TC-1933

ENGINEERING TEST PLAN FOR US/UK HIGHER ACTINIDES IRRADIATIONS TESTS

Hanford Engineering Development Laboratory

HANFORD ENGINEERING DEVELOPMENT LABORATORY
Operated by Westinghouse Hanford Company
P.O. Box 1970 Richland, WA 99352
A Subsidiary of Westinghouse Electric Corporation
Prepared for the U.S. Department of Energy
under Contract No. DE-AC14-76FF02170

DOE Richland, WA

Operated by
Westinghouse
Hanford Company
for the U.S. DOE

A Subsidiary of
Westinghouse Electric
Corporation

Contract No.
DE-AC14-76FF02170

P.O. Box 1970
Richland, WA 99352

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Legends removed
per S. Berglin
M. Kenney
by
M. Bostan
2/13/90

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Hanford Engineering Development Laboratory

J.A. Basmajian

March 1981

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ENGINEERING TEST PLAN

FOR

US/UK HIGHER ACTINIDES

IRRADIATIONS TESTS

by

J. A. Basmajian

March 1981

APPROVALS

J. A. Basmajian 3/6/81
J. A. Basmajian
Principal Engineer
Absorber & Ceramics Development

MB Parker 3-16-81
M. B. Parker, Manager
Absorber & Ceramics Development

R F Borisch 3/18/81
R. R. Borisch, Manager
Component Materials
Chairman - Design
Review Committee

W H Caplinger 3/17/81
W. H. Caplinger, Manager
Fuels Quality Engineering

E T Weber 3/23/81
E. T. Weber, Manager
Core Technology & Safety

HANFORD ENGINEERING DEVELOPMENT LABORATORY
Richland, Washington
Operated by
WESTINGHOUSE HANFORD COMPANY
a Subsidiary of Westinghouse Electric Corporation

UNITED STATES DEPARTMENT OF ENERGY UNDER CONTRACT DE-AC14-76FF02170

ENGINEERING TEST PLAN
US/UK HIGHER ACTINIDES IRRADIATIONS TEST

By
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March 1981

ACKNOWLEDGMENT

Portions of this document and some features of the test design represent contributions from Dr. S. Raman and H. L. Adair of the Oak Ridge National Laboratory.

CONTENTS

ENGINEERING TEST PLAN
US/UK HIGHER ACTINIDES
IRRADIATIONS TESTS

I. OBJECTIVE

The objective of the Higher Actinides Irradiations Program is to verify the neutronic and irradiation performance of americium and curium oxides in a fast reactor. The data obtained from the irradiation will be used to assess the basic neutronics parameters for actinide elements and determine the irradiation potential of the oxides of ^{241}Am and ^{244}Cm. This information has application in breeder reactor physics, fuel cycle analysis and assessment of waste management options.

The irradiation test program is a cooperative effort wherein the US is supplying the completed irradiation test pins, while the UK will perform the irradiation in their Prototype Fast Reactor (PFR).

Postirradiation examination and data analyses will be conducted on a cooperative basis, with some examinations performed in the UK and others in the US.

II. AUTHORITY

This irradiation program is covered under the US/UK Agreement, signed May 10, 1979, entitled Performance of Higher Actinides in Liquid Metal Cooled Breeder Reactors; henceforth, "Higher Actinides Agreement."

Resource for conduct of the program are provided under the Physics Budget and Reporting Catagory (B&R) #AF-15-40-10-4 of the Office of Reactor Research and Technology.

III. SCOPE

The irradiation experiment will be performed in the UK Prototype Fast Reactor (PFR) at Dounreay. There will be four pins irradiated in a demountable subassembly cluster (DMSA). Two pins will be identical, containing three actinide sections, a physics sample section and four dosimetry sections. One pin will contain three actinide sections and three dosimetry sections, and one pin will contain one physics sample section and two dosimetry sections. The expected data from the experiment will be used to quantitatively determine the amounts of actinide isotopes generated from specific precursors and actinide oxide materials irradiation behavior relevant to recycling of fissionable higher actinides in fast spectrum reactors.

The US effort will consist of fabrication of pellets and physics specimens, dosimetry, fabrication of the test pins, and the postirradiation analyses of dosimetry and physics samples. Postirradiation examination for actinide oxide pellet behavior will be performed in the UK using plans developed jointly by US and UK.

IV. SUMMARY DESCRIPTION OF THE EXPERIMENT

Four pins will be designed, fabricated and irradiated. The pins will be 0.230-inches in diameter and, in their completely assembled state, will be 88.75-inches long. The criteria for the design of the pins, and the operating conditions follow:

a. There will be four pins irradiated, and three pins will contain one sample of each of three compositions; namely,

$^{241}Am_2O_3$

$^{244}Cm_2O_3$

$(^{241}Am_6,\ ^{244}Cm,\ \overline{La}_7)_2O_3$

$\overline{La}$, is a mixture of stable Lanthanide elements of La, Ce, Nd, Sm in the mass ratio 1:2:3:1.

b. The actinide samples will be approximately 1-inch long.

c. There will be physics samples and dosimeters in the pins.

d. All pins will have the same axial sequence of actinide samples.

e. Design will ensure ease of postirradiation examination and maintenance of identification (unique numbers on all pin sections).

f. The cladding components of the pin will be D9 alloy.

g. Physics samples will be doubly encapsulated; their location in the physics sample section being defined by ORNL.

h. Design analysis will use best available estimates for heat generation, based on UK supplied flux data and fission cross sections from HEDL files.

i. Actinide oxide pellets are to have single encapsulation with a minimum cladding thickness of 0.030-inches.

j. The center temperature of actinide oxide pellets will not exceed 2200°C based on a one-sigma upper bound, at peak heat generation.

k. The center temperature of actinide oxide pellets will not be below 1000°C based on a best estimate calculation.

l. The center temperature of actinide oxide pellets will preferably exceed 1000°C based on a best estimate calculation.

m. Outer cladding temperature over actinide oxide pellet sections will be near 550°C.

n. Pins will be designed for 180 full power day (FPD) minimum time in PFR.

o. Physics specimens will be designed to operate at temperatures that keep the primary containment free from chemical attack and melting.

V. ORGANIZATIONAL RESPONSIBILITIES

1. Core Technology and Safety has overall programmatic responsibility for the US portion of the Higher Actinide Agreement. HEDL is specifically responsible for design, analyses, US/UK interfaces, and shipping.

2. Core Technology and Safety, with the assistance of Weld Engineering of Test Pin Fabrication is responsible for pin fabrication, fabrication data, maintaining archives and spare components.

3. Fuels Quality Engineering is responsible for review of the Engineering Test Plan, drawings, and Nonconformance Reports.

4. Fuels Quality Control is responsible for receiving inspection of components, final inspection, surveillance during HEDL fabrication and final release.

5. ORNL is responsible for oxide pellet fabrication, physics specimen fabrication and ORNL dosimetry fabrication. Post-irradiation analysis of the physics specimens will be ORNL's responsibility.

6. HEDL Irradiation Environment will be responsible for the HEDL dosimetry design, procurement, and postirradiation analysis of same.

7. The UKAEA will be responsible for the final pin assembly, placement in the demountable subassembly, irradiation, disassembly after irradiation and conducting the postirradiation examinations.

VI. FABRICATION AND ASSEMBLY SPECIFICATION

A. FABRICATION SPECIFICATIONS

The specifications necessary for the fabrication of the actinide oxide pellets and the pins are shown on the drawings and in Appendix A. The specification provided by ORNL for the physics specimens is given in Appendix D. Dosimetry specifications are presented in Appendix D.

B. ASSEMBLY PROCEDURES

The procedures for assembly of the pins, and the integration of the physics specimens and dosimeters into their respective encapsulation will be issued by Core Technology and Safety, with assistance from Weld Engineering.

The pins shall be made to conform to the dimensional requirements of Drawings H-3-46955 and H-3-4-6956.

C. SUBASSEMBLY LOADING

The assembly of the pins into the demountable subassembly shall be in accordance with detailed irradiation plans agreed to jointly by the US and UK as to location in the subassembly and location of the subassembly. All normal handling of the pins at PFR shall be done in accordance with UK procedures.

1. Actinide Pellets

Actinide pellets shall be produced by techniques which will meet the requirements of the specification in Appendix A.

2. Insulator Pellets

Insulator pellets should conform to the geometry and dimensions of Drawing H-3-46955 and shall be made of tungsten.

3. Metallic Components

All metallic components shall conform to Drawings H-3-46955 and H-3-46956. The metallic components shall be fabricated to meet applicable RDT or ASTM Standards.

a. Tubing

Cladding shall conform to the geometry and dimensions of Drawings H-3-46955 and H-3-46956 and applicable ordering data.

b. Bar Material (End Caps)

All solid pieces shall conform to the geometry and dimensions of Drawings H-3-46955 and H-3-46956 and shall meet the appropriate Specifications.

c. Plenum Spring

The plenum spring shall conform to the dimensions and specifications given on Drawings H-3-46955 and H-3-46956. It shall meet the appropriate Specification.

4. Supporting Procedures

Actinide test pin fabrication shall be in accordance with procedures provided by HEDL Core Technology and Safety.

All welds, unless otherwise specified, shall be made to the requirements of RDTF6-2T, Section 8, Category 10."

D. CODES AND STANDARDS

The codes and standards listed here are by reference a part of this Engineering Test Plan (ETP) and shall be applied in part or in its entirety as specified either in this ETP, Appendix C "Quality Assurance Index," on applicable drawings or in material specifications.

RDT-F2-2	Quality Assurance Program Requirements
RDT-F6-2T	Welding of Reactor Core Components and Test Assemblies
RDT-M-3-28T	Austenitic Stainless Steel and Superalloy Tubing for BRP Core Components.
ANSI/ASME N45.2-1977	Quality Assurance Program Requirements for Nuclear Facilities.

VII. CHARACTERIZATION REQUIREMENTS

Each Actinide Oxide Pellet Section, Physics Specimen Section, and each Dosimetry Section shall have the following information provided and recorded.

A. ACTINIDE OXIDE PELLET SECTION (H-3-46955, H-3-46956 and Appendix A)

1. Chemical composition
2. Isotopic composition
3. Column length $\pm$ 0.01-in. (0.025 cm)
4. Actinide oxide Wt $\pm$ 0.005 gm
5. Pin diameter (every 1/8-in. over pellets)

B. PHYSICS SPECIMENS AND DOSIMETRY PIN SECTION (H-3-46955 and H-3-46956 and Appendix D)

1. Chemical composition
2. Isotopic composition where applicable
3. Specimen weight $\pm$ 0.0001 gm
4. Isotopic weight $\pm$ 0.0001 gm
5. Specimen Identification Code

VIII. DATA PACKAGE REQUIREMENTS

HEDL will document the "as built" data and quality assurance records.

a. Certification that cladding tubing, and a piece of all other metal components, are kept in archives. Dosimetry, physics samples, etc., certifications will not be kept in archives. Actinide oxide pellets will be kept as archives with suitable records. Archives on physics samples are ORNL option.

b. Welding procedures, results of tests, and two representative samples of each type of weld with two inches of clad attached will be supplied to the UK.

c. Radiographs of each closure weld in 0°, 36°, 72°, 108° and 144° orientations will be supplied to the UK.

d. Helium leak check procedures, sensitivity and certification will be furnished to the UK.

e. Complete metrology records will be furnished to the UK.

f. Weights of the pins, less welds the UK makes, will be furnished to the UK.

g. One radiograph of each pin showing arrangement of all internal components will be furnished to the UK.

h. All Nonconformance Reports will be furnished to the UK.

i. Procurement specifications, inspection results and chemical analyses of all metallic sample components will be furnished to the UK.

j. Copies of the Chemical Analyses of all actinide and physics samples will be furnished to the UK.

IX. QUALITY ASSURANCE PROCEDURES

In accordance with established procedures, quality assurance will be provided. Procedures will be in accordance with Section QAI-1-3, dealing with development and testing programs, of the Westinghouse Hanford Quality Assurance Manual, WHAN-M-2. Those areas where quality assurance will be provided are checked on the attached QA Requirements Index (Appendix C). The quality assurance planning and audit data, quality assurance category, and hold points for the test items are detailed in Appendix C. Additionally, at least one informal and one formal design review will occur.

