U.S. DEPARTMENT OF ENERGY

PNNL-77150

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

Fluence Capsules for the SAM-2 Experiment MPO# 00239221/SOW-17494, Rev. 0

Analyst: Lori P. Darnell Date: 9-22-20

Technical Reviewer: LR Greenwood Date: 9-22-20

Quality Engineer: Samuel A. Adams Date: 09/23/2020

September 2020

Acceptance by Receiving Organization:

Accepted by: _____ Date: _____

Pacific Northwest
NATIONAL LABORATORY

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Tel: (509) 375-5301
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MSIN: P7-22
Larry.greenwood@pnnl.gov

September 22, 2020

BEA, Idaho National Laboratory
1765 North Yellowstone Highway
Attention: MPO No. 00230949 (Douglas Stacey)
Idaho Falls, ID 83415

Subject: Fabrication of Neutron Fluence Monitors on MPO #00239221 /SOW-17494- Rev. 0 – PNNL Project #: 77150

Dear Mr. Anderson,

Fabrication of the neutron fluence monitors has been completed and the capsules are included with this letter along with the documentation in accordance with your subject contract. There were no significant problems during the manufacture of the monitors and all monitors meet the specifications in your contract. The documentation of the capsules is described below.

1. Summary sheet listing all capsules with ID codes and wire weights
2. Certificate of conformance
3. Certificates for each type of material used to fabricate the capsules.
4. Calibration of the balance and digital calipers used for the capsule measurements.
5. Results of the helium leak testing and certificate for the helium leak standard.
7. Quality Assurance Project Plan–77150, Rev. 0, Quality Assurance Project Plan for Fluence Capsules for the SAM-2 Experiment
8. Test Instruction, TI-PREP-77150, Rev. 0, Test Instruction for Preparation of Fluence Wire Capsules – Project 77150
9. Test Instruction, TI-FAB-75643, Rev. 0, Tests and Inspections for Reactor Dosimetry Capsules - Project 77150

The capsule ID codes are stamped on the bottom of each monitor and can be seen using a low power microscope. If you have any questions regarding the monitors or documentation, please let me know. We will look forward to receiving notice of your acceptance of the capsules. We would like to discuss any needs that you may have for capsule fabrication or analysis in the future.

Sincerely,

L R Greenwood

Larry Greenwood, Ph. D.

Enc – Monitors and documents as described above

Summary of Neutron Fluence Monitors with ID Codes and Wire Weights

The fluence capsules are nominally 0.39" long with an O.D. of 0.05"

Capsule ID (Stamped on bottom)	Fe Weight (mg)	Ti Weight (mg)	Nb Weight (mg)	0.116% Co-Al Weight (mg)	Final Capsule Weight After Sealing (mg)
1F	2.516	1.195	2.165	0.695	49.619
97	2.613	1.264	1.863	0.830	48.352
9J	2.610	1.191	2.281	0.863	48.197
A5	2.247	1.349	2.053	0.709	50.630
5U	2.599	1.246	2.252	0.736	51.054
1I	2.144	1.187	1.928	0.708	50.863
77	1.918	1.168	2.514	0.675	51.485
7F	1.866	1.085	2.388	0.637	48.429

Note: The final capsule weights are listed after sealing. These weights include the fluence wires and vanadium capsule, a vanadium wire added for electron beam welding and a small loss of weight during welding. The capsules met all size requirements in the SOW, as documented in the attached tests and inspections.

Items: Neutron Fluence Capsules as specified on MPO #00239221 /SOW-17494- Rev. 0

Certificate of Conformance

We certify that all materials used for the preparation of the neutron fluence monitors conform to the requirements in the referenced purchase specifications.

The quality requirements for the subject work are documented in the Memorandum Purchase Order (MPO) 00239221 and SOW 17494, Rev. 0 as prepared by the INL client.

We further certify that all work on the neutron fluence capsules was performed according to the approved technical procedures and test instructions in accordance with the referenced purchase specifications in MPO00239221. The fabrication of the monitors is fully documented in this report.

Lori P. Darnell 9-22-20

L. P. Darnell Analyst

L R Greenwood 9-22-20

L. R. Greenwood, PhD Project Manager

Samuel A. Adams Digitally signed by Samuel A. Adams Date: 2020.09.23 15:29:02 -07'00'

S. A. Adams Quality Engineer

Pacific Northwest National Laboratory
Radiochemical Processing Laboratory
902 Battelle Boulevard
Richland, WA 99352

MATERIALS RESEARCH CORPORATION

Orangeburg, New York 10962 • 914 359-4200 • "Cable MATRESCO"

MATERIALS CERTIFICATION
PLEASE FORWARD WITH MATERIAL

Customer Order No. Y2Y-444-07448 Our Order No. 27799

1. Material 99.999% Iron Wire

2. Form and Condition 0.020" dia.

3. Processing Description 99.999% - Material cold swaged and drawn to size, annealed and spooled after cleaning.

4. Analyses —

Mg - 3
Cu - 2
Mn - 2

All others less than <1 by emission spec.

5. Inspection

The results shown on this report are correct to the best of my knowledge and belief.

MATERIALS RESEARCH CORPORATION

2/15/72
Date

By J. Galuszka

REACTOR EXPERIMENTS, INC.

963 TERMINAL WAY SAN CARLOS, CALIFORNIA 94070

MATERIAL DATA SHEET

Material: Titanium Thickness: --- Purity: 99.917%

Catalog No.: 614 Diameter: .020" Lot No: 139W

CHEMICAL ANALYSIS

Impurity	Maximum Percent	Impurity	Maximum Percent	Impurity	Maximum Percent	Impurity	Maximum Percent
Al	.0001						
Cu	.001						
Cr	.003						
Fe	.008						
Mn	.02						
Mg	.001						
Si	.005						
V	.005						
O	.03						
N	.002						
H	.008						

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE
Institute for Reference Materials and Measurements

CERTIFIED REFERENCE MATERIAL IRMM–526

CERTIFICATE OF ANALYSIS

NIOBIUM		
	Mass fraction	
	Certified value [1] [mg/kg]	Uncertainty [2] [mg/kg]
Ta	0.30	0.09

1) The certified value is the unweighted mean of the means of 6 accepted sets of data, each set being obtained in a different laboratory and/or with a different method of determination. The value is traceable to the International System of Units (SI).
2) The uncertainty is a 95 %/95 % tolerance interval.

This certificate is valid for five years after purchase.

Sales date:

The minimum amount of sample to be used is 100 mg.

DESCRIPTION OF THE SAMPLE

IRMM-526 consists of niobium metal with a nominal purity of > 99.994 %. Tantalum, present as impurity and causing spectral interference during application, is certified. The material is available as:

- IRMM-526A: 0.02 mm thick foil (20 cm^2 units)
- IRMM-526B: 0.1 mm thick foil (20 cm^2 units).
- IRMM-526C: 0.5 mm diameter wire (1 m units)

Brussels, May 1997
Latest revision: May 2007

Signed:

Prof. Dr. Hendrik Emons
Unit for Reference Materials
EC-JRC-IRMM
Retieseweg 111
2440 Geel, Belgium

U. S. Department of Commerce
Maurice H. Stans
Secretary

National Bureau of Standards
A. V. Astin, Director

Certificate of Analysis

Standard Reference Material 953
Neutron Density Monitor Wire[a]

(Cobalt in Aluminum)

The standard is provided as a reference source of a cobalt in aluminum alloy to serve as a neutron density monitor wire standard. Accurate determination of thermal neutron densities is essential for irradiation tests in order to obtain a basis for comparison of densities among reactors, to apply the data in the design of reactors, to understand the mechanisms of radiation damage and to use in neutron activation analysis.

SRM No.	Description	Cobalt, percent by weight
953	Neutron Density Wire (Co in Al)	0.116[b] ± 0.002[c]

[a] The neutron density monitor material is in the form of wire 0.5 mm in diameter and is available in one meter lengths, (approx. 0.6 g), or in multiples thereof (continuous length).

[b] The reported value is the average of the results of three analytical methods.

[c] The uncertainty limit reported is the sum of the following (in weight percent): 1. 0.0009, the estimated limit of the systematic error in the average; 2. 0.0010, the estimated limit of the inhomogeneity at the 95% confidence level, for samples $\geq$4 mg; and, 3. 0.0004, the estimated limit of the random error in the average at the 95% confidence level.

Fourteen 10-meter lengths of wire equally spaced over the entire lot of selected material were analyzed. The methods used, the weight (w) of samples taken from each of the fourteen lengths, the standard deviation (S_x) of a single measurement due to imprecision of the analytical method, the number of determinations (N) made by each method, and the average obtained ($\overline{X}$) are given below.

Method	w (mg)	S_x	N	$\overline{X}$
1	50–60	0.0013	14	0.1161
2	4–5	0.0007, 0.0005[a]	5 sets of 6	.1158
3	20	0.0015	41	.1173

[a] The standard deviation was 0.0007 within a set and 0.0005 between sets.

The analytical methods used were:

Methods 1 and 2: Two variations of a spectrophotometric method using 2,3-quinoxalinedithiol (E. R. Deardorff and R. W. Burke).

Method 3: Activation analysis, nondestructive, relative to cobalt metal foil and cobalt metal powder (T. E. Gills and D. A. Becker).

The use of gamma-ray spectroscopy to measure the induced radioactivity of this material is recommended. Gross counting will result in systematic errors due to the presence of ^{24}Na (from the n,α reaction on ^{27}Al) and ^{198}Au (from the n,γ reaction on a small amount of gold impurity present, estimated at ~5 ppm).

Washington, D. C. 20234
March 12, 1969

W. Wayne Meinke, Chief
Office of Standard Reference Materials

(over)

This standard has been established to provide a homogeneous and well-characterized neutron density monitor primarily for use in irradiation test programs on reactor materials. It will also be useful for the nuclear activation analyst in monitoring neutron densities and mapping neutron density variations in irradiation containers and in various irradiation locations within reactors. The 0.1 percent cobalt in aluminum monitor material was chosen as having (1) a well-known neutron cross section in all neutron energy ranges, (2) a suitable half life, and (3) a well-characterized energy spectrum and decay scheme.

The material for SRM 953 was prepared at the Materials Research Corporation, Orangeburg, New York, with special melting, casting and fabrication procedures designed to produce a large quantity of material of the highest possible homogeneity.

Extensive homogeneity testing was performed at the NBS laboratories in Gaithersburg, Maryland, and the material was found to be satisfactory for its intended use as a neutron density monitor.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of J. R. DeVoe.

The technical and support aspects involved in the preparation, certification and issuance of this Standard Reference Material was coordinated through the Office of Standard Reference Materials by R. E. Michaelis.

ANALYTICAL METHOD USED FOR CERTIFICATION

- Electrochemical separation (ECS)
- Spark source mass spectrometry (SSMS)
- Inductively coupled plasma mass spectrometry (ICP-MS)
- Neutron activation analysis (NAA)

PARTICIPANTS

- AEA Technology, Harwell (GB)
- Centre National de la Recherche Scientifique, Centre d'Etudes et de Recherches par Irradiation, Orléans, et Laboratoire d'Analyse par Activation, Pierre Sue (CEA-CNRS), Saclay (FR)
- European Commission, Joint Research Centre, Central Bureau for Nuclear Measurements (CBNM), Geel (BE)
- Forskningscenter Risø, Roskilde (DK)
- Max-Planck-Institut für Metallforschung – Institut für Werkstoffwissenschaften, Dortmund (DE)
- Studiecentrum voor Kernenergie / Centre d'Etude de l'Energie Nucléaire (SCK/CEN), Mol (BE)
- Universiteit Gent – Instituut voor Nucleaire Wetenschappen (INW), Gent (BE)

SAFETY INFORMATION

The usual laboratory safety precautions apply.

INSTRUCTIONS FOR USE

This Nuclear Reference Material (NRM) is intended to be used in neutron metrology to monitor fast neutron fluence rates and fluences. The reaction of interest is ^{93}Nb(n,n')^{93m}Nb.
Before use it is recommended that the samples are cleaned (ultrasonically for 5 minutes) in an adequate bath, e.g. trichlorotrifluoroethane, rinsed with distilled water and dried in a dust-free atmosphere.

STORAGE

The material can be stored at room temperature.
However, the European Commission cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially of opened samples.

LEGAL NOTICE

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(b) assume any liability with respect to, or for damages resulting from, the use of any information, material, apparatus, method or process disclosed in this document save for loss or damage arising solely and directly from the negligence of IRMM or any of its subsidiaries.

NOTE

A technical report on the production of IRMM-526 is available on the internet (http://www.irmm.jrc.be). A paper copy can be obtained from IRMM on request.

European Commission – Joint Research Centre
Institute for Reference Materials and Measurements (IRMM)
Retieseweg 111, 2440 Geel (Belgium)
Telephone: +32-(0)14-571.722 - Telefax: +32-(0)14-590.406

INDUSTRIAL TESTING LABORATORY SERVICES, LLC
635 Alpha Drive – RIDC Park
Pittsburgh, PA 15238 Phone: 412.963.1900 Fax: 412.963.1926
e-mail: itlslabs@cs.com website: itls-labs.com

TEST REPORT
L2603

June 13, 2002

Purchase Order No: 4500090790

To: Westinghouse Electric Co. LLC
Drawer R
Columbia, SC 29520

Attn: Warren Mauterer

Material received: One (1) sample of Vanadium Wire

Specifications: 10CFR Part 21, 10CFR50 Appendix B apply

RESULTS

Specimen	V (wt. %)
Wire	99.89%

Industrial Testing Laboratory Services, LLC

Anthony B. Freda
Manager, Mechanical Testing & Metallurgical Services

THE ANALYTICAL CHEMISTRY DIVISION
SPECTROCHEMICAL LABORATORY
BUILDING 9734, Y-12 TELEPHONE 3-7168

Request No 29836

REQUEST AND REPORT OF SPECTROGRAPHIC ANALYSIS

CHARGE NO.	SAMPLE
3675-2	Met. 640 Capsule #11

SUBMITTED BY	BUILDING NO.	PHONE	DATE
H. L. Adair	3037	3-6005	8 Sept. 1976

COPY REPORT TO	BUILDING NO.

COMPOSITION OF SAMPLE IF KNOWN: Vanadium Capsule.

TYPE OF ANALYSIS DESIRED: ☐ Qualitative, ☐ Semi-quantitative, ☐ Quantitative, ☐ Photoelectric ☒ SSMS.

