INL/MIS-21-61303-Revision-0

Yttrium Hydride Post-Irradiation Examination Plan

February 2021

Changing the World's Energy Future

Mahmut Nedim Cinbiz, Chase N Taylor, Thomas A Johnson

Idaho National Laboratory

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Prepared for the
U.S. Department of Energy
Under DOE Idaho Operations Office
Contract DE-AC07-05ID14517

Document ID: PLN-6268
Revision ID: 0
Effective Date: 02/08/2022

Plan

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EXECUTIVE SUMMARY

The Department of Energy Office of Nuclear Energy's Microreactor program aims to provide infrastructure and a qualification bed for the development of micro nuclear reactors. One key need is the development and qualification of high-temperature moderators that reduce fuel loading and ensure their compact design for flexible operational purposes. Because microreactors must be compact to offer a flexible operational purpose, metal hydrides are expected to be used as neutron moderator materials due to their high hydrogen number density. Los Alamos National Laboratory (LANL) is developing qualified fabrication techniques for metals hydride, especially yttrium hydride (YH_x) which provides advantages over other metal hydride candidates. Qualification of YH_x involves the fabrication process, thermomechanical properties, and performance which must be met for the deployment of microreactors. In the support of the qualification process metal hydride, LANL has developed fabrication techniques based in direct hydriding and powder metallurgy. For both manufacturing techniques, fresh material properties of metal hydrides have been reported in the literature. In order to establish the performance limits and the qualification parameters, an irradiation campaign has been initiated at Idaho National Laboratory (INL). The irradiation campaign aims to investigate the critical irradiated YH_x properties as a function of irradiation temperature and fabrication method. The first phase of the irradiation campaign includes the effect of the operating temperature under neutron irradiation which provides initial data on the metal hydrides and informs the manufacturing processes. A drop-in irradiation experiment for use in the INL Advanced Test Reactor (ATR) has been designed. This document provides the post-irradiation examination (PIE) plan of the ATR irradiated YH_x. The objective of the PIE plan is to serve as a qualification basis for YH_x as a neutron moderator material by understanding the impacts irradiation has on the physical and thermophysical properties, to establish a link between fabrication and the irradiated YH_x properties, to institute a PIE strategy for other solid-state neutron moderator candidates, and to inform microreactor fuel performance and safety codes.

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ACRONYMS

AL	Analytical Laboratory
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
ATF	Accident Tolerant Fuel
ATR	Advanced Test Reactor
BSE	Backscattered Secondary Electron
DE	destructive
DOE	Department of Energy
DOE-NE	Department of Energy Nuclear Energy
DSC	differential scanning calorimetry
EBSD	Electron backscatter diffraction
EDS	Energy dispersive spectroscopy
FASB	Fuels and Applied Science Building
GDOES	Glow- Discharge Optical Emission Spectroscopy
HFEF	Hot Fuel Examination Facility
IMCL	Irradiated Materials Characterization Laboratory
INL	Idaho National Laboratory
IRC	INL Research Center
LANL	Los Alamos National Laboratory
LECO	Laboratory Equipment Corporation
LFA	Laser-flash analysis
LI	Laboratory Instruction
MFC	Materials and Fuels Complex

NDE	non-destructive
NRAD	Neutron Radiography (Reactor)
PAS	Positron Annihilation Spectroscopy
PCA	Performer Controlled Activity
PGS	precision gamma scanner
PI	Principal Investigator
PIE	Post-irradiation examination
QA	Quality Assurance
SE	Secondary Electron
SEM	Scanning electron microscopy
SME	Subject Matter Expert
STAR	Safety and Tritium Applied Research
TEM	Transmission electron microscopy
VEM	Visual Examination Machine
WGS	Waste Generation Service
WPS	Welding Procedure Specification (American Welding Society)
XRD	X-ray diffraction
YH_x	yttrium hydride

1. INTRODUCTION

1.1 Background

An overarching goal of the Department of Energy Office of Nuclear Energy (DOE-NE) microreactor program is to develop technologies for the deployment of civilian microreactors by stakeholders [1]. Microreactors are expressed as advanced transportable nuclear reactors operating at low power (<20MWth) but high temperatures (>600°C), as well as plug-and-play and inherently safe designs. One prerequisite of a microreactor is the compactness, such that a truck can transport the reactor under safe conditions with the current road infrastructure [1,2]. The compactness of these reactors can only be attainable by use of dense material components for the essentials of the nuclear core, such as fuel, core heat removal components, reflectors, and moderators. Among these essentials, the largest contribution to the compactness is offered by use of solid moderators which benefits from light atomic weight elements, such as hydrogen, carbon, and beryllium [2]. Among these, hydrogen-bearing materials, from the lowest critical mass standpoint, such as metal hydrides are superior to other options. Noting that, factors other than critical mass should be considered for a specific reactor design.

Yttrium- or zirconium-based metal hydrides have been down-selected due to their neutronic performance. In addition to the neutronic perspective, maintaining hydrogen within the metal hydride is critical at high-operating temperatures of advanced reactors. Yttrium hydride is, therefore, a proposed moderator material that offers better hydrogen retention than zirconium hydrides due to higher retention and thermal stability of hydrogen in the metal [3].

The irradiated materials properties of metal hydrides, in this case yttrium hydride (YH_x), must be assessed for the qualification of these moderators. Material testing and inspection processes must illustrate that the effects of dimensional and property changes on thermophysical and mechanical properties do not cause any significant changes on the microreactor safety, and the moderating power is maintained within design limits. Thus, the effect of irradiation on the thermophysical and mechanical properties must be determined. This post-irradiation examination (PIE) plan specifically aims to determine these properties for YH_x following Advanced Test Reactor (ATR) irradiation.

1.2 Summary of LANL-MOD-1 Irradiations

The Los Alamos National Laboratory (LANL)-MOD-1 irradiation is a drop-in experiment that aims to examine the irradiation performance of YH_x and investigate any dimensional changes and determine the thermophysical properties of irradiated YH_x. Details of the LANL-MOD-1 irradiations are described in the Idaho National Laboratory (INL) reports of "TFR-1049 LANL-MOD-1 Irradiation in the ATR NEFT Small B Positions" [4] and "DP-157 LANL-MOD-1 Experiment Data Package." [5] Irradiation campaign uses one ATR basket that contains three stacked ATR fixtures as shown in Figure 1. Dimensions of the important components are listed in Table 1 as well.