X. SHIPPING AND INSTALLATION

All pin components will be packaged and shipped in accordance with procedures developed by HEDL-ORNL-DOE. Installation in PFR at UK will be in accordance to their established procedures, which will be transmitted to HEDL. There will be special shipping and handling procedures made specifically to provide coverage for the isotopes involved. These procedures will be developed by HEDL-ORNL-DOE, and will supersede those areas in TC-256 where necessary.

XI. RECORDS

All data required by the Engineering Test Plan, the Quality Assurance procedures, and the procedures used in fabrication, assembly and irradiation shall be kept in controlled notebooks, follower cards, and appropriate file folders. These records will be retained by HEDL Core Technology and Safety in 326 Building until irradiations are complete and postirradiation analyses are final, or for 5 years. Copies of the pertinent information shall be available to appropriate persons at HEDL, ORNL, UK and DOE.

XII. ARCHIVE SAMPLE REQUIREMENTS

Archive samples shall be retained in HEDL archive repositories as indicated below:

Actinide Pellets

Three pellets of each actinide oxide will be retained in 327 Building storage.

Archive pellets should be handled in the same manner as pellets used in the test, and will be sealed in a tube in the same manner as and concurrent with fabrication of test pins.

Cladding

Two feet of cladding tubing will be retained in 326 Building.

Bar Stock

Six inches of bar stock will be retained in 326 Building.

Insulator Pellets

Two insulator pellets will be retained in 326 Building.

Plenum Springs

One plenum spring will be retained in 326 Building.

Miscellaneous

Six-inch pieces of all dosimetry tubes and physics specimen heat transfer tubing will be retained in 326 Building.

XIII. POSTIRRADIATION EXAMINATIONS

The pins shall, after removal from PFR, be nondestructively examined in the UK as follows:

1. Visual examination
2. Neutron radiography
3. Gross γ (gamma) scan
4. Eddy current trace

After these nondestructive examinations, the UK will disassemble the pins, ship the dosimetry and physics specimen sections to the US, and hence to HEDL or ORNL, for further destructive examinations.

The actinide pin sections will be examined in the UK in accordance with techniques jointly determined by US and UK. Detailed plans will be formalized prior to initiation of irradiation at PFR.

XIV. DOCUMENTATION

A. QUALITY RECORDS

1. Component inspection data and material certification and overchecks will be maintained by HEDL Core Technology and Safety for a period of five years after postirradiation analyses are complete.

2. Follower cards will be maintained by HEDL Core Technology and Safety.

3. Prior to irradiation, a fabrication data package will be published by HEDL Core Technology and Safety.

B. REPORTS

Reports describing the results and conclusions drawn from the irradiation test results will be issued by US and UK. It is expected that HEDL Irradiation Environment and ORNL will submit reports on dosimetry and physics specimen analyses, respectively. HEDL, ORNL, and UK will report on the actinide pin performance jointly.

APPENDIX A

US-UK HIGHER ACTINIDES IRRADIATION IN PFR

ACTINIDE OXIDE PELLET SPECIFICATIONS

US-UK HIGHER ACTINIDES IRRADIATION IN PFR
ACTINIDE OXIDE PELLET SPECIFICATIONS

1. COMPOSITION

1.1 CHEMICAL FORMULA

Pellet Type	Actinide Oxide
I	$^{244}Cm_2O_3$
II	$^{241}Am_2O_3$
III	M_2O_3

$M = {}^{241}Am_6\ {}^{244}Cm\ \overline{La}_7$

Where $\overline{La}$ corresponds to a mixture of stable lanthanide elements of La, Ce, Nd, Sm in the mass ratio 1:2:3:1.

1.2 FISSILE ACTINIDE CONTENT

1.2.1 For Types I and II pellet, the ratio of Fissile Actinides to Total Metal shall be greater than 85%.

1.2.2 For Type III pellets, the ratio of Fissile Actinides to Total Metal shall be $0.45\ ^{+0.05}_{-0.03}$.

1.3 ISOTOPIC CONTENT

The ^{244}Cm isotope shall constitute more than 85% of the curium in pellets for Type I and III. The ^{241}Am isotope shall constitute more than 85% of the americium in Types II and III pellets.

1.4 OXYGEN-TO-METAL RATIO

The O/M ratio shall be 1.50 ± .02.

1.4.1 O/M shall be determined by a thermal reduction procedure approved by HEDL.

1.4.2 O/M shall be maintained after measurement by handling and storing pellets in an inert gas atmosphere prior to final enclosure in fuel pin sections.

1.5 IMPURITY CONTENT

1.5.1

Impurity	Level (μg/g)
Fe + Ni + Cr	3000
Al + Ca	750
Boron	20
Carbon + Sulfur	500
Cadmium	20
Chlorine + Fluorine	65
Europium, Gadolinium Dysprosium	1000
Moisture	50
Total Off-Gas (STP - cc/g)	0.09
Total Impurities (Other Lanthanides and Actinides are not to be included.)	5000

1.5.2 The analysis methods for carbon, sulfur, moisture, and total off-gas shall conform with those specified in RDT F-11-1T, "Analytical Chemistry Methods for Mixed Oxide Fuel."

1.5.3 Any carbon impurity shall be distributed in a uniform manner within the pellet. Specifically, carbon impurity levels in the pellet at and within 1 mm of the surface should not exceed twice the limit for the bulk sample. For hot-pressed pellets, a procedure for elimination of high levels of carbon on the surface must be demonstrated to HEDL's satisfaction.

2.0 MICROSTRUCTURAL

2.1 HOMOGENEITY

2.1.1 Coprecipitated powder shall be used for each actinide oxide type.

2.1.2 Compositional uniformity of primary actinides in Type III pellets shall be determined by shielded microprobe or other techniques, with equivalent resolution, for information only.

2.2 GRAIN SIZE

Pellets shall be fabricated so as to contain less than 1 Vol. % second phase. Ceramographic examination on one sibling pellet from each actinide type shall be conducted for information only. Process variables shall be adjusted to produce a grain size greater than 5μm. The grain size shall be determined for information only.

2.3 STABILITY OF POROSITY

A thermal test shall be performed to qualify the pellet fabrication process on each pellet type for microstructural stability. At least one pellet of each type shall exhibit a volume change of less than 2% when subjected to a temperature of 1600°C for two hours in an inert atmosphere.

2.4 CRYSTAL STRUCTURE

The crystal structure of the major phase shall be characterized by X-ray diffraction for information only.

3.0 PELLET CHARACTERISTICS

3.1 DENSITY

The pellet's bulk density shall be 90 ± 5% T.D. Mensuration and weight method shall be used to determine the density.

a. The theoretical density for Cm_2O_3 (Type I) shall be assumed to be 11.67 gm/cm^3.

b. The theoretical density for Am_2O_3 (Type II) shall be assumed to be 11.89 gm/cm^3.

c. The theoretical density for M_2O_3 (Type III) shall be assumed to be 9.40 gm/cm^3.

3.2 PELLET WEIGHT

The weight of each pellet shall be determined to ± 0.5% (2σ).

3.3 PELLET SIZE/SHAPE

3.3.1 Pellets shall be right circular cylinders with the ends normal to the pellet axis to within ± 0.005-in.

3.3.2 The length-to-diameter ratio of each pellet shall be less than 3.

3.3.3 Pellet length shall be 0.150 ± 0.015-in.

3.3.4 Pellet Diameter

3.3.4.1 Type II And Type III Pellets

Pellet diameter shall be 0.150 ± 0.001-in. (0.381 cm). At 70°F to 100°F, pellets shall pass through a 0.151-in. diameter ring gauge. Gauge length shall not be less than 1.25 times the maximum pellet length.

3.3.4.2 Type I Pellet

Pellet diameter shall be 0.148 $^{+0.002}_{-0.001}$ inch (0.376 cm). Measured at 70°F to 500°F, pellets shall pass through a 0.151-in. diameter ring gauge as follows:

With pellets equilibrated with a plate of stainless steel of metal of comparable thermal conductivity in an argon or helium atmosphere for a period of 30 minutes with the ambient gas temperature of 70°F to 100°F, the pellets will pass through the 0.151-in. diameter ring gauge (same as shown in 3.3.4.1) which also was equilibrated with the plate for the same length of time.

3.4 PELLET COLUMN LENGTH

The pellet column length shall be 1 $\pm$ 0.1-in. (2.54 $\pm$ 0.25 cm).

3.5 PELLET DEFECTS

Defective volume shall not constitute more than 2.0% of pellet volume as determined from 1X visual examination in the presence of a HEDL representative. The following are considered defects: cracks, chips, laminations, hour-glassing and barrelling.

3.6 ARCHIVE PELLETS

Three pellets of each actinide oxide type shall be provided as archive samples. Archive samples shall be handled in the same manner as pellets to be irradiated.

4.0 SAMPLING PLAN

	Specification	Pellet	Technique	Sample	Quantity Required	Performer
1.1	Metallic (Major)	III		Powder	--	ORNL
1.2	Fissile Content	I,II,III	Spark Source	Powder	--	ORNL
1.3	Isotopic Content	I,II,III	Spark Source	Powder	--	ORNL
1.4	O/M	I,II,III	Thermal Reduction	Pellet	1	ORNL
1.5	Impurity	I,II,III	Spark Source	Pellet	1	ORNL
		I,II,III	Carbon/Sulfur	Pellet	*	ORNL
		I,II,III	Moisture	Pellet	1**	HEDL
		I,II,III	Off-Gas	Pellet	1	HEDL
2.1	Homogeneity	III	Microprobe	Pellet	1	HEDL
2.2	G. S./Structure	I,II,III	Metallography		*	HEDL
2.3	Stability	I,II,III	Specification	Pellet	1	ORNL
2.4	Crystal Structure	I,II,III	X-Ray	Pellet	*	HEDL
3.1	Density	I,II,III	Metrology	100%	--	ORNL
3.2	Weight	I,II,III	Balance	100%	--	ORNL
3.3.1	Perpendicularity	I,II,III		100%	--	ORNL
3.3.2	L/D	I,II,III		100%	--	ORNL
3.3.3	Length	I,II,III		100%	--	ORNL
3.3.4	Diameter	I,II,III	Ring Gauge	100%	--	ORNL
3.4	Column Length	I,II,III		100%	--	ORNL
3.5	Defects	I,II,III		100%	--	ORNL
3.6	Archives	I,II,III		3 Pellets	3 Pellets	HEDL

*Multiple measurements made off of one pellet.
**Pellets will also be used as archives.

O/M DETERMINATION PROCEDURE

FOR HIGHER ACTINIDE PELLETS

1. Weigh a pellet in an inert atmosphere to an accuracy of 0.1 mg.

2. Place pellet in a platinum boat in a SiO_2 muffle tube. Vacuum out gas the pellet muffle and gas lines to 10^{-4} Torr. (In place of vacuum out gassing, the gas lines should be heated to approximately 110°C between the cold traps and muffle using a heat gun and with flowing gas through the lines).

3. Heat the pellet to 800°C for 4 hours in flowing dry (liquid N_2 cold trapped) He-6% H_2 high purity gas mixture. Gas flow rate should be 300 to 500 ml/min.

4. Cool in the same flowing atmosphere.

5. Weigh pellet in an inert atmosphere to an accuracy of 0.1 mg.

6. Repeat steps 2 through 5 to assure equilibration to O/M = 1.50. If weight change is greater than 0.2 mg during the second thermal reduction cycle, then repeat until weight change is less than 0.2 mg.