ELEMENTS DESIRED: Survey Esp. Ta.

GENERAL ANALYSIS (values in wt ppm)

Ag	<1	In		Sc		Si	200.		
Al	15	Ir		Si	200.	Sn			
As		K	3	Sn		Sr			
Au		Li		Sr		Ta	10		
B	2,	*Mg	≤100	Ta	10	Te			
Ba	<3	Mn	2	Te		Th			
Be		Mo	<2	Th		Ti	3		
Bi		Na	<1	Ti	3	Tl			
Ca	1	Nb	40	Tl		U			
Cd	<3	Ni	1	U		V	Major.		
Co	≤0.2	Os		V	Major.	W	20.		
Cr	≤2	P	10	W	20.	Zn	0.5		
Cs		Pb	<3	Zn	0.5	Zr	1		
Cu	1	Pd		Zr	1	Ca	5		
Fe	20	Pt		Ca	5	S	10		
Ga		Rb		S	10				
Ge		Re							
Hf	<3	Rh							
Hg		Ru							
		Sb							

RARE EARTH ANALYSIS (values in _____)

Sc			
Y			
La			
Ce			
Pr			
Nd			
Sm			
Eu			
Gd			
Tb			
Dy			
Ho			
Er			
Tm			
Yb			
Lu			
Th			

METAL ANALYSIS (values in _____)

Type alloy _____

Cr			
Ni			
Fe			
Mo			
Co			
Nb			
Ta			
Mn			
V			
Ti			
Cu			
Al			
Mg			
Sn			
Pb			
Zn			
Bi			
Si			

Explanation of Analysis: * Mg is interfered with by 5% V.

Symbols Used: P-Present; T-Trace; < - less than; > - greater than; nd-not detected; no analyses made in all other cases.

☒ SSMS

☐ Qualitative Analysis - Estimate only as follows: M-major; m-minor; t-trace.

☒ Semi-Quantitative Analysis - The values reported are visual estimates taken from a standard plate and using a common graphite matrix. These values are to be interpreted as approximations only. Actual value should be within the range times ½ to times 2.

☐ Quantitative Analysis - The values reported are obtained by visual comparison of the sample with standards similarly prepared. Precision is about ± 50% of the amount present.

☐ Densitometric Analysis - The values reported are obtained by precise analytical spectrochemical methods. Precision of the method varies but is of the order of ± 10% or better.

☐ Photoelectric Analysis - Rapid electronic method. Precision _____ %. Log # 1743

ANALYSIS PERFORMED BY	APPROVED BY	PLATE NO.	DATE REPORTED
DLD, JCF	J. C. Franklin	5940	9 Sept 1976

COMMENTS

CN-1003

Established 1974

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A2LA ACCREDITED
CERTIFICATE #1550.01

Battelle Pacific N.W. Natl. Lab
902 Battelle Blvd.
Richland, WA 99354

Report Number: BATN0377843190820

A2LA ACCREDITED
CERTIFICATE OF CALIBRATION WITH DATA

INSTRUMENT INFORMATION

Item	Make	Model	Serial Number	Customer ID	Location
Microbalance	Cahn	C33	77843	WD55353	RPL 514
Units	**Readability**	**SOP**	**Cal Date**	**Last Cal Date**	**Cal Due Date**
mg	0.001	QC012	8/20/19	2/14/19	8/2020

FUNCTIONAL CHECKS

ECCENTRICITY
Test Wt: 50 Tol: 0.003
As-Found: Pass: ☑ Fail: ☐
As-Left: Pass: ☑ Fail: ☐

LINEARITY
Test Wt: 100x2 Tol: 0.005
As-Found: Pass: ☑ Fail: ☐
As-Left: Pass: ☑ Fail: ☐

STANDARD DEVIATION
Test Wt: 200 Tol: 0.001

1. 200.001	5. 200.000	9. 200.003
2. 200.001	6. 200.002	10. 200.002
3. 200.000	7. 200.003	**Result**
4. 200.001	8. 200.004	0.0013

ENVIRONMENTAL CONDITIONS
☐ Good ☑ Fair ☐ Poor
Temperature: 25.0°C

A2LA ACCREDITED SECTION OF REPORT

Standard	As-Found	As-Left	Expanded Uncertainty
200	200.001	200.001	0.0067
150	150.002	150.002	0.0067
100	100.002	100.002	0.0034
50	50.000	50.000	0.0031
20	20.001	20.001	0.0030
10	10.000	10.000	0.0030

CALIBRATION STANDARDS

Item	Make	Model	Serial Number	Cal Date	Cal Due Date	NIST ID
Weight Set	Rice Lake	30 kg-1mg	S751	2/15/19	2/2020	20182428

Permanent Information Concerning this Equipment:
Applied tolerance = 0.010 @ span of 200g.

Comments/Info Concerning this Calibration:
8/19 PO #479560 As found / as left within tolerance. RH=18.4%

Report prepared/reviewed by: R.B. Service Manager **Date:** 9-5-19

Technician: R. Hintz **Signature:**

The uncertainty is calculated according to the ISO Guide to the Expression of Uncertainty in Measurement and includes the uncertainty of standards used combined with the observed standard deviation and readability of the unit under test. The uncertainty is expanded with a k factor of 2 for an approximate 95% level of confidence. Instruments listed above were calibrated using standards traceable to the National Institute of Standards and Technology (NIST). Calibration data reflect results at the time and location of calibration. Calibration data should be reviewed to insure that the instrument is performing to its required accuracy. Calibrations comply with ISO/IEC 17025 and ANSI/Z540-1-1994 quality standards.

Established 1974

QUALITY CONTROL SERVICES

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(503) 236-2712 • FAX (503) 235-2535 • www.qc-services.com

ACCREDITED
Calibration Services
Certificate Number: 1550.01
Laboratory code: 115953

Battelle Pacific N.W. Natl. Lab
902 Battelle Blvd.
Richland, WA 99354

Report Number: BATN0377843200818

A2LA ACCREDITED
CERTIFICATE OF CALIBRATION WITH DATA

INSTRUMENT INFORMATION

Item	Make	Model	Serial Number	Customer ID	Location
Microbalance	Cahn	C33	77843	WD55353	RPL 514

Units	Readability	SOP	Cal Date	Last Cal Date	Cal Due Date
mg	0.001	QC012	8/18/20	8/20/19	8/2021

FUNCTIONAL CHECKS

ECCENTRICITY
Test Wt: 50 Tol: 0.003
As-Found: Pass: ☑ Fail: ☐
As-Left: Pass: ☑ Fail: ☐

LINEARITY
Test Wt: 100x2 Tol: 0.005
As-Found: Pass: ☑ Fail: ☐
As-Left: Pass: ☑ Fail: ☐

STANDARD DEVIATION
Test Wt: 200 Tol: 0.001

1. 200.000	5. 200.002	9. 200.001
2. 199.998	6. 199.999	10. 200.000
3. 200.001	7. 200.000	Result
4. 199.996	8. 200.002	0.0018

ENVIRONMENTAL CONDITIONS
☐ Good ☑ Fair ☐ Poor
Temperature: 22.1°C

A2LA ACCREDITED SECTION OF REPORT

Standard	As-Found	As-Left	Expanded Uncertainty
200	199.984	200.000	0.0072
150	149.988	149.999	0.0072
100	99.989	99.998	0.0042
50	49.995	50.002	0.0040
20	19.996	20.000	0.0039
10	10.001	10.001	0.0039

☒ Accepted ☐ Rejected / Per Clause(s): 175b
No indication of Out-Of-Tolerance condition(s)
AQSS & OOT Reviewer

CALIBRATION STANDARDS

Item	Make	Model	Serial Number	Cal Date	Cal Due Date	NIST ID
Weight Set	Rice Lake	30 kg-1mg	S751	4/21/20	4/2021	20200533

Permanent Information Concerning this Equipment:
Applied tolerance = 0.020 @ span of 200g.

Comments/Info Concerning this Calibration:
8/20 PO #530367 As found / as left within tolerance. RH=56.3%

Report prepared/reviewed by: D. Thompson **Date:** 8/31/20

Technician: R. Hintz **Signature:**

The uncertainty is calculated according to the ISO Guide to the Expression of Uncertainty in Measurement and includes the uncertainty of standards used combined with the observed standard deviation and readability of the unit under test. The uncertainty is expanded with a k factor of 2 for an approximate 95% level of confidence. Instruments listed above were calibrated using standards traceable to the National Institute of Standards and Technology (NIST). Calibration data reflect results at the time and location of calibration. Calibration data should be reviewed to insure that the instrument is performing to its required accuracy. Calibrations comply with ISO/IEC 17025 and ANSI/Z540-1-1994 quality standards.

Kootenai Bldg. 34
MD 1025, PO Box 968
Richland, WA 99352-0968
Phone (509) 377- 8444
FAX (509) 377-8219

Calibration Certificate

PNL-13357600:1579876223

ENERGY NORTHWEST
STANDARDS LABORATORY

ASSET INFORMATION

Asset Number: PNL-13357600
Manufacturer: MITUTOYO
Model: CD-6 IN CSX
Description: CALIPER
Serial Number: 13357600
Release Number: NEW
Ref/PO Number: PCD-504635

Customer
PNNL
790 6th Street
Richland, WA 99354

Location: PNL

ACCREDITED
Certificate# 2724.01

CALIBRATION INFORMATION

Date Received: 22-Jan-2020
Calibration Date: 24-Jan-2020
Due Date: 24-Jan-2021
Limited: N
Calibration Specification: ISO/IEC 17025 Accred
Procedure/Rev: Calipers ID/OD/NIB (SLI 28-161 Rev. 3.6)

Temperature: 68 F
Humidity: 38 %
Condition: PASS
As-Found: In-Tolerance
As-Left: In-Tolerance

STANDARDS USED

Asset No.	Due	Manufacturer/Model	Asset No.	Due	Manufacturer/Model
43696	11/13/2020	AMP .0390-.0440	0065272	3/1/2021	COLLINS MICRO FLAT CO. INC. 3' x 14'
0065751	1/29/2020	FLUKE 2626-S	002-41-05-001	7/12/2020	MITUTOYO 515-560
0067925	10/23/2020	STARRETT RC88AA/AC11.A			

☒ Accepted ☐ Rejected / Per Clause(s): 175b
No indication of Out-Of-Tolerance condition(s)
AQSS & OOT Reviewer

NOTES/CONDITIONS:

Energy Northwest Standards Laboratory (ENSL) is accredited in accordance with the recognized International Standard ISO/IEC 17025:2005, (2017) General Requirements for the Competence of Testing and Calibration Laboratories. This laboratory also meets the requirements of ANSI/NCSL Z540-1-1994 and any additional program requirements in the field of calibration. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated January 2009). In addition, the standards and calibration program of the Energy Northwest Standards Laboratory complies with the applicable requirements of 10 CFR50 Appendix B and ASME NQA-1. This calibration complies with Energy Northwest Standards Laboratory Quality Manual, QM-1 Rev.11 (09 January 2019).

This calibration is traceable to the SI through the National Institute of Standards and Technology (NIST), accepted intrinsic standards, or ratio type measurements. The uncertainties were computed in accordance with the ISO Guide to the Expression of Uncertainty in Measurement (GUM). A coverage factor of approximately 2.0 sigma (k=2.0) has been applied to the standard uncertainty to express the expanded uncertainty at a 95% confidence level. The unaccredited test points, where applicable, are marked with a "^", or clearly identified. Functional tests (Pass/Fail) are not accredited. No statement of compliance with specifications is made or implied on this certificate. Uncertainties reported in base units at time of test where applicable and do not take into account transit, usage, drift over time, or other factors affecting stability. The results stated in this report relate only to item identified in the Asset Information portion of this report. This Report may not be reproduced, except in full, without the permission of the Energy Northwest Standards Laboratory.

Standards Laboratory Management — 1/28/2020 Date

J Larson (Josh)
Certified By

ORIGINAL

ENERGY NORTHWEST
STANDARDS LABORATORY

Calibration Report

PNL-13357600:1579876223

Asset Number	Manufacturer	Model	Serial No.
PNL-13357600	MITUTOYO	CD-6 IN CSX	13357600
Found/Left Data	**Cal Date**	**Performed by**	
FOUND-LEFT	24-Jan-2020	J Larson (Josh)	

Remarks: For this report where applicable: percentages are to be read as percent of reading unless noted otherwise; IV is defined as the indicated value; MV is defined as measured value; FS is defined as full scale; *L* is the numerical value of the nominal length of the device measured in inches; *D* is the diameter of the device in inches; and *R* is the resolution of the unit under test; GB indicates guardbanding; TAR represents test accuracy ratio and are informative and not used for decisions.

Certified to specifications listed below. Uncertainty per scope: (20 + 7L) µin + (0.6 x resolution) µin

The recording of false, fictitious or fraudulant statements or entries on this document may be punishable as a felony under Federal Statutes.

Test Description	True Value	Lower Limit	Indicated Value	Upper Limit	Error (% of Tol)	TAR/ GB
Calibrated to tolerances of ±0.001in for						
linearity and depth, and parallelism within 0.0005in.						
LINEARITY TEST						
OUTSIDE MEASUREMENT						
1.0000 In	1.00000	0.9990	1.0000	1.0010	0	Pass
2.0000 In	2.00000	1.9990	2.0000	2.0010	0	Pass
4.0000 In	4.00000	3.9990	4.0000	4.0010	0	Pass
6.0000 In	6.00000	5.9990	6.0000	6.0010	0	Pass
LINEARITY TEST						
INSIDE MEASUREMENT						
1.0000 In	1.00000	0.9990	1.0000	1.0010	0	Pass
2.0000 In	2.00000	1.9990	2.0000	2.0010	0	Pass
4.0000 In	4.00000	3.9990	4.0000	4.0010	0	Pass
6.0000 In	6.00000	5.9990	6.0000	6.0010	0	Pass
PARALLELISM						
Back = 0.044in						
Center = 0.044in						
Front = 0.044in						
Deviation from parallel within 0.0005"						
						Pass
DEPTH MEASUREMENT						
1.0000 In	1.00000	0.9990	1.0000	1.0010	0	Pass

END OF TEST DATA

ORIGINAL

PNL-13357600

ENERGY NORTHWEST

CALIBRATION SERVICES PNNL SALES ORDER

Energy Northwest Standards Laboratory www.energy-northwest.com/products/calibration

Mailing Address: PO Box 968
Mail Drop 1025
Richland, WA 99352-0968

Shipping Address: Energy Northwest
Standards Lab
Attn: Colin Carson
76 North Power Plant Loop
Richland, WA 99354-0968

Phone: (509) 377-8515 / 509-377-8444 Fax: (509) 377-8219 E-mail: ENSLSalesOrders@energy-northwest.com

Customer Information (Please Print)

CompanyName: PNNL

Address: P.O. Box 999

RECEIVED JAN 22 2020 By

Taxable: ☐ Yes ☒ No

If no, please supply tax exempt form.