Disk and cylindrical YH_x specimens were stacked in these LANL capsules (see Figure 1). Additional titanium-zirconium-molybdenum (TZM) disk-shaped sheets were placed in between RUS, Glow-Discharge Optical Emission Spectroscopy (GDOES), and laser-flash analysis (LFA) samples. These thin TZM sheets will also be used as samples. At the bottom of a portion of the LANL capsule, differential scanning calorimetry (DSC) specimens were surrounded by a TZM ring.

LANL capsules were made of TZM alloy, and they were sealed via welding under inert gas pressure.

Each of the three ATR fixtures consists of two LANL capsules and melt wires between two capsules (see Figure 1). Assembly steps of an ATR fixture are shown in Figure 2. LANL capsules and melt wires were inserted into a half piece of a clam shell ATR fixture, and the opposite piece of the clam shell ATR fixture was placed onto the other half. After this step, the ATR fixture was clamped from each side. The three clamped clam shells were slid inside a tube. This ATR basket was welded and, finally, was inserted into the B2 position of ATR. In total, six LANL capsules are subjected to one ATR cycle, 169A.

Table 1. Dimensions of components in a single LANL capsule loaded into an ATR fixture (INL-DWG-822579). Each ATR fixtures consist of two LANL capsules.

Component	Material	Quantity	Inner diameter (mm)	Outer diameter (mm)	Height (mm)	Depth (mm)
Sample Column	YH_x(RUS)	3	12.5	N/A	10	N/A
	YH_x(GDOES)	4	12.5	N/A	2	N/A
	YH_x(TEM)	1	12.5	N/A	2	N/A
	YH_x(LFA)	2	12.5	N/A	2	N/A
	YH_x(DSC)	6	5.0	N/A	1.5	N/A
	TZM sheet	5	12.5	N/A	0.51	N/A
	TZM ring	1	12.5	5	9	N/A
Capsule	TZM	2	14.0	13.5	63.6	N/A
Capsule lid	TZM	4	13	12	2	1.5
ATR Fixture	SS304L	1	Variable	14.43	174.9	N/A
Core holder	SS304L	1	21.01	18.47	543.3	N/A

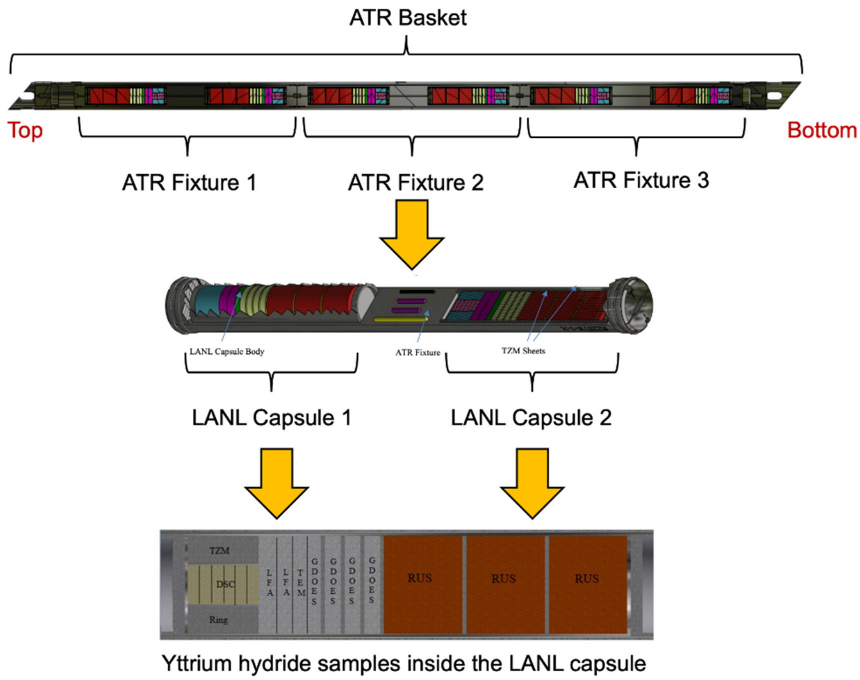

Figure 1. Arrangement of ATR basket, ATR fixtures, LANL capsules, and yttrium hydride sample. In total, six LANL capsules are subjected to irradiations in one ATR basket. Melt wires are not shown in this schematic.

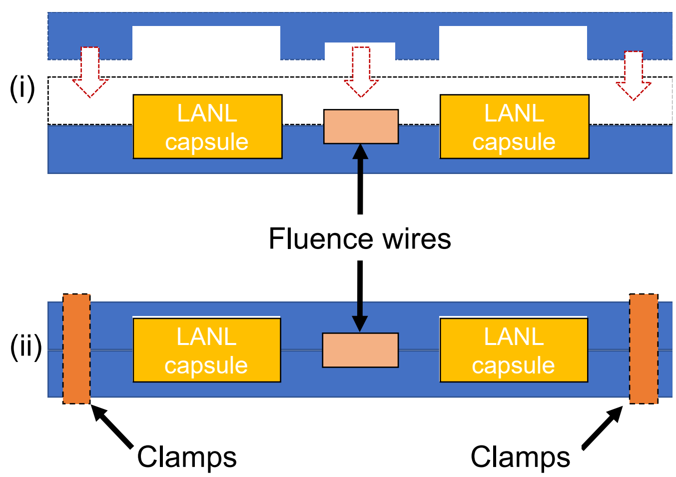

Figure 2. Schematics for an ATR fixture assembly steps.

Figure 3 shows the predicted temperatures of each ATR fixture during irradiation. Temperature predictions and other engineering data are reported in "INL-ECAR-4820 Thermal and Structural Safety Compliance Analysis for LANL-MOD-1 Experiment in the B-2 Position in ATR." [6] From the top to the bottom of the basket, the average temperature of YH_x samples is 600°C, 800°C, and 700°C, respectively. The maximum temperature gradient within each ATR fixture is calculated as 21°C. The ATR fixture is filled with a Ar-He gas mixture up to 760 Torr (1 atm). In addition, the LANL cans were fabricated with a varying Ar-He gas composition of 10 Torr hydrogen. The hydrogen was included to help maintain the given YH_x stoichiometry in the samples.