7. Calculate initial O/M from total weight change assuming final O/M is 1.50.

8. Examine the pellet visually.

A P P E N D I X B

DRAWING LIST

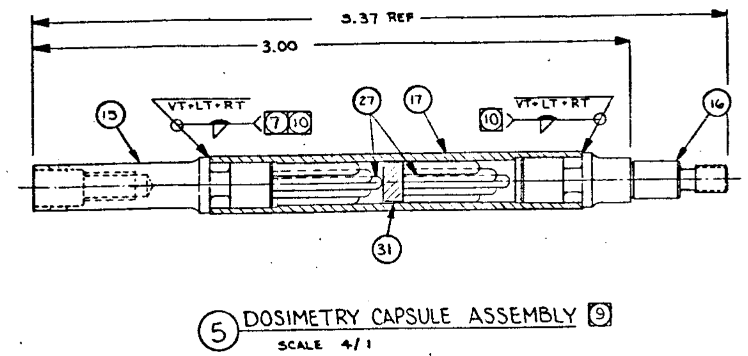
H-3-46955 - Sheet 1

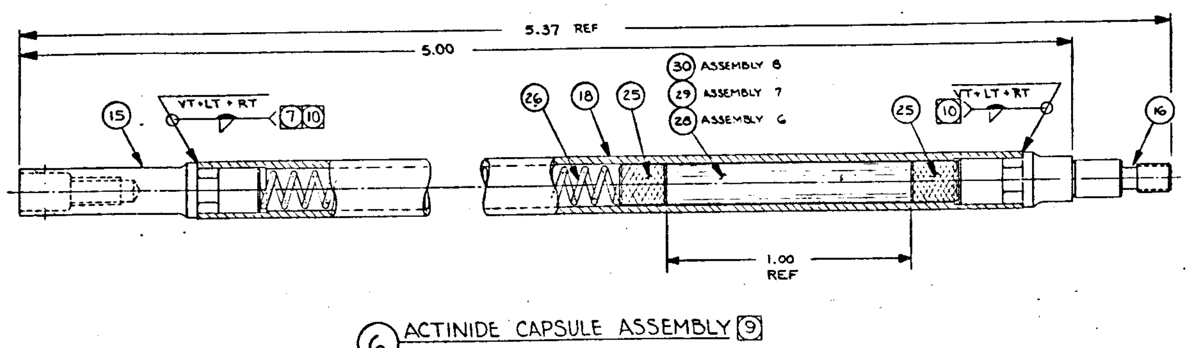
H-3-46955 - Sheet 2

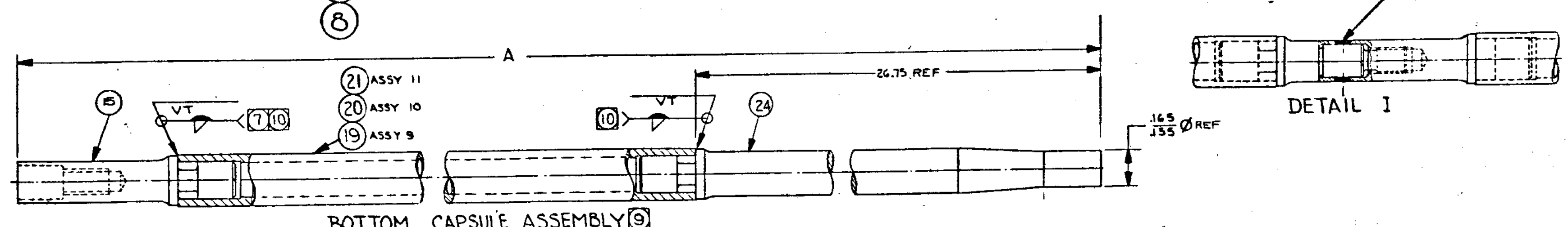
H-3-46956

General Notes (Continued)

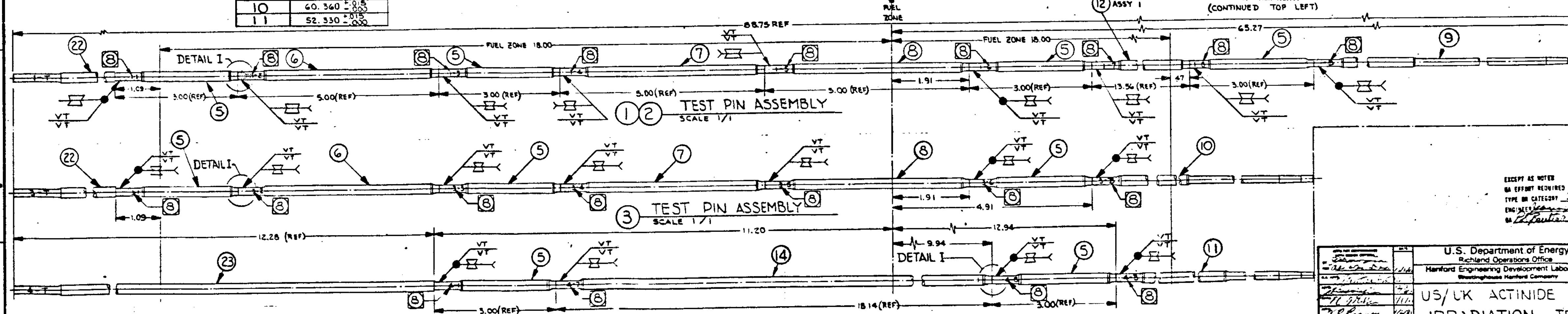
7. CLOSURE WELD AFTER HELIUM FILLING
8. ASSEMBLY IDENTIFICATION NUMBERS WILL BE LOCATED ON THE TOP OF PARTS PER IDENTIFICATION CHART. CHARACTER HEIGTH .10
9. TO BE STRAIGHT WITHIN .004 IN ANY 4.00 LENGTH i.e. .008 T.I.R. OVER FUEL ZONE, REST OF TUBE 1.0 IN 600.0
10. WELD DIA NOT TO EXCEED .231 MAX DIA.







PART Nº	DIMENSION "A"
9	43.800 +.015 −.000
10	60.360 +.015 −.000
11	52.330 +.015 −.000



QTY											PT NO.	DESCRIPTION	MAT'L/REF	QA
										X	1	TEST PIN ASSEMBLY		
									X		2	TEST PIN ASSEMBLY		
								X			3	TEST PIN ASSEMBLY		
							X				4	TEST PIN ASSEMBLY		
						X	2	3	4	4	5	DOSIMETRY CAPSULE ASSEMBLY		
					X			1	1	1	6	ACTINIDE CAPSULE ASSEMBLY (Cm_2O_3) TYPE I		
				X				1	1	1	7	ACTINIDE CAPSULE ASSEMBLY (Am_2O_3) TYPE II		
			X					1	1	1	8	ACTINIDE CAPSULE ASSEMBLY (Mixtures) TYPE III		
		X							1	1	9	BOTTOM CAPSULE ASSEMBLY		
	X							1			10	BOTTOM CAPSULE ASSEMBLY		
X							1				11	BOTTOM CAPSULE ASSEMBLY		
										1	12	PHYSICS CAPSULE ASSEMBLY	H-3-46956-1	
									1		13	PHYSICS CAPSULE ASSEMBLY	H-3-46956-2	
							1				14	PHYSICS CAPSULE ASSEMBLY	H-3-46956-3	
1	1	1	1	1	1	1					15	UPPER END CAP	SH 2	
			1	1	1	1					16	LOWER END CAP	SH 2	
						1					17	CLADDING TUBE DOSIMETRY	SH 2	
			1	1	1						18	CLADDING TUBE ACTINIDE	SH 2	
		1									19	CLADDING TUBE BOTTOM CAPSULE	SH 2	
	1										20	CLADDING TUBE BOTTOM CAPSULE	SH 2	
1											21	CLADDING TUBE BOTTOM CAPSULE	SH 2	
								1	1	1	22	TOP FITTING	SH 2	
							1				23	TOP FITTING	SH 2	
1	1	1									24	BOTTOM FITTING	SH 2	
			2	2	2						25	INSULATOR PELLET	SH 2	
			1	1	1						26	SPRING	SH 2	
						AR					27	DOSIMETRY SPECIMEN	SUPPLIED BY CUSTOMER	
					AR						28	ACTINIDE PELLETS (TYPE I)	SUPPLIED BY CUSTOMER	
				AR							29	ACTINIDE PELLETS (TYPE II)	SUPPLIED BY CUSTOMER	
			AR								30	ACTINIDE PELLETS (TYPE III)	SUPPLIED BY CUSTOMER	
						1					31	SPACER .145 DIA × AS REQD L	SST	III

GENERAL NOTES: (UNLESS OTHERWISE SPECIFIED)

1. DIMENSIONING & TOLERANCING PER ANSI Y14.5. TOLERANCES: .XX± .01, .XXX± .005. ANGULAR ± 1°.
2. ALL MACHINED SURFACES 63
3. MACHINE TOOL RADII .01 MAX.
4. BREAK ALL SHARP EDGES & REMOVE ALL BURRS.
5. ALL MATERIAL SHALL BE AS SPECIFIED OR EQUAL QUALITY WITH WRITTEN APPROVAL OF PURCHASER.
6. WELDS & WELDER TO BE QUALIFIED AS PER HDT F 6-2T PERFORM NONDESTRUCTIVE EXAM AS PER HDT F 3-6T INSPECT PER HDT. F 6-2T
   - RT – PARA 4.2.7.4
   - LT – PARA 4.2.7.2
   - VT – PARA 4.2.7.1

(CONTINUED TOP LEFT)

IDENTIFICATION MARKS (NOTE 8)										
ASSEMBLY	DOSIMETRY (5)	ACTINIDE (6)	DOSIMETRY (5)	ACTINIDE (7)	ACTINIDE (8)	DOSIMETRY (5)	PHYSICS (12)(13)(14)	DOSIMETRY (5)	TOP FITTING (22)(23)	BOTTOM FITTING (9)(10)(11)
1	1-1	1-2	1-3	1-4	1-5	1-6	1-7	1-8	1-T	1-B
2	2-1	2-2	2-3	2-4	2-5	2-6	2-7	2-8	2-T	2-B
3	3-1	3-2	3-3	3-4	3-5	3-6			3-T	3-B
4	4-1						4-7	4-8	4-T	4-B

EXCEPT AS NOTED
QA EFFORT REQUIRED
TYPE OR CATEGORY II
ENGINEER
QA

U.S. Department of Energy
Richland Operations Office
Hanford Engineering Development Laboratory
Westinghouse Hanford Company