Phone/Fax: 375.7300

Contact: Lisa M. Stoetzel

Payment Type: ☐ Net 30 PO; ☒ Visa; ☐ MC

PNNL P-Card Order #: PCD-504635

Certificate/Calibration Requirements
Type of Calibration:
☐ **Manufacturer** (ANSI Z540-1)
☐ **Customer / Limited** (ANSI Z540-1)
☒ **Accredited** (ISO 17025) *See Note

Certificate of Conformance: ☒ Yes ☐ No

Notice of Discrepancy: ☒ Yes ☐ No

*Note: Energy Northwest Standards Laboratory Statement of Compliance:
Energy Northwest Standards Laboratory (ENSL) employs a shared risk approach to statements of compliance. Unless directed otherwise, ENSL will provide ISO/IEC 17025 Accredited Calibrations testing to manufacturer's specifications, customer's specifications, or government/industry standards. The Calibration Certificate/Report will report the calibration condition (In-Tolerance, Out-Of-Tolerance, Pass/Fail) without factoring in the measurement uncertainties to determine compliance to those specifications or standards and will list those measurement uncertainties.

See P-Card Agreement for Terms & Conditions.

Authorized Customer Name and Title (Please print): Lisa M. Stoetzel, Technician

Email address: lisa.stoetzel@pnnl.gov

Phone: 375.7300

N/A
Signature

1/16/2020
Date

Lab Acceptance (Name / Date): _____

ENSL Sales Order Number / Date: _____

Item #	Quantity	Description ✓	Price Each
1	1	Mitutoyo Digital Slide Caliper, Model CD-6"ASX, 6 inch, Resolution 0.0005 Inch, S/N 13357600 ✓ JJ Jan -2020	$189.58
2	1	Mitutoyo Digital Slide Caliper, Model CD-6"CSX, 6 inch, Resolution 0.0005 Inch, S/N 08459791 - Expedite	$303.33
3	1	Starrett Outside Micrometer, Catalog No. 230RL, Readability: 0.001 inch x 1 inch, S/N 230-1	$252.78
		NEW JJ Calibrations, Inc. 503.786.3005 Cert 694275 Cal 02/12/2019 Due 02/12/2020 S/N 13357600 ID# 20180016	

ORIGINAL

ENERGY NORTHWEST

ENERGY NORTHWEST

CALIBRATION SERVICES PNNL SALES ORDER

Energy Northwest Standards Laboratory www.energy-northwest.com/products/calibration

Mailing Address: PO Box 968
Mail Drop 1025
Richland, WA 99352-0968

Shipping Address: Energy Northwest
Standards Lab
Attn: Colin Carson
76 North Power Plant Loop
Richland, WA 99354-0968

Phone: (509) 377-8515 / 509-377-8444 Fax: (509) 377-8219 E-mail: ENSLSalesOrders@energy-northwest.com

ENSL Quote Provided By Name: Terry L. Daffe Date: 1/16/2020

ORIGINAL

Asset#

PNL-13357600_01242020

Barcode

MEMORANDUM

Battelle
The Business of Innovation

Date:	**September 18, 2020**	Project No.:	**77150**
To:	**Larry Greenwood**		
From:	**Andrew Carney** Andrew M Carney Digitally signed by Andrew M Carney Date: 2020.09.21 11:35:24 -07'00'	Internal Distribution:	**File/LB**
Subject:	**Helium Leak Testing of INL Vanadium Capsules**		

Larry,

The helium leak testing of 20 INL vanadium capsules has been completed. All 20 capsules were found to have an average normalized helium leak rate value of 1.46 x 10^{-9} atm cc/sec. The leak test was performed using a Pfeiffer HLT550 (serial number 40106410) helium leak detector and a calibrated helium leak (serial number 6627) supplied by Vacuum Technology Incorporated. Testing was performed on 9/02/2020. Helium leak testing was done following PNNL technical procedure RPL-HE-001, Rev. 0, *Dosimetry Capsule Helium Leak Testing*. A record of the analysis was recorded in LRB 61427 on page 93. Work package NG0120 has been charged for the analysis.

Helium Soak

Capsules were placed in a helium bomb chamber that was evacuated to a system pressure of 1.5 x 10^{-6} Torr. The chamber was subsequently pressurized with 2650 Torr of helium from 8/27/2020 – 09/02/2020. Chamber pressure was 2046 Torr prior to the capsules being removed from the chamber on 09/02/2020. All capsules were allowed to degas for 1 hour per procedure before leak testing.

Leak Rate Analysis

Leak rate analysis was performed by taking alternating measurements of the calibrated leak and the capsules' leak rates after establishing the detector's background leak rate. The calibrated leak rate, 2.01 x 10^{-8} atm cc/sec (01/29/15), for the standard (6627) corrected for a 0.1% depletion per year, 2.00 x10^{-8} atm cc/sec, and the observed rate was 2.3 - 2.4 x10^{-8} atm cc/sec. The capsules' leak rates were measured as a set. An observed rate greater than 1 x 10^{-8} atm cc/sec was designated as the threshold for a capsule leak, and any capsule observed with this leak rate or greater was rejected.

All capsules had a leak rate $\leq$1 x 10^{-8} atm cc/sec.

If you have any questions, please contact me at 509-375-6433.

Capsules tested	Average normalized value of accepted capsules (atm cc / sec)	Capsules accepted	Capsules rejected
20 capsules (A7, T2, 41, 1I, V1, X2, 77, 61, 7F, A1, 05, 1F, 50, 97, 5D, B5, 9J, A5, 9C, 5U)	1.46E-09	A7, T2, 41, 1I, V1, X2, 77, 61, 7F, A1, 05, 1F, 50, 97, 5D, B5, 9J, A5, 9C, 5U	None

Concurrence: Richard M Cox Digitally signed by Richard M Cox Date: 2020.09.21 11:42:33 -07'00' Date:_____

September 2, 2020

Adjusted Calibrated leak rate	2.00E-08	Ratio leak standard	0.845					
	Measured Helium leak rate (atm cc/sec)	Normalized Value	Average Normalized Value	STDEV for Normalized Value	Capsules tested Id.	Average Normalized Leak rate (atm cc / sec)	Capsules accepted	Capsules rejected
System Blank	2.9E-09	2.4E-09			A7, T2, 41, 1I, V1, X2, 77, 61, 7F, A1, 05, 1F, 50, 97, 5D, B5, 9J, A5, 9C, 5U	1.46E-09	20	0
	2.5E-09	2.1E-09	2.14E-09	2.97E-10				
	2.2E-09	1.9E-09						
Calibrated Std leak (6627)	2.3E-08							
	2.4E-08	2.4E-08	0.845					
	2.4E-08							
Chamber blank	2.2E-09	1.9E-09						
	1.9E-09	1.6E-09	1.38E-09	2.97E-10				
	1.5E-09	1.3E-09						
INL Capsules (All)	1.9E-09	1.6E-09						
	1.7E-09	1.4E-09	1.46E-09	1.29E-10				
	1.6E-09	1.4E-09						
Chamber blank	1.6E-09	1.4E-09						
	1.4E-09	1.2E-09	1.18E-09	1.69E-10				
	1.2E-09	1.0E-09						
Calibrated Std leak (6627)	2.4E-08							
	2.3E-08	2.3E-08	0.857					
	2.3E-08							
System Blank	1.2E-09	1.0E-09						
	1.0E-09	8.4E-10	8.98E-10	1.00E-10				
	9.9E-10	8.4E-10						

Calibration Data		
Leak Rate	2.01E-08	atm-cc/s
Depletion Rate	-0.1%	/year
Calibration Date:	January 29, 2015	
Years Since Cal	5.6	
Adjusted Leak Rate	2.00E-08	atm-cc/s
		[(Depletion % + 1) ^ years since Cal * Cal Leak Rate]

Calibration Leak (6627)

4/26/2019	6/13/2019	9/2/2020
2.6E-08	2.4E-08	2.3E-08
2.7E-08	2.3E-08	2.4E-08
2.6E-08	2.3E-08	2.4E-08
2.5E-08	2.3E-08	2.4E-08
2.5E-08	2.2E-08	2.3E-08
2.4E-08	2.2E-08	2.3E-08

5/2/2019	6/25/2019
2.3E-08	2.2E-08
2.3E-08	2.2E-08
2.4E-08	2.3E-08
2.5E-08	2.3E-08
2.4E-08	2.3E-08
2.4E-08	2.3E-08

5/3/2019	8/29/2019
2.4E-08	2.8E-08
2.4E-08	2.7E-08
2.4E-08	2.6E-08
2.6E-08	2.4E-08
2.6E-08	2.4E-08
2.6E-08	2.4E-08

5/9/2019	1/27/2020
2.4E-08	2.3E-08
2.4E-08	2.3E-08
2.4E-08	2.4E-08
2.4E-08	2.4E-08
2.4E-08	2.4E-08
2.4E-08	2.4E-08

Average 2.41E-08

Signature / date Andrew Carey 9/18/2020

Technical Review / date Richard M Cox Digitally signed by Richard M Cox Date: 2020.09.21 11:42:53 -07'00'

8/27/2020

Capsules placed in chamber and evacuated to 1.5×10^{-6} torr. Chamber pressurized to 2650 torr He at 11:30 AM 8/27/2020.

9/02/2020 Helium pressure over capsules 2046 torr. Capsules removed from chamber and allowed to degas for one hour.

- Capsules: A7, T2, 41, 1I, V1, X2, 77, b1, 7F, A1, 05, 1F, 50, 97, 5D, B5, 9J, A5, 9C, 5U

System Blank: 2.9×10^{-9}
2.5×10^{-9}
2.2×10^{-9}

System Blank: 1.2×10^{-9}
1.0×10^{-9}
9.9×10^{-10}

Calibration Leak (6627): 2.3×10^{-8}
2.4×10^{-8}
2.4×10^{-8}

Chamber Blank: 2.2×10^{-9}
1.9×10^{-9}
1.5×10^{-9}

All Capsules: 1.9×10^{-9}
1.7×10^{-9}
1.6×10^{-9}

Chamber Blank: 1.6×10^{-9}
1.4×10^{-9}
1.2×10^{-9}

Calibration Leak (6627): 2.4×10^{-8}
2.3×10^{-8}
2.3×10^{-8}

Project No. 77150 Date of Work 9/02/2020

Entered by Andrew Carney Date 09/02/2020

Disclosed to and Understood By

Signed 1) Richard M Cox Digitally signed by Richard M Cox Date: 2020.09.21 11:43:19 -07'00' Date

2) Date

vti Vacuum Technology INCORPORATED

ilac-MRA A2LA ACCREDITED
Calibration Laboratory
Certificate No. 1707.01

CERTIFICATE OF CALIBRATION

Calibration Report Number: 6627-ACAL-COMP-1-85437
Customer: PACIFIC NORTHWEST NATIONAL LABORATORY

790 6TH Street
Richland, WA USA 99354

Customer Purchase Order Number: 250729 **Order Number:** 13479

MODEL NUMBER AND/OR DESCRIPTION	SERIAL NUMBER
CLP-8-He-4FVCR-110CC ✓	6627 ✓

CALIBRATION RESULTS

The unit specified was calibrated using the procedure defined below and conforms to the requirements of the customer's purchase order.

METHODS AND EQUIPMENT

The unit was calibrated by a traceable comparison against a primary transfer standard on an Automated AERO VAC Mass Spectrometer System (AutoCal) using Calibration Procedure VTI-QAP-11-001, Rev E.

Procedures used are in accordance with ISO/IEC 17025:2005, ISO/IEC Guide 25, Military Standard 45662A, and ANSI/NCSL Z540-1-1994, which supersedes Military Standard 45662A.

MEASUREMENT TRACEABILITY

All measurement and test equipment (M&TE) as well as all primary transfer standards are calibrated traceable to NIST. The Calibrated Leak Lab maintains all relevant calibration records for the M&TE and primary transfer standards used. Spinning Rotor Gauges and Capacitance Manometers are calibrated traceable to NIST. Temperature measurement devices and reference volumes are traceable through accredited calibration sources. Time measurements are traceable through the U.S. Naval Observatory Master Clock.

MEASUREMENT UNCERTAINTY

The total estimated measurement uncertainty for this calibration, as reported on Page 2 of this Certificate of Calibration, reflects a coverage factor of 2 (k=2). The reported estimated measurement uncertainty is calculated in accordance with NIST Technical Note 1297, 1994 Edition, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results.*

Greg Swift
Calibration Lab Technician

Kimberly L Thomas
Calibration Lab Approver
Date: 29-Jan-2015

☑ Accepted ☐ Rejected / Per Clause(s): 188

AQSS Reviewer AS Coffey 2-12-15

Vacuum Technology
INCORPORATED

CERTIFICATE OF CALIBRATION

Vacuum Technology Inc.
1003 Alvin Weinberg Drive
Oak Ridge, TN 37830
Phone:(865)481-3342

Calibration Laboratory
Certificate No. 1707.01

Calibration Report Number
6627-ACAL-COMP-1-85437

Customer	Order ID
PACIFIC NORTHWEST NATIONAL LABORATORY	13479

Purchase Order	Recalibration ID
250729	

Accu-Flow Calibrated Leak

Model #: CLP-8-He-4FVCR-110CC
Element : 7740 Pyrex — Serial #: 6627
Fill Gas: Helium — Fill Pressure: 3177 Torr
Volume: 110cc

Calibration Data

LR = 2.01×10^{-8} atm-cc/s into vac. at 23.2°C
n = 8.30×10^{-13} mol/s (±6.49 %),(k=2)
Depletion Rate: 0.1% Per Year
Calibration Date: Jan 29, 2015
Temp Coeff: 4.0% per °C

Warning!!
Do not exceed 100°C during bakeout!

