BNWL-994

UC-25

2-

PROMETHIUM-147 RADIOISOTOPE
APPLICATION PROGRAM
AMSA HEAT SOURCE
FINAL REPORT

March 1969

AEC RESEARCH &
DEVELOPMENT REPORT

DATE	BY	REMARKS

BNWL-994

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

PACIFIC NORTHWEST LABORATORY

RICHLAND, WASHINGTON

operated by

BATTELLE MEMORIAL INSTITUTE

for the

UNITED STATES ATOMIC ENERGY COMMISSION UNDER CONTRACT AT(45-1)-1830

3 3679 00061 2541

BNWL-994

UC-25, Metals, Ceramics,
and Materials

PROMETHIUM-147 RADIOISOTOPE APPLICATION PROGRAM
AMSA HEAT SOURCE
FINAL REPORT

By

N. C. Davis and D. W. Brite
Fuels and Materials Department
Chemistry and Metallurgy Division

March 1969

FORM 1
2000

BATTELLE MEMORIAL INSTITUTE
PACIFIC NORTHWEST LABORATORY
RICHLAND, WASHINGTON 99352

Printed in the United States of America
Available from
Clearinghouse for Federal Scientific and Technical Information
National Bureau of Standards, U.S. Department of Commerce
Springfield, Virginia 22151
Price: Printed Copy \$3.00; Microfiche \$0.65

PROMETHIUM-147 RADIOISOTOPE APPLICATION PROGRAM
AMSA HEAT SOURCE
FINAL REPORT

N. C. Davis and D. W. Brite

ABSTRACT

A promethium-147 fueled 60 W thermal source was fabricated for the Air Force for use in heating a navigation device for the Advanced Manned Strategic Aircraft (AMSA) Program. A high strength impact resistant capsule was designed, and a capsule fabrication process was developed. A series of dummy (Sm_2O_3 stand-in for $^{147}\text{Pm}_2\text{O}_3$) capsules were fabricated, impact tested, and destructively evaluated to demonstrate process quality and to determine impact strength. Process and quality control inspection procedures were established and the $^{147}\text{Pm}_2\text{O}_3$ fueled capsule was produced and delivered to the Air Force for laboratory testing and flight demonstration of AMSA navigational equipment.

TABLE OF CONTENTS

LIST OF FIGURES	vii
LIST OF TABLES	ix
INTRODUCTION.	1
SUMMARY	3
Capsule Development	3
Capsule Qualification	4
AMSA Radioisotope Capsule	5
CAPSULE DEVELOPMENT PHASE I	6
Design.	6
Powder Preparation.	8
Pressing and Sintering.	8
Cladding Components	12
Welding Development	17
Primary Welds	18
Secondary Welds	19
Alternate Process Development	23
QUALIFICATION CAPSULES PHASE II	25
Impact Tests and Postimpaction Evaluation	25
AMSA HEAT SOURCE FABRICATION PHASE III	29
Promethium Purification	29
Powder Preparation.	31
Pressing and Sintering.	32
Assembly of AMSA Heat Source Capsule	43
HANDLING AND SHIPPING	49
REFERENCES	55
ACKNOWLEDGEMENTS.	56
APPENDIX.	A-1

LIST OF FIGURES

1	AMSA Thermal Power Capsule	7
2	Sm_2O_3 Pellets	9
3	Split Sleeve Die	10
4	AMSA Pellet and Cladding Assembly Components	12
5	Electron Beam Welder	17
6	Typical Tantalum Electron Beam Seal Weld	18
7	L-605 Phase II Secondary Cladding Weld, Showing Optimized Penetration	22
8	HERF Die and Punches	23
9	Double Clad Capsule High-Energy Rate Formed, Ready for Final Machining	24
10	45° Impact Failure	26
11	45° Impact Failure as Sectioned	27
12	AMSA Capsule After Impact Against Concrete at 678 ft/sec	28
13	AMSA Radioisotope Encapsulation Process	30
14	$^{147}\text{Pm}_2\text{O}_3$ Powder Container	33
15	$^{147}\text{Pm}_2\text{O}_3$ Modified Die	34
16	$^{147}\text{Pm}_2\text{O}_3$ Glove Box with Press Attached	36
17	$^{147}\text{Pm}_2\text{O}_3$ Sintering Furnace	36
18	Promethium Pellet No. 1 as Pressed	37
19	Promethium Pellet No. 2 as Sintered	38
20	Promethium Pellet No. 1 Recovery Attempt	40
21	Promethium Pellet No. 2 as Sintered	41
22	Promethium Pellet No. 3 as Assembled	43
23	AMSA Capsule Inner Clad Welded	44
24	Inner Tantalum Weld, Test Capsule	45
25	Nondestructive Test Profile of Fueled Capsule	46
26	Outer L-605 Weld Test Capsule	47
27	Two Views of Final AMSA Fueled Capsule	50
28	Radiation Mapping	51
29	2R ICC Specified Container with Holding Fixture	52
30	Shipping Barrel	52

31	Radiation and Temperature Profile of Shipping Container	53
32	Radiation and Temperature Profile of 2R Container	54
A-1	Gamma Spectrum of 60 W, $^{147}\text{Pm}_2\text{O}_3$ AMSA Capsule	A-7

LIST OF TABLES

1	Sm ₂ O ₃ Pellets Pressing and Sintering Data	11
2	Primary Cladding Inspection Data	13
3	Secondary Cladding Inspection Data	14
4	Final Assembly Data on Sm ₂ O ₃ Capsules	15
5	Impact Test Data and Postimpaction Evaluation	20
6	Sm ₂ O ₃ Pellets - AMSA Phase II	34
7	¹⁴⁷ Pm ₂ O ₃ Pellets	39
A-1	Types of Quality Assurance Inspections	A-1
A-2	Fueled Capsule Component Test Results	A-1
A-3	Fueled Capsule Pellet Test Results	A-4
A-4	Fueled Capsule Assembly Test Results	A-5
A-5	AMSA Fueled Capsule Final Test Results	A-6
A-6	AMSA Capsule Calorimeter Data	A-8

PROMETHIUM-147 RADIOISOTOPE APPLICATION PROGRAM
AMSA HEAT SOURCE
FINAL REPORT

N. C. Davis and D. W. Brite

INTRODUCTION

Battelle-Northwest (Battelle Memorial Institute/Pacific Northwest Laboratory) was requested to design and fabricate a radioisotope heat source for an Air Force application involving the Advanced Manned Strategic Aircraft (AMSA). In this application, a promethium-147 heat source was proposed for use in a thermal preconditioner to maintain the navigation system at an elevated temperature.

The program required close coordination among personnel from the Air Force, AC Electronics (overall systems responsibilities), AEC Division of Isotope Development, Battelle-Northwest (capsule responsibility), and Sandia Laboratory (safety analyses and testing).

This document is the final report on the AMSA capsule development and fabrication program conducted by Battelle-Northwest.

The AMSA Radioisotope Capsule program was divided into three phases. The objectives of the program are listed under each phase as follows:

Phase I Capsule Development

- Prepare six to 10 Sm_2O_3 fueled capsules using two candidate fabrication processes: pressing and sintering, and high energy-rate forming.
- Impact test the Sm_2O_3 fueled capsules at Sandia to determine design and process suitability.

Phase II Capsule Qualification

- Refine capsule design, process parameters, and evaluation techniques based on Phase I results.
- Prepare 20 to 30 Sm_2O_3 fueled capsules.
- Impact test 20 Sm_2O_3 fueled capsules at Sandia for final qualification.

Phase III AMSA $^{147}\text{Pm}_2\text{O}_3$ Capsule

- Fabricate and deliver to the Air Force - one, 65 thermal watt $^{147}\text{Pm}_2\text{O}_3$ heat source for use in the AMSA application.

The major divisions in this final report correspond to the major divisions in the program (i.e., Phases I, II, and III). A final section has been added to illustrate handling and shipping procedures for the final heat source. Process development of the test capsules is discussed, destructive evaluation of the impact capsules is summarized, and special emphasis is placed on the Phase III final fabrication and quality assurance of the promethium-fueled heat source capsule.

SUMMARY

CAPSULE DEVELOPMENT

Promethium-147 was selected as the most desirable isotope due to low radiation dose rate with minimal shielding, fuel stability, and adequate power density in the designed size. The allowable design flexibility permitted a heavy wall, double clad pellet assembly that could be fabricated either by slip-fit assembly of sintered pellets or by High Energy Rate Forming (HERF) of the assembly.

The final capsule design and development resulted in a nearly-spherical (dome ends for impact resistance), 2 in. diameter and 2 in. long heat source with double containment cladding. The inner (primary) clad was fabricated from tantalum to provide radiation shielding, and the outer (secondary) clad was machined from L-605 (cobalt alloy) bar stock for high strength impact resistance. Preliminary process development and impact test results indicated that either the slip-fit assembly process or the high energy rate forming (HERF) process would be suitable in this fabrication program. The slip-fit assembly method was selected since the process was easier to develop and the simple approach was considered adequate for this application. The usual advantages of the HERF method, e.g., intimate contact between core and all claddings and higher core densities, were not considered necessary for this application.