US/UK ACTINIDE IRRADIATION TEST PIN ASSEMBLY

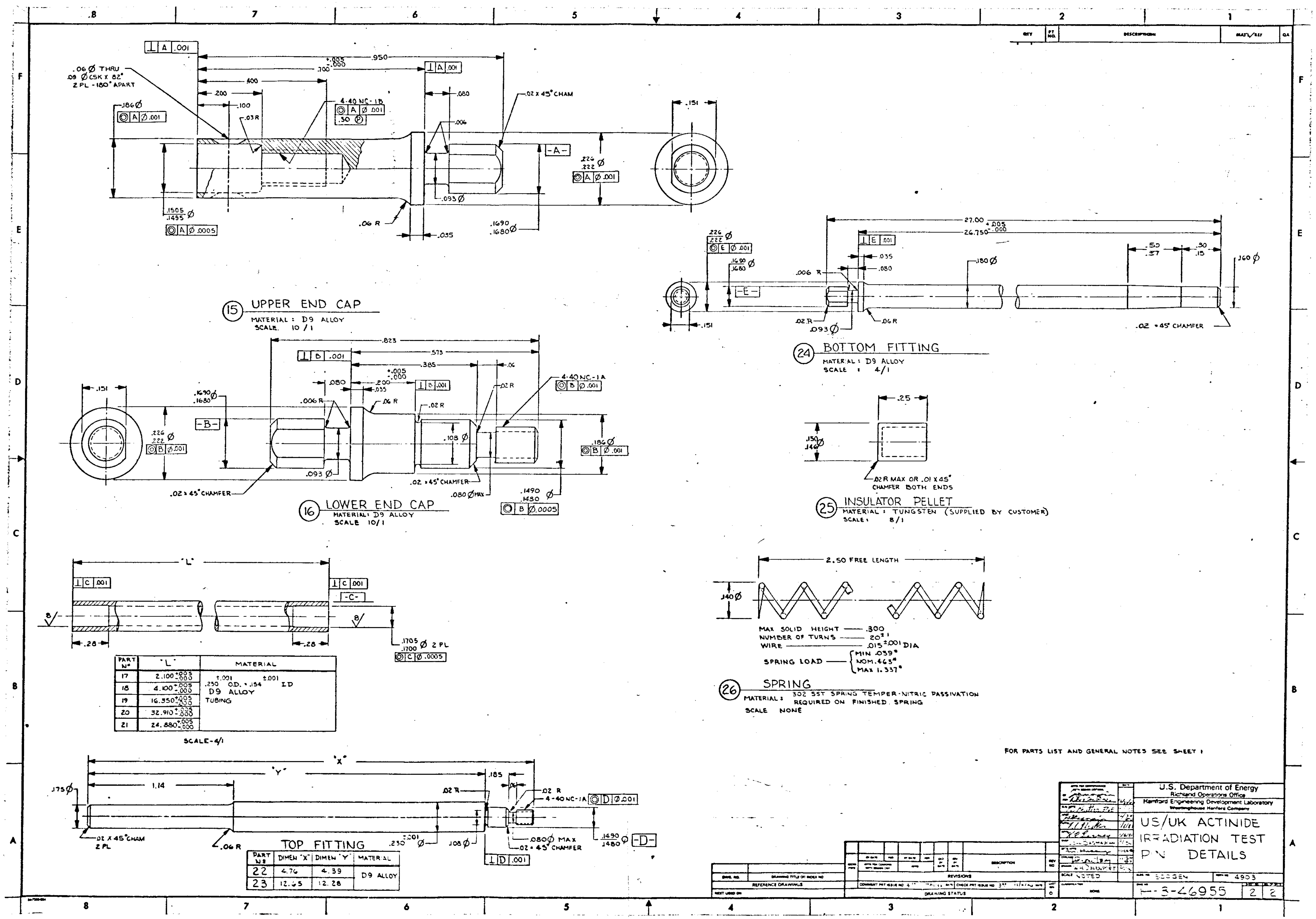
W H DAVENPORT

SCALE NOTED

300GEN | 4903

H-3-46955 | 1 | 2

REFERENCE DRAWINGS: NONE



PART Nº	"L"	MATERIAL
17	2.100 +.005 -.000	.250 ±.001 O.D. x .154 ±.001 I.D D9 ALLOY TUBING
18	4.100 +.005 -.000	
19	16.350 +.005 -.000	
20	32.910 +.005 -.000	
21	24.880 +.005 -.000	

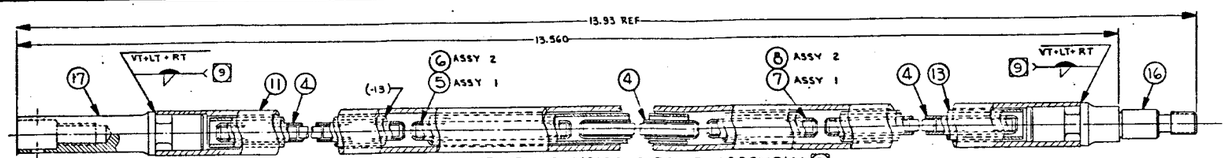
PART Nº	DIMEN "X"	DIMEN "Y"	MATERIAL
22	4.76	4.39	D9 ALLOY
23	12.65	12.28	

U.S. Department of Energy
Richland Operations Office
Hanford Engineering Development Laboratory
Westinghouse Hanford Company

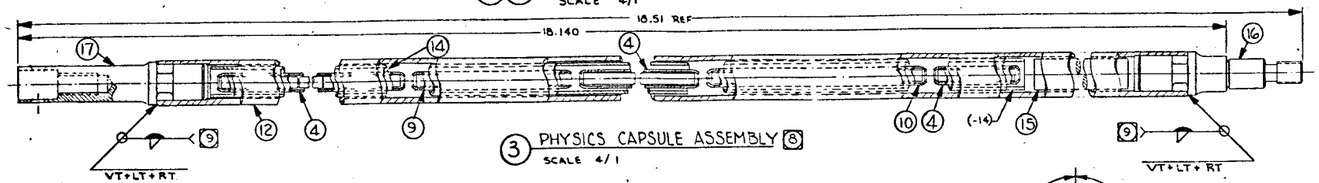
US/UK ACTINIDE IRRADIATION TEST PIN DETAILS

SCALE NOTED

H-3-46955 2 2

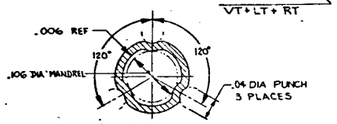


1 2 PHYSICS CAPSULE ASSEMBLY [8]
SCALE 4/1

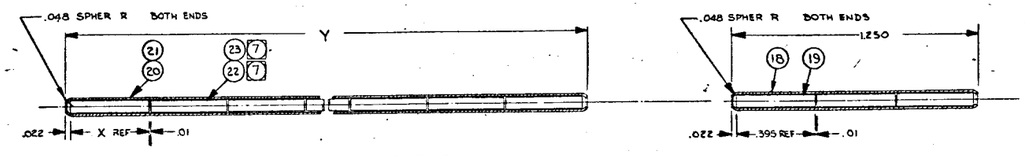


3 PHYSICS CAPSULE ASSEMBLY [8]
SCALE 4/1

QTY							PT NO.	DESCRIPTION	MAT'L/SPEC	QA
						1	1	PHYSICS CAPSULE ASSEMBLY 1		
						1	2	PHYSICS CAPSULE ASSEMBLY 2		
						1	3	PHYSICS CAPSULE ASSEMBLY 3		
					3	3	4	DOSIMETRY TUBE ASSEMBLY		
						1	5	PHYSICS TUBE ASSEMBLY 1		
					1		6	PHYSICS TUBE ASSEMBLY 2		
						1	7	PHYSICS TUBE ASSEMBLY 3		
					1		8	PHYSICS TUBE ASSEMBLY 4		
						1	9	PHYSICS TUBE ASSEMBLY 5		
						1	10	PHYSICS TUBE ASSEMBLY 6		
					1	1	11	CLADDING TUBE		
					1		12	CLADDING TUBE		
					1	1	13	LINER TUBE		III
					1		14	LINER TUBE		III
					1		15	SPACER		III
					1	1	16	LOWER END CAP		
					1	1	17	UPPER END CAP		
				1			18	DOSIMETRY TUBE-.095 O.D.×.013 WALL×12.56 L	304 SST	III
				3			19	DOSIMETRY SPECIMEN	SUPPLIED BY CUSTOMER	
		1	1	1	1		20	PHYSICS TUBE-.095 O.D.×.013 WALL×4.106 L	304 SST	III
1	1						21	PHYSICS TUBE-.095 O.D.×.013 WALL×5.306 L	304 SST	III
		13	13	13	13		22	PHYSICS SPECIMEN [7]	SUPPLIED BY CUSTOMER	
15	15						23	PHYSICS SPECIMEN [7]	SUPPLIED BY CUSTOMER	



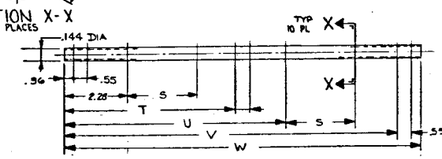
SECTION X-X
10 PLACES



PHYSICS TUBE ASSEMBLY

PART NO	DIMEN X	DIMEN Y
5	.300	4.10
6	.300	4.10
7	.300	4.10
8	.300	4.10
9	.395	5.30
10	.395	5.30

4 DOSIMETRY TUBE ASSEMBLY
SCALE 4/1

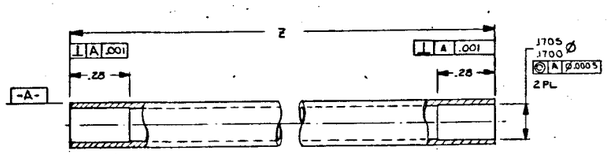


LINER TUBE

PART NO	DIMEN S	DIMEN T	DIMEN U	DIMEN V	DIMEN W	MATERIAL
13	2.10	5.75	7.67	11.14	12.06	.148 OD.×.015 WALL 304 SST TUBING
14	3.50	6.97	8.90	13.58	14.50	

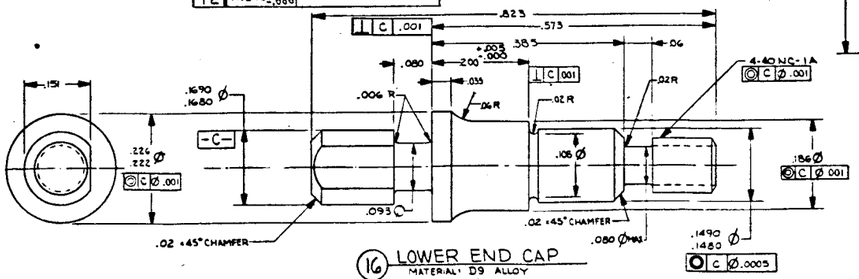
GENERAL NOTES: (UNLESS OTHERWISE SPECIFIED)

1. DIMENSIONING & TOLERANCING PER ANSI Y14.5. TOLERANCES: .XX ± .01, .XXX ± .005, ANGULAR ± 1°.
2. ALL MACHINED SURFACES 63 √
3. MACHINE TOOL RADII .01 MAX.
4. BREAK ALL SHARP EDGES & REMOVE ALL BURRS.
5. ALL MATERIAL SHALL BE AS SPECIFIED OR EQUAL QUALITY WITH WRITTEN APPROVAL OF PURCHASER.
6. WELDS & WELDER TO BE QUALIFIED AS PER WPS FS-2T PERFORM NONDESTRUCTIVE EXAM AS PER RDT F 3-6T INSPECT PER: RDT FS-2T AS SHOWN BELOW.
   - VT - PARA 4.2.7.4
   - LT - PARA 4.2.7.2
   - VT - PARA 4.2.7.1
7. PHYSICS SPECIMENS PT 22 & 23 SHALL BE SELECTIVELY LOADED PER ENGINEERING TEST PLAN.
8. TO BE STRAIGHT WITHIN .006 IN ANY 4.00 LENGTH.
9. WELD DIA NOT TO EXCEED .231 MAX DIA

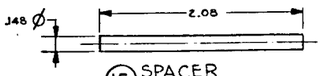


CLADDING TUBE

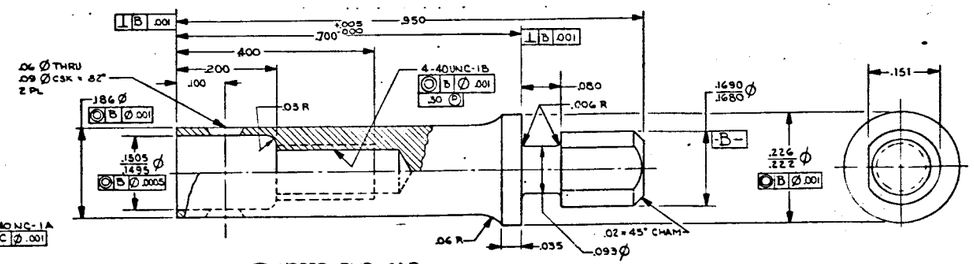
PART NO	DIMEN Z	MATERIAL
11	12.660 +.035 −.000	.230 +.001 −.000 O.D. × .154 +.001 −.000 I.D. D9 ALLOY TUBING
12	17.240 +.035 −.000	



16 LOWER END CAP
MATERIAL: D9 ALLOY
SCALE 10/1



15 SPACER
MATERIAL: 316 SST
SCALE 2/1



17 UPPER END CAP
MATERIAL: D9 ALLOY
SCALE 10/1

EXCEPT AS NOTED
QA EFFORT REQUIRED
TYPE OR CATEGORY II
ENGINEER
QA

DRAWING NO.	DRAWING TITLE OR INDEX NO.	REVISIONS	DESCRIPTION
REFERENCE DRAWINGS		COMMENT PKT ISSUE NO. 3	CHECK PKT ISSUE NO. 3
NEXT USED ON	M-3-46955	DRAWING STATUS	

U.S. Department of Energy
Richland Operations Office
Hanford Engineering Development Laboratory
Westinghouse Hanford Company

US/UK ACTINIDE IRRADIATION TEST PIN PHYSICS CAPSULE ASS'Y

SCALE	DRAWN	APPROVED
NOTED		

M-3-46956 1 1

A P P E N D I X C

QUALITY ASSURANCE PROGRAM INDEX

QUALITY ASSURANCE PROGRAM INDEX

Test Plan No. _____ Title US/UK JOINT ACTINIDES IRRADIATION IN PFR

COMPONENT	QA Level (See QAI 1-1)	REMARKS
Actinide Oxide Pellet	II	
Dosimetry	III	Specs
Physics Capsules	III	Specs
Components in contact with coolant	II	

Task Requester: DOE-RRT - Physics Branch; P. B. Hemmig

Test Performer: HEDL Core Technology & Safety; E. T. Weber

Test Fabricator: HEDL Core Technology & Safety; Weld Engineering - W. F. Brown

Test Operator: UK; PFR

Development Project Engr. (DPE): J. A. Basmajian, HEDL Absorber and Ceramics Development

Cognizant Quality Engr. (CQE): P. S. Beutler

Hold Points: The following Hold Points shall be observed. All required inspection and testing shall be completed prior to proceeding beyond any hold point. Any nonconformances shall be resolved per Section II of WHAN-M-2 before further processing.

