific Northwest National Laboratory and composition measurements provided by the Y12 security complex. The carbide volume fraction and ^{235}U enrichment then was used to provide a direct estimation of the effective density of the final U-10Mo fuel as a function of carbon concentration in the final U-10Mo alloy.⁽¹⁾

A standard procedure is not available for measuring the density of uranium alloys or metal alloys in general. Hence, the purpose of this document is to provide guidelines for preparing U-Mo specimens and performing density measurements, based upon the Archimedes principle, ASTM D792-13 (Standard Test Methods for Density and Specific Gravity of Plastics by Displacement) and ASTM B311-13 (Standard Test Method for Density of Powder Metallurgy Materials Containing less than 2% Porosity) Standards.^(2,3)

In the procedure, typical specimens (i.e., solids) will be sheared to small rectangular foils, disks, or pieces of metal. A mass balance, a solid density determination kit, and a liquid of known density can be used to determine the density of U-10Mo specimens using the Archimedes principle. By measuring the density of the U-10Mo sample, it is possible to predict the porosity present in the sample.

2.0 Sample Receipt and Identification

Upon receipt of a U-Mo sample, an entry should be made in the Sample Log. The Sample Log entry should contain the sample identification number, sample description, and other relevant information to ensure positive sample identity and traceability. The log entry also should note the number of specimens (to be sectioned) required per sample and the type of preparation required. This information will be

determined by the cognizant engineer. The sample and/or sample container shall be marked with a sample identification number and tagged. The location of the sample also should be recorded in the Sample Log.

3.0 Sample Sectioning for Uranium–Molybdenum Density Measurement

For performing density measurements on a given U-Mo sample (foil/casting), we recommend that at least three different regions be selected and marked for sectioning into three (recommended) specimens per U-Mo sample. ASTM D792 suggests a minimum of two specimens per sample.⁽²⁾ Per ASTM D792, the test specimen shall be a single piece of material with a size and shape suitable for the testing apparatus, provided its volume is not less than 1 cm³. ASTM B311 recommends a specimen mass less than 10 g. For the highest precision, the test specimen should have a minimum mass of 5 g.⁽³⁾ Make sure to refer to the mass balance specifications, and choose a specimen mass that gives the best linearity and reproducibility. For example, the Mettler AE100 balance has a weighing range of 0 to 100 g, readability of 0.1 mg; reproducibility of 0.1 mg, linearity of ± 0.2 mg, and linearity relative to 10 g of ± 0.1 mg).

Proper cutting requires the correct selection of abrasive type, bonding, and size, as well as proper cutting speed, load, and coolant. Make sure to choose appropriate sectioning/cutting equipment based on sample dimensions (primarily the thickness).

This section of the procedure applies to shearing, cutting, sectioning, or machining radioactive samples in the fume hood or glove box using a low-speed saw, band saw, or bench shear. A contamination area or high contamination area may be created during the sectioning of the various materials. Sectioning operations are strictly limited to the fume hood or glove box.

3.1 Equipment/Supplies

- Bench shear
- Buehler IsoMet low-speed saw
- UKAM Smart Cut 6001 GP (for larger pieces)
- Water
- Terry towel (or similar absorbent material)
- Lubricating solution.

3.2 Preparation of the Cutting Equipment

1. Verify that the cutting equipment is working properly. Refer to its manual for instructions related to its operation.
2. In the fume hood, the equipment should be placed in such a way that the radioactive material is at least 6 inches from the front sill of the fume hood and that the air flow baffles at the rear of the fume hood are not blocked.
3. Prepare the fume hood or glove box by covering the area around the equipment with absorbent material to control the spread of contamination. For example, a terry towel or similar absorbent material may be placed around the equipment.

4. If appropriate, provide lubrication for the cutting blade or surface.
 - For the saw blade, either fill water in the lubrication pan or place a wet towel into the pan to lubricate the blade and collect the cutting residue. A paper towel also can be folded and inserted, so it can wipe the blade and minimize spraying of lubricant from the sample during sawing.

3.3 Retrieving Materials for Sectioning

Unpack and transfer the samples to the fume hood or glove box. If contamination is suspected or if levels of contamination are not known, the innermost or sample package of concern may be opened in the fume hood, glove box, or other contamination area. Make sure appropriate procedures are followed while working with radioactive materials.