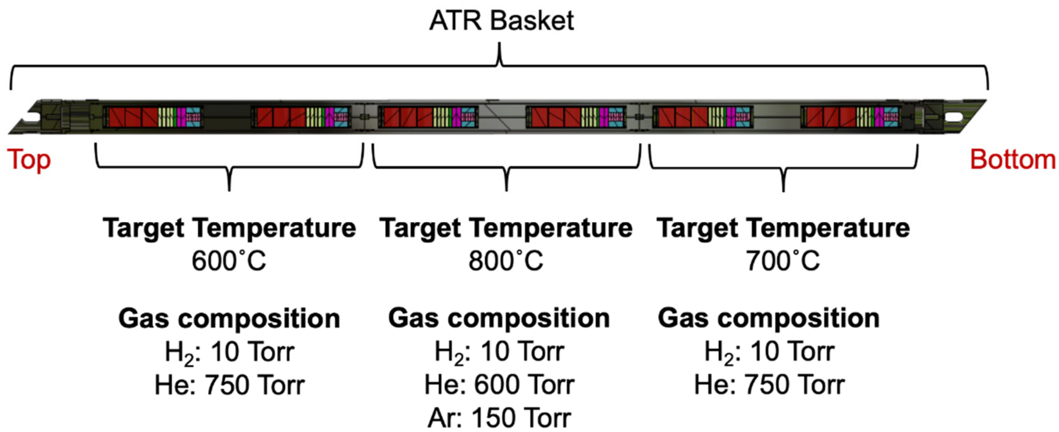

Figure 3. Predicted irradiation temperatures and gas compositions of LANL capsules.

1.3 Initial Condition of LANL Capsules Prior to Irradiation

LANL capsules were assembled at LANL facility. After installing specimens into TZM capsule, capsules were sealed with a lid made of TZM using an orbital arc-welder. Assembled capsules are shown in Figure 4, except capsule 2B. A surface oxidation on the capsule lids was observed after the welding process. Capsules (1,2,3A,4,5, and 6) passed the helium gas leak check. The welding of the capsule 2B successively failed, and the helium gas leak check implied the presence of a crack. The crack at the weld section was also detected via optical microscopy as shown in Figure 5. The crack extended through the wall thickness of the TZM capsule. The samples in this can were fabricated via powder metallurgy technique.

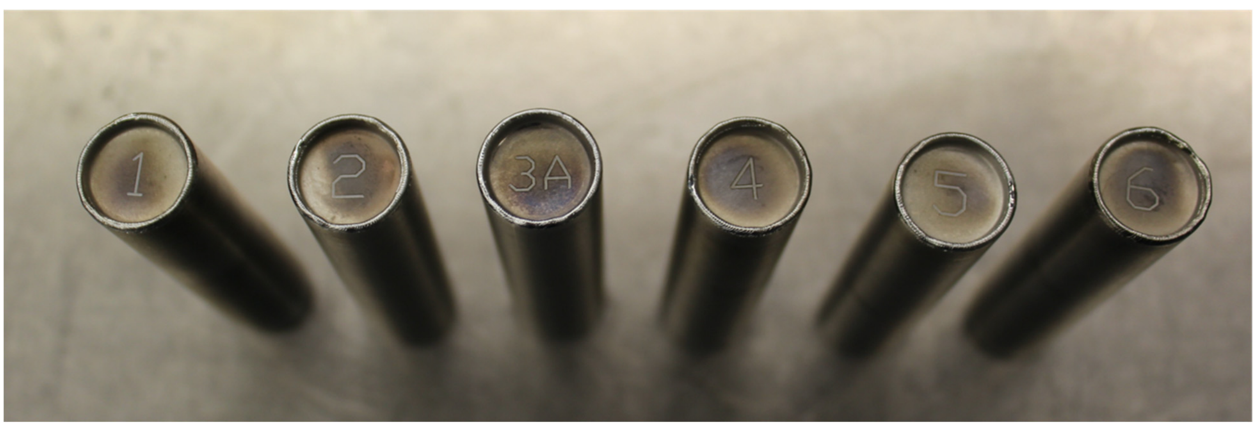

Figure 4. Picture of assembled capsules, except the capsule 2B.

During ATR irradiation, this location is expected to act as an open path for the gas communication between the samples in this can and the ATR fixture's free volume. The final step in the weld assembly of the ATR basket involves a WPS weld, which seals the basket under the Ar He gas mixture. During this process, the ATR basked gas will infiltrate the cracked 2B capsule during the final weld, and the 2B capsule will not contain the desired H_2 gas. A new thermal analysis has been performed for this "as-built" configuration, and the new projected temperatures are shown in

Table 2. The cracked and air-filled capsule scenario is considered as the worst situation. Under that condition, the average temperature of the capsule increases 10-15˚C for all capsules other than 2B. The average temperature of the capsule 2B increases from 699˚C (intact condition) to 769˚C. Since installation will be performed under inert atmosphere, the cracked and evacuated condition of the capsule 2B is considered as the most likely situation during the irradiation period. The arrangement of individual LANL capsules and the defected capsule (2B) is also illustrated in Figure 6.

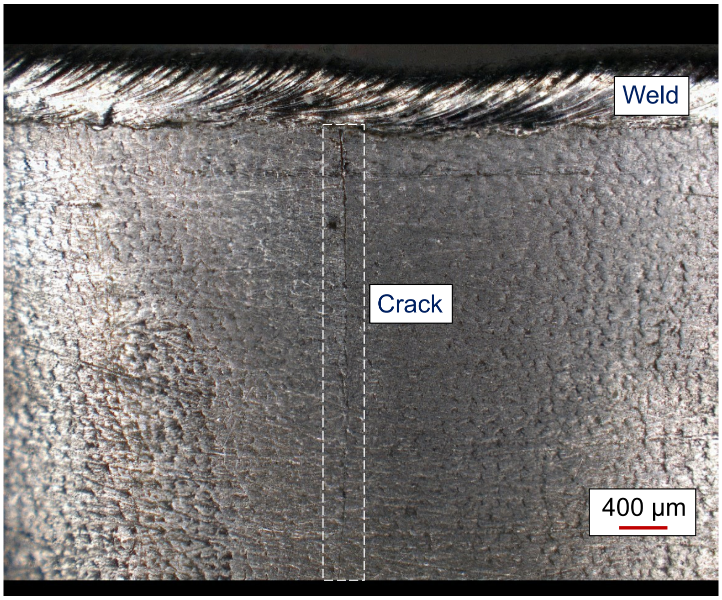

Figure 5. A crack detected after welding of the LANL Capsule 2B during optical inspections, the crack initiated via welding process.