HOLD POINT DESCRIPTION	PROVISIONS FOR RELEASE
Receiving Inspection (Metallic Components)	Signed-Off Release Cards From FQC
ETP required at this point	
Receipt of Pin Sections	Performer, CQE
(Documentation Certifications)	
Dosimetry	Performer, CQE
Assembly	FQC, Performer, Weld Engineering
Readiness Review	Performer, CQE, FQC
Final Review Prior to Release	Acceptance By Performer, Weld Engineer FQC

A. QUALITY ASSURANCE REQUIREMENTS:

- Check the Q.A. Elements which apply to the test program.
- Line out any referenced HEDL procedures that do not apply.
- Add any other procedures that do apply.
- When no HEDL procedure is noted, refer to the applicable standard indicated.
- Use space provided to indicate the appropriate paragraph number of the Test Plan which explains any deviation from normal procedures or explains additional requirements.
- For any selected element which is denoted by an asterisk (*), either describe in the test plan how the element will be implemented, or provide a separate plan for it, as appropriate.

Q.A. ELEMENT	APPLICABLE STANDARDS/PROCEDURES				REMARKS	TEST PLAN PARA. NO.
	HEDL	RDT F2-2	ANSI 45.2	OTHER		
1. Management and Planning						
(x) 1.1 Responsibility & Authority	P/P 14-01	2.3.1	1.3			
( ) 1.2 Training and Indoctrination		2.3.2	2.0			
( ) 1.3 Personnel Qualification	WHAN-M-15	2.3.3	10.0			App.C,Sec.
(x)*1.4 Quality Records	(1) 14-1	2.4.2	7.0,18.0			*IX,XI
( )*1.5 Quality Status Reports		2.4.3	3.0			*
( ) 1.6 Corrective Action	(1) 12-1	2.6	17.0			
( ) 1.7 Engineering Holds	WHAN-M-20 (EI-32)	2.7	4.0			
( ) 1.8 Unusual Occurrence Reporting	P/P 10-01	2.8	17.0			
2. Design and Development						
(x) 2.1 Design Criteria		3.3.1	4.1			
(x)*2.2 Codes or Standards Assigned	WHAN-M-20 (EI-26) MG-22, (1) 13-1	3.3.2	1.2			*Sec. VI,D

(1) Refer to Quality Assurance Manual WHAN-M-2 Section

Q.A. ELEMENT	APPLICABLE STANDARDS/PROCEDURES HEDL	RDT F2-2	ANSI 45.2	OTHER	REMARKS	TEST PLAN PARA. NO.
2. Design and Development (Cont'd)						
( )*2.3 Engineering Studies		3.3.3	4.2			*
( ) 2.4 Parts, Mat'l & Processes	MG-22	3.3.4	4.1			
(x)*2.5 Design Description	WHAN-M-20 (EI-6)	3.3.5				*Sec IV
(x)*2.6 Specs., Dwgs. & Instruction		3.3.6	5.0			*App. A&B
(x)*2.7 Identification		3.3.7	9.0			*
(x) 2.8 Accept. Criteria		3.3.8				
(x)*2.9 Interface Control	WHAN-M-20 (EI-6)	3.3.9	4.2			*Sec. V
(x) 2.10 Doc. Review & Approval	P/P 17-02	3.4.1	4.1			
( ) 2.11 Document Control	P/P 17-02 WHAN-M-20 (EI-23)	3.4.2	4.1 7.0			
( )*2.12 Dwg. Control Lists	P/P 17-01	3.4.3	7.0			*
(x)*2.13 Design Review (x) Formal ( ) Informal	WHAN-M-20 (EI-23)	2.5	4.3			*Sec. IX
( )*2.14 Item Qualification		3.7	4.3			*
( )*2.15 Quality Records	WHAN-M-20 (EI-6) (1) 14-1	3.8	4.1, 18			*

(1) Refer to Quality Assurance Manual WHAN-M-2 Section

Q.A. ELEMENT	APPLICABLE STANDARDS/PROCEDURES HEDL	RDT F2-2	ANSI 45.2	OTHER	REMARKS	TEST PLAN PARA. NO.
3. Procurement						
( ) 3.1 Procurement Planning	(1) 4 MG-5	4.2	5.0			
(x) 3.2 Procurement Requirements	(1) 4 MG-5	4.3	5.0			
(x) 3.3 Document Review	(1) 4-1 MG-5	4.4	5.0			
(x) 3.4 Evaluation & Selection of Suppliers	(1) 4-2	4.5	5.0			
( ) 3.5 Contract Change Control	(1) 4-1 MG-5	4.6.1	5.0			
( ) 3.6 "As-Built" Verification	(1) 4 MG-5	4.6.2	18.0			
( ) 3.7 Meas. & Test Equip. Control	(1) 9-1	4.7	13.0			
( ) 3.8 Source Surveillance Insp.	(1) 4-3	4.8	8.0			
(x) 3.9 Rec. Inspection (x) Mat'l Overchecks	(1) 4-4 (1) 4-5	4.9	8.0			
( ) 3.10 Nonconforming Item Control	(1) 11	4.10	16.0			
( ) 3.11 Received Item Control	(1) 6-1 MG-52 MG-78 NFCL-38	4.11	9.0 14.0			
(x) 3.12 Alloy Verification	(1) 4	4.13				*VIII,A

(1) Refer to Quality Assurance Manual WHAN-M-2 Section

Q.A. ELEMENT	APPLICABLE STANDARDS/PROCEDURES HEDL	RDT F2-2	ANSI 45.2	OTHER	REMARKS	TEST PLAN PARA. NO.
4. Fabrication						
(x) 4.1 Insp. & Test Plan	(1) 8-1 MG-51	5.3	6.0			
(x) 4.2 Mat'l Ident. & Control	MG-52 MG-78 (1) 6-1	5.4	9.0			
(x) 4.3 Fab. & Assy. Processes (x) Work Instructions (x) Assy. Procedures (x) Follower/Traveler Cards ( ) Assy. Logbooks	(1) 3-1	5.5.1	6.0			
(x) 4.4 Process Qualification (x) Welding ( ) Chemical Analysis ( ) Other	(1) 7-1 MG-31 MG-28	5.5.2				
(x) 4.5 Nondestructive Exam.	WHAN-M-15 MG-25 (1) 7	5.5.3	10.0			
(x) 4.6 Cleaning	MG-19	5.5.4	14.0			*VI,B
(x) 4.7 Insp. & Test Requirements	(1) 8-1	5.6.1	11., 12.			
(x) 4.8 Insp. & Test Procedures	(1) 8-2	5.6.2	11., 12.			
(x) 4.9 Completed Item Insp. & Test	(1) 8-6	5.6.3	11., 12.			
(x) 4.10 Insp. Status Indication	(1) 8-02	5.6.4	15.0			
( )*4.11 Certification		5.6.5				*

(1) Refer to Quality Assurance Manual WHAN-M-2 Section

Q.A. ELEMENT	APPLICABLE STANDARDS/PROCEDURES HEDL	RDT F2-2	ANSI 45.2	OTHER	REMARKS	TEST PLAN PARA. NO.
4. Fabrication (Cont'd)						
( )*4.12 Document Control	WHAN-M-20	5.7	7.0			*
(x) 4.13 Meas. & Test Equip. Calib.	(1) 9-1 (1) 9-2	5.8	13.0			
( ) 4.14 Statistical Q.C. & Anal.		5.9				
( ) 4.15 Control of Nonconform. Items	(1) 11	5.20	16.0			
( ) 4.16 Corrective Action	(1) 12-1	5.11	17.0			
(x) 4.17 Handling, Storage & Shipping	(1) 8-13 (1) 10-1	5.12	14			*
(x) 4.18 Quality Records	(1) 14-1	5.13	16			*Sec. XIV
( ) 4.19 Alloy Verification	(1) 4	5.15				*
5. Testing						
(x)*5.1 Test Requirements & Objective	WHAN-M-20 (EI-6)	3.6.1	15.0			*Sec. I
(x) 5.2 Test Planning	WHAN-M-20 (EI-6) (1) 8-3	3.6.2	15.0		Satisfied by Test Pland, Including QAPI	
( )*5.3 Test Procedures	(1) 8-1	3.6.3	15.0			*
(x)*5.4 Test Item Characterization, Identification, Control and Disposition		3.6.4				*Sec. VII
( )*5.5 Test Equip. & Fluids Control		3.6.5				*
( )*5.6 Data Acquis. Equip. & Method		3.6.6				*

(1) Refer to Quality Assurance Manual WHAN-M-2 Section

Q.A. ELEMENT	APPLICABLE STANDARDS/PROCEDURES HEDL	RDT F2-2	ANSI 45.2	OTHER	REMARKS	TEST PLAN PARA. NO.
5. Testing (Cont'd)						
( )*5.7 Readiness Reviews	WHAN-M-20 (EI-6)	3.6.7				*_____
(x)*5.8 Failure Analysis		3.6.8				*Sec. XIV,8
( )*5.9 Test Records, Analy., & Reports	WHAN-M-20 (EI-6)	3.6.9	18.0			*_____
( )*5.10 Test Results Review	WHAN-M-20 (EI-6)	3.6.10	18.0			*_____
( )*5.11 Test Records		3.6.11	18.0			
6. Quality Audits						
( ) 6.1 Quality Audits	(1) 15	8.0	19		Review Test Data Records	

(1) Refer to Quality Assurance Manual WHAN-M-2 Section

A P P E N D I X D

PHYSICS SPECIMEN SPECIFICATIONS

LOADING SCHEME, AND DOSIMETRY

SPECIFICATIONS FOR PHYSICS AND DOSIMETER SPECIMENS

H. L. Adair - ORNL

1.0 CAPSULE BLANK SPECIFICATIONS

1.1 COMPOSITION

All capsule blanks and lids are made from high purity vanadium metal that was purchased from Materials Research Corporation. A Spark Source Mass Spectrographic analysis (SSMS) of the material is shown in Table 1.

1.2 LOT IDENTIFICATION

The vanadium lot purchased from NRC (5.5 Kg) has been identified at Batch MRC 79-1.

1.3 DIMENSIONS

The vanadium capsule dimensions for both physics and dosimeter specimens are shown in tables A.1.1 and A.1.2.

TABLE 1

SSMS ANALYSIS OF VANADIUM FROM BATCH MRC 79-1

Element	Weight (ppm)
Ag	5
Ag	2
B	5
Ca	0.3
Fe	100
K	1
Mg	10
Mo	50
Na	2
Nb	10
Ni	20
P	20
Si	300
Ta	100
Ti	20
V	Major
W	40
Zr	10
S	100
F	0.5

2.0 PHYSICS AND DOSIMETER MATERIAL SPECIFICATIONS

2.1 IDENTIFICATION

The batch numbers and enrichments of these materials are shown in Table 2.

2.2 COMPOSITION

The materials will be characterized by spark source mass spectrographic, isotopic and elemental analyses.

2.3 WEIGHT (MASS)

The weight of material loaded into each capsule will be within the range given in Table 3.

The primary method of determining the mass of the physics and dosimeter materials will be by physical weighing. The weight of material per capsule will be reported to the nearest microgram and the 2σ uncertainty of each weight will be reported. A substitution weighing scheme (Appendix 2) will be used.