3.4 Sectioning

1. Specimens should be sectioned from the sample as marked for performing density measurements. The location of the cut section with respect to the original sample should be recorded in the laboratory notebook.
2. Position the sample in the cutting fixture to cut specimens where marked. Clamp the sample to be sectioned in the sample chuck.
3. Before operating the equipment to section the material, make sure the blade can be lubricated continuously. Make sure the lubricant is contained by adjusting the blade speed to prevent splashing and spraying from the blade. A shield can be used to control the spread of contamination. ALARA (as low as reasonably achievable) principles should apply to minimize radiation dose to workers.
4. Turn on the cutting equipment and gently lower the sample onto the rotating blade (or if applicable, by gently lowering the blade onto the sample).
5. When operating the saw, decrease the speed of the blade, hold onto the cut section, and lift the fulcrum arm to prevent a back-slash cut to the sample when the blade has cut almost completely through the sample.
6. When operating the bench shear, make sure to use appropriate gloves and keep fingers away from the cutting blade.
7. Multiple cuts may be required to get the required number of specimens from a sample. Store specimens inside properly labeled containers.

3.5 Job Completion and Cleanup

1. Once sectioning is complete, remove the sample from the chuck. Collect all sectioned pieces. Clean the work area to reduce/remove contamination and arrange for a radiological survey. Based on the survey findings, determine if contamination is as low as can be reasonably achieved or if further cleaning is required. After cleaning, store the sample and sectioned pieces (specimens) in a clean, clearly labeled bag/containers. Transfer, if needed. Record the identification number and location of the cut pieces in the Sample Log.
2. Place any unused pieces (labeled bag) in a shielded area of the fume hood, glove box, or other shielded location. Record the identification number and location of these pieces in the Sample Log.

3. Place any waste material in a waste container in the fume hood or glove box. If the waste material has a high dose rate (as defined by a Radiological Control Technician), segregate it from the other waste material and shield it until the material can be removed for disposal.
4. Clean the equipment, chuck, and tools by washing and wiping the contaminated areas.
5. When using the saw, remove excess liquids from the saw pan by blotting up the liquids with an absorbent material. If using a damp towel to assist in the lubrication, dispose it using an absorbent material in a radioactive waste bag clearly marked with a description of the contents, the source of the material, and the contamination level.
 - Rinse the pan with water, and blot up the rinse water.
 - Remove the pan, and place it in a plastic bag.
6. Remove the absorbent material placed around the equipment and dispose of it in the fume hood or glove box waste container. Wipe the exterior surface of the equipment and surrounding area with a damp towel.
7. The cleaned equipment may be stored by placing it in a back corner of the fume hood or glove box.

4.0 Sample Acid Cleaning Prior to Density Measurements

U-Mo samples should be free from oil, grease, and other foreign matter. U-Mo samples typically oxidize when exposed to air for few hours. Hence, prior to density measurements, they must be cleaned to remove all oxides formed on the surface. Nitric acid is typically used to remove uranium oxidation products because of its orders-of-magnitude higher reactivity with uranium oxides compared to metallic uranium. The primary objective of the acid cleaning procedure is to remove all oxide products and expose the bare U-Mo metal so the mass of the solid can be measured accurately. Acid cleaning should be performed in a fume hood, according to the operational procedure. Density measurements should be performed immediately after acid cleaning.

4.1 Equipment/Supplies

- Concentrated nitric acid
- Deionized water
- Ethanol
- Containers for acid bath and water
- Fine-tip wire brush
- Compressed air.

4.2 Acid Cleaning Uranium-Molybdenum Samples

1. Prepare 35% nitric acid using deionized water. *Never add water to concentrated acid.*
2. Pour 35% nitric acid into an appropriately sized container.
3. Place the U-Mo specimens into the acid solution using tongs made of Teflon or coated with Teflon.

4. A fine-tip wire brush also can be used to lightly remove oxidation products on the surface of U-Mo specimens during acid cleaning.
5. Remove the U-Mo specimens from the acid solution after removing all oxidation products from the surfaces.
6. Transfer U-Mo specimens immediately into a glass container filled with deionized water, and move gently to accomplish a quick rinse.
7. Remove U-Mo specimens from the glass container and place them on a poly wipe to dry.
8. As an alternative approach, the U-Mo specimens can be rinsed with ethanol to decrease the drying time. Store specimens in appropriately labeled containers.
9. After cleaning, transfer the acid solution and water to an appropriate rad-waste container.