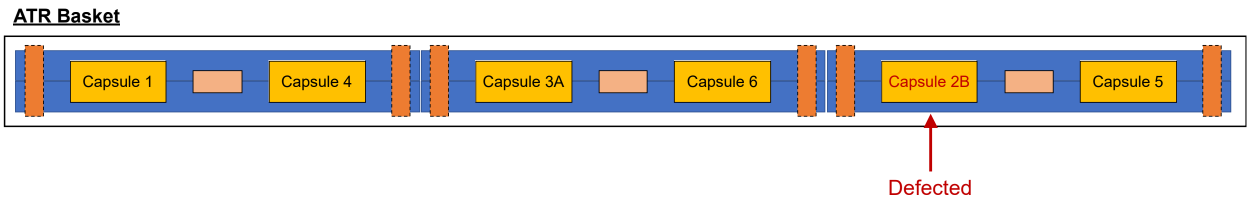

Figure 6. Arrangement of LANL capsules in the ATR basket and location of the defected capsule.

Table 2. Layout of LANL capsules in each ATR fixture. The original temperatures are shown as intact condition, as well as the projected temperatures for the as-built assembly under two potential scenarios. The "Evacuated cracked capsule" scenario is considered the most likely scenario.

Average Capsule Temperature (°C)

	Condition of Capsule 2B		
Capsule	Intact	Cracked and Evacuated (Best scenario)	Cracked and Air filled (Worst scenario)
1	600	601	609
4	601	602	610
3A	801	801	811
6	800	803	813
2B	699	756	769
5	700	706	715

1.4 Objective of the Post-Irradiation Examinations

The objective of this plan is to document data collection and handling requirements for the non-destructive (NDE) and destructive (DE) PIE of YH_x. Data collected from NDE and DE examinations will be utilized for one or more of the activities shown below.

1. Serve as a qualification basis for YH_x as a neutron moderator material, by understanding the impacts irradiation has on the physical and thermophysical properties;

2. Establish a link between manufacturing path and the irradiated YH_x properties;

3. Establish a PIE strategy for other solid-state neutron moderator candidates, and;

4. Inform microreactor material performance and safety codes.

1.5 Scope of Work

The scope of PIE work includes receipt of the materials at the Hot Fuel Examination Facility (HFEF) located at the Materials and Fuels Complex (MFC). Upon receipt, the experiment assembly will be examined visually and via the Neutron Radiography. Sequentially, the ATR basket and ATR fixtures will be disassembled in the HFEF hot cell to allow for collection of the LANL capsules. LANL capsules will be transferred to the Fuels and Applied Science Building (FASB) to collect individual pieces (96 samples, 120 total pieces). Samples identity will be tracked in a unique (serialized) storage container. The YH_x samples have not been individually engraved. In order to minimize sample/material substitution errors, at no time shall two 'like' samples (i.e., samples having the same geometry) be allowed to be out of their sample containers and in the same container. Radiation surveys of a subset (at a minimum) of samples will be performed to help determine in what facility the samples can be safely examined. After housekeeping the samples, NDE and DE will be executed.

2. REQUIREMENTS

Because requirements for the solid moderator qualification are currently not known, fuel qualification PIE requirements will be emulated in an effort to best capture what may be required for moderator qualification. Applicable moderator qualification requirements have been identified in "INL/EXT-13-30238, Chapter 2." [7] Data produced in this plan must be compliant with the American Society of Mechanical Engineers (ASME) Nuclear Quality Assurance (NQA)-1, Part 1, Quality Assurance Program.

This PIE activity may use more than one facility. Therefore, all necessary work control documents will be completed as necessary and approved prior to commencing any testing activities. Experimental work will be conducted under an approved Laboratory Instruction (LI) or Performer Controlled Activity (PCA) depending on the risk involved in performing the activity. Both LIs and PCAs will be utilized for conducting work associated with this test plan.

Equipment operators shall have backgrounds in engineering and/or appropriate knowledge, training, and experience to safely conduct the tests. Equipment operators shall meet the training requirements of the specified facilities.

Documentation of verification and/or calibration results is provided as part of the test record. Calibration/calibration-check acceptance criteria established is ±2% error as compared to literature values for the standard tested as provided by the instrument vendor. Selection of measurement and test equipment and its calibration status will be documented in the respective test plan, laboratory notebook, work support record, INL's initial calibration (i.e., the Standards and Calibration Laboratory's records and equipment-management system) database, or a combination of these records.

Due to radiological contamination concerns, some equipment in radiological hoods and glove boxes may only be calibrated prior to installation. In these instances, redundant measurement equipment should be used whenever possible. Potential sources of uncertainty and error in the measurement shall be identified, documented, and minimized as appropriate.

3. PRECAUTIONS AND LIMITATIONS

All activities, at a minimum, shall be performed in accordance with LWP-21220, “Work Management.” [8] Further instruction on work scope execution is provided by this test plan.

Samples to be examined in this PIE plan must be handled with care due to the brittle nature of the samples. Because hydrogen is bound to the samples, hydrogen release and oxidation can occur. Precautions to avoid any unexpected or unplanned hydrogen release must be taken. This will require careful consideration before polishing samples and any heat treatments.

4. PERSONNEL

This PIE plan may include one or more principal investigators (PI), subject matter expert (SME), and performers/operators. Personnel must read and understand the relevant topic in this plan and the plan’s overarching objectives.

- *Principal Investigator (PI)* must be knowledgeable in the NDE and DE activities. The PI must have technical understanding of the Micro Nuclear Reactor program goals, interpretation and use of the generated data, knowledge of Quality Assurance (QA) requirements. Responsibilities of the PI are (i) development of test plans, (ii) specification of test instructions and requirements, (iii) data analysis and reporting.

- *Subject Matter Expert (SME)* must be the expert on the relevant NDE or DE technique. SME must be familiar with the physical operation and limits of the relevant technique’s equipment. SME must be familiar with the sample requirements and data-acceptance requirements of the relevant technique. Responsibilities of SME are (i) implementation of test plans, (ii) overseeing measurement equipment’s calibration for testing conducted in accordance with the test plan, (iii) verifying performers/operators meet the training requirements of the relevant technique, (iv) supervision of the equipment’s periodic operation and calibration verifications, completion of data review, data analysis, and reporting.