VTI Recalibration Service
Phone:(865)481-3342
Email:sales@vacuumtechnology.com
For permeation leaks, the isolation valve should be left open when not in use to prevent saturation of the glass element.

Pressure Measurement Equip.		Volume Standard	
Manufacturer	-	Manufacturer	-
Model#	-	Model#	-
Serial#	-	Serial#	-
Report # :	-	Report # :	-
Cal Date :	-	Cal Date :	-
Cal Due :	-	Cal Due :	-

Primary Transfer Flow Standard		Temperature Measurement Equip.	
Manufacturer	VTI	Manufacturer	Athena
Leak Model#	GPP-8-1P-He-4FVCR-110CC-IHS	Model#	16-C-B-B-B-30-00 w/101105
Leak Serial#	903	Serial#	1322000547
Report # :	903- Dec-2014	Report # :	1000356132
Cal Date:	December 3, 2014	Cal Date :	May 13, 2014
Cal Due :	December 3, 2015	Cal Due :	May 13, 2015
Cal Temp (°C):	24.6		
Coeff (%/°C) :	4.0%		
Gas Type	-He		
Q (atm-cc/s)	2.10E-08		

ANALYTICAL REPORT

Holston Gases Inc.
545 W. Baxter Ave.
Knoxville,Tn 37921
Phone# 865-573-1917

Batch# 112114JC

Ultra High Purity Helium

This gas meets the requirements of Federal Specification BB-H-1168 and AWS A5.32

Date: 11-24-2014

Component	Nominal	Actual	Method
Helium	99.999%	99.999%	
O2	< 1 ppm	< 1 ppm	Gow-Mac 580 Series GC
N2	< 5 ppm	< 1 ppm	Gow-Mac 580 Series GC
CO	< 0.5 ppm	"ND"	Gow-Mac 580 Series GC
ME	< 0.5 ppm	"ND"	Gow-Mac 580 Series GC
H2O	< 2 ppm	< 1 ppm	Meeco AquaVolt

Serial Numbers in Batch:

4505224Y	4126589Y
4550936Y	LX-400185
N-295160	W-382298
4525210Y	4432479Y
5123176Y	300-1132
L462458	4525170Y
AF-100152	4090054Y
LX-1441	4525224Y
5123138Y	LX-420311
LX-657964	300-0819
5123140Y	WC-789295
1106304Y*	C5368018

Note: (*) Indicates cylinder tested.

Issued By: Curtis J. Briggs

PACIFIC NORTHWEST NATIONAL LABORATORY

QUALITY ASSURANCE PROJECT PLAN for

The SAM-2 EXPERIMENT

Product Line Number: 0053 - Nuclear Science and Legacy Waste

Project Number: 77150

July 2020

Approvals:

Project Manager and Primary Point of Contact:	Lawrence R Greenwood Digitally signed by Lawrence R Greenwood Date: 2020.07.20 08:11:25 -07'00'	
	Larry R. Greenwood	Date
Quality Engineer:	Samuel A. Adams Digitally signed by Samuel A. Adams Date: 2020.07.20 08:16:32 -07'00'	
	Samuel A. Adams	Date

PROJECT 77150 QUALITY ASSURANCE PLAN

Title: Fluence Capsules for the SAM-2 Experiment
Product Line Number: 0053 - Nuclear Science and Legacy Waste
Project Number: 77150

The approach for meeting the identified Quality Assurance (QA) program requirements for project 77150 is provided below. This project has a funding expiration date of 10/30/2020; delivery of Task 1 dosimeters is expected by about October 15, 2020.

Background

This statement of work (SOW) applies to the fabrication and analysis of flux wire capsules that will be used in the SAM-2 experiment. This experiment is a drop-in design slated for irradiation in the Advance Test Reactor (ATR) at Idaho National Laboratory (INL). The 8 neutron fluence capsules will be used to determine the neutron exposure that the specimens have received.

For Task 1 Pacific Northwest National Laboratory (PNNL) will prepare eight (8) neutron fluence monitors for irradiation in the ATR. Task 2 will involve analysis port-irradiation; the monitors will be returned to PNNL for analysis in FY21 after irradiation. The analysis report will include the neutron fluences and radiation damage calculations for all irradiated positions in the experiment.

Task 1

Fluence Wire Capsules

A total of ten (eight and two spares) fluence monitor capsules are required. All of the capsules will measure 0.047 to 0.054" OD x 0.420" maximum length and shall be made of pure vanadium (99.8%). All of the capsules will contain Fe, Ti, Nb and 0.1% Co-Al wires. Each capsule shall be stamped or engraved with a unique identification number, electron beam welded in a vacuum or sealed by a laser in inert gas, and helium leak tested. An acceptable helium leak test rate will be less than or equal to 1x10-7 standard cc/second.

Each fluence wire capsule described above shall contain one wire segment of each of the following four materials:

- 99.99% minimum purity iron (Fe) wire
- 99.92% minimum purity titanium (Ti) wire
- Niobium (Nb) wire Reference Material. 526C from the Institute for Reference Materials and Measurements (IRMM)
- 0.116% cobalt (Co)-aluminum (Al) wire from the National Institute of Standards and Technology (NIST)

Required supplies, measuring and test equipment, vendor calibration of M&TE and standards for user-calibrated M&TE are already in place and in routine use. No sub-contracts are necessary.

Task 2

Following irradiation of the eight fluence wire capsules in the ATR at INL, INL shall load the capsules into a shipping cask and ship them to PNNL. INL shall ship the capsules to PNNL within 90

days following disassembly of the experiment post-irradiation. PNNL shall remove the irradiated fluence wire capsules and ship the empty cask back to INL.

Work to be performed by PNNL includes:

1. Open the capsules and gamma count each wire to determine the activation product activities.

2. Dissolve the niobium wires in acid and prepare aliquots on filter paper for x-ray analysis to determine the Nb-93m activity.

3. Determine the saturated activation rates using irradiation history data provided by INL. The activities will be used to calculate nuclear activation rates using neutron spectral and reactor power history data provided by INL.

4. Calculate neutron fluences by adjusting the neutron spectrum using a least squares procedure (STAYSL and associated computer codes). Radiation damage parameters including displacements per atom (dpa) and gas production will be calculated using the adjusted neutron spectra.

5. Deliver a letter report summarizing the activities of the eight fluence wire capsules, the calculated neutron fluence of the flux wires, measured activity data, and damage parameters (dpa, gas production), calculated using the SPECTER computer code.

The measured neutron fluences determined by analysis of the flux wires with the appropriate neutron energy range will be used to compare with calculations from physics analyses.

All work is required to be performed according to approved procedures and following an approved Quality Assurance Plan that meets applicable NQA-1 2008/NQA-1A-2009 addenda quality standards.

Task 1 Deliverable

Each fluence wire capsule fabrication shall be documented. All documentation shall be supplied with the fluence wire capsule shipment. Documentation shall include:

1. Certification of Conformance for meeting all requirements of the SOW. The Certificate of Conformance will contain the following information:
   a. Identification of the purchased material or equipment, such as by the purchase order number
   b. Identification of the specific procurement requirements met by the purchased material or equipment, such as codes, standards, and other specifications. This may be accomplished by including a list of the specific requirements or by providing, on-site, a copy of the purchase order and the procurement specifications or drawings, together with a suitable certificate. The procurement requirements identified shall include any approved changes, waivers, or deviations applicable to the subject material or equipment.
   c. Identification of any procurement requirements that have not been met, together with an explanation and the means for resolving the non-conformances.
   d. Signature or otherwise authenticated by a person who is responsible for this quality assurance function and whose function and position are described in the Purchaser's or Supplier's quality assurance program.

2. Material certifications
3. Capsule dimensions
4. Fluence wire masses
5. Helium leak test results
6. Date that each measurement/test was performed
7. Calibration certificates for each measurement/test tool. Calibration certificates shall include:
   a. Traceability to NIST
   b. Accuracy
   c. Date of calibration
   d. Calibration due dates

Task 2 Deliverable

Deliver a letter report summarizing the activities of the eight fluence wire capsules, the calculated neutron fluence of the flux wires, measured activity data, and damage parameters (dpa, gas production), calculated using the SPECTER computer code.

1.0 Organization

1.1 Quality Assurance Project Plan

This Quality Assurance Project Plan (QAPP), 77150, Rev. 0, *Fluence Capsules for the SAM-2 Experiment* contains the project-level QA requirements for the administration of the PNNL QA Program for the INL project. As such, it is to be implemented by all participant project staff. This QAPP is prepared by the project Quality Engineer (QE) and approved by the project manager. Staff will be trained to this plan.

1.2 Scope

The quality requirements for this work are documented in the Memorandum Purchase Order (MPO) 00239221 (Effective Date: 7/15/2020 prepared by the INL client. The expiration/completion date for Task I is 10/30/2020. The MPO references a Statement of Work No. 17494, Revision No. 0 entitled, "Fluence Capsules for the SAM-2 Experiment," dated June 23, 2020.

Requirements

The MPO/SOW referenced quality standard is ASME NQA-1 2008 with NQA-1A-2009 addenda, except the following: Requirements 3, 9, 12, and 14.

Referenced ASTM documents included the following:

- ASTM E1297 - 08 Standard Test Method for Measuring Fast-Neutron Reaction Rates by Radioactivation of Niobium

- ASTM E261 - 10 Standard Practice for Determining Neutron Fluence, Fluence Rate, and Spectra by Radioactivation Techniques

- ASTM E526 - 08 Standard Test Method for Measuring Fast-Neutron Reaction Rates by Radioactivation of Titanium

QA Clauses identified in the MPO/SOW documents included:

Clause No. 241C - ASME NQA-1-2008 1a 2009 addenda: The supplier shall implement and maintain a quality system in accordance with the applicable elements of ASME-NQA-1-2008, 1a 2009 Addenda, “Quality Assurance Requirements for Nuclear Facility Applications."

Clause No. 422 - Metals/Piping - Actual Chemical and Physical Report:
2.1. The supplier shall provide CMTR for all material (metals and/or piping) delivered under this MPO. CMTR documentation shall include:

2.1.1. Identification of actual chemical and physical test results performed on a representative sample of the material heat/melt/lot.
2.1.2. Heat/melt/lot number.
2.1.3. Traceability to the material and/or end item(s) delivered.
2.1.4. Contractor MPO number.
2.1.5. Identification of Testing and Certifying organization.
2.1.6. Statement of test results certification.
2.1.7. Legibility and reproducible: each page of documentation supplied shall be photographically reproducible through two additional reproductions. Any illegible or unreproducible documentation shall be returned for replacement.
2.1.8. Applicable specification.

2.2 Physical marking/labeling of material is required to maintain CMTR traceability. As a minimum, marking shall include:

2.2.1. Specification designation.
2.2.2. Heat/melt/lot number.
2.2.3. Manufacturer identification.

2.3. Unless otherwise specified, CMTR documentation shall be delivered With Shipment (WS) to the final destination.

Clause No. 437 - Certificate of Conformance: The supplier shall certify that item(s) or service(s) delivered under this MPO conform(s) in all respects to the MPO requirements. Supplier certification shall be documented utilizing Contractor Form 540.04, "Certificate of Conformance," or supplier's standard Certificate of Conformance (C of C). Certifications shall be complete, accurate, legible, and reproducible. Incomplete or inaccurate certifications will be refused. Each certification shall be issued by the designated supplier certifying authority in accordance with established supplier certification procedures. The certification must be signed (electronic signature is acceptable) by an authorized company representative. Unless otherwise authorized, the supplier’s C of C shall be submitted WS, to the shipping destination.

Clause No. 723** - Inspection/Test Data:

4.1. Part, drawing, and specification number
4.2. Serial number
4.3. Heat/melt Identification of raw material
4.4. Lot identification of each item
4.5. Characteristic subject to inspection
4.6. Inspection sequence
4.7. Acceptance criteria and source
4.8. Inspection results
4.9. Examination method
4.10. Measure & Test Equipment (M&TE)
4.11. Inspection setup
4.12. Environmental conditions
4.13. Test personnel identification
4.14. Dated approval signature by supplier authorized representative.

****** PNNL was provided with direction to consider clauses that do not apply as NA. INL stated that their interest is in the documentation that you use to get your work done. Any test procedure, traveler and or inspection records that are generated and are applicable to the clause are useful. All clauses cited by the MPO and SOW that are applicable will be addressed in part or in whole as necessary.

A crosswalk document between the Analytical Support Operations (ASO) QA Plan and the ASME NQA-1-2008/1a 2009 criteria addresses ASO compliance to the requirements and this has been provided to INL.

The PNNL Radiochemical Processing Laboratory (RPL) was evaluated by INL in a desk audit in July 2016, a follow-up in December 2016, another in October 2017, and most recently in March 2019. The basis of the March 2019 audit was ASO-QAP-001, Rev. 11, *ASO QA Plan*, associated administrative and technical procedures and various QA Project Plans supporting assigned INL work.

Interfaces

Dr. Larry Greenwood is the PNNL Project Manager. Dr. Larry Greenwood is the primary point of contact. Samuel Adams will be the assigned Quality Engineer (QE). For INL, Klint Anderson will be the primary technical point of contact.

The activities required to fabricate the capsules under Task 1 will include the following:

1) Prepare QA Plan and any required procedures and test instructions
2) Issue QA Plan and any required procedures and test instructions
3) Assure that training has been assigned and completed for QA Plan and any required procedures
4) Fabricate and inspect the fluence capsules as noted above.
5) The capsules will be uniquely identified, electron beam sealed in a vacuum, and helium leak tested. Documentation will consist of the capsule and wire dimensions and weights, material certifications, and helium leak testing results.
6) PNNL will ship the capsules and supporting documentation including a Certificate of Compliance that the package submitted meets the requirements of the PO, capsule and wire dimensions, and weights, material certifications, inspection and helium leak test results.
7) PNNL will prepare the supporting data package according to requirements and specifications and submit to INL.

The activities required to perform testing under Task 2 will include the following steps. There is a gap between the time of capsule fabrication and return of irradiated capsules to PNNL for analysis by INL.