5.0 Definition of Density and Archimedes' Principle

The density, ρ , of a homogeneous body is the relationship of its mass to its volume or its mass per unit of volume. The unit of density is g/cm^3 . Note: $1 \text{ g/cm}^3 = 1 \text{ g/mL}$.

$$\text{Density} = \frac{\text{Mass}}{\text{Volume}}$$

According to DIN (German Engineering Standards) 1305, the term “weight” can be used instead of “mass.”

Archimedes' principle says that the apparent weight of an object immersed in a liquid decreases by an amount equal to the weight of the volume of the liquid that it displaces. Because 1 mL of water has a mass almost exactly equal to 1 g, if the object is immersed in water, the difference between the two masses (in grams) will equal (almost exactly) the volume (in mL) of the object weighed. By knowing the mass and the volume of an object, the density can be calculated.

6.0 Density Measurement Setup

The density of a solid can be measured using an analytical mass balance (with a minimum readability and reproducibility of 0.1 mg) and a kit for solid density measurements. The kit is used to measure the mass of a solid (i.e., a U-Mo specimen) in air and water. The analytical mass balance must be supported in a manner that eliminates mechanical vibrations and be shields it from air drafts. A glass beaker or other suitable transparent container should be used to contain the liquid so the presence of air bubbles can be easily discerned.

The Mettler AE200 balance and the Mettler Density Determination Kit for Solids (Part #33360) are examples of suitable equipment.

The following factors should be considered before measuring the density of a solid (e.g., U-10Mo specimens): liquid temperature, choice of a liquid of known density, air buoyancy, presence of air bubbles, immersion depth of the gem holder, surface tension of the liquid, and porosity of the material being tested. These factors are described in the following sections.

6.1 Temperature

In this setup, the density of a solid body is determined by using a liquid of known density. *The temperature of the liquid must always be taken into consideration when the density is to be determined with better than 1 % accuracy.* A thermometer readable to 0.1°C or better is required.⁽²⁾ Densities of water at different temperatures are shown in Table 1.

Table 1. Density of Distilled Water

(According to "Handbook of Chemistry and Physics" 66th Ed. 1985-1986, F4-F5)

Temperature (°C)	Density (g/ ml)	Temperature (°C)	Density (g/ ml)
15.0	0.9991	24.0	0.9973
15.5	0.9990	24.5	0.9972
16.0	0.9990	25.0	0.9970
16.5	0.9989	25.5	0.9969
17.0	0.9988	26.0	0.9968
17.5	0.9987	26.5	0.9966
18.0	0.9986	27.0	0.9965
18.5	0.9985	27.5	0.9964
19.0	0.9984	28.0	0.9962
19.5	0.9983	28.5	0.9961
20.0	0.9982	29.0	0.9959
20.5	0.9981	29.5	0.9958
21.0	0.9980	30.0	0.9956
21.5	0.9979	30.5	0.9955
22.0	0.9978	31.0	0.9953
22.5	0.9977	31.5	0.9952
23.0	0.9975	32.0	0.9950
23.5	0.9974		

In the case of solid bodies, the change in density caused by a change in temperature is generally so small that the temperature of a solid body (e.g., a U-Mo specimen) can be disregarded as far as the density determination is concerned.

6.2 Choosing a Liquid of Known Density

Typically, distilled water is used as a liquid with known density for measuring the density of a solid, such as the density standard. *However, for U-Mo specimens, it is recommended that water be avoided.* Hence, solutions such as 3M Fluorinert Electronic Liquid FC-43 can be used. Make sure the liquid does not contain dust or debris.

FC-43 is a clear, colorless, fully fluorinated liquid. Like other Fluorinert Electronic Liquid products, FC-43 is thermally and chemically stable, compatible with sensitive materials, nonflammable, and leaves essentially no residue upon evaporation.

According to the 3M Fluorinert Electronic Liquid FC-43 specification sheet, the liquid density is 1.860 g/cc (25°C). The following formula can be used to calculate the density of Fluorinert Electronic Liquid FC-43 at various temperatures.

$$\text{Density} \left(\frac{\text{g}}{\text{cc}} \right) = \frac{[1913 - 2.18 (T)]}{1000}$$

where, T is temperature in °C.

For all other liquids, the density at temperature T must be taken from published tables.

6.3 Air Buoyancy

Depending on physical conditions, the weight of a cubic centimeter of air ranges from 1.0 to 1.2 mg. Thus, any object that is being weighed in air is subject to this kind of buoyancy for each cubic centimeter of its volume. This means for a density of 1 g/cm³, an error of approximately 0.1% would occur if air buoyancy is not taken into consideration. *Thus, if density data with three or four decimal places are required, the result will have to be corrected for the air buoyancy.* The true density is about 0.001 g/cm³ more than the calculated density.