Both the production and evaluation of the many process and impact test samples required in the development and qualification of an isotope heat source are simplified by using a nonradioactive stand-in applicable to the final heat source fuel form. Samarium oxide was chosen as a stand-in for the highly radioactive promethium because its chemical and physical

properties are very similar. Another pertinent consideration in the selection of a stand-in is the fact that samarium-147 is the stable product formed when promethium-147 decays.

CAPSULE QUALIFICATION

In the capsule development phase, the fabrication and evaluation of 10 capsules resulted in the capsule design and fabrication process selection. An additional 30 capsules were fabricated during the qualification phase of the program. Twenty of these were impact tested at the Sandia Laboratories. The total test program has shown:

- The capsule may reasonably be expected to provide fuel containment for all except the most adverse impact conditions.⁽¹⁾
- Capsule terminal velocity as determined by calculations and experimental drop tests is approximately 340 ft/sec.⁽¹⁾
- A critical impact angle exists at an altitude of 45° from the cylindrical axis of the capsule. The critical angle was determined experimentally by rocket sled impact tests against a hard granite target.
- At or near the critical impact angle, capsules impacted against granite at velocities as great as 275 ft/sec. remained intact.
- At other impact angles (0°, 67-1/2°, and 90°) against the hard granite target, the capsule did not fail at impact velocities up to about 500 ft/sec.
- At the critical impact angle, using 6000 psi compressive strength concrete as the target, capsules tested at 400 ft/sec and at 675 ft/sec were only superficially scratched, although the concrete targets were destroyed.
- At a random impact angle against a soil target, a single test at 625 ft/sec also resulted in only superficial scratching of the capsule.

- Post-testing evaluations indicated a high degree of capsule integrity, and thus a sound design and reproducible fabrication processes. The only impact failures were attributed to the 40,000 psi compressive strength granite targets at or near the critical impact angle. All capsules survived impacts against concrete and soil targets.

AMSA RADIOISOTOPE CAPSULE

A detailed process and quality assurance plan based on process technology established during the qualification phase of this program was placed in effect for the fabrication of the promethium fueled AMSA heat source. In working with the radioactive isotope, contamination and radiation problems were minor, but fabrication difficulties were encountered due to the self-heating characteristics of promethium. Progressive improvements were made in three attempts to produce a suitable one-piece pellet for the AMSA capsule. However, in each case, thermal gradients within the pressed pellet caused it to fracture and the final assembly was made with a three-piece pellet. Although assembly of the capsule was made more difficult by having to stack the three-piece pellet, the physical integrity of the completed capsule was not compromised.

The AMSA process flow diagram and quality control procedures were followed very closely in making the double cladding assembly. After decontamination of the primary cladding was effected and the first weld was made, the capsule was non-smearable for the balance of the assembly process. A detailed quality control inspection of the fueled capsule indicated that its integrity was equivalent to that of the qualification impact test capsules. The high degree of reliability developed for the nondestructive testing procedures used in making this determination was achieved by careful destructive evaluation of all of the impacted test capsules and correlation of the observed cladding integrity with the nondestructive testing data.

CAPSULE DEVELOPMENT PHASE I

A promethium-147 heat source capsule was fabricated for the Air Force to be used in a Thermal Preconditioning Unit (TPU) within the Advanced Manned Strategic Aircraft (AMSA). The TPU, a piece of auxiliary support equipment for an Inertial Measuring Unit (IMU) was designed to provide an improved navigation system reaction capability. The TPU reduces the time interval between the application of power to the navigation system and the attainment of specified performance by maintaining the navigation system at an elevated temperature. To accomplish this function, a promethium-147 source of 65 thermal watts initial power was designed, developed, and flight qualified.

DESIGN

The basic capsule dimensions were fixed by the TPU design, while the shape, including the dome ends, was designed to optimize impact resistance (Figure 1). Promethium-147 met the basic requirements of geometry, weight, and safety for the application and was considered a desirable isotope due to:

- A very low dose rate with only minimal shielding (beta emitter).
- Appropriate half-life.
- Adequate power density of the stable sesquioxide fuel form.
- Minimal design problems due largely to a demonstrated capability of producing heat sources of this size.
- Projected availability for potential scaleup requirements.

Tantalum, because of its shielding characteristics and its relative ease of fabrication was selected as the inner cladding. Tantalum radiation shielding 0.060 in. in thickness reduced the dose rate to 10 to 30 mR/hr at 1 m. A uniform 0.200 in. thick wall of L-605 alloy for the secondary outer cladding was

Neg 0681559

FIGURE 1. AMSA Thermal Power Capsule

selected to maximize impact strength within limits dictated by the total capsule size, by the fuel core volume, and by shielding requirements. This high strength cobalt alloy has outstanding corrosion resistance.

Another pertinent consideration in the capsule design was the weld location. The primary and secondary welds were offset from each other to minimize any possible shear plane of weakness through the capsule (Figure 1). The primary weld, intended only as a contamination seal, was offset from the center of the capsule. The secondary weld, which required the optimum impact resistance, was located at the center of the longitudinal axis of the capsule. This center location,

because of the capsule's dome end shape, afforded the greatest protection regardless of impact attitude.

This particular design resulted in an overall capsule size 2 in. in diameter and 2 in. long, with dome ends on a 0.645 in. outside radius. Dimensions of the inner capsule were chosen to allow adequate radiation protection and sufficient clearance for easy assembly while providing the maximum practical fuel volume. The resulting volume for the isotope, approximately 37 cm^3 , was adequate to provide the specified 65 W of thermal power using freshly purified promethium-147 oxide at a density with relative ease to 90 to 95% of theoretical density.

POWDER PREPARATION

Samarium oxide was used in all the development and qualification prototype capsules. A comparison between the physical and chemical characteristics of samarium oxide and promethium oxide indicated that processing results of both materials would be comparable except for the decay heating of the latter. Purity of the samarium, as received, was 99.0%. Prior to its use in the capsule development program, the oxide was subjected to further purification treatment similar to that used in preparing high purity promethium-147.

Additional powder processing included a consolidation step consisting of pressing the powder into pellets and then crushing them into agglomerated powder. The powder was then calcined for at least 4 hr at 1100°C to stabilize the monoclinic form. After this treatment, the powder showed a tap density of approximately 30% of theoretical. Theoretical density of monoclinic Sm_2O_3 is 7.74 g/cm^3 .

PRESSING AND SINTERING

The original fuel core concept was based on a stack of $^{147}\text{Pm}_2\text{O}_3$ pellets. These pellets could be pressed and sintered with relative ease to 90 to 95% of theoretical density.

The first series of 10 Sm_2O_3 capsules in Phase I was produced by the use of stacked pellets. Since handling problems were anticipated with the radioactive pellets, effort was made to increase the size of the individual pellets and thus minimize the number required for the 65 W thermal power source. Sm_2O_3 capsule loads consisting of two pellets were produced in Phase I and, in Phase II, one-piece double dome pellets were produced (Figure 2).

Neg 0672194-3

FIGURE 2. Sm_2O_3 Pellets (stand-in fuel)

The punch and die set (Figure 3) includes a tapered split sleeve which makes it possible to press and eject pellets without the addition of an organic binder.

Neg 0670660-8

FIGURE 3. *Split Sleeve Die*

A pressure of 65 tsi in the particular die produced pellets of approximately 75% TD. Pellets of this density would sinter to the correct length and diameter to fit in the capsule cladding. It was determined that 12 hr at 1350 to 1400 °C would reduce the pressed pellets at least 7% in diameter and 6% in length, and result in a final density of approximately 95% TD.