- *Process Engineer/Performer/Operator* must have background in science and engineering. Performer/Operator must be trained on safe and accurate execution of relevant tests and activities under SME’s supervision, in accordance with training requirements indicated in applicable work control document, test standards, and methods. Responsibilities of the performer/operator are (i) configuration control of relevant equipment, (ii) ensuring the operational checks of the relevant equipment, (iii) completion of experimental instructions, records registered laboratory notebooks, and (iv) printed or electronic data management and archival.

5. PRE-EXAMINATION SURVEY OF FRESH YTTRIUM HYDRIDES

Fresh YH_x will be obtained from LANL. These samples will be used for performing baseline experiments to gain experience with the metal hydrides which are brittle in nature and oxidation-resistant.

- Testing is required to identify if hydrogen is emitted from the unirradiated YH_x while stored in a low hydrogen partial pressure atmosphere (i.e., argon atmosphere).

- Mockup LANL capsules will be obtained from LANL to practice at INL's mockup shop to develop a capsule cutting procedure and associated equipment.

- Handling of irradiated YH_x samples is challenging due to the brittle nature of the metal hydrides. Therefore, possible jigs, storage, and transfer methods will be investigated with MFC's remote engineering support group.

6. YTTRIUM HYDRIDE POST-IRRADIATION EXAMINATION ACTIVITIES

This PIE plan involves NDE and DE activities for the assessment of the neutron irradiated YH_x properties to. All NDE, DE, and base-line examination activities must be ASME NQA-1 Part 1 certification compliant starting from the shipping cask acceptance to the final dissemination of PIE data.

6.1 Cask Shipment

The shipping cask will be transferred from ATR to HFEF using BR-3 cask which is handled using existing standard procedures. Once the shipping cask arrives at HFEF, visual examination will be performed to investigate any defects on the shipping cask. Pictures will be taken using a camera from all sides of the cask. Cask will be mated with the HFEF hot cell, and the irradiation basket will be unloaded from the shipping cask.

6.2 Non-Destructive Examinations

NDE activities will be performed at HFEF's facilities. Steps of the NDE are enumerated below.

1. Visual examination of the ATR basket

2. Neutron radiography of the ATR basket

3. Retrieval of ATR fixtures from the ATR basket

4. Retrieval of LANL capsules from ATR fixtures

5. Visual and dimensional inspection of LANL capsules

6. Weight measurements of LANL capsules

7. Disassembly of LANL capsules

8. Individual labeling and bagging of the YH_x and TZM samples

9. Radiation surveys of the samples

10. Optical inspection of samples

11. Volume measurements of samples

12. Weight measurements of samples

13. Disposal of the structural (TZM cans and basket).

6.2.1 Visual examination of the basket

After the ATR basket is unloaded from the shipping cask, the exterior of the ATR basket will be inspected to identify any significant damage via transportation. Visual inspection by digital photography will be performed at HFEF Window 4M (or 8M is Visual Examination Machine [VEM]). Digital photo-documentation will be provided for the basket at a maximum of three viewing angles (e.g., 0°/120°/240°). Photo-documentation of the basket will consist of one macro image spanning the full length of 21.39 in. (543.3 mm). In addition, higher magnification images at points of interest, such as lower and upper end-plug welds, will be documented. The axial position of fuel rod features is determined by a scale bar incorporated into the photo fixture. The inspection should occur with the PI present to allow for additional areas of interest to be identified by the PI. The PI will review the collected images to identify the adequacy of the images and to identify if any additional areas of interest exist. Further inspection may be performed with the Visual Examination Machine at HFEF Window 8M.

6.2.2 Neutron radiography of the basket

Neutron radiography will be performed using the Neutron Radiography (NRAD) Reactor facility which is located in HFEF at Window 4M. Neutron radiography of the basket will be performed and documented in consultation with the PI on Form (FRM)-1411. The transmitted data will include neutron radiographs, the applicable FRM-1411 and loading order photographs. Neutron radiography will be provided for the basket at a minimum of one azimuthal orientation. The axial position is determined from a gadolinium treated scale (marking spacing 0.025 in. and 1 mm) incorporated in the utilized NRAD rodlet fixture. Test shots will be performed before neutron radiography to verify system operation and to ensure that test parameters result in acceptable image quality. The quality of the final images is validated by a certified neutron radiographer and system engineer and documented on the Neutron Radiography Validation Checklist. Neutron radiography data will be digitized at high-resolution. Certification records for the scale bars used during radiography will be included in the data package.

6.2.3 Retrieval of ATR fixtures from the ATR basket

The ATR basket (see INL-DWG-822576) containing three ATR fixtures will be disassembled by a process engineer by following standard sectioning procedures; locations of ATF fixtures will be recorded and tracked. To ensure no visible surface defect, each ATR fixture will be visually inspected and digital photo-documentation will be provided for each ATR fixtures at minimum of three viewing angles (e.g., 0°/120°/240°).

6.2.4 Retrieval of LANL capsules from the ATR fixtures

Each ATR fixture will be cut to open to retrieve LANL capsules by a process engineer by keeping the identifications of the ATR fixtures and LANL capsules. The cutting methodology will be decided after the neutron radiography analysis of the basket. Each LANL capsule will be identified and stored in a bag or a container.

Warning

Capsule 2B has a crack. Take extra care and precaution while retrieving capsule 2B.

6.2.5 Visual and dimensional examination of LANL capsules

Digital photographs of LANL capsules will be collected through visual examination machine (VEM). As needed, higher magnification pictures will also be captured at a minimum of three viewing angles (e.g., 0°/120°/240°) under the PI's supervision. Each capsule will be optically inspected using VEM at window 8M. Photographs will be collected from a minimum of three viewing angles (e.g., 0°/120°/240°), and the whole LANL capsule (2.5 inches) length will be captured.

Each LANL capsule will be subjected to the diameter profile measurement. Axial scan profilometry will be collected on each LANL capsule. Diameter data at intervals will be recorded as agreed between the process engineer and the PI. At least four axial scans at a minimum of four angles (0°, 45°, 90°, 135°) will be recorded for each rodlet. The length of each LANL capsule will be measured via caliper or optical microscopy located at Met-Box hot cell at HFEF.