8) Review prepared QA Plan and existing required procedures and test instructions and develop or revise any that are necessary.
9) If the QA plan requires revision, issue QA Plan and any required procedures and test instructions
10) Assure that training has been assigned and completed for QA Plan and any required procedures
11) Receive the samples.
12) Prepare the samples for analysis by extracting them from the aluminum fuel holder.
13) Perform gamma spectroscopy analyses of the wires to determine activities of Sc-46, Mn-54, Nb-94, and Fe-59. The Nb wires will be dissolved and thin mounts prepared for X-ray measurement of Nb-93m.
14) The measured activities will be used to calculate nuclear activation rates using neutron spectral and reactor power history information provided by INL.
15) Neutron fluences will be calculated by adjusting the neutron spectra using a least squares procedure (STAYSL PNNL and associated computer codes). Radiation damage parameters (dpa, gas production) will be calculated using the adjusted neutron spectra and the SPECTER computer code.
16) Prepare the data package according to requirements and specifications and submit to INL.

Applicable and excluded NQA-1 requirements for the planned scope of work transmitted to PNNL RPL from INL are the following basic requirements:

1.0 Organization
2.0 Quality Assurance Program
3.0 Excluded - Design Control
4.0 Procurement Document Control
5.0 Instructions, Procedures and Drawings
6.0 Document Control
7.0 Control of Purchased Items and Services
8.0 Identification and Control of Items
9.0 Excluded -Control of Processes
10.0 Inspection
11.0 Test Control – basic, test requirements and test records
12.0 Excluded -Control of Measuring and Test Equipment
13.0 Handling, Storage, and Shipping
14.0 Excluded -Inspection, Test and Operating Status
15.0 Control of Nonconforming Items – basic, identification and segregation
16.0 Corrective Action
17.0 Quality Assurance Records, basic, generation of records and storage
18.0 Audits

Responsibilities and authorities of PNNL staff working in the Radiochemical and Processing Laboratory (RPL) and those staff responsible for achieving and maintaining quality are identified in this document and in technical implementing procedures. An integrated approach to management is applied at PNNL and specifically in the PNNL RPL where the work will be performed.

1.3 Roles and Responsibilities

1.3.1 Cognizant Space Manager

A Cognizant Space Manager (CSM) and a delegate CSM are assigned to each hazardous space in the RPL as part of the PNNL Integrated Operations System (IOPS). RPL CSMs play a key role in hazard mitigation in the RPL and are delegated responsibility from their line manager and the RPL Manager for the following RPL-unique CSM responsibilities: CSM responsibilities are the following:

- Authorization of day-to-day laboratory operations.
- Determination if work hazards are adequately mitigated via procedure or by other hazard mitigation procedure, practice, and/or permit prior to the start of work by working with the researcher-provided information and using the appropriate subject matter experts for assistance, as needed.
- Tracking and managing radioactive material inventories within their assigned locations using the Radioactive Material Tracking (RMT) system as specified in procedure, RPL-ADMIN-002, current revision, *Radioactive Material Tracking*.
- Maintain radioactive material inventory limits with the applicable control ranges and conducting periodic evaluations of radioactive material inventory holdings including completion of a certification form per procedure RPL-SA-001, current revision, *Radioactive Material Inventory Tracking* Instruction.

1.3.2 Project Manager

The Project Manager is the Single Point of Contact between the funding client and the RPL research organization. The Project Manager has responsibility to interface directly with the client, negotiate the scope of work, and meet the client's quality, cost, and schedule requirements. Specific responsibilities related to the RPL include:

- Managing projects per requirements.
- Obtaining CSM approval for work proposed in RPL laboratory spaces.
- Identifying project risk(s).
- Communicating project expectations to project staff with regards to operations and safety.
- Determining what procedures, test plans and test instructions apply to the work.
- Assigning training.
- Obtaining and documenting technical reviews.
- Monitoring project activities.

1.3.3 Assigned Quality Engineer

The Quality Engineer (QE) assigned to this project provides independent oversight and reviews project requirements with the Project Manager, prepares and revises this QA plan, and ensures that requirements that apply to the work are satisfied. The assigned QE ensures that the analysis reports meet all requirements specified in the MPO.

1.3.4 Technician/Analysis Staff

RPL staff assigned to fabricate capsules and perform helium leak testing are required to work to a test instruction that documents steps performed to assemble and inspect capsules prior to shipping. Capsules are stamped or engraved with a unique identification number and sealed using an electron beam welder.

RPL staff assigned to perform gamma and x-ray analyses for fluence capsule samples are required to follow reviewed and approved procedures to perform work as specified in work practices, by use permits, and any other applicable work permits. Staff are required to ensure that measuring and test equipment used is adequately calibrated and that the results of analyses are documented and defensible and of known quality to meet project objectives. Staff are trained to the QA Plan and procedures.

2.0 QUALITY ASSURANCE PROGRAM

2.1 INL requirements are defined in the document, Memorandum Purchase Order (MPO)00239221, Effective Date:07/15/2020. The expiration/completion date is 10/30/2020. The MPO references a Statement of Work No.17494, Revision No. 0 entitled, "Fluence Capsules for SAM-2 Experiment," dated June 23, 2020.

2.2 This project-specific QA Program for PNNL Project 77150 is being implemented for this project. PNNL's applicable policies and procedures will be followed as listed in the sections below.

The ASO QA Plan, ASO-QAP-001, Rev. 11 (or most recent revision), *Analytical Support Operations Quality Assurance Plan)* is supported by the following applicable administrative procedures:

Table 2. ASO Administrative Procedures

Procedure Number	Procedure Title
PNL-ASO-052	*Balance Performance Checks*
PNL-ASO-058	*ASO Data Reporting*
PNL-ASO-062	*Standards*
PNL-ASO-065	*ASO Control Charting*
PNL-ASO-066	*Pipette Performance Check – Determination of Delivery Volume*
PNL-ASO-070	*Sample Management: Overview*
PNL-ASO-071	*Sample Management: Receipt and Inspection*
PNL-ASO-072	*Sample Management: Labeling, Log-in, and Work Authorization*
PNL-ASO-073	*Sample Management: Storage and Security*
PNL-ASO-074	*Sample Management: Distribution and Transfer of Unprocessed and Processed Samples*
PNL-ASO-075	*Sample Management: Disposition and Waste Disposal*
PNL-ASO-076	*ASO Records Management*
PNL-ASO-077	*ASO Document Control*
PNL-ASO-078	*ASO Training Plan*
PNL-ASO-079	*ASO Occurrences, Deficiencies and Nonconformances*
PNL-ASO-080	*ASO Instrument Software, Multiple- and Single-use Spreadsheets*
PNL-ASO-081	*ASO Procedure Development, Review and Approval Process*

The following technical procedures are proposed for use, but alternative procedures may also be used and those will be clearly defined in the reported results:

Table 3. ASO Technical Procedures

Procedure Number	Procedure Title
RPG-CMC-450	*Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)*

Procedure Number	Procedure Title
RPL-OP-001	*Routine Research Operations*

RPL staff expected to support this work have been identified as the following:

Table 4. Staffing for Project 75643 and QE Organizational Independence

Role	Assigned Staff	Organization
Project Manager	L Greenwood	D9H63
ASO Administrator	T Trang Le	D9H63
Fabrication	L Darnell	D9H63
Technical POC, gamma spectrometry /Technical Reviewer	L Greenwood	D9H63
Quality Engineer	S. Adams	PQ400
Analysis Staff GEA	Michael Cantaloub/ B Pierson/T Trang Le	D9H63
Technical Reviewers	L Greenwood/B Pierson/ C Soderquist	D9H63

Technical reviewers cannot review work they performed. Staff may be analysts for one data set and not for the following set, so the pool of knowledgeable technical reviewers may vary from sample batch to sample batch. Technical reviewers must be knowledgeable of the work they are reviewing. The staffing plan is to facilitate and ensure that staff participants are trained to applicable procedures.

Project-specific training is assigned by the Project Manager. At a minimum staff will be trained to the QA plan and applicable procedures for the work being performed. Training of staff may take the form of briefings, reading assignments, on-the-job training or mentoring. The Project Manager or the project QE may issue training assignments for QA and technical implementing procedures.

3.0 DESIGN REQUIREMENTS

This basic requirement is not applicable as no designs are required.

4.0 PROCUREMENT CONTROL

4.1 This basic requirement is applicable and quality-related procurements if needed, would be anticipated to support this work. However, required measuring and test equipment, vendor calibration of M&TE and standards for user-calibrated M&TE are already in place and in routine use.

When quality-related procurement actions are necessary, procurement control at PNNL is described in the following PNNL HDI workflow: *Acquire Product or Service.*

4.2 When needed, the assigned project QE shall approve all procurements containing QA requirements which must be processed via the HDI workflow, *Acquire and Product or Service.* The Project Manager will also review and approve these procurements.

Procurement planning at PNNL involves both the Project Manager and QE to determine if there are any applicable QA requirements or specifications; this includes any planned software purchases. Procurement of items and services that do not need specified QA requirements may be

processed using a PNNL Purchasing Card (P-Card) or through the Business to Business (B2B) Program. Items and services on the P-Card Prohibited Purchases and Purchases with Exceptions list) shall be purchased by use of a purchase requisition. The B2B program is a streamlined electronic purchasing mechanism for PNNL staff to purchase commercial, off-the-shelf items at competitive prices from pre-selected suppliers.

4.3 External suppliers of balance calibration services on a PNNL-wide basis involves the use of a vendor on the PNNL Evaluated Supplier Listing as specified by the Acquisition Quality Procedure AQP-03, *Pre-Award and At Site Evaluations Surveys and At Site Evaluations*. Submittals from calibration vendors are reviewed to determine their compliance with the procurement document technical and QA requirements in accordance with implementing procedure AQP-06, *Review of Supplier/Subcontractor Submitted Documents.*

5.0 INSTRUCTIONS, PROCEDURES, AND DRAWINGS

5.1 Content, review and approval of RPL procedures and TIs are guided by requirements specified in the ASO procedure, PNL-ASO-081, *ASO Procedure Development, Review and Approval Process.* This procedure specifies the format, review and approval process and issuing for implementing procedures used for work in RPL. The review and approval process addresses technical, quality and safety reviews to assure that concerns associated with performing work in a Category 2 nuclear facility are addressed. Procedures are screened and reviewed by a Safety Evaluation Screening process. Existing procedures to perform the work have been identified above.

5.2 This QA Project Plan, technical procedures and any necessary test instructions provide the acceptance criteria for determining that ASO analytical prescribed requirements defined by procedures listed in Table 2 have been met and that the tests have been satisfactorily accomplished.

6.0 DOCUMENT CONTROL

Once RPL procedures and test instructions are approved, the Project Manager determines the delivery method to assure that procedures and test instructions are available to staff and that only the most current revision is available for use. Changes to procedures must meet the requirements and follow the processes specified in PNL-ASO-081, *ASO Procedure Development, Review and Approval Process.* The most current versions of ASO procedures are available on the ASO SharePoint. ASO procedures are controlled under the ASO procedure, PNL-ASO-077, *ASO Document Control*. Test instructions, when used, are prepared, reviewed and approved before use and are considered as controlled documents.

7.0 CONTROL OF PURCHASED ITEMS AND SERVICES

This requirement is addressed by PNNL processes defined by the procurement system discussed in Section 4, Procurement Document Control above.

8.0 IDENTIFICATION AND CONTROL OF ITEMS

As noted above for Task 1 RPL will fabricate capsules with a unique identification number and ship these to INL.

RPL will receive the irradiated capsules returned from INL. Incoming analysis samples will be assigned a unique RPL identification (ID) number. All received samples will be evaluated for condition upon receipt and an Analytical Service Request (ASR) form completed and submitted to ASO Lead for

authorization to perform work. Chain of Custody forms, if provided, will be completed and returned to INL. All remaining sample material will be disposed of by RPL as radioactive waste.

9.0 CONTROL OF PROCESSES

This basic requirement is not applicable to the scope of work. Capsules are sealed using an electron beam welder, but this is not a welding process per se and only requires a special skill to perform this task.

10.0 INSPECTION

Task 1
Fabricated capsules will be inspected for conformance to required dimensions, visual inspection after sealing, a helium leak test and the bubble test.

Task 2
Capsules will be shipped from INL to RPL and will be inspected by PNNL upon receipt to evaluate sample container integrity and condition. If there are any concerns identified, INL will be immediately notified via the RPL Project Manager. Chain of Custody will be maintained by recording time and date of receipt and Chain of Custody forms, if provided to RPL, will be returned to INL. The capsule samples shall be controlled to prevent damage or loss and to minimize deterioration.

11.0 TEST CONTROL

This basic requirement is addressed by following project test instructions, and the administrative and technical procedures as noted above. Acceptance criteria for calibration verification and quality control samples are defined in procedures. Data reports contain a narrative discussing quality control sample results and the data report reports the sample and quality control sample results.

Excel spreadsheets are used for data reduction and reporting. Within the ASO QA Program multiple-use spreadsheets are developed, reviewed and approved for use. Locked cells are used for algorithms and entered or transcribed data are checked by technical and QE reviewers. Single-use spreadsheets require 100% technical review. Instrument software control and spreadsheet review and approval are addressed in the procedure, RPL-ASO-080, *ASO Instrument Software, Multiple- and Single-use Spreadsheets*.

12.0 CONTROL OF MEASURING AND TEST EQUIPMENT

12.1 Measuring and test equipment (M&TE) is controlled through the PNNL property management system. PNNL's calibration control system is explained in the HDI subject area, *Calibration* and addresses both vendor- and user-calibrated M&TE. When M&TE is used, manufacturer, model and serial number are recorded, as well as calibration status. Pedigrees to calibration standards are procedurally-controlled and traceable to certified materials. As previously discussed, a qualified vendor provides annual balance calibration services documented by applying a calibration sticker to the unit specifying serial number, calibration date and calibration due date. Calibration Reports are available and will be placed in the project record. M&TE needed for the work will be selected based on the type, range, accuracy, and tolerance needed and listed in the test instruction. Only M&TE within a valid calibration period will be selected for use.

12.2 No M&TE software is being developed or modified as part of the scope of work. Measuring and test equipment that contains software will be recalibrated when updates affecting the calibration are made to the software.

13.0 HANDLING, STORAGE, AND SHIPPING

Task 1
PNNL will complete all work and deliver the fluence wire capsules to INL after award of the MPO. Goods will be shipped to:

Idaho National Laboratory
1765 North Yellowstone Highway
Idaho Falls, Idaho 83415

Task 2
Capsules will be shipped from INL to RPL and will be inspected upon receipt to evaluate sample container integrity and condition. If there are any concerns identified, INL will be immediately notified via the RPL Project Manager. Chain of Custody will be maintained by recording time and date of receipt and Chain of Custody forms, if provided to RPL, will be returned to INL. The capsule samples shall be controlled to prevent damage or loss and to minimize deterioration.