6.4 Air Bubbles

Water is a poor wetting liquid. Hence, when using water without a wetting agent, it is possible for air bubbles to adhere to the submerged solid body or gem holder, which is the wire basket that holds the specimen. *Because of their buoyancy, air bubbles could affect the results.* A bubble 1 mm in diameter would cause a buoyancy of 0.5 mg, while a bubble 2 mm in diameter would have a buoyancy of as much as 4 mg.

Precautionary measures that should be taken are listed below:

- Degrease solids that are resistant to solvents.
- Periodically clean the gem holder and sinker; do not touch immersible parts with your hands.
- To loosen air bubbles that might stick to it, gently shake the gem holder when first immersing it in the liquid before suspending it from hook.
- Use wetting agents or organic solvents as auxiliary liquid (e.g., Kodak Photo-Flo; Pervitro 75% Mettler #72409).

Note: The change of density caused by the addition of a wetting agent to distilled water is negligible. If, for example, 0.1 mL of a wetting agent with a density of 1.2 g/cm³ is added to 250 mL of water, the overall density changes by 0.001 g/mL.

6.5 Immersion Depth of the Gem Holder

The submersible part of the gem holder has a diameter of 0.8 mm and is made of corrosion-resistant wire. With a liquid density of approximately 1 g/cm³, the resulting buoyancy is approximately 5 mg for each 10 mm that is submerged. Because the gem holder remains submerged while the solid body is weighed in air and, with electronic balances, the immersion depth does not change from one weighing to the next (in spite of the difference in weight), *the buoyancy of the gem holder remains constant and can thus be disregarded. Note: Do not change the liquid level.*

The change in liquid level caused by submerging the solid body can be disregarded in most cases. A solid body with a volume of 1 cm^3 causes the liquid to rise by about 0.5 mm. This is equivalent to a buoyancy of about 0.15 mg (i.e., a density error of 0.00015 g/cm^3).

6.6 Surface Tension of the Liquid

Because the liquid adheres to the suspension wire, an apparent weight increase occurs. A force of up to 3 mg can act on the gem holder due to the wire being submerged in the water. *However, any influence on the result will be virtually eliminated by having the gem holder submerged during both weighings (i.e., in air and in water).* For very high accuracy requirements, reducing the surface tension would constitute an additional precautionary measure (see Section 6.4).

6.7 Porous Bodies

Because submerging porous bodies in a liquid do not generally cause a 100% displacement of air within the pores, errors will occur as a consequence. Thus the density of the body can only be approximated.

7.0 Calibration of Analytical Mass Balance

For a mass balance, regular calibration is critical. A test weight should be used to verify proper functioning of the mass balance before using the setup for density measurements.

7.1 Equipment

- Analytical mass balance such as Mettler Toledo AE200
- Test weight (e.g., 5 g)
- Tweezers.

7.2 Procedure

1. Make sure the mass balance is free from debris.
2. Verify that the calibration of the mass balance is current.
3. Close all the doors of the mass balance and tare it. Make sure it reads all zeros.
4. Place a test weight on the mass balance and record the mass. Repeat this step three times. Make sure the readings are within the acceptable tolerance.
5. Refer to Appendices A and B for more information.

8.0 Example – Analytical Mass Balance Verification

This section provides an example of an analytical mass balance verification (Mettler Toledo AE200) using a 5 g test weight obtained from Rice Lake Weighing System (5 g Weight OIML Class E2 [Part #29766]). Refer to Appendix A for Test Weight Calibration Certificate from the vendor. The following measurements were made using the information provided in the Appendix B. A similar table should be entered in the laboratory notebook.

Date	Reading 1	Reading 2	Reading 3	Average	Standard Deviation
4/8/2016	10.0006 g	10.0006 g	10.0006 g	10.0006 g	0.0

9.0 Density Measurement Setup

The density of a solid can be measured using an analytical mass balance (with a minimum readability and reproducibility of 0.1 mg) and a kit for solid density measurements. The kit is used to measure the mass of a solid (e.g., a U-Mo specimen) in air and water.

In this document, the density measurement setup description is based on the Mettler AE200 balance and Mettler Density Determination Kit for Solids (Part #33360). For other balances and density determination kits, please refer to the applicable manual for setup instructions. The numbers below refer to the solid density measurement kit components shown in the Figure 1.