Table 1 summarizes the dimensional changes resulting from sintering the Sm_2O_3 pellets. In this as well as in subsequent tables, the identification system was continued (with No. 11 forward) to include all the pellets fabricated for the qualification phase of the program. The capsule assembly number noted in Table 1 applies to the final capsule identification number

TABLE 1. *Sm₂O₃ Pellets Pressing and Sintering Data*

Pellet No.	Pressed Dimensions, in.		Sintering Data		Final Dimensions, in.		Weight, g	Capsule Assembly No.
	Diameter	Length	Time, hr	Temp., °C	Diameter	Length		
1								1
2								2
3								3
4								4
5								5
6								6
7								7
8								8
9								9
10								10
11*	1.549	1.533	6	1350	1.445	1.459	249.7	25
12*	1.549	1.515	6	1350	1.452	1.446	249.6	24
13*	1.550	1.533	6	1350	1.445	1.450	249.5	21
14*	1.549	1.524	6	1350	1.446	1.441	249.9	26
15*	1.550	1.524	6	1350	1.452	1.455	249.2	28
16*	1.549	1.528	6	1350	1.448	1.450	249.5	30
17	1.549	1.524	9	1350	1.438	1.445	249.9	36
18*	1.549	1.539	9	1350	1.439	1.454	249.7	29
19*	1.549	1.525	9	1350	1.440	1.443	256.6	22
20	1.550	1.524	9	1350	1.440	1.432	249.3	34
21	1.549	1.524	9	1350	1.440	1.442	250.0	14
22	1.549	1.521	9	1350	1.441	1.454	250.0	12
23	1.551	1.521	21	1350	1.435	1.439	249.9	13
24	1.552	1.521	21	1350	1.443	1.440	250.0	23
25	1.550	1.524	21	1350	1.436	1.444	250.0	27
26	1.551	1.517	21	1350	1.443	1.445	250.0	32
27	1.553	1.515	21	1350	1.442	1.440	250.0	35
28	1.551	1.513	21	1350	1.443	1.450	250.1	16
29	1.552	1.521	14	1400	1.433	1.427	249.9	17
30	1.552	1.522	14	1400	1.435	1.428	249.8	31
31	1.551	1.524	14	1400	1.433	1.428	249.9	20
32	1.553	1.520	14	1400	1.433	1.427	249.9	15
33	1.551	1.520	14	1400	1.432	1.426	249.8	33
34	1.551	1.520	14	1400	1.432	1.426	250.0	19
35*	1.551	1.523	14	1400	1.434	1.432	249.6	37
36	1.552	1.524	14	1400	1.430	1.433	250.2	11
37	1.551	1.526	12	1450	1.430	1.440	249.3	18
38	1.552	1.533	12	1450	1.438	1.446	249.8	40
39	1.551	1.522	12	1450	1.435	1.438	249.3	39
40**	1.570	1.477	12	1450	1.443	1.400	246.0	Test Weld
41	1.570	1.483	12	1450	1.447	1.425	246.0	Test Weld
42	1.570	1.507	12	1450	1.440	1.440	246.0	Test Weld

* Pellets with broken domes. Other pellets were one-piece. However, micro cracks were evident in many of the single-piece pellets.

** Phase III - larger diameter die

used throughout the program. All additional tables in this report containing component data and final impact test results carry the same capsule assembly number. These tables thus provide a complete fabrication record of the components used in each capsule, as well as a final postimpaction evaluation.

CLADDING COMPONENTS

Both the tantalum primary cladding and the L-605 alloy secondary cladding were machined from bar stock. Cladding and pellet assembly components are shown in Figure 4. Dimensional inspection data of primary and secondary claddings are given in Tables 2 and 3, respectively. Final assembly data on all Sm_2O_3 developmental capsules are given in Table 4.

Neg 0671707-2

FIGURE 4. AMSA Pellet and Cladding Assembly Components

TABLE 2. Primary Cladding Inspection Data

Inner Clad No.	Dimensions, in.				Wall, in.	Weight, g	Capsule Assembly No.
	Outside Diameter	Outside Length	Diameter	Length			
1	1.598	1.595	1.458	1.456	0.077	180.3	1
2	1.597	1.652	1.457	1.510	0.075	183.3	2
3	1.595	1.600	1.456	1.457	0.082	184.2	7
4	1.592	1.591	1.457	1.455	0.079	183.1	9
5	1.597	1.648	1.459	1.506	0.072	177.4	3
6	1.600	1.597	1.458	1.457	0.075	182.4	8
7	1.596	1.596	1.459	1.460	0.080	166.2	10
8	1.595	1.643	1.460	1.510	0.073	168.4	4
9	1.596	1.645	1.458	1.509	0.074	179.3	5
10	1.594	1.655	1.457	1.508	0.072	173.7	6
11	1.595	1.601	1.460	1.461	0.071	165.0	14
12	1.596	1.599	1.461	1.459	0.071	162.9	12
13	1.597	1.604	1.459	1.463	0.071	167.2	13
14	1.593	1.599	1.459	1.460	0.070	155.5	11
15	1.596	1.604	1.460	1.460	0.073	166.1	23
16	1.597	1.600	1.459	1.460	0.071	165.0	16
17	1.596	1.699	1.458	1.459	0.071	164.8	17
18	1.595	1.600	1.459	1.455	0.074	165.6	27
19	1.590	1.600	1.460	1.458	0.074	162.3	15
20	1.598	1.601	1.458	1.458	0.074	169.0	20
21	1.598	1.600	1.460	1.455	0.073	170.5	22
22	1.598	1.601	1.460	1.457	0.073	170.5	18
23	1.596	1.600	1.460	1.463	0.072	167.5	25
24	1.598	1.603	1.459	1.459	0.073	172.0	24
25	1.597	1.602	1.461	1.459	0.073	167.5	35
26	1.597	1.603	1.460	1.458	0.073	171.0	26
27	1.597	1.602	1.459	1.460	0.071	170.0	29
28	1.598	1.601	1.459	1.457	0.072	171.0	19
29	1.596	1.600	1.460	1.455	0.073	170.5	21
30	1.598	1.601	1.459	1.460	0.071	171.5	30
31	1.597	1.601	1.460	1.458	0.072	171.0	31
32	1.596	1.601	1.459	1.460	0.071	170.5	37
33	1.596	1.600	1.459	1.458	0.071	168.5	33
34	1.597	1.600	1.459	1.460	0.071	166.5	34
35	1.597	1.600	1.460	1.459	0.072	171.0	32
36	1.596	1.599	1.460	1.460	0.070	172.5	28
37	1.596	1.600	1.459	1.458	0.072	168.5	36
38	1.596	1.600	1.460	1.459	0.071	168.5	Test Weld
39	1.595	1.599	1.461	1.456	0.072	170.0	39
40	1.598	1.598	1.460	1.459	0.070	170.0	40
41	1.596	1.597	1.458	1.457	0.072	165.6	Test
42	1.599	1.599	1.464	1.459	0.069	160.7	
43	1.600	1.598	1.462	1.458	0.070	160.8	
44	1.600	1.600	1.463	1.459	0.069	158.9	
45	1.598	1.595	1.460	1.458	0.067	164.4	
46R	1.599	1.597	1.461	1.460	0.071	166.3	

TABLE 3. *Secondary Cladding Inspection Data*

Capsule Assembly No.	Dimensions, in.				Wall, in.		Weight, g
	Outside		Inside		Maximum	Minimum	
	Diameter	Length	Diameter	Length			
1	2.005	2.004	1.601	1.601	0.206	0.192	375.6
2*	2.000	2.070	1.600	1.655	0.203	0.200	524.9
3*	2.001	2.075	1.598	1.653	0.202	0.200	528.9
4*	2.000	2.072	1.596	1.658	0.210	0.200	527.2
5*	2.000	2.071	1.599	1.648	0.207	0.198	524.9
6*	1.999	2.076	1.602	1.660	0.210	0.201	525.5
7	2.000	1.999	1.600	1.601	0.205	0.199	374.1
8	2.001	2.006	1.598	1.602	0.208	0.198	374.5
9	2.000	2.003	1.601	1.601	0.203	0.199	372.5
10	2.003	2.001	1.600	1.604	0.205	0.200	373.9
11	1.999	2.007	1.600	1.607	0.200	0.180	362.5
12	2.000	2.004	1.599	1.602	0.201	0.200	375.0
13	1.998	2.005	1.598	1.602	0.202	0.199	373.0
14	1.998	2.007	1.600	1.603	0.202	0.199	374.5
15	1.999	2.003	1.601	1.601	0.203	0.199	373.5
16	1.999	2.003	1.598	1.604	0.200	0.199	374.7
17	1.999	2.002	1.600	1.602	0.200	0.199	375.9
18	1.999	2.004	1.600	1.604	0.201	0.198	375.2
19	1.998	2.003	1.602	1.604	0.200	0.198	375.6
20	1.998	2.006	1.600	1.604	0.201	0.199	374.6
21	1.999	2.005	1.600	1.604	0.201	0.199	372.8
22	1.999	1.997	1.599	1.601	0.201	0.195	374.0
23	1.998	2.001	1.601	1.602	0.200	0.199	374.5
24	1.998	2.000	1.600	1.605	0.199	0.196	374.5
25	1.999	2.001	1.601	1.604	0.200	0.198	347.0
26	1.999	1.998	1.601	1.601	0.201	0.196	373.5
27	1.999	2.002	1.601	1.605	0.199	0.198	373.5
28	1.998	1.999	1.600	1.604	0.198	0.197	375.0
29	1.998	2.001	1.600	1.608	0.198	0.196	375.5
30	1.998	2.002	1.600	1.607	0.199	0.196	376.0
31	1.999	2.000	1.599	1.604	0.200	0.197	375.0
32	1.998	2.002	1.600	1.609	0.199	0.196	375.0
33	1.997	1.998	1.600	1.601	0.200	0.197	374.5
34	1.998	2.000	1.601	1.606	0.199	0.197	374.5
35	1.998	2.001	1.600	1.608	0.199	0.196	375.5
36	1.998	2.002	1.600	1.607	0.198	0.197	375.0
37	1.998	2.000	1.601	1.605	0.199	0.196	373.5
38	1.998	2.000	1.601	1.607	0.199	0.196	376.5
39	1.998	2.002	1.600	1.608	0.198	0.197	375.7
40	1.998	2.002	1.600	1.608	0.198	0.197	374.5

* Capsule Assemblies No. 2 through 6 were processed by an alternate High Energy Rate forming process. The final dome ends were machined after HERF.