6.2.6 Weight measurement of LANL capsules

LANL capsule weight will be measured using the balance at HFEF's hot cell. The small balance measuring 0-300g range with accuracy of ±0.5g will be performed. Measurements will be recorded and verified according to the procedure to be developed by the HFEF Process Engineer and PI.

6.2.7 Disassembly of the LANL capsules

A procedure to disassemble LANL capsules will be developed during the pre-examination surveys. If there is available equipment to measure internal pressure and gas composition of LANL capsules, the amount of gas release from each capsule will be measured using precision gamma scanner (PGS) equipment at HFEF. Each YH_x and TZM disc foil sample will be tracked and identified using unique numbering or barcode system. Samples will be secured in containers which ensure the integrity of the brittle samples. If extensive cracking is observed in the samples, the NDE will be re-arranged to immediately proceed to DE.

Warning

After this point YH_x samples must be handled very carefully due to their brittle nature. Any jigs for handling may be designed and used.

NOTE: *All YH_x samples will be subjected to weight measurements, optical inspections, and dimensional measurements. Based on the radioactive activation of the samples, one or more facility will be identified to perform weight measurements, optical inspections, density measurements, and dimensional measurements.*

6.2.8 Radiation survey of samples

Each sample will undergo a gamma-beta radiation survey, and the dose will be recorded by a health physicist using the facility standards.

6.2.9 Optical Inspection and dimensional measurements of samples

Optical inspection will be performed at either HFEF or available facilities (Irradiated Materials Characterization Laboratory [IMCL], FASB, or others). Optical microscopy will be performed to determine any observable surface defects and measurement of diameter of samples. Sample thickness will be measured using height gauges or calibrated micrometers. Calibration records must be included in the measurement documentation.

6.2.10 Volume measurements of samples

Physical volume of YH_x DSC samples (the total number of samples is 36) will be measured using the gas pycnometer available at HFEF's hot cell. Because DSC samples fit in the gas pycnometer chamber, the pycnometer will be used only for DSC samples. Calibration of the instrument is performed daily as needed using a stainless-steel ball with a nominal value of 1 as a vender supplied standard. Work will not proceed until the measurement of the calibration ball is within 3 decimal places of 1. Measurement of a single specimen will be repeated twelve times.

6.2.11 Mass measurement of samples

The weight of the retrieved samples will be measured at HFEF or other available facilities. Measurements will be recorded and verified according to the procedure to be developed by the facility Process Engineer and PI. Sample masses will be measured twelve times using a high-resolution balance with a sensitivity of 0.8 mg. Calibration processes for the scale/balance must be included in the sample weight documentation.

NOTE: *Density of the DSC samples will be calculated via: "**density = mass / volume**" by the Process Engineer or PI. Calculated density values will be reported by showing the mass of the sample, each of the measured volume, the measurement temperature and the standard deviation. Standard deviation will be calculated via error propagation.*

6.2.12 Disposal of ATR basket and fixtures

The shipping basket may be deconned and returned to ATR for re-use. If the contamination levels on the shipping basket are excessive, the basket will be disposed of per Waste Generation Service (WGS) direction (likely low-level waste). The ATR basket will also be disposed of per WGS direction. The TZM capsules disposition is under review as these components may be retained for further PIE.

6.2.13 Fluence wires

Fluence wires will be secured in separate bags and be transported to the Pacific Northwest National Laboratory from MFC for the analysis.

6.2.14 Melt wires

Melt wires will be secured in separate bags and be transported to the relevant facility at MFC for analysis.

7. DESTRUCTIVE EXAMINATIONS

NOTE: *DE can be performed at one or more facilities, depending on the activation levels of the samples.*

To enable the YH_x moderator qualification for micro nuclear reactors, phase identifications, mechanical properties and thermophysical properties of fresh and irradiated YH_x must be determined. Measured properties provide the linkage with the manufacturing process qualification as well.

1. Phase identifications embody x-ray diffraction (XRD), metallography, and hydrogen content.
2. Mechanical properties of interest are micro or nano hardness measurements and elastic properties.
3. Thermophysical properties encompass the physical density, linear thermal expansion, thermal diffusivity, heat capacity, and hydrogen diffusivity.
4. Selected samples will be subjected to GDOES to survey the possible hydrogen redistribution in the YH_x specimens during the irradiation.

7.1 Phase Identifications

7.1.1 X-ray diffraction

X-ray diffraction patterns of YH_x samples irradiated at 600, 700, and 800°C will be collected at ambient temperature. X-ray peaks will be indexed. Samples with 12.5 mm diameter and 2 mm thick will be used. The thickness of the sample will be reduced to 1.5 mm via polishing equipment located at HFEF or IMCL.

7.1.2 Hydrogen content measurements

Hydrogen content of selected samples will be measured using the existing LECO system at Analytical Laboratory (AL). Due to the high levels of H in the samples and potential saturation of the instrument detector, the calibration and measurement processes will be studied to develop a testing process. The calibration samples may be manufactured by INL or LANL. Selected YH_x (GDOES) samples will be crushed into powder. The total mass will be measured. Then these samples will be mixed with a certified material to dilute amount of hydrogen and avoid saturation of the thermal conductivity detector. Mixed samples will be subject to Laboratory Equipment Corporation (LECO) measurements to determine the hydrogen content. After measurements, the hydrogen concentration will be back-calculated to determine initial YH_x sample's hydrogen concentration. The PI will provide the identity of each of the samples prior to the testing. The calibration and process methods must be provided with the measurement data.

7.1.3 Metallography

Prior to the metallography, selected samples will be mounted into epoxy, grind, and mirror-polish based on the appropriate laboratory standards, LI, and under the guidance of American Society for Testing and Materials (ASTM) E3-11(2017) [9]. For metallography YH_x transmission electron microscopy (TEM) samples will be used. The PI will provide the identity of each of the samples prior to the metallographic examinations.

NOTE: *Sample mounts will be compliant with the electron microscopy equipment located at INL facilities.*

Mounted YH_x samples will be characterized using optical microscopy and scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS) at IMCL, the Electron Microscopy Laboratory (EML), or the INL Research Center (IRC). In addition, electron backscatter diffraction (EBSD) pattern will be collected to determine the grain orientations.

Optical microscopy will be taken at magnifications of 25x and 50x. If necessary, the PI can suggest higher magnification tests during the optical microscopy. Full cross section mosaics will be compiled from macrographs. The montage will be performed from partially overlapping images. The amount of overlapping should be at least 15%. Average grain size will be determined via ASTM standards, and any imperfection, defect, second phase will be recorded.