14.0 INSPECTION, TEST, AND OPERATING STATUS

This basic requirement is applicable to the scope of work.

15.0 CONTROL OF NONCONFORMING ITEMS

Task 1
Capsules will be uniquely identified, electron beam sealed in a vacuum, visually and microscopically inspected, helium leak tested, and bubble tested. Fabricated capsules not passing any inspection attribute will be removed from the population and discarded.

The procedure PNL-ASO-079, *ASO Occurrences, Deficiencies and Nonconformances* will be followed. If any issues arise, an Occurrence Report form will be used to document the issue and if evaluated as a significant condition adverse to quality, elevated to a Nonconformance or Deficiency Report as appropriate and corrective and preventive actions tracked to conclusion. Refer to the Corrective Action section below.

Task 2
This basic requirement is applicable to the scope of work at the point when the capsules are delivered to RPL from INL as discussed in Section 13 above. If any issues arise, an Occurrence Report form will be used to document the issue and if evaluated as a significant condition adverse to quality, elevated to a Nonconformance or Deficiency Report as appropriate and corrective and preventive actions tracked to conclusion. Refer to the Corrective Action section below.

16.0 CORRECTIVE ACTION

The corrective action process for documenting issues of concern and determining if significant conditions adverse to quality exist will be controlled via the use of Occurrence Report forms, Deficiency Report forms and Nonconformance Report forms as applicable conclusion as per the procedure, RPL-ASO-079, *ASO Occurrences, Deficiencies and Nonconformances*. The Project Manager and QE will evaluate occurrences for conditions adverse to quality and to determine if the occurrence is a deficiency or nonconformance. Deficiencies and nonconformances are tracked by the project QE to closure and submitted to records. When necessary the ASO Project Manager will inform the INL POC of any concerns or conditions**.**

17.0 QUALITY ASSURANCE RECORDS

17.1 Analytical Support Operations (ASO) staff comply with the HDI work flows, *File and Maintain Project Records and Manage Project Records* and the ASO procedure, PNL-ASO-076, *ASO Records Management*. The Project Manager defines project records and QA records to be maintained in the File Plan which functions as an index to the record file. PNNL has a Records Management system to electronically manage project records. A project Share Drive location is used to store .pdf copies of procedures reviewed and approved test plans and instructions, as well as completed plans and instructions, data packages, and reports.

17.2 For Task 1 the completed TI and attachments (supporting documentation: capsule and wire dimensions, and weights, material certifications, inspection, helium leak and bubble test results and the Certification of Conformance are the records to be submitted to ASO records.

17.3 For Task 2 the data summary report when applicable, will reference any deficiency, nonconformance or occurrence reports, any audit and surveillance reports, the data generated by measuring instruments. Final data reports will be prepared and will have a cover page, a narrative and a data summary. This report and the system files for each analysis will be submitted to ASO records.

17. 4 Final data reports shall be reviewed by the Project Manager and the project QE prior to submittal to the client. This review is to verify that the records are legible and traceable to project deliverables and that project objectives have been met.

18.0 AUDITS

Surveillances will be performed as requested by the Project Manager and when work-in-progress is evaluated against procedures. When completed tests and data packages and project reports are reviewed, a QE Memorandum will be prepared.

Test Instruction for
Preparation of Fluence Wire Capsules - Project 77150

Revision 0

Effective Date: Upon last signature

Author:	Lawrence R Greenwood — Digitally signed by Lawrence R Greenwood Date: 2020.07.20 08:43:35 -07'00'	Larry R. Greenwood, Project Manager	Date
Technical Reviewer:	Lori P. Darnell	Lori P. Darnell	7/20/20 Date
Quality Assurance Reviewer:	Samuel A. Adams — Digitally signed by Samuel A. Adams Date: 2020.07.20 14:57:42 -07'00'	Samuel A. Adams, Quality Engineer	Date

INTRODUCTION

Test instructions (TIs) are prepared to direct the specific dosimetry fabrication requirements required by the Idaho National Laboratory (INL) client. This TI provides the requirements for the fabrication of ten (eight for delivery with 2 spares) fluence capsules. All of the vanadium capsules contain Fe, Ti, Nb and 0.116% Co-Al wires. Each capsule has a unique identifier code. The laser-etched identification codes and the number of wires required is given on the sample loading sheets attached to this test instruction.

The following documents (current versions) are applicable to this activity:

ASO-QAP-001, Rev. 11, *Analytical Support Operations (ASO) QA Plan*
Quality Assurance Project Plan – 77150 (Rev. 0), Fluence Capsules for the SAM-2 Experiment
PNL-ASO-052, Rev. 3, *Balance Performance Checks*
RPL-OP-001, Rev. 15, *Routine Research Operations*
TI-FAB-77150, Rev. 0, *Tests and Inspections for Reactor Dosimetry Capsules- Project 77150*

PREREQUISITES

Training

Fabricators and the Technical Reviewers must be familiar with the process of inspecting capsules, acceptance testing, and all steps required by this procedure. Technicians will be trained in capsule fabrication and acceptance testing if needed through mentoring. Because this TI is completed as it is performed; no training to the TI is needed.

Data Recording Forms

Data forms will be used to record data. The data forms for this activity are attached.

No fields are to be incomplete when used. Upon completion, data forms require completeness reviews by the Fabricator, as well as technical and quality reviews. Both Fabricators and technical reviewers must sign and date forms before they can be deemed to be complete. The quality review is performed in a review of all the fabrication records for manufacturing, assembling, examination, and inspection and documented in a surveillance report.

MEASUREMENT AND TEST EQUIPMENT

Calibration

Equipment used in fabrication of dosimeter capsules is listed in Table 1. Unique IDs of capsules and calibration status of M&TE used for acceptance testing will be recorded before use. Fabrication equipment requiring prior calibration for wires includes calipers.

Only accepted materials are available to the Fabricator. Project inspectors will use calibrated measuring devices when applicable.

Fabrication staff will ensure that only the correct materials are available for use.

This TI will result in a handoff of materials to the next follow-on process for capsule sealing/closure and final tests and inspections (TI-FAB-77150, Rev. 0).

Required Materials

The materials specified in Table 1 are applicable to this TI:

Table1. Wires required for Project 77150

Vanadium Capsules	Quantity	Description
The capsules shall be made of pure vanadium (99.8% minimum V): Tube with an overall capsule size of outer diameter (OD) = 0.047 inches to 0.054 inches and 0.420-inch maximum length.	Quantity = At least 10 (8 for delivery + 2 spares, one of each type) to ensure that 8 are available for shipping	1. 99.99% minimum purity iron (Fe) wire (MRC Y2Y-444-07448) 2. 99.92% minimum purity titanium (Ti) wire (RE 614) 3. Niobium (Nb) wire; Reference Material 526C from the Institute for Reference Materials and Measurements (IRMM) 4. Cobalt (Co)-Aluminum (Al) alloy wire: 0.116% Co-Al wire SRM-953 from the National Institute of Standards and Technology (NIST)
Total	**10**	

Additional fabrication materials include: Calibrated balances, cotton or plastic gloves and ethanol or acetone

Post-sealing inspection supplies include:

Plastic bottles/jars labeled with the type of monitor being fabricated.
Microscope with at least 15X magnification
Inspection block
Calibrated caliper or micrometer

Table 1. Measuring and Test Equipment for Fabrication

Manufacturer	Model Number	Description	Serial Number (if available)	Property Number (if available)	Location	Calibration Required?	Frequency of Required Calibration
Thermo	Orion Cahn C-31	Analytical Balance	75242	WD 24315	RPL, 55	Yes, annual by metrology and verification check with use	Annual, daily verification 200 g mass weight
Thermo	Orion Cahn C-33	Analytical Balance	77843	WD 55353	RPL, 514	Yes, annual by metrology and verification check with use	Annual, daily verification 200 g mass weight
Mitutoyo	CD-6"-ASX	Digital Slide Caliper	13357600	NA	RPL, 514	Yes, annual by metrology	Annual
Mitutoyo	CD-6"-ASX	Digital Slide Caliper	084599791	NA	RPL, 514	Yes, annual by metrology	Annual

WORK INSTRUCTIONS:

Each step will be **initialed and dated as completed**. Required forms are attached. The TI requires a completeness review by the Fabricator (Step 17), and technical and quality review when complete.

Initial	Date	
LPD	7/27/20	1. Locate and identify all materials as specified above in order to fabricate the fluence capsules. See the requirements and specifications table above for selection of vanadium capsule lengths. Capsules containing Fe, Nb, Ti, and 0.1% Co-Al wires should be up to 0.420" in length. If there are any questions or concerns, direct these to the Project Manager.
LPD AMM	7/27/20 7.27.20	2. Inspect the vanadium capsules to make sure that the capsules are free of any defects and have a unique ID stamp on the bottom or side of the capsule.
LPD AMM	7/27/20 7.27.20	3. Clean the capsules in acetone or ethanol using a plunger wire to make sure that the capsules do not contain any extraneous material from machining. Allow the capsules to dry before further use. AMM 8-11-20
LPD AMM	7/28/20 8-11-20	4. Select vanadium capsules according to the instructions in the table above. Weigh each capsule and place the capsules into an aluminum block with numbered holes and record the unique ID number on the prepared weighing form.

Initial	Date	
LPD	7/28/20	5. Cut sections of the selected fluence wire in varying lengths according to the direction of the Project Manager, ensuring that the wire lengths are at least 1/16".
LPD LPD LPD LPD LPD AMM	7/28/20 8/3/20 8/4/20 8/5/20 8/10/20 8/11/20	6. Ensure that the performance of the balance needed to weight wires in the next step has been verified by performing the daily calibration performance check as per the procedure, PNL-ASO-052, Rev. 3, *Balance Performance Checks* Was the daily balance performance check acceptable? (Yes) or No (Circle one)

Initial	Date	
LPD AMM	8/4/20 8.11.20	7. Weigh each wire and record the weight on the weighing form. Place the wire inside the vanadium capsule.
LPD	8/5/20	8. Cut approximately 1/16" length sections of vanadium wire for all of the capsules or select prepared wire segments to plug the open end of the vanadium capsule.
LPD AMM	8/5/20 8.5.20	9. Weigh the vanadium wires and record the weights on the weighing form. AMM 8.11.20
LPD AMM	8/5/20 8.5.20	10. Push the vanadium wire into the top of each capsule leaving about 1/64" at the top for the sealing. The wires should fit snugly so they will stay in place for transport to the sealer. Crimp in place using small pliers. AMM 8.11.20
LPD AMM	8/10/20 8.11.20	11. Weigh the final, loaded capsule and record the weight on the weighing sheet. Compare the final loaded weight with the sum of the weights of the individual components. If the difference is outside the expected tolerance range, unload the capsule and reweigh all the components to ensure that the weights agree. **Note:** If there are any questions regarding the required tolerance range(s), obtain this information before completing this step.
LPD	8/20/20	12. Once all of the capsules have been prepared, load them into the copper chill blocks leaving about 1/32" exposed for the sealing. Record the unique chill block number on the weighing sheets so that the capsule identifications will be maintained.
LPD	8/20/20	13. Notify the Project Manager that the capsules have been prepared and are ready for sealing and submit the weighing sheets and a copy of the balance log to the project manager for approval prior to sealing.
LPD LPD	9/8/20 9/18/20	14. After sealing, perform the tests and inspections as specified in TI-FAB-001, current revision. On completion of the tests, submit the test and inspection records to the Project Manager for review and approval.

LPD	9/18/20	15. Prepare plastic pill vials and labels with the unique capsule identifications and final, sealed weights. Insert the capsules into the vials and notify the Project Manager that the capsules are ready for shipment to the client.
LPD	9/21/20	16. Review the completed TI to ensure that it is complete, legible, and that error corrections were corrected using a single line-out that was initialed and dated, and that if warranted, that an explanation for error corrections is present.
LRL	9/21/20	17. The Project Manager will perform a technical review of the data and then arrange for the Quality Engineer assigned to this project to review all project records to ensure compliance with the QA Project Plan and Memorandum Purchase Order (MPO) and Statement of Work (SOW).

Quality Control

Quality control requirements are addressed by using calibrated M&TE within the specified calibration interval and ensuring that technical and Quality Engineer reviews of data are performed and that all identified issues are resolved. Reported data must be traceable to supporting data.

Records

Records include the weighing form, a copy of the Balance Performance Check Log form for the time interval the balance was used for this work, and a copy of the balance Calibration Certificate from the supplier providing RPL balance calibration services to demonstrate that the balance was within its calibration interval.