TABLE 4. Final Assembly Data on Sm_2O_3 Capsules

AMSA Assembly No. 1*	Inner Clad No. 2**	Pellet No. 3***	Final Dimensions, in.		Weight, g	Comments
			Diameter	Length		
						NOTE: HERF = High Energy Rate forming
1	1	1	2.005	2.004	793.9	Phase I
2	2	2	-	-	-	HERF and sectioned
3	5	3	2.001	2.001	766.3	HERF and tested
4	8	4	2.000	1.951	769.8	HERF and tested
5	9	5	2.000	1.912	756.2	HERF and tested
6	10	6	1.999	1.918	770.3	HERF and tested
7	3	7	2.000	1.999	779.8	
8	6	8	2.001	2.006	773.8	
9	4	9	2.000	2.003	782.5	
10	7	10	2.003	2.001	786.2	
11	14	36	1.999	2.007	-	Destructive evaluation
12	12	22	2.000	2.004	789.5	Phase II
13	13	23	1.998	2.005	791.5	
14	11	21	1.998	2.007	791.0	
15	19	32	1.999	2.003	790.5	
16	16	28	1.999	2.003	794.0	
17	17	29	1.999	2.002	795.3	
18	22	37	1.999	2.004	796.0	
19	28	34	1.998	2.003	797.0	
20	20	31	1.998	2.006	799.0	
21	29	13	1.999	2.005	789.5	
22	21	19	1.999	1.997	-	Destructive evaluation
23	15	24	1.998	2.001	-	Destructive evaluation
24	24	12	1.998	2.000	781.0	
25	23	11	1.999	2.001	-	Destructive evaluation
26	26	14	1.999	1.998	781.2	
27	18	25	1.999	2.002	791.0	
28	36	15	1.998	1.999	786.0	Reject
29	27	18	1.998	2.001	785.0	
30	30	16	1.998	2.002	790.1	
31	31	30	1.999	2.000	796.2	
32	35	26	1.998	2.002	788.0	
33	33	33	1.997	1.998	793.2	
34	34	20	1.998	2.000	-	Destructive evaluation
35	25	27	1.998	2.001	786.1	Reject
36	37	17	1.998	2.002	787.0	
37	32	35	1.998	2.000	790.0	
38	-	-	-	-	-	Not loaded
39	39	39	1.998	2.002	771.0	
40	40	38	1.998	2.002	785.0	

* This is the final No. assigned to capsule (same as outer clad No., Table 3)

** Inner clad No. (Table 2)

*** Pellet No. (Table 1)

The cladding components were cleaned ultrasonically using trichlorethylene as a degreaser with a final rinsing in absolute ethyl alcohol. All parts were dried using a hot air gun. Clean, white cotton gloves were used for all subsequent handling steps through final welding. These assembly steps and procedures using Sm_2O_3 pellets were developed with particular concern for the potential problems in performing the final radioisotope encapsulation.

A 1 to 2 mil thick tantalum foil liner was inserted around the inside diameter of the inner clad to protect the weld zone after pellet assembly and during welding. This technique, due to the potential for smearable contamination, was critically important.

Inspection points were established and data recorded to maintain quality as well as to correlate process steps with impact test results. In the developmental phase, minor dimensional discrepancies did not necessarily cause rejection, but all imperfections were recorded for subsequent process evaluation.

Tantalum and L-605 alloy bar stock were inspected by dye penetrant inspection of discs cut from the ends of the bars. Dye penetrant inspection of machined components was required prior to use to eliminate use of faulty material.

Other inspections during fabrication were used as controls on process development and quality assurance. More than the required number of capsules were fabricated for impact tests so that representative capsules would be available for destructive evaluation throughout the process. These capsules were first examined by a nondestructive, ultrasonic flaw detection technique, and the results were confirmed by metallographic examination of weld areas. Although only a limited number of capsules were sectioned prior to impact testing, all of the

impacted capsules were sectioned for final evaluation. Excellent correlation existed between nondestructive test results and observed weld microstructures. The high reliability of the ultrasonic test procedures, and the results of impact tests of capsules having known small voids or defects established the welding acceptance criteria for the final Phase III $^{147}\text{Pm}_2\text{O}_3$ capsule.

WELDING DEVELOPMENT

Development of seal welding by electron beam equipment (Figure 5) may be separated into three major areas:

- Desired penetration,
- Elimination or minimization of defects, and
- Reproducibility.

Neg 0662358-3

FIGURE 5. Electron Beam Welder

Primary Welds

The seal weld in the tantalum primary cladding is mainly for contamination containment. Release of radioisotopic material during welding is highly undesirable because of equipment contamination, formation of weld inclusions, and degradation of joint integrity. High penetration welds, therefore, could not be used.

Welding experience during Phases I and II of the program indicated that penetration equal to 50% of the wall thickness was a desirable compromise of the various factors. Parameters for achieving the typical weld shown in Figure 6 were as follows:

<u>Beam Potential (kV)</u>	<u>Beam Current</u>	<u>Transverse Beam Deflection</u>	<u>Face Speed</u>
135 (no load)	7.5 mA	16 mils	30 ipm

Defects and reproducibility were not problems because of minimum penetration requirements.

Neg 567-1028

36X

FIGURE 6. *Typical Tantalum Electron
Beam Seal Weld*

Secondary Welds

Initial problems in development of electron beam welds for the L-605 secondary cladding were (1) elimination of excess voids, and (2) reproducible penetration.

Initial test welds were made on joints having a 15 mil step, 100 mils from the inner surface. A 30 mil deflection of the finely focused beam resulted in fully-penetrated welds approximately 43 mils wide.

Postimpaction evaluation of Phase I capsules 1 through 10 (see Table 5) showed three problems:

- Penetration was neither adequate nor consistent.
- Large root voids existed.
- An undesirable "tooth root" weld configuration existed.

A few of the Phase I outer cladding welds exhibited good penetration and an absence of voids. The parameters for these welds, (unintentionally differing from others in beam focus) appeared desirable for subsequent welding. The requisite change in parameters consisted of defocusing the beam by shifting its focal point upward (consequently increasing spot size at the face) and reducing the transverse deflection to 16 mils. Although the new parameter resulted in welds of approximately the same width as those previously made, penetration and weld geometry were perfected and voids were minimized. A typical example of the final weld design utilizing a 15 mil step 15 mils from the inner surface is shown in Figure 7.

Optimized parameters for Phase II electron beam welding of L-605 alloy were:

<u>Beam Potential (kV)</u>	<u>Beam Current</u>	<u>Transverse Beam Deflection</u>	<u>Face Speed</u>
142.5 (no load)	13.5 mA	0.016 in.	24 ipm

TABLE 5. Impact Test Data and Postimpaction Evaluation

AMSA Capsule No.	Impact Angle	Velocity ft/sec	Target Material	Results	Postimpaction Weld Inspection and Comments
1	22-1/2	414	LV granite	intact	90% pent. - excellent weld
2	not tested	-	-	-	Destructive evaluation
3	22-1/2	400	ES granite	intact	80% pent. - weld root voids
4	45	396	LV granite	intact	45% pent. - weld reject
5	0	396	ES granite	intact	95% pent. - excellent weld
6	67-1/2	398	ES granite	intact	95% pent. - excellent weld
7	45	406	ES granite	rupture	50% pent. - reject
8	0	403	ES granite	intact	70% pent. - reject
9	67-1/2	407	ES granite	intact	40% pent. - reject
10	90	410	ES granite	intact	75% pent. - weld root voids
11	not tested	-	-	-	Destructive evaluation
12	0	494	ES granite	intact	90% pent. - excellent weld
13	22-1/2	494	ES granite	rupture	90% pent. - excellent weld
14	45	479	ES granite	rupture	85% pent. - excellent weld
15	67-1/2	479	ES granite	intact	95% pent. - excellent weld
16	0	485	ES granite	intact	95% pent. - small voids
17	22-1/2	485	ES granite	rupture	95% pent. - excellent weld
18	45	487	ES granite	rupture	95% pent. - small voids
19	45	381	ES granite	rupture	95% pent. - excellent weld
20	90	487	ES granite	intact	95% pent. - ~0.030 in. void
21	45	329	ES granite	intact	95% pent. - excellent weld
22	not tested	-	-	-	Destructive evaluation
23	not tested	-	-	-	Destructive evaluation
24	22-1/2	429	ES granite	intact	90% pent. - excellent weld
25	not tested	-	-	-	Destructive evaluation
26	35	388	Es granite	rupture	90% pent. - 0.010-0.015 in. voids
27	45	429	ES granite	rupture	95% pent. - excellent weld
28	30	630	soil	intact	95% pent. - excellent weld
29	45	285	ES granite	rupture	85% pent. - excellent weld
30	55	388	ES granite	rupture	85% pent. - excellent weld
31	35	374	ES granite	intact	95% pent. - excellent weld
32	45	374	ES granite	rupture	95% pent. - excellent weld
33	45	323	ES granite	rupture	85% pent. - 0.010-0.015 in. voids
34	45	392	concrete	intact	90% pent. - excellent weld
35	not tested	-	-	-	Destructive evaluation
36	not tested	-	-	-	Wind tunnel test - WPAFB
37	45	312	ES granite	rupture	95% pent. - excellent weld
38	45	285	ES granite	intact	95% pent. - 0.030 in. void
39	-	-	-	-	Not loaded
40	45	678	concrete	intact	85% pent. - small voids
	45	650	concrete	intact	Not sectioned