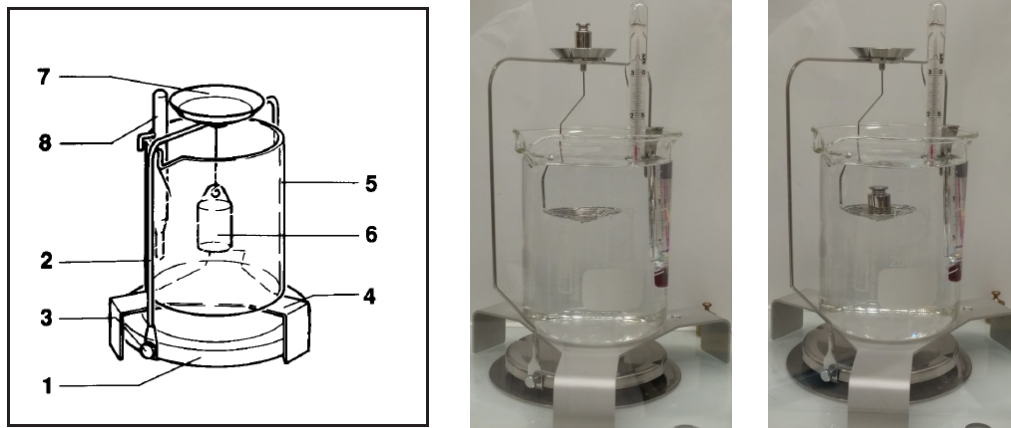


Figure 1. Mettler AE200 Balance with Density Determination Kit for Solids (Part #33360)

1. Locate the Mettler Density Determination Kit for Solids (Part #33360)
2. Remove the weighing pan (#1) and then attach the bracket (#2) to it using a clamping screw (#3).
3. Place the weighing pan (1) with the attached bracket (2) back in its original location.
4. Place the bridge (#4) inside balance housing across the weighing pan (#1).
5. Make sure that the weighing pan (#1) and bracket (#2) are not in contact with the bridge (#4).
6. Place a 250 mL beaker (#5) filled with a liquid of known density on the bridge (#4).
7. Make sure there is sufficient liquid of known density so that solid body after being placed in the wire basket of the gem holder (6) will be covered by at least 10 mm of liquid.

8. Attach the gem holder (#6). Watch for air bubbles, especially ones on the wire basket.
9. Make sure that the suspension (#7) (i.e., the gem holder) is hanging freely in the center of the beaker (#5) without touching the sides.
10. Attach the thermometer (#8) to the beaker.
11. Make sure the beaker (5) and thermometer (8) are not in contact with the bridge (4).

10.0 Density Measurement Operating Procedure

10.1 Equipment

- Analytical mass balance (e.g., Mettler Toledo AE200)
- Solid density determination kit (e.g., Mettler Part #33360)
- Density standard (e.g., 5 g standard with a measured density report from the vendor [e.g., Rice Lake Weighing System Weight Kit 5g OIML Class E2 Part #29766])
- A liquid of known density (distilled water for the density standard; liquid such as 3M Fluorinert Electronic Liquid FC-43 for U-Mo samples)
- Tweezers.

10.2 Operating Procedure

First, use a density standard (with a known density, as a solid body) and measure its density by following the steps listed below. Compare the measured density value with the value shown in the Density Standard Report to verify the operation of the density measurement setup. After successful verification, use the test specimen (solid) to measure its density.

1. Tare balance to read exactly zero.
2. Make sure the solid body is completely dry.
3. Place the solid body (e.g., density standard or test specimen) in the upper cup of gem holder.
4. Record the weight displayed by the balance (in grams). This is the weight, A , of the solid body when weighed in the air.
5. Again tare the balance (with the solid in the upper cup).
6. Remove the solid body from the upper cup and place it in the lower wire basket (in liquid) carefully using tweezers.
7. The balance now displays buoyancy, P , of the solid body (preceded by a minus sign).
8. Record the temperature of liquid.