Capsule ID	Preparation and Weight Sheet for Capsules Containing Fe, Ti, and Nb wires								
	Balance SN: 77842 73 LRL 9-22-20								
	Last Calibration Date: 8/20/2019				Next Calibration Due: 8/20/2020				
	A Vanadium	B Fe	C Ti	D Nb	E 0.116%CoAl	F Vanadium	G = Sum A to E	H	Absolute Value of H-G
	Capsule Weight (mg) Step 4	Wire Weight (mg) Step 7	Wire Weight (mg) Step 7	Wire Weight (mg) Step 7	Wire Weight (mg) Step 7	Wire Weight (mg) Step 9	Sum of Previous six columns (mg)	Final Load Capsule Weight (mg) Step 11	Compare Calculated Sum Weights to Final Loaded Capsule weights Agreement within tolerance? (0.1 mg) Y/N Step 11
A7	40.824	2.302	1.207	2.252	0.722	2.977	50.284	50.253	0.031, Yes, Short Cap
T2	39.958	2.149	1.414	1.829	0.808	3.055	49.213	49.207	0.006, Yes, Short Cap
Y1	40.163	2.065	1.153	2.242	0.813	2.280	48.716	48.726	-0.010, Yes, Short Cap
1F	40.297	2.516	1.195	2.165	0.695	2.848	49.680	49.718	-0.038, Yes
5B	42.256	2.013	1.221	1.902	0.702	2.832	50.926	50.920	0.006, Yes
97	39.420	2.613	1.264	1.863	0.830	2.533	48.523	48.492	0.031, Yes
5D	43.558	2.136	1.299	2.069	0.830	2.854	52.746	52.720	0.026, Yes
B5	41.147	2.406	1.253	1.801	0.712	2.680	49.999	50.003	-0.004, Yes
9J	38.378	2.610	1.191	2.281	0.863	3.035	48.358	48.302	0.056, Yes
A5	40.991	2.247	1.349	2.053	0.709	3.536	50.885	50.879	0.006, Yes
9C	42.525	2.010	1.579	1.827	0.705	3.609	52.255	52.205	0.050, Yes
5U	41.161	2.599	1.246	2.252	0.736	3.228	51.222	51.233	-0.011, Yes
1I	41.956	2.144	1.187	1.928	0.708	3.036	50.959	50.956	0.003, Yes
V1	41.727	2.003	1.133	1.844	0.728	2.555	49.990	49.978	0.012, Yes
X2	41.242	2.043	1.603	2.168	0.745	2.664	50.465	50.431	0.034, Yes

Analyst: Lori P. Darnell Date: 9-22-20

Technical Reviewer: LRLeeuwen Date: 9-22-20

QE Reviewer: Samuel A. Adams Digitally signed by Samuel A. Adams Date: 2020.09.23 15:31:13 -07'00' Date: _____

	Preparation and Weight Sheet for Capsules Containing Fe, Ti, and Nb wires								
	Balance SN: 77843								
	Last Calibration Date: 8/20/2019				Next Calibration Due: 8/20/2020				
Capsule ID	A Vanadium	B Fe	C Ti	D Nb	E 0.116%CoAl	F Vanadium	G = Sum A to E	H	Absolute Value of H-G
	Capsule Weight (mg) Step 4	Wire Weight (mg) Step 7	Wire Weight (mg) Step 7	Wire Weight (mg) Step 7	Wire Weight (mg) Step 7	Wire Weight (mg) Step 9	Sum of Previous six columns (mg)	Final Load Capsule Weight (mg) Step 11	Compare Calculated Sum Weights to Final Loaded Capsule weights Agreement within tolerance? (0.1 mg) Y/N Step 11
77	42.284	1.918	1.168	2.514	0.675	3.004	51.563	51.587	-0.024, Yes
61	40.169	1.878	1.099	2.477	0.651	2.559	48.833	48.787	0.046, Yes
7F	39.888	1.866	1.085	2.388	0.637	2.760	48.624	48.645	-0.021, Yes
A1	41.782	2.053	1.113	2.817	0.853	3.052	51.670	51.660	0.010, Yes
O5	40.946	2.151	1.088	2.439	0.651	2.900	50.175	50.156	0.019, Yes

Analyst: Lori P. Darnell Date: 9-22-20

Technical Reviewer: LR Steward Date: 9-22-20

QE Reviewer: Samuel A. Adams Digitally signed by Samuel A. Adams Date: 2020.09.23 15:31:52 -07'00' Date: _____

0.375

Preparation and Weight Sheet for Capsules Containing Fe, Ti, and Nb wires + Co-Al LRT 8/18/20

Balance SN: 77843

Last Calibration Date: 8-20-19 Next Calibration Due: 8/20/20 → 8/18/2021

	Capsule ID	A Vanadium	B Fe	C Ti	D Nb	E 0.116%CoAl	F Vanadium	G = Sum A to E	H	Absolute Value of H-G
		Capsule Weight (mg) Step 4	Wire Weight (mg) Step 7	Wire Weight (mg) Step 7	Wire Weight (mg) Step 7	Wire Weight (mg) Step 7	Wire Weight (mg) Step 9	Sum of Previous six columns (mg)	Final Load Capsule Weight (mg) Step 11	Compare Calculated Sum Weights to Final Loaded Capsule weights Agreement within tolerance? (0.1 mg) Y/N Step 11
1	A7	40.824	2.302	1.207	2.252	0.722	2.977	50.284	50.253	.031, yes, Short cap
2	T2	39.958	2.149	1.414	1.829	0.808	3.055	49.213	49.207	.006, yes, Short cap
3	Y1	40.163	2.065	1.153	2.242	0.813	2.280	48.716	48.726	-.010, yes, Short cap
4	1F	40.297	2.516	1.195	2.165	0.695	2.848	49.680	49.718	-.038, yes
5	5B	42.256	2.013	1.221	1.902	0.702	2.832	50.926	50.920	.006, yes
6	97	39.420	2.613	1.264	1.863	0.830	2.533	48.523	48.492	.031, yes
7	5D	43.558	2.136	1.299	2.069	0.830	2.854	52.746	52.720	.026, yes
8	B5	41.147	2.406	1.253	1.801	0.712	2.680	49.999	50.003	-.004, yes
9	9J	38.378	2.610	1.191	2.281	0.863	3.035	48.358	48.302	.056, yes
10	A5	40.991	2.247	1.349	2.053	0.709	3.536	50.885	50.879	.006, yes
11	9C	42.525	2.010	1.579	1.827	0.705	3.609	52.255	52.205	.050, yes
12	5U	41.161	2.599	1.246	2.252	0.736	3.228	51.222	51.233	-.011, yes
13	11	41.956	2.144	1.187	1.928	0.708	3.036	50.959	50.956	.003, yes
14	V1	41.727	2.003	1.133	1.844	0.728	2.555	49.990	49.978	.012, yes
15	X2	41.242	2.043	1.603	2.168	0.745	2.664	50.465	50.431	.034, yes

Analyst: Lori L. Darrell Date: 8/18/20 8.20.20

Technical Reviewer: [signature] Date:

QE Reviewer: Samuel A. Adams Digitally signed by Samuel A. Adams Date: 2020.09.23 15:33:23 -07'00' Date:

~~0.375~~ AMM 8.11.20

Preparation and Weight Sheet for Capsules Containing Fe, Ti, and Nb wires + Co Al AMM 8-18-20

Balance SN: 77843

Last Calibration Date: 8/20/19 → 8/18/20 Next Calibration Due: 8/20/20 + 8/18/21

	Capsule ID	A Vanadium	B Fe	C Ti	D Nb	E 0.116%CoAl	F Vanadium	G = Sum A to E	H	Absolute Value of H-G
		Capsule Weight (mg) Step 4	Wire Weight (mg) Step 7	Wire Weight (mg) Step 7	Wire Weight (mg) Step 7	Wire Weight (mg) Step 7	Wire Weight (mg) Step 9	Sum of Previous six columns (mg) 51.563 8.11.20	Final Load Capsule Weight (mg) Step 11	Compare Calculated Sum Weights to Final Loaded Capsule weights Agreement within tolerance? (0.1 mg) Y/N Step 11
16	77	42.284	1.918	1.168	2.514	0.675	3.004	~~51.563~~ AMM	51.587	-0.024 Y
17	61	40.169	1.878	1.099	2.477	0.651	2.559	48.833	~~48.887~~	0.046 Y
~~18~~	7F	39.888	1.866	~~1.189~~	2.388	0.637	12.760	48.624	48.645	-0.021 Y
19	A1	41.782	2.053	1.113	2.817	0.853	3.052	51.670	51.660	0.010 Y
20	D5	40.946	2.151	1.088	2.439	0.651	2.900	50.175	50.156	0.019 Y
									~~48.787~~	
AMM 8.11.20				1.085 AMM 8.11.20					48.787 AMM 8.11.20	

Analyst: L.P. Darnell Date: 8.11.20 8-11-20

Technical Reviewer: _____ Date: _____

QE Reviewer: Samuel A. Adams Digitally signed by Samuel A. Adams Date: 2020.09.23 15:33:58 -07'00' Date: _____

Tests and Inspections for Reactor Dosimetry Capsules - Project 77150

Effective Date: Upon last signature

Approvals:

Project Manager:	Lawrence R Greenwood Digitally signed by Lawrence R Greenwood Date: 2020.07.20 08:42:36 -07'00'	
	Larry R. Greenwood	Date
Technical Reviewer:	Lori P. Darnell	7/20/20
	Lori P. Darnell	Date
Quality Engineer:	Samuel A. Adams Digitally signed by Samuel A. Adams Date: 2020.07.20 14:59:33 -07'00'	
	Samuel A. Adams	Date

Hazard Mitigation

The fluence capsules when fabricated are not irradiated and capsules are inspected after the sealing process is completed. Therefore, there are no hazards other than work with boiling water, which does not require special mitigation.

Work Instructions

The electron beam sealing system in the Applied Process Engineering Laboratory (APEL) or the laser sealing system in building 3410 will be used to seal fluence capsules for RPL projects. Inspection approaches to ensure that capsules pass project-specified requirements are addressed in this procedure. Attachment 1 is the example sealing request, sealing request completion, and sealing acceptance forms.

Visual Microscopic Inspections

Prior to initiation of work and following capsule sealing, all capsules are visually inspected under a microscope with at least 15X magnification. The purpose of the inspection is to (1) verify the capsule ID code stamped on the bottom, (2) ensure the absence of any visible flaws or cracks, especially after sealing, and (3) assess the general condition of the capsule seal (e.g., size, penetration).

Dimensional Checks

The length and outside diameter of each capsule is measured with a micrometer, calipers or dimensional gauge to ensure that the capsules meet the client's specifications. The measurements are conducted after final sealing since the sealing process may lead to some dimensional changes.

Weighing

The capsules are weighed on calibrated balances to at least three (3) significant figures at several points during the fabrication process including the initial empty weight, the weight with the wires loaded, and the final weight after sealing. Balance performance checks are conducted and documented prior to each weighing. The weight check must be within the tolerance specified for the assigned mass weight on the Balance Performance Check Log form. The weight sheets include the M&TE number of the balance and calibration interval (Date last calibrated / next calibration due) for traceability. Capsules that show an unusually high weight loss on sealing are rejected.

RPL balances are calibrated at least annually by Quality Control Services, Inc. (Portland, OR) a PNNL evaluated supplier. Balance calibration certificates are received and maintained as records and calibration stickers showing the valid calibration interval are placed on each unit.

Helium Leak Testing

Helium leak testing is performed using a Pfeiffer helium leak detector using procedure, RPL-HE-001, Rev. 0, *Dosimetry Capsule Helium Leak Testing*. Prior to measurements, the leak detector is calibrated with a helium leak standard. Prior to testing, the capsules are placed in a chamber, which is then evacuated, and backfilled with helium gas at a pressure of about 1500 Torr for at least 18 hours. [NOTE: If the capsules are sealed with a laser in a helium filled glovebox, then helium soaking is not required prior to leak testing.] The chamber is then evacuated and connected to the helium leak detector. Helium outgassing measurements are then performed and compared directly to the helium leak standard. Typical results indicate an outgassing rate for the capsules of $< 1 \times 10^{-8}$ atm-cc/second. The requirement is that the helium leak test for the capsules is not to exceed 1.0E-07 std cc/sec. A helium leak testing report is prepared to document the testing and results and must have the he leak calibration information attached.

- Completed Chain of Custody and Sealing Request and Completion form
- Inspection Worksheet & Sealing Acceptance Summary Worksheet
- Inspection Worksheet Project 77150 (INL) Fluence Capsules
- He Leak Test report
- He Leak Calibration documentation

Attachment 1

Chain of Custody for Capsule Sealing Page 1 of 1

I. Sealing Request

Number and list of capsules to be sealed: 20
A7, T2, Y1, 1F, 5B, 97, 5D, B5, 9J, A5, 9C, 5U, 1I, V1, K2, T1, 61, 7F, A1, 05

Date:	Time:	Relinquished by:	Date:	Time:	Received by:
8/20/20	11:45	Lori P. Darnell	20 AUG 2020	11:45	
Date:	Time:	Relinquished by:	Date:	Time:	Received by:
20 AUG 2020	10:00		08/21/20	10:10	Lori P. Darnell
Date:	Time:	Relinquished by:	Date:	Time:	Received by:

II. Sealing Request Completion

Number of capsules sealed: 20

The sealing was performed by _____ on 25 AUG 2020
Signature Date

Distribution: Larry Greenwood, Project Manager/ Project File; Project QE

Attachment 2
INSPECTION WORKSHEET & SEALING ACCEPTANCE SUMMARY WORKSHEET

Capsules Inspected:

Length Specification:

Maximum of 0.420"

What method(s) was/were used?