TABLE 5. (contd)

AMSA Capsule No.		Postimpaction Inner Clad Condition	Inspection Fuel Pellet Condition	Type of Fuel Core
1		Minor damage	Fractured	Multi pellet stack
2	not tested	-	-	Multi pellet stack
3		Excellent	Fractured	Multi pellet stack
4		Excellent	Fractured	Multi pellet stack
5		Excellent	Fractured	Multi pellet stack
6		Severe damage	Fractured	Multi pellet stack
7		Rupture	Fractured	Multi pellet stack
8		Excellent	Fractured	Multi pellet stack
9		Severe damage	Fractured	Multi pellet stack
10		Severe damage	Fractured	Multi pellet stack
11	not tested	-	-	Multi pellet stack
12		Excellent	Minor damage	Intact pellet - one piece*
13		Rupture	Minor damage	Intact pellet - one piece
14		Rupture	Fractured	Intact pellet - one piece
15		Severe damage	Fractured	Intact pellet - one piece
16		Excellent	Minor damage	Intact pellet - one piece
17		Rupture	Fractured	Intact pellet - one piece
18		Rupture	Fractured	Intact pellet - one piece
19		Rupture	Fractured	Intact pellet - one piece
20		Excellent	Minor damage	Intact pellet - one piece
21		Minor damage	Fractured	Broken dome pellet
22	not tested	-	-	Broken dome pellet
23	not tested	-	-	Intact pellet - one piece
24		Minor damage	Minor damage	Broken dome pellet
25	not tested	-	-	Broken dome pellet
26		Rupture	Fractured	Broken dome pellet
27		Rupture	Fractured	Intact pellet - one piece
28		Rupture	Fractured	Broken dome pellet
29		Rupture	Fractured	Broken dome pellet
30		Excellent	Fractured	Broken dome pellet
31		Rupture	Fractured	Intact pellet - one piece
32		Rupture	Fractured	Intact pellet - one piece
33		Excellent	Fractured	Intact pellet - one piece
34	not tested	-	-	Intact pellet - one piece
35	not tested	-	-	Intact pellet - one piece
36		Rupture	Fractured	Intact pellet - one piece
37		Rupture	Fractured	Broken dome pellet
39		Excellent	Fractured	Intact pellet assembly
40		Not sectioned		Intact pellet assembly

* Intact pellets were one-piece. However, micro-cracks were evident in many of them.

NOTES: Impact angle between normal to target face and cylindrical axis of capsule.

All capsules preheated to nominal 300 °F.

Target material included Escondido and Las Vegas granite and 6,000 psi concrete and soil.

Soil test 28 was repeated as test 28A into granite.

Postimpaction comments are a result of microscopic examination of outer welds (36X) after impact tests.

Neg 567-849A and B

36X

FIGURE 7. *L-605 Phase II Secondary Cladding
Weld, Showing Optimized Penetration*

ALTERNATE PROCESS DEVELOPMENT

Capsules 2, 3, 4, 5, and 6 were fabricated by an alternate process using high-energy rate forming (HERF). This process appeared to have potential for improving impact resistance. The pressed and sintered cores were seal-welded into a double-clad tantalum and L-605 billet. The billet was then heated to 1100 °C and consolidated by HERF using pressures in excess of 200,000 psi. The HERF punch and die components are shown in Figure 8. The HERF consolidation process effected intimate interfacial contact between the billet components and increased the billet diameter slightly (Figure 9). Because of this increase in diameter, it was necessary to remove as much as 0.025 in. to 0.030 in. from the outer cladding thickness

Neg 0670660-4

FIGURE 8. HERF Die and Punches

during final machining. This loss of wall thickness apparently did not degrade the impact resistance, since these capsules did not rupture during impact testing (Table 5). Although the HERF process could probably have been perfected to yield as-formed shapes to more exacting dimensions, fewer process variables were involved in the slip-fit and weld assembly process. The program time schedule dictated adoption of the simpler approach. The slip-fit system was considered completely adequate for this isotope application.

Neg 0670692-2

FIGURE 9. *Double Clad Capsule High-Energy Rate Formed,
Ready for Final Machining*

QUALIFICATION CAPSULES PHASE II

The Phase II capsule qualifications for this program overlapped considerably into the final evaluation of the design and development Phase I. The basic design established at the beginning of the program did not change. Program time schedules did not allow major changes or experimentation, however, the only questionable areas after the completion of Phase I were process welding problems and improved detection of defects by nondestructive testing methods.

By means of an extensive review of postimpaction weld inspections (Table 5), the nondestructive ultrasonic test was refined to identify both the degree of weld penetration and voids as small as 0.010 in. in size.

The process flow was revised to delete the alternate HERF process and to add the nondestructive testing inspections. With this plan in effect, the qualification Phase II capsules were fabricated. The same lot of samarium was used for producing all core pellets (Table 1) and the same heat of material was used for all of the cladding components (Tables 2 and 3). The chemical analyses of these materials are included in the detailed quality control summary of the fueled capsule in the Appendix of this report.

IMPACT TESTS AND POSTIMPACTION EVALUATION

Table 5 (Impact Test Data and Postimpaction Evaluation) provides an overall summary of the individual tests. This table provides information regarding impact conditions, post-impaction inspection, and fabrication history for every capsule. The AMSA capsule number is a control number that can be referenced to previous Tables 1 through 4 for pellet, inner and outer cladding, and assembly data.

Details of the impact testing are covered in W. J. Dalby's Sandia impact test report.⁽¹⁾ Fuel containment except in the most adverse impact conditions, can be expected. A typical example of an impact failure is shown in Figures 10 and 11. When impacted against a granite target at, or near the critical impact angle (45° from the cylindrical axis of the capsule), and at a velocity exceeding 80% of terminal velocity, a capsule may be expected to fail. These conditions involve the extremely hard (40,000 psi compressive strength) Escondido granite. The impact deformed surface area increases as the impact attitude is shifted in either direction from the critical 45° position. The increased surface area results in a reduced impaction energy absorption per unit area.

Neg 0671777-25

FIGURE 10. 45° Impact Failure

Neg 0671797-37

FIGURE 11. 45° Impact Failure as Sectioned

The performance of the capsule against 6,000 psi concrete and soil targets at the critical impact angle (Table 5) presents an entirely different set of test results. These capsules remain intact at velocities as high as 678 ft/sec or approximately twice the terminal velocity. A capsule after impact at this velocity against 6,000 psi concrete is shown in Figure 12.

Neg 0683430-1

FIGURE 12. *AMSA Capsule After Impact Against
Concrete at 678 ft/sec*

AMSA HEAT SOURCE FABRICATION PHASE III

At the completion of the impact qualification testing evaluation, a complete review of the process technology and quality control requirements was made. A AMSA radioisotope encapsulation, process description, and quality control plan was prepared (Figure 13).

Process fabrication steps were frozen. All the pertinent quality control data covering test results of the 14 types of inspections indicated on the flow chart (Figure 13) are exhibited either in the following test or accumulated in the appendix.

PROMETHIUM PURIFICATION

The rare earth elements resulting from nuclear fission and decay are isolated from Hanford Purex Plant waste by utilizing a series of successive precipitation processes.^(2,3) After storage to allow for decay of the gamma emitting ^{148}Pm , the rare earth crude cut is further refined by solvent extraction,⁽³⁾ thermal concentration, and "acid kill"⁽⁴⁾ steps prior to transfer to the Pacific Northwest Laboratory's Hot-Cell Facility.

Isolation of promethium from its adjacent rare earth neighbors and from all other impurities is accomplished at BNW by a displacement cation exchange process⁽⁵⁾ utilizing an ammonia buffered solution of the complexing agent DTPA* as the separating agent. The separation is conducted in a seven-column ion-exchange facility installed, together with associated tanks, valves, and instrumentation, in a heavily shielded remotely operated cell.

With the very high ^{144}Ce and ^{154}Eu separation factors achieved in the ion-exchange process, purified promethium does not require extensive shielding from the final conversion

---

* [(Carboxymethylimino)bis (ethylenenitrilo)] tetra-acetic acid.