TABLE 2

BATCH NUMBERS AND ENRICHMENTS OF MATERIALS
USED FOR PHYSICS AND DOSIMETER SPECIMENS

Physics Specimen Materials

Isotope	Batch Number	Enrichment %
Th-230	256A	89.46
Th-232	SNM-4151	100
Pa-231	PA-F-1	100
U-233	240A	99.86
U-234	M9	99.887
U-235	264C	99.887
U-236	201DMR	89.2
U-238	Q1	99.964
Np-237	NP24HP	100
Pu-238	208HP-014	99.4
Pu-239	453B	99.11
Pu-240	SHIP-1068	99.9
Pu-241	307A	97.79
Pu-242	290A	99.744
Pu-244	297C	87
Am-241	79AMB4	100
Am-243	SHIP-1018	99.987
Cm-243	1011	55
Cm-244	CMP-576	92.94
Cm-246	C59SHIP	55
Cm-248		97

DOSIMETER MATERIALS

U-235	99.89
U-238	99.999
Np-237	$\geq$99.99
Pu-239	99.10
Cu-63	(1)
Co-59 (0.1% Co-MgO)	(1)

(1)Normal materials that have a chemical purity $\geq$99.9.

TABLE 3

WEIGHT OF OXIDE FOR PHYSICS AND DOSIMETER SPECIMEN MATERIALS PER CAPSULE

Isotope	Amount of Oxide Materials Per Capsule (Mg)
Physics Specimen Materials	
Th-230	3.5-4.0
Th-232	20.0-25.0
Pa-231	2.5-4.0
U-233	9.0-10.0
U-234	4.5-5.0
U-235	9.0-10.0
U-236	12.0-12.5
U-238	12.0-12.5
Np-237	13.5-14.0
Pu-238	3.5-4.0
Pu-239	9.0-10.0
Pu-240	13.5-14.0
Pu-241	5.5-6.0
Pu-242	2.0-2.5
Pu-244	2.5-3.0
Am-241	11.0-14.0
Am-243	11.0-14.0
Cm-243	1.0-1.2
Cm-244	11.0-14.0
Cm-246	11.0-14.0
Cm-248	2.0-2.5
DOSIMETER MATERIALS	
U-235	1.0-1.5
U-238	1.0-1.5
Np-237	1.0-1.5
Pu-239	1.0-1.5
Cu-63	1.0-1.5
Co-59 (0.1% Co - MgO)	1.0-1.5

3.0 PHYSICS AND DOSIMETER CAPSULE SPECIFICATIONS

3.1 CAPSULE DIMENSIONS

The materials will be encapsulated in vanadium capsules detailed in Appendix 1.

3.2 WELDING

The capsules will be welded closed by tungsten inert-gas (TIG) fusion welding. The capsules will be welded in a helium atmosphere.

3.3 HEAT TREATMENT

Each finished capsule will be heated to 800°C and held at this temperature for one hour.

3.4 NONDESTRUCTIVE EXAMINATIONS

a) A microscopic examination will be made of each capsule for visible cracks under a magnification of 10X. A capsule will be rejected if any crack is seen.

b) Each capsule will be leak tested under vacuum in an ethylene glycol solution. The observance of any leak, as evidenced by bubbles, will be the basis for rejecting a capsule.

c) The primary weight of the actinide materials will be verified by gamma counting where applicable. The gamma counts per milligram will be compared for each set of physics and dosimeter specimens. An average count per milligram will be obtained and all specimens with a counts per milligram variation ~3% from the average count will be rejected.

3.5 WEIGHT (MASS)

The gross weight of each finished capsule will be obtained by physical weighing. Each weight will be reported the nearest 0.001 mg.

3.6 IDENTIFICATION

Each finished capsule will be identified by the dot code system shown in Table 4. There will be three sets of 35 vanadium capsules. Each capsule set will be identified by a number on the bottom of the vanadium capsule.

The vertical ordering of the physics and dosimeter capsules is shown in Table 5.

3.7 CLEANING, PACKAGING, AND SHIPPING

Each finished capsule will be cleaned as follows:

a) Ultrasonically cleaned in benzene for 30 minutes.
b) Rinsed in alcohol
c) Ultrasonically cleaned in alcohol for 30 minutes.
d) Dried under a heat lamp.

Steps a) through d) are used initially to degrease the capsules after the capsules are loaded and welded, steps a) through c) are used for decontamination.

TABLE 4

VANADIUM CAPSULE DOT CODE IDENTIFICATION SYSTEM

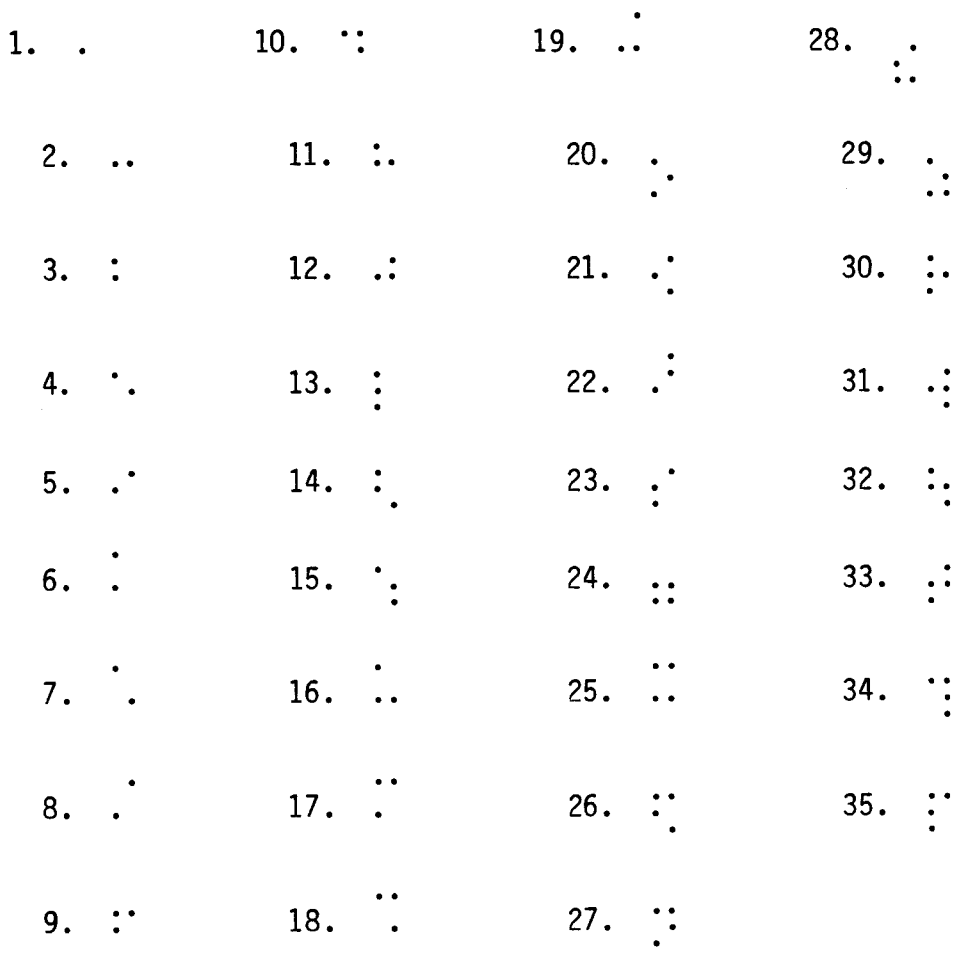


TABLE 5

VERTICAL ORDERING OF PHYSICS SPECIMENS AND DOSIMETER CAPSULES FOR FP1, FP2, AND FP4*

Position	Capsule Material
1	Dos
2	Dos
3	Dos
4	Cm-248
5	Cm-246
6	Cm-246
7	Np-237
8	Cm-244
9	Cm-244
10	Cm-243
11	U-238
12	Am-243
13	Am-243
14	Am-241
15	Am-241
16	Pu-242
17	Dos
18	Dos
19	Dos
20	Pu-244
21	Pu-240
22	Pu-240
23	Pu-239
24	Pu-241
25	Th-232
26	U-236
27	U-234
28	U-235
29	Pa-231
30	Pu-238
31	Th-230
32	U-233
33	Dos
34	Dos
35	Dos

*The physics specimen capsule lengths for FP1 and FP2 are 0.762 cm (0.300 in.). All physics specimen capsule lengths for FP4 are 1.003 cm (0.395 in.).

4.0 QUALITY ASSURANCE MEASURES

4.1 QUALITY CONTROL PROCEDURES

Procedures have been established to verify the quality of the physics specimens and dosimeters. These procedures are described in Appendix 2 and include detailed procedures for:

a) Elemental, spark source mass spectrographic, and isotopic analyses;

b) weighing procedures including statistical procedures for establishing weighing uncertainties;

c) loading physics and dosimeter materials;

d) TIG welding;

e) heat treating specimens;

f) capsule leak testing;

g) gamma counting;

h) dimension measurements; and

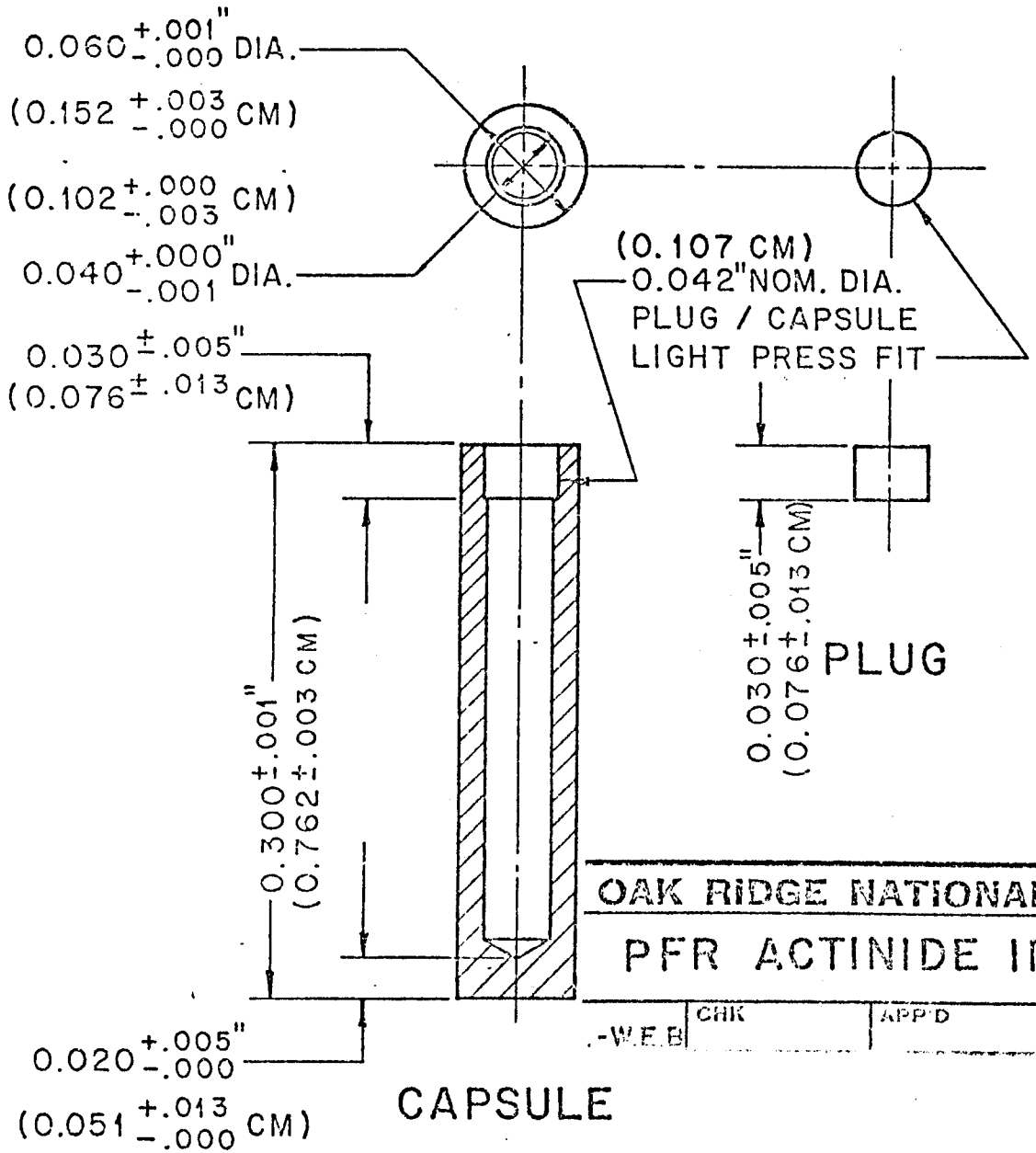
i) cleaning, packaging and shipping actinide materials.