Test	Date Performed	Analyst	Criteria	Summary of Results
Visual microscopic inspection	7/28/20 9/8/20	LPD LPD	Verify unique capsule ID code Verify absence of visual flaws	100/100 % Pass 0 % Fail
Dimensional Checks	9/18/20	LPD	O.D. 0.047-0.054" Length: Max 0.420"	100 % Pass 0 % Fail

Distribution: Larry Greenwood, Project Manager/ Project File; Project QE

9 LRH 9-22-20

Inspection Worksheet Project 77150 (INL) Fluence Capsules

Visual Microscopic Inspection				**Dimensional Check**			**Weight Check**			**Other Tests**	
Microscope SN: N674872				Caliper/Micrometer SN: 13357600			Balance SN: 77843			Helium Leak: ≤1.0E-7 std cc/second	
Fluence Wires: Fe, Ti, Nb, and Co-Al				Last Calibration Date: 1/24/2020 Next Calibration Due: 1/24/2021			Last Calibration Date: 8/18/2020 Next Calibration Due: 8/18/2021			Bubble Test: Steady stream, or growing	
Capsule ID Code and wire type	**Verified ? Y/N**	**Absence of flaws? Y/N**	**General Obs: seal size, penetration**	**Length Max 0.420 inches**	**Outside Diameter 0.047 - 0.054 inches**	**Meets Specs: (see above) Y/N**	**Weight with Dosimeters Loaded (mg)**	**Final Weight after Sealing (mg)**	**Acceptable? (< 0.3 mg) Y/N**	**Helium Leak Test OK? Y/N**	**Bubble Test OK? Y/N**
Analyst /Date:	LPD 7/28/20	LPD 7/28/20	LPD 7/28/20	LPD 9/18/20	LPD 9/18/20	LPD 9/18/20	LPD 8/18/20	LPD 9/18/20	LPD 9/18/20	AC 9/2/20	LPD 9/8/20
A7	Yes	Yes	Good	.3820	.0490	YES	50.253	50.108	.145 YES	YES	YES
T2	Yes	Yes	Good	.3845	.0505	YES	49.207	49.030	.177 YES	YES	YES
Y1	Yes	Yes	Good	.3735	.0530	YES	48.726	48.581	.145 YES	YES	YES
1F	Yes	Yes	Good	.3890	.0570	YES	49.718	49.619	.099 YES	YES	YES
5B	Yes	Yes	Good	.3920	.0495	YES	50.920	50.799	.121 YES	YES	YES
97	Yes	Yes	Good	.3890	.0505	YES	48.492	48.352	.140 YES	YES	YES
5D	Yes	Yes	Good	.3930	.0510	YES	52.720	52.472	.248 YES	YES	YES
B5	Yes	Yes	Good	.3755	.0500	YES	50.003	49.933	.070 YES	YES	YES
9J	Yes	Yes	Good	.3870	.0505	YES	48.302	48.197	.105 YES	YES	YES
A5	Yes	Yes	Good	.3880	.0500	YES	50.879	50.630	.249 YES	YES	YES
9C	Yes	Yes	Good	.3915	.0495	YES	52.205	52.120	.085 YES	YES	YES
5U	Yes	Yes	Good	.3950	.0500	YES	51.233	51.054	.179 YES	YES	YES
1I	Yes	Yes	Good	.3910	.0490	YES	50.956	50.863	.093 YES	YES	YES
V1	Yes	Yes	Good	.3945	.0500	YES	49.978	49.782	.196 YES	YES	Fail
X2	Yes	Yes	Good	.3930	.0500	YES	50.431	50.330	.101 YES	YES	YES

Analyst: Lori P. Darnell Date: 9-22-20

Technical Reviewer: LR Leonard Date: 9-22-20

QE Reviewer: Samuel A. Adams Digitally signed by Samuel A. Adams Date: 2020.09.23 15:34:58 -07'00' Date: _____

9 Cont, LRD 9-22-20

Inspection Worksheet Project 77150 (INL) Fluence Capsules

Visual Microscopic Inspection				**Dimensional Check**			**Weight Check**			**Other Tests**	
Microscope SN: N674872				Caliper/Micrometer SN: 13357600			Balance SN: 77843			Helium Leak: ≤1.0E-7 std cc/second	
Fluence Wires: Fe, Ti, Nb, and Co-Al				Last Calibration Date: 1/24/2020 Next Calibration Due: 1/24/2021			Last Calibration Date: 8/18/2020 Next Calibration Due: 8/18/2021			Bubble Test: Steady stream, or growing	
Capsule ID Code and wire type	**Verified ? Y/N**	**Absence of flaws? Y/N**	**General Obs: seal size, penetration**	**Length Max 0.420 inches**	**Outside Diameter 0.047 - 0.054 inches**	**Meets Specs: (see above) Y/N**	**Weight with Dosimeters Loaded (mg)**	**Final Weight after Sealing (mg)**	**Acceptable? (< 0.3 mg) Y/N**	**Helium Leak Test OK? Y/N**	**Bubble Test OK? Y/N**
Analyst /Date:	LPD 7/28/20	LPD 7/28/20	LPD 7/28/20	LPD 9/18/20	LPD 9/18/20	LPD 9/18/20	AMM 8/11/20	LPD 9/18/20	LPD 9/18/20	AC 9/2/20	LPD 9/8/20
77	Yes	Yes	Good	.3875	.0490	YES	51.587	51.485	.102 YES	YES	YES
61	Yes	Yes	Good	.3865	.0495	YES	48.787	48.654	.133 YES	YES	FAIL
7F	Yes	Yes	Good	.3840	.0495	YES	48.645	48.429	.216 YES	YES	YES
A1	Yes	Yes	Good	.3750	.0515	YES	51.660	51.604	.056 YES	YES	YES
O5	Yes	Yes	Good	.3825	.0495	YES	50.156	50.061	.095 YES	YES	YES

Analyst: Lori P. Darnell Date: 9-22-20

QE Reviewer: Samuel A. Adams Digitally signed by Samuel A. Adams Date: 2020.09.23 15:35:39 -07'00' Date: _____

Technical Reviewer: LR Greenwood Date: 9-22-20

Inspection Worksheet Project 75643 (INL) Fluence Capsules

Visual Microscopic Inspection				**Dimensional Check**			**Weight Check**			**Other Tests**	
Microscope SN: N674872				Caliper/Micrometer SN: 13357608			Balance SN: 77843			Helium Leak: ≤1.0E-7 std cc/second	
Fluence Wires: Fe, Ti, Nb, and Co-Al				Last Calibration Date: 1-24-2020 Next Calibration Due: 1-24-2021			Last Calibration Date: 8/18/2020 Next Calibration Due: 8/18/2021			Bubble Test: Steady stream, or growing	
Capsule ID Code and wire type	**Verified ? Y/N**	**Absence of flaws? Y/N**	**General Obs: seal size, penetration**	**Length Max 0.420 inches**	**Outside Diameter 0.047 - 0.054 inches**	**Meets Specs: (see above) Y/N**	**Weight with Dosimeters Loaded (mg)**	**Final Weight after Sealing (mg)**	**Acceptable? (< 0.3 mg) Y/N**	**Helium Leak Test OK? Y/N**	**Bubble Test OK? Y/N**
Analyst /Date:	~~LPD~~ 7/28/20	~~LPD~~ 7/28/20	~~LPD~~ 7/28/20	~~LPD~~ 9/18/20	~~LPD~~ 9/18/20	~~LPD~~ 9/18/20	~~LPD~~ 8/18/20	~~LPD~~ 9/18/20	~~LPD~~ 9/18/20	AC yes	~~LPD~~ 9/8/20
A7	y	y	Good	.3820	.0490	yes	50.253	50.108	.145 yes	yes	yes
T2	y	y	Good	.3845	.0505	yes	49.207	49.030	.177 yes	yes	yes
Y1	y	y	Good	.3735	.0530	yes	48.726	48.581	.145 yes	yes	yes
1F	y	y	Good	.3890	.0510	yes	49.718	49.619	.099 yes	yes	yes
5B	y	y	Good	.3920	.0495	yes	50.920	50.799	.121 yes	yes	yes
97	y	y	Good	.3890	.0505	yes	48.492	48.352	.140 yes	yes	yes
5D	y	y	Good	.3930	.0510	yes	52.720	52.472	.248 yes	yes	yes
B5	y	y	Good	.3755	.0500	yes	50.003	49.933	.070 yes	yes	yes
9J	y	y	Good	.3870	.0505	yes	48.302	48.197	.105 yes	yes	yes
A5	y	y	Good	.3880	.0500	yes	50.879	50.630	.249 yes	yes	yes
9C	y	y	Good	.3915	.0495	yes	53.205	52.120	.085 yes	yes	yes
5U	y	y	Good	.3950	.0500	yes	51.233	51.054	.179 yes	yes	yes
11	y	y	Good	.3910	.0490	yes	50.956	50.863	.093 yes	yes	yes
V1	y	y	Good	.3945*	.0500*	yes *	49.978 *	49.782 *	.196 yes *	yes *	Fail *
X2	y	y	Good	.3930	.0500	yes	50.431	50.330	.101 yes	yes	yes

- Short caps fail
- Fail *

Analyst: Lori P. Darnell Date: 9/18/20

QE Reviewer: Samuel A. Adams Digitally signed by Samuel A. Adams Date: 2020.09.23 15:36:20 -07'00' Date: _____

Technical Reviewer: LPDarnell Date: 9/21/20

Inspection Worksheet Project 77150 (INL) Fluence Capsules

Visual Microscopic Inspection				**Dimensional Check**			**Weight Check**			**Other Tests**	
Microscope SN: N674872				Caliper/Micrometer SN: 13357600			Balance SN: 77843			Helium Leak: ≤1.0E-7 std cc/second	
Fluence Wires: Fe, Ti, Nb, and Co-Al				Last Calibration Date: 1-24-2020 Next Calibration Due: 1-24-2021			Last Calibration Date: 8/18/2020 Next Calibration Due: 8/18/2021			Bubble Test: Steady stream, or growing	
Capsule ID Code and wire type	**Verified ? Y/N**	**Absence of flaws? Y/N**	**General Obs: seal size, penetration**	**Length Max 0.420 inches**	**Outside Diameter 0.047 - 0.054 inches**	**Meets Specs: (see above) Y/N**	**Weight with Dosimeters Loaded (mg)**	**Final Weight after Sealing (mg)**	**Acceptable? (< 0.3 mg) Y/N**	**Helium Leak Test OK? Y/N**	**Bubble Test OK? Y/N**
Analyst /Date:	LPD 7/28/20	LPD 7/28/20	LPD 7/28/20	LPD 9/18/20	LPD 9/18/20	LPD 9/18/20	8/11/20 AMM	LPD 9/18/20	LPD 9/18/20	AL	LPD 9/8/20
77	y	y	Good	.3875	.0490	yes	51.587	51.485	.102 yes	yes	yes
Fail * 61	y	y	Good	.3865 *	.0495 *	yes *	48.787	48.654 *	.133 yes *	yes *	Fail *
7F	y	y	Good	.3840	.0495	yes	48.645	48.429	.216 yes	yes	yes
A1	y	y	Good	.3750	.0515	yes	51.660	51.604	.056 yes	yes	yes
05	y	y	Good	.3825	.0495	yes	50.156	50.061	.095 yes	yes	yes

Analyst: Lori P. Darnell Date: 9/18/20

QE Reviewer: Samuel A. Adams Digitally signed by Samuel A. Adams Date: 2020.09.23 15:37:00 -07'00' Date: _____

Technical Reviewer: LPDarnell Date: 9/21/20

Balance Performance Check Log ✓ RAP 8/19/20

Pacific Northwest National Laboratory

Cahn C-33

Balance Information
Identificaiton (e.g. WD34567)
77843/WD55353
Location (e.g. Building/Room)
RPL / 514
Calibration Due Date
Aug-21

Check Weight (CW) Information	#1	CW Units	#2	CW Units	#3	CW Units	#4	CW Units
CW Identification	13060 200 mg							
CW Assigned Mass (+Units):	200.004	mg		g		g		
Method for Assigning Diff/Range[a]:	Option 1		Option 1		Option 1			
CW Difference ± Mass (+Units):								
Or Range Low Mass (+Units) &:	199.992	mg		g		g		g
Range High Mass (+Units):	200.016	mg		g		g		g

(a) Enter "Option #1" or "Option #2" from Procedure TTP-7-072, Table 1. For factors >3, append factor in parentheses, e.g., Option #1 (4)

Date	Lab Staff Initials	Check Weight ID or #	Check Weight Measured Mass (+Units)	Meets Acceptance Criterion (Within Diff/Range)? (Y/N)	Comments/Corrective Action
8/18/2020	RG	13060 200mg	200.000		Weights taken to establish assigned mass
8/18/2020	RG	13060 200 mg	200.012		
8/18/2020	RG	13060 200 mg	199.996		
8/18/2020	RG	13060 200 mg	200.004		
8/18/2020	RG	13060 200 mg	200.010		
9-10-20	LRL	11	200.007		
9/18/20	ZPD	11	200.001	yes	

> **Complete all column entries.**

> **For corrections, place single line through incorrect entry, enter correction and initial/date.**

> When full, forward to the TTP M&TE Custodian; replace with new Log.

> Following recalibration, lineout/ initial/date unused rows and forward to TTP M&TE Custodian; replace with new Log.

> To calibrate or recalibrate a balance contact the TTP M&TE Custodian.

> If additional check weights are used on balance, enter appropriate information on another sheet and append to this Log form.

Samuel A. Adams Digitally signed by Samuel A. Adams Date: 2020.09.23 15:38:01 -07'00'

Reviewer Signature _____ **Date** _____

Balance Performance Check Log

✓ ZnP 8/21/19

ic Northwest National Laboratory

Cahn C-33

Balance Information
Identificaiton (e.g. WD34567)
77843/WD55353
Location (e.g. Building/Room)
RPL / 514
Calibration Due Date
Aug-20

Check Weight (CW) Information	#1	CW Units	#2	CW Units	#3	CW Units	#4	CW Units
CW Identification	13060 200 mg							
CW Assigned Mass (+Units):	200.004	mg		g		g		
Method for Assigning Diff/Range[a]:	Option 1		Option 1		Option 1			
CW Difference ± Mass (+Units):								
Or Range Low Mass (+Units) &:	199.992	mg		g		g		g
Range High Mass (+Units):	200.016	mg		g		g		g

(a) Enter "Option #1" or "Option #2" from Procedure TTP-7-072, Table 1. For factors >3, append factor in parentheses, e.g., Option #1 (4)

Date	Lab Staff Initials	Check Weight ID or #	Check Weight Measured Mass (+Units)	Meets Acceptance Criterion (Within Diff/Range)? (Y/N)	Comments/Corrective Action
8/20/2019	RG	13060 200mg	200.011		Weights taken to establish assigned mass
8/20/2019	RG	13060 200 mg	199.992		
8/20/2019	RG	13060 200 mg	200.014		
8/20/2019	RG	13060 200 mg	200.002		
8/20/2019	RG	13060 200 mg	200.000		
9-17-19	LRH	"	200.006	Y	
24 Nov 19	BP	"	200.002	Y	
2dec.19	BP	"	200.001	Y	
7 JAN 20	LRH	"	200.009	Y	
1/20/20	LRH	"	200.000	Y	
1/21/20	LRH	"	199.998	Y	
1/29/20	LRH	"	200.0005	Y	
3/4/20	LRH	"	200.001	Y	
7/22/20	LRH	"	199.999	Y	
7/28/20	LRH	"	200.0004	Y	
8/3/20	LRH	"	200.0007	Y	
8/14/20	LRH	"	200.0004	Y	lined out ∅ LRH 8/18/20
				LRH	
8/15/20	LRH	"	200.0001	yes	
8/18/20	LRH	"	199.998	Y	
8.31.20	AMM	"	199.995	yes	

ZnP 9/9/20

Recalibrated 8/18/2020 -rg

1. 200.000
2. 200.012
3. 199.996
4. 200.004
5. 200.010

> **Complete all column entries.**

> **corrections, place single line through incorrect entry, enter correction and initial/date.**

> en full, forward to the TTP M&TE Custodian; replace with new Log

> Following recalibration, lineout/ initial/date unused rows and forward to TTP M&TE Custodian; replace with new Log.

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> If additional check weights are used on balance, enter appropriate information on another sheet and append to this Log form.

Reviewer Signature Karl N. Pool **Date** 8/19/20