1. Dimension Measurements	6. Metallography	11. Decon. Smear Check
2. Dye Penetrant	7. Isotopic Analysis	12. Radiation Mapping
3. Impurity Analysis	8. X-Ray Diffraction	13. Gamma Spectrometry
4. Density Determination	9. Radiography	14. Calorimetry
5. He Leak Check	10. Ultrasonic Testing	

Neg 0674164

FIGURE 13. AMSA Radioisotope Encapsulation Process

to the sesquioxide. The promethium product solution, collected as it flows from the final purification column, is transferred to the lightly shielded glove box facilities for oxide conversion to the oxide.

Radiochemical analysis, corrected to November 3, 1967, indicated the AMSA $^{147}\text{Pm}_2\text{O}_3$ capsule to contain:

- ^{147}Pm : 168,200 Ci
- ^{146}Pm : $0.25 \pm 0.2 \times 10^{-6}$ Ci/Ci ^{147}Pm or 0.042 Ci
- ^{148}Pm : Ci ^{148}Pm /Ci $^{146}\text{Pm} \leq 0.04$ or <0.002 Ci
- ^{154}Eu : 3×10^{-9} Ci/Ci ^{147}Pm or 5×10^{-4} Ci
- ^{144}Ce : $<10^{-9}$ Ci/Ci ^{147}Pm or $<2 \times 10^{-4}$ Ci

The ^{147}Sm daughter, although constituting the principal impurity, was less than 1% on the date of promethium purification. Non-rare earth impurities spectrographically detected were 0.02% Al, $<0.001\%$ Fe, 0.005% Mg, and $<0.005\%$ Si and, in aggregate, constituted less than 1% of the oxide. X-ray analysis of the treated oxide confirmed the crystal structure to be monoclinic.

POWDER PREPARATION

The purified $^{147}\text{Pm}_2\text{O}_3$, after the conversion and heat treatment, is difficult to control due to its extremely small particle size (approximately 1 micron). This physical characteristic of the promethium powder, as with the stand-in Sm_2O_3 , makes it difficult to press to the optimum density for sintering. In the case of $^{147}\text{Pm}_2\text{O}_3$, the radiation exposure to glove box operators increases with spread of the highly mobile $^{147}\text{Pm}_2\text{O}_3$ powder around the glove box. The micronized powder can thus create a difficult exposure problem. This problem is reduced when the powder is consolidated by a prepressing (slugging) operation. Upon crumbling this material to a specified particle size range, a relatively dustless, free-flowing powder is obtained which may then be pressed a

second time into the shape and volume required to produce the final sintered density needed for assembly.

The parameters to produce the most favorable powder load for the AMSA prepressing die appeared to be 25 tons/in.² (tsi) in increments of 35 to 40 g of $^{147}\text{Pm}_2\text{O}_3$. This would produce a density of 55 to 60% TD dependent on the increment powder load. These pellets were crushed and screened through a 12 mesh stainless steel screen to produce a granular powder with a tap density of approximately 30% TD.

A special calibrated powder container was fabricated to transfer the $^{147}\text{Pm}_2\text{O}_3$ powder from the screening step to the die loading operation. This container (Figure 14) had a volume sufficient to contain the required powder for a die load. By means of the slide release at the bottom of this container, the die could be loaded with a minimum of powder loss, thus reducing the spread of ^{147}Pm throughout the glove box.

PRESSING AND SINTERING

The die design used for the stand-in Sm_2O_3 pellet fabrication was modified for use in the pressing of the promethium pellets. This punch and die set (Figure 15), utilizing a tapered and split sleeve, was very successful in producing highly dense Sm_2O_3 pellets for the impact test capsules.

An increase in die diameter was predicted on the basis of earlier experience indicating a lower pressed density and a greater radial reduction during the sintering of promethium compared to samarium. The average diameter reduction with Sm_2O_3 during the present program was approximately 7%, while the length reduction was slightly greater than 5%. These data, summarized and reproduced from Part I of this report, are shown in Table 6. Previous experience with $^{147}\text{Pm}_2\text{O}_3$ pellets

Neg 0672551-1

FIGURE 14. $^{147}\text{Pm}_2\text{O}_3$ Powder Container

having diameters 25% less than the AMSA pellet and lengths of 0.25 in. showed diameter reductions of 9% and length reductions on the order of 7%.

By correlating this experience with the Sm_2O_3 sintering data from the present program, it appeared that greater shrinkage would be experienced with the $^{147}\text{Pm}_2\text{O}_3$ AMSA pellet during sintering. Since the Sm_2O_3 AMSA pellets were assembled in the inner cladding, with extra tolerance on the diameter and minimum clearance axially, a decision was made to increase the die sleeve diameter.

Neg 0672642-1

FIGURE 15. $^{147}\text{Pm}_2\text{O}_3$ Modified Die

TABLE 6. Sm_2O_3 Pellets - AMSA Phase II

Run* No.	Pressing Data			Time, hr	Temp., °C	Sintering Data			
	Pressure, tsi	Dimensions				Diam	Length	Reduction, %	
		Diam	Length					Diam	Length
R-1	64.5	1.550	1.526	6	1350	1.450	1.450	6.4	4.9
R-2	64.5	1.550	1.524	9	1350	1.440	1.445	7.1	5.2
R-3	64.5	1.550	1.524	21	1350	1.440	1.445	7.1	5.2
R-4	64.5	1.552	1.523	14	1400	1.433	1.430	7.7	6.1

* Run No. represents average dimension of six pellets per run.

Diameter variation = ± 0.001 in.

Length variation = ± 0.010 in.

Run R-4 - porosity determination

As pressed 75% TD

As sintered 95% TD

The die was accordingly modified by increasing the internal diameter by 0.020 in. The back-up plugs, to produce the dome shaped ends, were remade to fit the new die bore.

After the die was modified to the larger internal diameter, three test runs were made using typical pressing conditions. The 200 ton press was attached by use of an extension from the shielded gloved enclosure (Figure 16). Sm_2O_3 was pre-pressed, screened, and loaded into the die by use of the special powder container. The results of these experiments are tabulated as follows:

Pellet No.	Pressing Data			Sintering Data					
	Pressure, tsi	Dimensions, in.		Time, hr	Temp., °C	Dimensions, in.		Reduction, %	
		diam	length			diam	length	diam	length
SM-1	63	1.570	1.477	12	1450	1.443	1.400	8.0	5.2
SM-2	58	1.570	1.483	12	1450	1.448	1.425	7.8	3.9
SM-3	53	1.570	1.507	12	1450	1.440	1.440	8.2	4.4

All three of these pellets were sintered within the heat zone in a single furnace run (Figure 17). Although micro-cracks were noted at the transition point where the straight cylinder blended into the radii for the dome ends, the pellets were intact and sintered reductions were suitable for assembly. These pellets were used for test weld capsules. These pellets, comparable to the previous Phase II pellets, showed a slightly greater radial reduction and a slightly smaller length reduction. Although the applied pressure should have a direct relationship to the final reduction during sintering, this effect was probably obscured by thermal gradients within the furnace. The center position in the furnace probably accounts for the better sintering results in the SM-1 pellet.

The first attempt to press $^{147}\text{Pm}_2\text{O}_3$ as a single, one-piece pellet failed. To duplicate the optimum-process parameters developed with the Sm_2O_3 powder, the die was assembled with the required quantity of powder and 63 tsi was

Neg 0672936-1

FIGURE 16. $^{147}\text{Pm}_2\text{O}_3$ Glove Box with Press Attached

Neg 0672575

FIGURE 17. $^{147}\text{Pm}_2\text{O}_3$ Sintering Furnace

applied. The die sleeve was quickly ejected and an apparently good pellet (Figure 18) was produced. On closer examination, the upper dome was found to be merely resting in position. Removal of the upper dome revealed large concentric and radial cracks in the lower core of the pellet. Upon sintering, these cracks were found more severe rather than to have healed or closed (Figure 19).

The maximum diameter at the break in the pellet was 1.511 in. Several dimensions from 0.020 to 0.030 in. oversized were noted at the broken end of the pellet. Although this end of the pellet was flared due to the internal cracks, the balance of the pellet body was well within the dimensional

Neg 0672642-5

FIGURE 18. *Promethium Pellet No. 1 as Pressed*

Neg 0672642-1

FIGURE 19. *Promethium Pellet No. 2 as Sintered*

limits required for assembly. The length reduction, with the broken dome in place, was satisfactory. Disregarding the cracking problem, the sintering results indicated a satisfactory degree of sintering reduction (Table 7).

At this point in the program, several alternate courses of action were considered to produce a satisfactory pellet for the AMSA heat source. These were:

- Utilize the existing pellet,
- Press and sinter another pellet and modify conditions to hopefully achieve a solid pellet, and
- Use an assembly of wafers to reduce thermal gradients and resultant stresses during processing.

The first two of the preceding courses of action were considered to be the most desirable for initial efforts.