9. Divide the weight, A, recorded from Step 4 by the buoyancy, P, recorded from Step 7, and then multiply this intermediate result by the density, ρ_0 , of the auxiliary liquid (at the given temperature T obtained in Step 8).

$$\text{Density of solid body} = \left(\frac{A}{P}\right) \times \text{density of known liquid}$$

10. The final result is the density of the solid body, ρ . The unit of measurement is the same as that used for density of the auxiliary liquid, ρ_0 (e.g., g/cm^3 [identical to g/mL]).
11. If the direct buoyancy reading is not used, P can also be obtained from A – B. The balance will display B instead of P if it was not tared after result A was displayed or if it was tared with the solid body removed.
12. Repeat the above steps (after drying the specimen completely after each measurement) to get at least six readings.
13. Refer to Appendix B and Appendix C for more information.

10.3 Example with Buoyancy Indication

Following is an example calculation for determining the density of a coin using distilled water.

Measurement values: A = 3.011 g; P = 0.336 g; Temperature = 25.5°C

Density ρ_0 of the distilled water: 0.997 g/cm^3 or 0.997 g/mL

$$\text{Density of coin} = \left(\frac{3.011}{0.336}\right) \times 0.997 = 8.93 \text{ g/ml}$$

11.0 Example – Density Measurement with a Density Standard

This section provides an example of the density measurement setup verification (Mettler Toledo AE200 and a Mettler Solid Density Determination Kit # 33360) using a density standard obtained from a vendor (5 g density standard/test weight from Rice Lake Weighing System; OIML Class E2 Part #29766) in water. Refer to Appendix A and C for the Test Weight Calibration Certificate and Density Report, respectively, that were obtained from the vendor. Distilled water was used as the liquid of known density. The measurements shown below were made using information provided in the Appendix D.

Reading #	Temperature of the liquid (°C)	Weight of Solid in Air (A) in grams	Buoyancy (P) in grams	A/P	Density of Solid in g/cc = (A/P)*Liquid Density
1	22	4.9990	0.6240	8.0112	7.9936
2	22	4.9991	0.6247	8.0024	7.9848
3	22	4.9992	0.6268	7.9757	7.9582
4	22	4.9992	0.6264	7.9808	7.9633
5	22	4.9992	0.6258	7.9885	7.9709
6	22	4.9991	0.6255	7.9922	7.9746

Average measured density of density standard (six readings): 7.9742 g/cc; standard deviation: 0.0132 g/cc

Density (per the Vendor Report): 7.9787 g/cc (uncertainty: 0.0068 g/cc)

12.0 Example – Density Measurement of U-Mo Specimen

This section provides an example of the U-Mo specimen density measurement using a Mettler Toledo AE200 balance and a Mettler Solid Density Determination Kit # 33360. 3M Fluorinert Electronic Liquid FC-43 was used as the liquid with known density. The following measurements were made using the information provided in the Appendix E.

According to 3M specification sheet, the density of FC-43 at various temperatures can be determined as follows:

$$\text{Density (g/cc)} = [1913 - 2.18 (T)]/1000$$

where T is the temperature of the liquid in °C.

Specimen # DUM 53

Reading #	Temperature of the liquid (°C)	Weight of Solid in Air (A) in grams	Buoyancy (P) in grams	A/P	Density of Solid in g/cc = (A/P)*Liquid Density
1	22.4	6.8053	0.7477	9.1016	16.9670
2	22.4	6.8054	0.7476	9.1030	16.9695
3	22.6	6.8054	0.7476	9.1030	16.9655
4	22.6	6.8056	0.7477	9.1020	16.9638
5	22.6	6.8056	0.7476	9.1033	16.9660
6	22.6	6.8055	0.7475	9.1043	16.9681

Average measured density of density standard (six readings): 16.9667 g/cc

Standard deviation (six readings): 0.0020 g/cc

13.0 Predicting Porosity from Uranium-Molybdenum Density

Porosity in a U-Mo sample can be calculated by determining the theoretical density and measured density of the U-Mo alloy. The theoretical density of U-Mo alloy can be calculated as follows:

$$\frac{1}{\text{Theoretical Density}} = \left(\frac{\text{Mass Fraction of U}}{\text{Density of U}} \right) + \left(\frac{\text{Mass Fraction of Mo}}{\text{Density of Mo}} \right) + \left(\frac{\text{Mass Fraction of C}}{\text{Density of C}} \right)$$

where:

U = Uranium; Density = 19.1 g/cc

Mo = Molybdenum; Density = 10.28 g/cc

C = Carbon; Density = 2.26 g/cc

Example: A 100 g sample of U-10Mo alloy with 1000 ppm (weight) of carbon

The theoretical density can be determined as follows:

The following designation can be used for the simplicity: U90-xM10Cx (where x is the weight fraction of carbon that can vary).