4.2 RECORD SYSTEM

All data generated during the fabrication of physics and dosimeter specimens (including the analysis, testing and examination of materials) is recorded in a permanent type record system that provides traceability of all finished specimens back to the specific materials from which the capsules were fabricated. Identification of materials by lot is maintained throughout the record system. The record system has provisions for documenting any unusual observations made during fabrication of specimens and any changes in procedures used.

TABLE A.1.1

VANADIUM CAPSULE FOR ACTINIDE OXIDE PHYSICS SPECIMENS



NOTES:

1. MATERIAL - VANADIUM
2. CLOSED CAPSULE VOLUME
   3.19×10^{-4} CU. IN.
   5.231×10^{-3} CU. CM.
3. CAPSULE I.D. / O.D. TO BE CONCENTRIC W/IN 0.0015 IN. (0.004 CM)

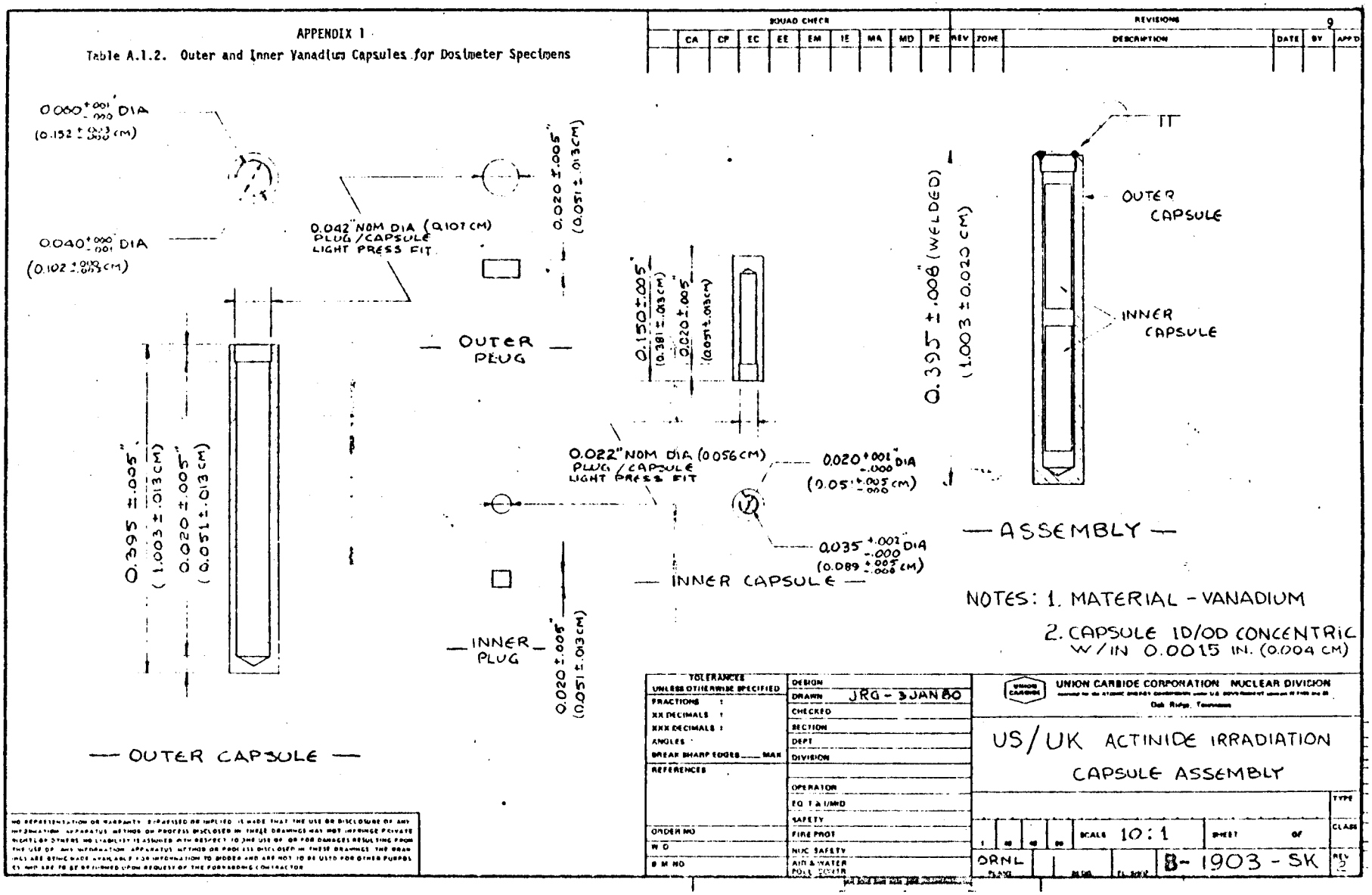
OAK RIDGE NATIONAL LABORATORY Solid State Division

PFR ACTINIDE IRRADIATION CAPSULE

-W.E.B	CHK	APP'D	DATE 7/79	SCALE 10=1	SSD-A-1612-R2

TABLE A.1.2

OUTER AND INNER VANADIUM CAPSULES FOR DOSIMETER SPECIMENS



APPENDIX 2

A.2.1 Elemental, Spark Source Mass Spectrographic and Isotopic Analyses Procedures

a. Elemental Analyses by Isotope Dilution Mass Spectrometry (IDMS). Quantitative elemental analyses of the actinide oxides are obtained by adding a known amount of an enriched isotope (spike) of the element in the sample being analyzed. Complete isotopic homogenization between the sample and the spike is essential. This is attained by dissolving the sample in an acid solution and evaporating the solution to dryness. The sample is redissolved in a weak acid solution and an aliquot is pipetted onto a mass spectrometer filament. The filament is heated by d.c. current and the ions formed are accelerated through a magnetic field. After an acceptable ion beam intensity has been reached and stabilized, the ratio of the intensities of the known spike nuclide to the sample nuclide is measured. The amount of the sample nuclide present is then calculated from the isotope intensity ratios of the spike sample, sample/spike mixture, and the known concentration of the spike.

b. General Procedure for Spark Source Mass Spectrographic Analyses.

1. SAMPLE PREPARATION*

a. Metals

Form metal samples into electrodes approximately 0.4 mm diameter and 19 mm long either by machining or by cutting with a diamond saw. Clean the metal rods in teflon beakers with acetone and then with the proper acid to dissolve the surface layers of the sample. Rinse the cleansed metal rods thoroughly with deionized water. The sample is now ready to be loaded into the instrument for pump down.

*Samples must be prepared and loaded in a clean room environment.

b. Powdered Conductors

(Note: Degrease metals in the form of chips, turnings, and chunks prior to grinding.) Load powdered conductors into cylindrical naphthalene molds and press isostatically into sample rods by applying a pressure of 2.11 x 10^7 kg/m^2 for one minute. After the rod has been pressed, dissolve the naphthalene mold with acetone. Pump and dry the rod in a vacuum oven at 140°C to remove naphthalene and other hydrocarbons. The sample is now ready to be loaded into the instrument for pump down.

c. Non-Conducting Powders and Insulators

1) If the insulator is a powder, mix a weighed portion of the sample with a weighed portion of 99.999% silver powder. Load the mixture into a cylindrical naphthalene mold and press isostatically into a sample rod. After the rod has been pressed, dissolve thenaphthalene mold with acetone. Pump and dry the rod in a vacuum oven at 140°C to remove naphthalene and other hydro-carbons. The sample is now ready to be loaded into the instrument for pump down. 2) If the insulator is a rod, spark it counter to a gold probe or use a much higher (approximately 60 kV) radio frequency spark thatn normally is used.

d. Plastics

Analyze plastic samples by one of the following two methods: 1) Ash the samples in a low temperature asher, mix the ash with silver, and press the mixture isostatically into rods as described in the preceeding Step 1.c. 1) for an insulating powder. 2) If the plastic is present in large pieces, insert a conducting wire into the plastic and generate the spark through the plastic around the wire.

2. SPARKING SAMPLES

Detailed instructions for operating the instrument are given in "Instruction Manual Mass Spectrograph MS-7," Associated Electrical Industries Ltd.

a. Regardless of the method of preparation, load the samples in the ion source of the mass spectrograph for analysis. Evacuate the ion source to a pressure of approximately 1×10^{7} mm Hg. If it is necessary to measure carbon, oxygen, and nitrogen in the sample, bake the source at 150°C for twelve hours. The ultimate pressure reached is about 2×10^{-9} mm Hg.

b. When the pressure is low enough, spark the samples so that an ion beam is generated. Instrument parameters are:

Accelerating Voltage	20 kV
R. F. Voltage	30 kV (60 kV for insulators)
Magnet Current	305 ma (105 ma for Li and B)
Source Pressure	1×10^{-7} mm Hg or less
Analyzer Pressure	1×10^{-8} mm Hg or less
Spark Repetition Data	10 to 300 pulse per second
Spark Duration	25 to 100 microseconds

c. The ion beam produced is measured electronically by intercepting 50 percent of the ion beam before separating according to mass to charge ratio. By use of the electronic monitor, a series of graded exposures is made on the photographic plate. The exposure range will cover the range of 1×10^{-13} to 1×10^{-6} coulombs.

d. Exposures needed for a specific detection limit are:

Detection Limit	Exposure in Coulombs
1 to 3 ppb atom	1×10^{-6}
10 ppb atom	1×10^{-7}
100 ppb atom	1×10^{-8}
1000 ppb atom	1×10^{-9}
1 ppb atom	1×10^{-9}
10 ppb atom	1 x 10-10

Average detection limit has been about 2 parts per billion atom for a 1×10^{-6} coulomb exposure. Total elapsed time for a 1×10^{-6} coulomb exposure is approximately three hours.

3. Developing Plates

Remove the photographic plate from the instrument and transfer to the dark room. Develop the plate 3 minutes in D-19, short stop in dilute acetic acid, and fix 45 seconds in Kodak rapid fixer. Rinse the plate thoroughly with distilled water and place in an oven to dry for at least 10 minutes.

3. CALCULATION

1. Visual Estimation of Line Density

Visual estimation of line density is used for all low level impurities (~1.0 ppm atom) and many high level impurities. This type of interpretation usually gives data that is accurate within a factor of 2. This calculation is outlined as follows:

$$P.S. = \frac{E_{MIN}}{E_{MAX}} \times \frac{\%A_S}{100} \times \frac{\%I_S}{100} \times 10^6 \qquad (1)$$

where:

P.S. = plate sensitivity

E_{MIN} = the shortest exposure on the photographic plate in nanocoulombs

E_{MAX} = the longest exposure on the photographic plate in nanocoulombs

$\%A_S$ = concentration of chosen internal standard in atom percent

$\%I_S$ = isotopic abundance of the chosen isotope of the internal standard element

then,

$$C_i = P.S. \times \frac{E_{MAX}}{E_{DET}} \times \frac{100}{\%I_i} \qquad (2)$$

where:

C_i = concentration of impurity in ppm atom

P.S. = plate sensitivity

E_{MAX} = the longest exposure on the photographic plate in nanocoulombs

E_{DET} = the shortest exposure in nanocoulombs on which the impurity isotope can be detected

$\%I_i$ = isotopic abundance of the chosen impurity isotope

now,

$$W_i = C_i \times \frac{I_A}{M_A} \qquad (3)$$

where:

W_i = concentration of impurity in ppm weight

C_i = concentration of impurity in ppm atom

I_A = atomic weight of impurity

M_A = <u>average</u> atom weight of the matrix (Note: When mixed pressed samples are used, it is necessary to correct for the impurities present in the silver support material.)