TABLE 7. $^{147}\text{Pm}_2\text{O}_3$ Pellets*

Pellet No.	Pressing Data			Sintering Data					
	Pressure, tsi	Dimensions, in.		Time, hr	Temp., °C	Dimensions, in.		Reduction, %	
		diam	length			diam	length	diam	length
PM-1	63	1.580	1.550	30	1450	1.445	1.450	8.5	6.3
PM-2	63	1.580	0.900	30	1450	1.485	0.860	6.0	4.4
				24	1500	No change on rerun			
PM-3	50	1.580	1.588	30	1450	1.460	1.470	7.6	7.4

* These $^{147}\text{Pm}_2\text{O}_3$ pellets, either cracked or assembled in as many as three pieces, made the dimensional tolerances no more accurate than ± 0.010 in. in length, and ± 0.005 in. in diameter

Using a specially modified inner cladding, clamping pressure was applied to the oversize portion of the cracked pellet in an attempt to close the cracks, and thus reduce the pellet sufficiently for assembly. This effort was soon abandoned to prevent high radiation exposure to personnel. It soon became apparent that this pellet could be used only by machining out the inside of the primary cladding. Since this approach would result in a loss of shielding, this assembly (Figure 20) was held in reserve until the results of pressing and sintering a second pellet could be evaluated.

The second attempt to form a monolithic pellet for the AMSA capsule was also unsuccessful. The pellet body did not show cracking but, as with the first pellet, the dome ends broke off. Severe internal cracking in the first pellet was believed to be caused by a buildup in the core temperature after the pellet was pressed due to self-heating of the $^{147}\text{Pm}_2\text{O}_3$. The core temperature induced greater thermal expansion in the hotter central portion than in the outer surface. This effect was minimized in the second attempt to press the pellet. The die was loaded and only slight pressure (approximately 1000 psi) was applied to consolidate the powder in the fixed form and then released. With the powder fixed in the die in a shape and volume similar to that of the final pressed pellet, it was allowed to stand for 1 hr before applying the final pressure of 63 tsi, so that temperature stabilization

Neg 0673936-2

FIGURE 20. *Promethium Pellet No. 1 Recovery Attempt*

could occur. Hopefully, after pressing of the pellet was completed and it was ejected from the die, thermal stresses would not build up, since the pellet core was already at a higher temperature than the surface at the time of maximum pressure application. The procedure was demonstrated to be partially effective, as shown by the complete lack of internal cracks in the body of Pm_2O_3 Pellet No. 2, (Figure 21). The problem of the broken domes remained. Additional experiments with Sm_2O_3 , in which three-part die plugs and split ring plugs were used with colloidal graphite die lubricant, demonstrated that sticking of the pellet to the die plug was not the cause of the

broken domes. A positive answer to this problem has not been determined. It is not a new problem, since earlier pellet pressing with the Sm_2O_3 stand-in produced several two- and three-piece pellets, while quite a few of the one-piece assemblies had microcracks. Since these multi-piece Sm_2O_3 assemblies did not significantly affect the impact integrity of the capsule (Table 5), this problem was given second priority in producing a fuel core for the AMSA capsule.

The sintering reduction of the second $^{147}\text{Pm}_2\text{O}_3$ pellet was not nearly as great as the first one. Table 7 shows that even the rerun at 1500 °C failed to reduce the pellet center section sufficiently for assembly. In a detailed review of

Neg 0673936-3

FIGURE 21. Promethium Pellet No. 2 as Sintered

prepowder preparation, it was determined that this powder had been prepressed in the "slugging" operation to a density 10 to 15% higher than that of the first pellet powder. Rather than using applied pressure as a criterion for forming these dome-shaped pellets, it became apparent that maintaining a precise length-to-diameter ratio before sintering was of prime importance to produce a pellet for assembly in the specific inner cladding dimensions. The second pellet could be used by sanding approximately 0.020 in. off the diameter. This pellet was retained as a possible lower density stand-by, and the first pellet was reprocessed chemically to provide powder for a third pellet.

The loaded die delay procedure developed for use with $^{147}\text{Pm}_2\text{O}_3$ Pellet 2 was followed in pressing the third and final AMSA pellet. This pellet (Figure 22) was fractured into an upper radiused end cap, a cylindrical body, and a lower radiused end cap. After sintering for 30 hr at 1450 °C and smoothing the laminarly fractured surfaces by sanding, the three pieces could be assembled into the AMSA inner tantalum cladding.

The weight of the Pm_2O_3 loaded in the assembly was 227 g. Process design parameters were based on the use of 235 g of Pm_2O_3 , corresponding to a heat output of 65 W 30 days after purification of the promethium. Heat output of the 65 W source would be reduced to 62.4 W on October 1, 1967, due to aging of the promethium. A slight weight loss in the three-piece pellet, sustained by chipping of the fractured surfaces, caused a further reduction of 2.1 W to a final assembled 60.3 wattage.

The proper length-to-diameter ratio of this third and final pellet was assured by using a positive stop during pressing to control the length of the pellet. As Table 7 shows, only 50 tsi was needed to reach this mechanical press stop as compared to the two previous Pm_2O_3 pellets, pressed at 63 tsi

Neg 0673374-1

FIGURE 22. *Promethium Pellet No. 3 as Assembled*

without using a mechanical stop. The final sintered length of this pellet indicates the planned length to be satisfactory for assembly.

ASSEMBLY OF AMSA HEAT SOURCE CAPSULE

The AMSA process flow and quality control chart (Figure 13) was followed very closely in making the double cladding assembly. As expected, only the inner cladding assembly offered significant problems in contamination control. After decontamination was effected and the first weld was made, the capsule remained nonsmearable for the balance of the assembly. This condition confirmed by double smear tests

using a dry cotton swab and measuring the contamination level on a mica window (beta detection) GM Counter every time the capsule was moved in a process step.

The electron beam weld closure of the inner tantalum cladding (Figure 23) was determined to be satisfactory by ultrasonic nondestructive testing. Destructive evaluation of the test welds, made before and after welding the fueled capsule, established weld penetration for an inner seal weld (Figure 24) to be optimum.

The same procedure was used on the final outer weld. The nondestructive evaluation was noted as satisfactory and was confirmed by destructive examination of the first test weld made immediately before the fueled capsule was welded. The

Neg 0673438-3

FIGURE 23. AMSA Capsule Inner Clad Welded

Neg 467-1711

FIGURE 24. *Inner Tantalum Weld, Test Capsule*

last test weld, made immediately after the fueled capsule was welded, showed penetration to be far less than the desired 85%. A recheck of the fueled capsule (Profile Figure 25) using a dual testing technique was made. Minimum weld penetration of 75% was noted in some areas. However, most of the circumferential weld exceeded the 85% desired.

A third test weld using welding parameters identical to those in the previous welds was made to confirm the non-destructive finding. Very slight differences between this weld, the first test weld, and the fueled capsule weld were noted by nondestructive examination of all three welds, and by destructive examination of the test welds. A photomicrograph of the first test weld, considered to be typical of the fueled capsule, is shown in Figure 26.

The promethium fueled capsule was subjected to the quality assurance inspections identified in Figure 13. These quality control inspection tests also are listed in

Neg 0681613-3

FIGURE 25. Nondestructive Test Profile of Fueled Capsule

Neg 567-1708 A and B

FIGURE 26. *Outer L-605 Weld Test Capsule*

Appendix Table A-1, and all pertinent test results are accumulated in Tables A-2 through A-5. These tables provide a complete review of the quality control procedures and test results.

HANDLING AND SHIPPING

Handling procedures for the AMSA $^{147}\text{Pm}_2\text{O}_3$ heat source capsule were recommended by Battelle-Northwest personnel. These procedures covered the detailed packaging required for the shipment of the fueled capsule, as well as recommended steps for optimum safety in removing the capsule from the shipping container, placement into and removal from the thermal preconditioner unit (TPU), and storage of the heat source capsule. All required handling tools were supplied with the capsule, and general radiological safety procedures used at Pacific Northwest Laboratory were reviewed.

Final photographs of the completed AMSA capsule are shown as Figure 27, and the radiation map of measured dosage is shown as Figure 28. The ICC approved specification - 2R container is shown with the special copper chill block holding fixture in Figure 29. This assembly, when loaded into the shipping barrel (Figure 30), was checked for detailed temperature and radiation measurements (Figures 31 and 32).

The AMSA promethium-147 capsule was delivered to the Air Force at Wright-Patterson Air Force Base in March 1968. The capsule, a 60 thermal watt heat source as of October 1, 1967, was held in storage until delivery of the Inertial Measuring Unit. A reduction to approximately 54 W occurred during this storage period. Flight and laboratory tests using the capsule were performed by the Air Force, and the capsule was returned to Battelle-Northwest in April, 1968.