% weight of C = weight ppm/10000 = 1000/1E4 = 0.1 %

% weight of Mo = 10 %

% weight of U = 89.9 %

$$\frac{100}{\text{Theoretical Density UMo alloy}} = \left(\frac{89.9}{19.1} \right) + \left(\frac{10}{10.28} \right) + \left(\frac{0.1}{2.26} \right) = 17.47 \text{ g/cc}$$

After calculating the theoretical density and measuring the density of the sample, it is possible to predict the porosity as follows:

$$\text{Porosity \%} = \frac{(\text{Theoretical Density} - \text{Measured Density})}{\text{Theoretical Density}} \times 100$$

14.0 Upon Completion of Density Measurement

1. After completing the last test of the day/session, make sure that U-Mo specimens are safely stored in the correct storage bag.
2. Make sure the work space is free of debris.
3. Make sure liquids are stored in properly labeled, leak-proof closed containers.
4. Properly dispose of used/contaminated liquid in leak-proof, closed containers with appropriate radiological labels.

5. Perform any needed housekeeping or waste removal.
6. Record changes in the location of the specimen(s) tested in the Specimen Transfer Log.

15.0 Waste Management

Only a minimum amount of waste will be generated under this operating procedure. Generated low-level waste may include potentially contaminated immersion water/fluid, gloves, and paper products.

16.0 References

1. Devaraj A, R Prabhakaran, VV Joshi, SY Hu, EJ McGarrah, and CA Lavender. 2016. *Theoretical Model for Volume Fraction of UC, 235U Enrichment, and Effective Density of Final U10Mo Alloy*. PNNL-SA-117284, Pacific Northwest National Laboratory, Richland, Washington.
2. ASTM D792-13. 2013. Standard Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement. ASTM International, West Conshohocken, Pennsylvania. www.astm.org
3. ASTM B311-13, 2013. Standard Test Method for Density of Powder Metallurgy (PM) Materials Containing Less Than Two Percent Porosity. ASTM International, West Conshohocken, Pennsylvania. www.astm.org.

Appendix A

Test Weight Calibration Certificate from the Vendor

Appendix A

Test Weight Calibration Certificate from the Vendor

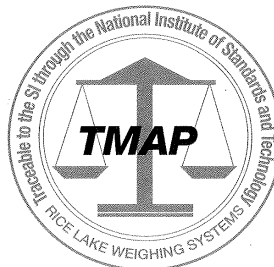


ANSI/NCSL Z540-1-1994; Part 1 & ISO/IEC 17025 Accredited

Contractor: **Battelle Northwest**
PO Box 999
Richland, WA 99352

Purchase Order #: **CC**
Client: **Battelle Northwest**
Address: **Receiving Bldg 1166**

City & State: **Richland, WA 99352**
Date Received: **13 APR 2016**
Date Calibrated: **22 APR 2016**
Temperature Range: **21.35 °C**
Pressure Range: **734.6 mmHg**
Relative Humidity Range: **48 %**
Air Density: **1.1531 mg/cm³**
Traceable Report #: **2425548**
NIST Certificate #: **684/286541-15, 684/284451-14**
Tested By: **12**
Procedure: **Mass Dissemination (reference HB 952)**



Primary Standard Calibration Date: **03/16/15, 11/26/13** Due: **03/15/19, 11/24/17**
Description of Weights: **5 g Polished Stainless Steel Weight, OIML Class E2, S/N 6J14**
Although there are two NIST numbers, one or both may apply.

Nominal Value	Id.	Conventional Mass Corr.		Unc. ¹ K=2 (mg)	Tol. (mg)	Balance Used	Standard Set Used Calibrated/due MM-DD-YY/MM-DD-YY	Assumed Density (g/cm³)
		As Found (mg)	As Left (mg)					
5 g		0.0124	0.0124	0.0045	0.050	650Q	L595Q 04-11-16/07-11-16	7.9787

¹ Uncertainties apply to As Left values only

☐ This report contains data not covered by the NVLAP Accreditation if the box is checked.

Check with your local state agency for certification of compliance on Legal for Trade Items.
The weight tolerance class is referenced in the Description of Weights. Unless otherwise noted, weights tested meet the requirements of the class.
*The specifications for the weight classes can be found in NIST Handbook 105-1, ASTM E-617 or OIML R111.