2. <u>Measurement of Line Density</u>

When more accurate and precise values are required, it is necessary to measure line densities on the plate with a microphotometer. Line transmission is measured and the percent transmission is converted to line density with a previously determined emulsion calibration curve. From the measured line

densities, concentrations may be derived in two ways. If a standard with the same impurity elements as the sample is available, then

$$C_i = \frac{IU}{I_S} \times C_S \qquad (4)$$

where:

C_i = concentration of impurity in sample
I_U = density of impurity line in sample
I_S = density of impurity line in standard
C_S = concentration of impurity standard in sample

If the calculations are based on standard impurity elements other than those desired, the calculation becomes

$$C_i = \frac{D_i}{D_s} \times \frac{\%A_S}{100} \times \frac{\%I_S}{\%I_i} \times \frac{E_S}{E_i} \times \frac{S_S}{S_i} \times \frac{M_i}{M_s} \qquad (5)$$

where:

C_i = concentration of impurity in atom percent

D_i = density or intensity of impurity line

D_S = density or intensity of standard line

$\%A_S$ = concentration of standard in atom percent

$\%I_i$ = isotopic abundance of standard isotope

E_S = exposure in nanocoulombs at which standard line is measured

E_i = exposure in nanocoulombs at which impurity line is measured

S_S = relative sensitivity factor for standard element

S_i = relative sensitivity factor for impurity level

M_i & M_S = single to multiple charge ratio for the two elements in question (preferably 1)

3. PRECISION AND ACCURACY

The precision and accuracy of this method of analysis is widely variable. When visual estimation of line densities is used, the precision may vary from ±50 percent of the value to as much variation as a factor of 2 or 3. The accuracy may vary accordingly. If the microphotometer method is employed and suitable standards are available, the precision in 1.0 to 50 parts per million atom range is about ±25 percent with an accuracy of about ±15 to 20 percent. In the concentration range of 50 parts per million atom at a few atom percent the precision is about ±15 to 20 percent and the accuracy is approximately ±10 percent. When concentrations are below 1 part per million atom, precision is about a factor of 0.5 to 2.

c. General Procedure for Isotopic Analyses (Procedure Described is for Uranium; However, the Same General Procedure is Also Used for Other Actinide Materials)

(Uranium Isotopic Composition of Uranyl Nitrate Feed Solutions)

1. Scope

This method covers the determination of the isotopic composition of uranium in uranyl nitrate feed solutions.

2. Summary of Method

Thermal ionization mass spectrometry is based on the evaporation of positive ions from a heated surface. The ions are accelerated in the source by an electrostatic field, collimated into a ribbon beam, and injected into a magnetic field where they are separated by their mass. The ions are measured by a detector, and the mass spectrum can be obtained by scanning either the accelerating voltage or the magnetic field.

3. Equipment

A double magnetic deflection mass spectrometer with ion counting is used for the determination of the isotopic composition. The ions are accelerated by approximately 8 kV, and magnetically deflected through 2-90°, 30 cm radius stages. A slit between the two magnets allows only a single mass to be admitted into the second stage, resulting in a clean spectrum with little scattering.

The vacuum system is all metal except for a glass dome on the source envelope. Pumping is accomplished with an 80-liter ion pump on the source region and three 50-liter ion pumps on the analyzer region. The thermal ionization single-filament technique is used for all measurements. A "ferris wheel" arrangement allows for five samples to be installed into the source region at a time. Sample filaments are made from zone-refined rhenium sheet approximately 0.03 mm thick. A canoe shape about 0.5 mm wide and 6 mm wide is used.

Ions are detected by a 14-stage electron multiplier with copper-beryllium dynodes. A pulse preamplifier is mounted directly at the base of the multiplier and it feeds three wide-band amplifiers which transmit the pulses to a 400-channel analyzer. The pulses are accumulated in the 400-channel analyzer and then dumped onto a magnetic tape cartridge, which is then taken to an IBM 1130 computer for calculation of results.

4. Calibrations

The mass spectrometer is calibrated using NBS uranium isotopic standards. NBS U500 is used for determining the counting system dead time, and the "voltage correction" required because the accelerating voltage is swept. Other NBS standards are used for determining the accuracy and precision of the results.

5. Procedure

a. Place a drop of uranyl nitrate feed solution containing 10-100 ng uranium in a filament canoe, and dry under a heat lamp.

b. Install the filament in the mass spectrometer source, and evacuate to 10^{-5} torr.

c. Introduce benzene vapor into source region to a pressure of 10^{-4} torr. Heat filament to about 1500°C for 30 seconds. Remove benzene and continue evacuation.

This procedure reduces the oxide peak and alllows the determination to be made on the metal ions.

d. When source pressure reaches 10^{-7} torr, analyses may proceed.

e. Turn on accelerating voltage and voltage sweep panel. Turn up filament current until peak is located.

f. Focus on largest peak to obtain maximum intensity with good peak shape.

g. The 400-channel analyzer subgroups should be set so that each of the 50 channel subgroups scans the top of a peak. Set so that masses 233 to 240 are scanned.

h. Take required amount of data--usually 10 runs of 100 scans each. Data are automatically read onto magnetic tapes.

i. Take magnetic tape and required control cards to computer for calculation.

j. Check data, average results if more than one filament is run, and report results on "Isotope Analysis Report," UCN 1115.

A.2.2 Weighing Procedures Including Statistical Procedures for Establishing Weighing Uncertainties

As stated in Section 2.3, a substitution weighing scheme will be used to determine the weights of material per capsule. The recommended weighing scheme is that described by the National Bureau of Standards in a paper, "Design and Test of Standards of Mass" in Precision Measurement and

Calibration, Optics Metrology and Radiation Handbook 77, Vol. III, United States Department of Commerce (1961). In this scheme, three similar size specimens and a similar size standard weight are each weighed by themselves three times in a particular specified sequence. The balance reading is recorded each time. The zero reading is not recorded or adjusted, but is allowed to drift. This is because the weights of the unknown specimens are determined solely by differences between themselves and the standard.

The following FORTRAN program is used for data reduction. The final output is the three weights together with their 2σ uncertainties.

```
  1 I = 1
  5 J = 2
 10 K = 3
 20 READ (5, 30) TITLE
 30 FORMAT (20A4)
 40 READ (5, 50, END = 300) X, SEN, STD
 50 FORMAT (8F 10.5)
 60 A = SEN * (X(1) - X(2))
 70 B = SEN * (X(3) - X(4))
 80 C = SEN * (X(5) - X(6))
 90 D = SEN * (X(7) - X(8))
100 E = SEN * (X(9) - X(10))
110 F = SEN * (X(11) - X(12))
120 IF(ABS(X(2) - STD) . GE . 2.000) GO TO 300
130 W(I) = ((2.*A + B + C + D + E)/4.) + STD
140 W(J) = ((A + 2.* B + C - D + F)/4.) + STD
150 W(K) = ((A + C + 2.*C - E - F)/4.) + STD
```

```
160 R1 = A - (W(I) - STD)

170 R2 = B - (W(J) - STD)

180 R3 = C - (W(K) - STD)

190 R4 = D - (W(I) - W(J))

200 R5 = E - (W(I) - W(K))

210 R6 = F - (W(J) - W(K))

  C  U = UNCERTAINTY AT THE 95 PERCENT CONFIDENCE INTERVAL
     U = 3.182 (0.70711 * (SQRT((R1**2 + R2**2 + R3**2 + R4**2 +
         R5**2 1 + R6**2)/3.)

220 WRITE (6,230) TITLE

230  FORMAT (1H1, 'B DOS WGTS',//,1X,204A,//,
     1'SAMPLE MASS (MG)',/)

240  WRITE (6,250) I, W(I), J, W(J), K, W(K)

250  FORMAT (I7, F12.4,/,17,F12..4,/,17,F12.4(
     WRITE (6.255) SEN, STD, U

255  FORMAT ('SENSITIVITY = ',F7.4./,'STANDARD = ',F8.4,//,
     1' UNCERTAINTY =', 1PE12.5,/)

260  I = I + 3

270  J = J + 3

280  K = K + 3

290  GO TO 40

300  STOP

     END
```

All standard weights used in the dosimeter program are calibrated using Class M weight set M65829. This set was calibrated by Y-12 set M538000 which was calibrated by NBS set 232.09 - 212230 - A.

A.2.3 Loading Physics and Dosimeter Materials

Prior to loading the physics specimens, the vanadium capsules are weighed according to the weighting scheme detailed in A.2.2. Actinide oxide powders are loaded into the capsules using funneled powder loaders and the capsules are weighed again according to the substitution weighing scheme. The oxide powder weight is determined by difference.

Prior to loading the dosimeter specimens, small pieces of "oxide" wire (for the actinide dosimeter materials) or small pieces of metal wire or foil for the stable dosimeter materials are cut. Each piece is roughly weighed to verify that it is within the required weight range. The substitution weighing scheme is then used to determine the final weight and 2σ uncertainty. All physics and dosimeter specimens are loaded in helium.

A.2.4 TIG Welding

After the vanadium capsules are loaded with the desired dosimeter material, a vanadium plug is placed in the open end of each vanadium capsule. The capsules are transferred to a copper chill block in which approximately 0.5-0.8 mm of the top part of the capsules are exposed. The capsules are sealed using a power setting of approximately 4.5 amperes at 15 volts. The capsules are welded in a helium atmosphere.

A.2.5 Heat Treating Physics and Dosimeter Specimens

The welded capsules are placed in Al_2O_3 containers by batch inside a quartz tube. The tube is located in a clam shell furnace and is evacuated with a 0.14 m^3/min. roughing pump. After the tube is roughed to 10 microns, the furnace is closed and the specimens are heated to 800°C for one hour. After cooling, the dosimeters are removed and examined under a microscope at a setting of 10X. If any cracks are observed, the dosimeter is rejected.

A.2.6 Capsule Leak Testing

Each capsule undergoes a vacuum leak check to determine capsule integrity. Each vanadium capsule is placed in a small volumetric flask and completely immersed in an ethylene glycol solution. As the flask is evacuated, the vanadium capsule is observed to determine if air bubbles are present. If the capsule is leaking, air will be pumped out of the capsule and will result in air bubbles in the ethylene glycol solution. If air bubbles are observed, the capsule is rejected. This method will detect leaks as small as $\sim 10^{-4}$ cm^3/min.

A.2.7 Gamma Counting

In addition to weighing to determine the amount of actinide material present, each capsule is gamma counted. The gamma counts per milligram will be compared for each set of physics and dosimeter specimens. An average count per milligram will be obtained for each set and all specimens with a count per milligram variation $\geq$3% from the average count will be rejected.

A.2.8 Dimensional Measurements

Each completed capsule must meet the following specifications:

Material	Length (cm)	Diameter (cm)	Closed Hole Depth (cm)	Hole Diameter (cm)
Physics Specimens	0.762 (±0.020)	0.152 (+0.003 -0.000)	0.635 (+0.013 -0.025)	0.102 (+0.00 -0.003)
Dosimeter Inner Capsules	0.381 (±0.020)	0.089 (+0.005 -0.000)	0.279 (±0.025	0.051 (+0.005 -0.000)
Dosimeter Outer Capsules	1.003 (±0.020)	0.152 (+0.003 -0.000)	0.902 (±0.025	0.102 (+0.000 -0.003)

The hole depth and hole diameter are determined from radiographs. The capsule diameter is verified by dropping each capsule through a calibrated opening. A micrometer is used to verify final capsule lengths.

PROPOSED HEDL DOSIMETRY CAPSULE LOADING

D. L. Oberg - HEDL

Material	Approx. Mass (mg)	Approx. Length	(Dia.)
Fe	3.7	0.240 in.	0.035 in.
Ni	4.3	0.180 in.	0.035 in.
Cu	4.0	0.210 in.	0.035 in.
Ti	2.1	0.240 in.	0.035 in.
0.1% Ta/V	3.0	0.240 in.	0.035 in.
0.1% Sc_2O_3/MgO	1.1	0.180 in.	0.035 in.
0.1% Co/MgO	1.0	0.300 in.	0.035 in.
B	0.14	0.250 in.	0.050 in.
6Li	0.20	0.250 in.	0.050 in.
237Np	1.5	0.345 in.	0.050 in.
235U	1.5	0.125 in.	0.050 in.
239Pu	1.5	0.255 in.	0.050 in.

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