Further examination of the microstructure will be performed using SEM /EDS to survey defects that are invisible with a optical microscope. Secondary Electron (SE) and Backscattered Secondary Electron (BSE) images should be acquired as directed by the PI. At a specific low magnification (decided by the PI), the sample cross-section pictures will be collected, and montaged to show whole sample cross-section. EDS data will be collected from the edge of the sample, mid-radius, and center. Magnifications for EDS data acquisition will be decided by the PI.

7.2 Mechanical Properties

7.2.1 Elastic property measurement

If radiation levels and isotopic composition allow, the elastic properties will be determined at the INL's IF-603 facility using pulse-echo method by measuring the speed of sound in YH_x. Experiment matrix is shown in Table 3. For pulse-echo experiments, YH_x(RUS) samples will first be used. After the test, the same samples will be used in the push-rod dilatometry measurements. Another set of data will be collected after the push-rod dilatometer

Table 3. Experiment matrix for the elastic constant measurements. YH_x samples will be used. T_{irrad} is the irradiation temperature during drop-in experiments.

T_{irrad} (°C)	Hydriding method	# of samples	Sample Height (mm)	# of tests
600	Direct	3	10	5
600	Powder	3	10	5
700	Direct	3	10	5
700	Powder	3	10	5
800	Direct	3	10	5
800	Powder	3	10	5

7.2.2 Nanoindentation

Nanoindentation tests will be performed on YH_x(GDOES) samples by using the nanoindentation machine located at IMCL. These tests will be performed under International Standards Organization (ISO) 14577 Parts, 1, 2, 3, and 4. Prior to meaningful data collection, the indentation surface effects will be determined by changing the indent size in a systematic manner. For each sample, 50 indentations will be applied in an automatic operation. Half of the indents will be applied to the specimen surface using a Berkovich tip, and the other half will be conical or spherical tips. If conical or spherical tips are not present, all data will be collected using Berkovich tip. Table 4

Table 4. Experiment matrix for nano hardness measurements of YH_x (GDOES) samples.

T_{irrad} (°C)	Hydriding method	# of samples	Sample Height (mm)	# of data
600	Direct	1	2	50
600	Powder	1	2	50
700	Direct	1	2	50
700	Powder	1	2	50
800	Direct	1	2	50
800	Powder	1	2	50

7.3 Thermophysical Properties

7.3.1 General instructions

Thermophysical measurements will be performed under NQA-1 compliance. The relevant forms will be filled out by the PI or SME, and the PI will brief the operator prior to the tests. The operator shall follow the standard safety rules while performing tests. All certification documents for standards used shall be included in the data package.

Warning

Because YH_x is prone to oxidation and hydrogen release, the thermal property equipment like crucibles must be made of inert material such as yttria (yttrium oxide) or, at a minimum, they should be coated with yttria.

7.3.2 Physical density

Density of all samples will be calculated by measured mass divided by the calculated sample volume via taking dimensional measurements. Volume of the YH_x (DSC) samples will be compared to the pycnometer measured values of the same sample. Difference in the volume measurements will be recorded, and it will provide the degree of the uncertainty. Average density will be calculated by dividing the average mass by the average volume measured.

7.3.3 Linear thermal expansion

Linear thermal expansion will be determined using the push-rod dilatometry technique according to the ASTM E228-17 “standard test method for linear expansion of solid materials with a push-rod dilatometer.” [10] Push-rod dilatometry will be performed whether at AL or the thermophysical properties laboratory at Idaho Falls (IF)-603. For this activity, YH_x(RUS) samples will be used. Samples after pulse-echo measurements will be transferred to the relevant facility to perform push-rod dilatometry measurements. Samples will be subjected to single heating-cooling treatments, cycling heating-cooling treatments to determine the reversibility of hydrogen dissolution or escape from the hydride moderator. Prior to these tests, the effect of heating and cooling rate on as-fabricated YH_x will be investigated. Before and after certified dilatometry, a sample will be subjected to the experiments to verify the push-rod dilatometry equipment. In addition to that, a certified sample will be subjected to heating and cooling rate experiments to detect any possible variances on the data set. The experimental matrix is shown as in Table 5. Specimen mass will be measured before and after the push-rod dilatometry. If needed, extra tests will be performed to elucidate certain irradiation-induced phenomena that can occur during the tests.

Table 5. Experiment matrix for dilatometry. YH_x(RUS) will be used; see pulse-echo measurements.

T_{irrad} (°C)	Hydriding method	# of samples	Heating/Cooling rate (°C/min)	Maximum Temperature (°C)	# of cycles
600	Direct	1	2.5/2.5	600	1
600	Direct	1	2.5/2.5	800	1
600	Direct	1	2.5/2.5	800	2
600	Powder	1	2.5/2.5	600	1
600	Powder	1	2.5/2.5	600	2
600	Powder	1	2.5/2.5	600	4
700	Direct	1	2.5/2.5	700	1
700	Direct	1	2.5/2.5	800	1
700	Direct	1	2.5/2.5	800	2

T_{irrad} (°C)	Hydriding method	# of samples	Heating/Cooling rate (°C/min)	Maximum Temperature (°C)	# of cycles
700	Powder	1	2.5/2.5	800	1
700	Powder	1	2.5/2.5	1200	1
700	Powder	1	2.5/2.5	1200	2
800	Direct	1	2.5/2.5	800	1
800	Direct	1	2.5/2.5	800	1
800	Direct	1	2.5/2.5	800	2
800	Powder	1	2.5/2.5	800	1
800	Powder	1	2.5/2.5	1200	1
800	Powder	1	2.5/2.5	1200	2

7.3.4 Heat capacity

Heat capacity will be measured using differential scanning calorimetry experiments. Measurements will be performed according to ASTM E1269-11 "standard test method for determining specific heat capacity by differential scanning calorimetry." [11] Mass of samples will be measured before and after each test (Table 6).

Table 6. Experimental matrix of YH_x(DSC) samples. *700°C tests will be performed first to determine the effect of heating and cooling rate on the DSC signal.