Prepared By:
Rice Lake Weighing Systems
230 West Coleman Street, Rice Lake, WI 54868 • USA
TEL: 715-234-9171 • FAX: 715-234-6967 • www.ricelake.com
An ISO 9001 registered company

Page 1 of 1 Page

Dated **22 APR 2016**

Dan Demers Metrologist



This report is not to be used to claim product endorsement by Rice Lake Weighing Systems, NVLAP or any agency of the U.S. Government. This document shall not be reproduced, except in full, without the written approval of Rice Lake Weighing Systems' Metrology Laboratory. Uncertainties are assigned using NIST SOP 29 in conformance with the GUM and include uncertainty components for reference standard, process, air buoyancy correction and bias/drift. Not included are magnetism, handling and use. The coverage factor *k* provides a level of confidence of approximately 95 percent.

PN 64784 1

Appendix B

Mass Balance Verification

Appendix B

Mass Balance Verification

Mass Balance Calibration Current (Yes/No):

Mass Balance Readability (minimum: 0.1 mg):

Mass Balance Reproducibility (minimum: 0.1 mg):

Test weight (example: 5 g):

Date	Reading 1	Reading 2	Reading 3	Average	Standard Deviation

Appendix C

Density Standard – Report from the Vendor

Appendix C

Density Standard – Report from the Vendor

RLWS DENSITY REPORT

Contractor: **Battelle Northwest**
PO Box 999
Richland WA, 99352
CC

Purchase Order Number: **Battelle Northwest**
Client: Receiving Bldg 1166
Richland WA, 99352

Date Received: 13-Apr-2016
Date Calibrated: 15-Apr-2016
Temperature Range: 21.54 °C
Pressure Range: 732.4 mmHg
Relative Humidity Range: 50.8 % rh
Air Density Range: 1.1485 mg/cm³
Traceable Report Number: 2425548D
Tested By: 20
Procedure: WI05-0036

Description of Weights: 5 g Polished Stainless Steel Weight, OIML Class E2, S/N 6J14



Nominal Value	ID	Tested Density (g/cm ³)	Unc. K=2 (g/cm ³)	Balance Used	Standard Set Used Calibrated/Due MM-DD-YY/MM-DD-YY
5 g		7.9787	0.0068	636Q	C1018Q 12-17-15/12-15-17

Check with your local state agency for certification of compliance on legal-for-trade items.

Calibration At:



230 West Coleman Street • Rice Lake, WI 54868 • USA
TEL: 715-234-9171 • FAX: 715-234-6967

Page 1 of 1 Page

Dated 20 Apr 2016

Dan Demers Metrologist

This report is not to be used to claim product endorsement by Rice Lake Weighing Systems or any agency of the U.S. Government.
This document shall not be reproduced, except in full, without the written approval of Rice Lake Weighing Systems' Metrology Laboratory.

PN 67126 1/01

Appendix D

Density Measurement Setup Verification

Appendix D

Density Measurement Setup Verification

Date:

Mass balance calibration current (Yes/No):

Mass Balance verified using a test weight (Yes/No):

Mass balance readability (minimum: 0.1 mg):

Mass balance reproducibility (minimum: 0.1 mg):

Density standard (example: 5 g test weight with a density report):

Liquid used for density measurement:

Reading #	Temperature of the liquid (°C)	Weight of Solid in Air (A) in grams	Buoyancy (P) in grams	A/P	Density of Solid in g/cc = (A/P)*Liquid Density

Average measured density of density standard (six readings):

Standard deviation (six readings):

Appendix E

Uranium–Molybdenum Density Measurement

Appendix E

Uranium–Molybdenum Density Measurement

Mass balance calibration current (Yes/No):

Mass balance verified using a test weight (Yes/No):

Density measurement setup verified using a density standard (Yes/No):

U-Mo specimens cleaned using acid (Yes/No):

Mass balance readability (minimum: 0.1 mg):

Mass balance reproducibility (minimum: 0.1 mg):

Liquid used for density measurement:

Reading #	Temperature of the liquid (°C)	Weight of Solid in Air (A) in grams	Buoyancy (P) in grams	A/P	Density of Solid in g/cc = (A/P)*Liquid Density

Average density of U-Mo specimen (6 readings):

Standard deviation (six readings):



Pacific Northwest
NATIONAL LABORATORY

*Proudly Operated by **Battelle** Since 1965*

902 Battelle Boulevard
P.O. Box 999
Richland, WA 99352
1-888-375-PNNL (7665)

U.S. DEPARTMENT OF
ENERGY

www.pnnl.gov