T_{irrad} (°C)	Hydriding method	# of samples	Heating/Cooling rate (°C/min)	Maximum Temperature (°C)	Sample Height (mm)	# of cycles
600	Direct	1	10/10	600	2	1
600	Direct	1	10/10	600	2	2
600	Direct	1	10/10	800	2	1
600	Direct	1	10/10	1200	2	1
600	Powder	1	10/10	600	2	1
600	Powder	2	10/10	1200	2	1
600	Powder	2	10/10	1200	2	2
700*	Direct	1	5/5	700	2	1
700	Direct	1	10/10	700	2	1
700	Direct	1	20/20	1200	2	1
700	Powder	1	10/10	700	2	1
700	Powder	2	10/10	1200	2	1
700	Powder	2	10/10	1200	2	2

T_{irrad} (°C)	Hydriding method	# of samples	Heating/Cooling rate (°C/min)	Maximum Temperature (°C)	Sample Height (mm)	# of cycles
800	Direct	1	10/10	800	2	1
800	Direct	1	10/10	800	2	1
800	Direct	1	10/10	1200	2	1
800	Powder	1	10/10	800	2	1
800	Powder	1	10/10	1200	2	1
800	Powder	1	10/10	1200	2	2

7.3.5 Thermal diffusivity

Thermal diffusivity will be measured via LFA located at IMCL's thermal properties laboratory or the IRC laboratory. LFA measurements will be performed on samples as irradiated. In addition, selected samples that were subjected to DSC experiments will be subjected to LFA tests to inspect the effect of damage annealing effects on the diffusivity. The experimental matrix is shown below in Table 7. If needed, additional tests will be scheduled and performed. LFA tests will be conducted according to ASTM E1461-13 "standard test method for thermal diffusivity by laser flash method." [12]

Table 7. Thermal diffusivity experiments.

Irradiation Temperature (°C)	Hydriding method	# of samples	Heating/Cooling rate (°C/min)	Maximum Temperature (°C)	Sample Height (mm)	# of runs
600	Direct	2	10/10	1200	2	5
600	Powder	2	10/10	1200	2	5
700	Direct	2	10/10	1200	2	5
700	Powder	2	10/10	1200	2	5
800	Direct	2	10/10	1200	2	5
800	Powder	2	10/10	1200	2	5

8. SURVEY PIE ON HYDROGEN MIGRATION

Hydrogen migration under temperature-gradient in metal hydrides is an observed phenomena in metal hydrides, and it is expected to happen in YH_x when subjected to temperature-gradient. The systematic characterization of this phenomena is lacking due to the complexity of the experiments. Based on the Nuclear Regulatory Commission's safety evaluation for uranium zirconium hydride fuels, hydrogen migration must be assessed via proper techniques. Currently, INL does not have such a capability, but preliminary surveys of the hydrogen concentration gradient can be performed by GDOES technique. Thus, selected samples will undergo GDOES experiments to examine the possible hydrogen migration at the Safety and Tritium Applied Research (STAR) facility. For these experiments, samples from the temperature zones of 600 and 800°C will be picked depending on the status of the sample. Prior to GDOES experiments, samples undergo Positron Annihilation Spectroscopy (PAS) at room temperature.

9. SURVEY PIE ON TZM SHEETS

TZM sheets between YH_x specimens will be used for advanced PIE activities such as nanoindentation, TEM, and micromechanical tests. Additional samples may also be sectioned from available LANL capsules. Selected specimens will be subjected to PAS and GDOES examinations. For these activities, STAR and IMCL will be utilized.

10. BASE-LINE EXAMINATIONS OF FRESH YTTRIUM HYDRIDES

Base-line examinations of YH_x comprise all the destructive examinations to be performed on fresh YH_x samples. These experiments are expected to be performed at the facilities where DE will be performed. Including metallography, all thermophysical measurements will be performed on fresh samples to gain experience and determine the data reproducibility. All these experiments will be under the NQA-1 Part 1 compliance.

11. REFERENCES

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4. *Technical and Functional Requirements. LANL-MOD-1 Irradiation in the ATR NEFT Small B Positions. TFR-1049. Revision 1*. 2020, Idaho National Laboratory: Idaho Falls. p. 41.
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7. Miller, C., B. Durtschi, and K. Daum, *Base Monolithic Fuel: Research, Development, and Qualification Plan*. 2014: United States.
8. *Laboratory-wide Procedure Work Management LWP-21220 Revision 21220*. 2019, Idaho National Laboratory: Idaho Falls.
9. *ASTM International Standard Guide for Preparation of Metallographic Specimens ASTM E3-11 (Reapproved 2017)*. 2017: West Conshohocken, PA.
10. *ASTM International Standard Test Method for Linear Thermal Expansion of Solid Materials With a Push-Rod Dilatometer E228-17*. 2017: West Conshohocken, PA.
11. *ASTM International E1269-11(2018) Standard Test Method for Determining Specific Heat Capacity by Differential Scanning Calorimetry*. 2018, ASTM International: West Conshohocken, PA.
12. *ASTM International. E1461-13 Standard Test Method for Thermal Diffusivity by the Flash Method*. 2013:

Appendix A

Prior to the ATR irradiation campaign, mass of each capsule is recorded in the shown record sheet.

Date:

LANL-MOD-1 Capsule Mass Measurement Record Sheet

Sheet 1 of 2

Measurement Operator Name: Signature:

Measurement instrument ID:

Notice: Ensure the balance is calibrated or verified prior to measurement. Perform weight measurements in accordance of NQA-1 Part I QA.

Append the certifications of the reference weights to this record sheet

Reference weight measurements before sample measurement:

Measurement (g)	The largest reference mass	The smallest reference mass
1		
2		
3		

Notes: If necessary, take more than five (5) measurements of each or individual capsule

Measurement (g)	Capsule 1	Capsule 2B	Capsule 3A	Capsule 4	Capsule5	Capsule 6
1						
2						
3						
4						
5						

Date:

LANL-MOD-1 Capsule Mass Measurement Record Sheet

Sheet 1 of 2

Measurement Operator Name: Signature:

Measurement instrument ID:

Notice: Ensure the balance is calibrated or verified prior to measurement. Perform weight measurements in accordance of NQA-1 Part I QA.

Append the certifications of the reference weights to this record sheet

Reference weight measurements before sample measurement:

Measurement (g)	The largest reference mass	The smallest reference mass
1		
2		
3		

Notes: If necessary, take more than five (5) measurements of each or individual capsule

Measurement (g)	Capsule 1	Capsule 2B	Capsule 3A	Capsule 4	Capsule5	Capsule 6
1						
2						
3						
4						
5						