Neg 0673446-1

Neg 0673446-2

FIGURE 27. *Two Views of Final AMSA Fueled Capsule*

Inner Numbers Are 12 in. from Center of Capsule, in mr/hr
 Outer Numbers Are 18 in. from Center of Capsule

Neg 0681613-4

FIGURE 28. Radiation Mapping

Neg 0673109-1

FIGURE 29. *2R ICC Specified Container with Holding Fixture*

Neg 0673826-2

FIGURE 30. *Shipping Barrel*

All Readings with Barrel Closed, or Taken Immediately on Opening,
After Standing at Room Temperature for 64 Hours. 10/30/67

Temperature in Place with Lid B Removed
Steel Wool Removed to Make Measurements

- A. 245-265 °F - 100 mr/hr
- B. Side - 230 °F - 180 mr/hr
- C. 260 °F - 70 mr/hr
- D. Valve Cap 215 °F
- E. Supports - 200 °F

Neg 0681613-2

FIGURE 31. Radiation and Temperature Profile
of Shipping Container

2R Inner Container (Sealed) Removed from Shipping Drum after 64 Hours.
 Allowed to Stand 30 Min. in Air - Room Temp. - Sitting on Table Top
 (Non-Metallic) 10/30/67

A	Surface - 230 °F	100 mr/hr
B	Surface - 200 °F	180 mr/hr
C	Surface - 225 °F	70 mr/hr
D	Valve Caps - 140 °F	
E	Supports - 135 °F	

After 2R Spec. Container Opened, Immediately after Above Measurements were Made.

NOTES:

1. Temp. Top of Copper Block 245 °F
 Side of Copper Block 245 °F
2. Radiation Field to Hands in Screwing Eye Bolt into Top of Copper Block = 220 mr/hr
3. Temp. of AMSA Capsule Immediately After Removing Copper Chill Block - 330 °F
4. Temp. of AMSA Capsule after Sitting on Ceramic Surface for two Hours in Still Room Temperature Air-480 °F
 A Ring Gage 2.005" diam. Passed over Capsule at this Point with Minimum Interference - Length = 1.999" - wt. - 745.2 grams
5. Radiation = 1100 mr/hr with CP Beta Shield in Actual Contact with Capsule. (Uncorrected for Geometry of CP Chamber)

Neg 0681613-1

FIGURE 32. Radiation and Temperature Profile of 2R Container

REFERENCES

1. W. J. Dalby. 9312 AMSA Fuel Capsule Impact Tests, SC-DR-67-718. Sandia Corporation, Albuquerque, New Mexico, August 1967.
2. L. A. Bray and H. H. Van Tuyl. Laboratory Development of a Carrier-Precipitation Process for the Recovery of Strontium from Purex Wastes, HW-69534. Available from Clearinghouse for Federal Scientific and Technical Information, Springfield, Virginia, May 9, 1961.
3. W. V. DeMier, G. L. Richardson, A. M. Platt, and W. H. Swift. Development of Radioactive Waste Fractionization and Packaging Technology, HW-SA-2786. Available from Clearinghouse for Federal Scientific and Technical Information, Springfield, Virginia, January 1963.
4. L. A. Bray. Denitration of Purex Wastes with Sugar, HW-76973 Rev. Available from Clearinghouse for Federal Scientific and Technical Information, Springfield, Virginia, April 1963.
5. E. J. Wheelwright, F. P. Roberts, U. L. Upson, L. J. Kirby, T. R. Myers, and H. H. Van Tuyl. Ion-Exchange Separation of Kilocurie Quantities of High Purity Promethium, BNWL-318. Pacific Northwest Laboratory, Richland, Washington, December 1966.

ACKNOWLEDGEMENTS

The authors are indebted to the many co-workers at the Pacific Northwest Laboratory who performed much of the work reported on this document. Personal acknowledgements, identified with areas of major performance, are noted below. Special thanks are extended to the many contributors.

- R. J. Baker and C. E. Vogel   Program Management
- R. F. Boolen and D. E. DeWitt   Welding
- W. F. Riemath, G. O. Hammer, and
E. H. Shade   Process Development
Pressing, Sintering,
and Assembly
- R. H. Beauchamp   Metallography
- R. W. Steffen and F. M. Coffman   Nondestructive
Testing
- E. J. Wheelwright, F. P. Roberts
and U. L. Upson   Chemical Development,
Radiation Mapping,
and Calorimetry

Special recognition is extended, additionally, to W. J. Dalby, Sandia Laboratory, for his cooperation and effort in planning and conducting the many impact tests.

APPENDIX

TABLE A-1. *Types of Quality Assurance Inspections**

1. Dimension Measurements	8. X-ray Diffraction
2. Dye Penetrant	9. Radiography
3. Impurity Analysis	10. Ultrasonic Testing
4. Density Determination	11. Decontamination Smear Check
5. He Leak Check	12. Radiation Mapping
6. Metallography	13. Gamma Spectrometry
7. Isotopic Analysis	14. Calorimetry

---

* See Figure 13

TABLE A-2. *Fueled Capsule Component Test Results*

	<u>Inner Clad</u>	<u>Outer Clad</u>
1. Dimension Measurements	1.598 × 1.592 in. overall	1.999 × 1.997 in. overall
	$\frac{0.0605 \text{ in.}}{0.0615 \text{ in.}}$ wall	$\frac{0.199 \text{ in.}}{0.202 \text{ in.}}$ wall
2. Dye Penetrant*	No relevant indications	No relevant indications
3. Impurity Analysis	Certificate of Analysis Page A-2	Test Report Page A-3

---

* Dye Penetrant - ZY Glo-Post emulsifier technique consists of (1) soaking in penetrant 20 min, (2) emulsifying 4 min, and (3) washing dry and developing for black light read-out

FANSTEEL CERTIFICATION

FANSTEEL

FANSTEEL CORPORATION

NUMBER ONE TANTALUM PLACE
NORTH CHICAGO, ILLINOIS • 60064
Phone: 312/689-4900 TWX: 312/244-4530

CERTIFICATE OF ANALYSIS

Date **October 20, 1967**Customer **Battelle Memorial Institute**Purchase Order No. **B88-777**Material **Tantalum Rod 1.600" Diameter +0.020"-0.000" x 12" +1/8" -0.000"**Production No. **634-032688-0**Lot No. **69B2053**Quantity **15.551 Lbs.**

CHEMICAL ANALYSIS

LOT ANALYSIS

C	10- ppm
O	37 ppm
N	10- ppm
H	5- ppm
W	140 ppm
Cb	50 ppm
Zr	5- ppm
Mo	5- ppm
Ti	5- ppm
Fe	20 ppm
Ni	5- ppm
Si	5- ppm
Mn	5- ppm
Ca	5- ppm
Al	5- ppm
Cu	5- ppm
Sn	5- ppm
Cr	5- ppm
V	5- ppm
Co	5- ppm
Ta	Balance

Victor E. Davis
Quality Control Representative

UNIVERSAL-CYCLOPS SPECIALTY STEEL DIVISION

TERMS: 30 DAYS NET = DISCOUNT		BILL OF LADING NO.	ORDER DATE	SHIPPING & INVOICE DATE
☆ ON \$	IF PAID ON OR BEFORE	DIST. OFFICE - SALESMAN	YOUR ORDER NUMBER	INVOICE NO.
INDEX NO.	CUST CODE NO.	STATE	2/2/67	878-777-26712
T2752				INV-6915
SOLD TO		SHIPPED TO		
★ BATTTELLE NORTHWEST RICHLAND WASHINGTON 99352		BATTTELLE NORTHWEST RICHLAND WASHINGTON		

ROUTING AIR FREIGHT (RUSH) UNITED AIRLINES TO PORTLAND C/O WEST COAST AIRLINES TO
 COLL ☒ PPD F.O.B. BRIDGEVILLE F/E KOKOMO /RICHLAND WASH

PAPER WRAP		METAL TAG WITH CUST NO., SIZE, HEAT, TYPE AND PATENT NO 2,740,250	
INVOICE NO. INV.	PRICE & D	OTHER	TEST REPORT INSTR.
		1 T/R WITH SHIPMENT	ANAL. PHYSICALS NO. T/R
		3 T/R ATTN MR B J BEAVER	CERT. NOTARIZED
STOCK CONTROL NO.	QUAN. ORDERED PCS. OR WT.	SIZE AND DESCRIPTION	HEAT NO.
			QUAN. SHIPPED NET WEIGHT
			PRICE
			AMOUNT

UNITEMP L605 SOLUTION
TREATED CENTERLESS GROUND BAR

R53050 1 BAR
APPROX 1490

2" RD X ABT 12 FT R/L
ORDER COMPLETE

817569

TYPE	L605	AMS-5759B and O.E. B50T26-S4	2" round	T2752									
COND. & FINISH Hot rolled - solution treated - centerless ground													
HEAT NO.	C	MN	SI	S	P	CR	W	V	NI	MO	Co	Fe	
B17569	.14	1.51	.63	.010	.005	19.82	14.84		9.99		Bal.	1.86	
TEST COND.	HARDNESS	YIELD	TENSILE	% EL.	% R.A.	BENDS	OTHER PROPERTIES						
	BHN	.2%											
	241/255	75,000	145,000	57.9	52.1								
GR. SIZE	HARDENABILITY	TREATMENT				STRESS RUPTURE							
						STRESS-PSI	TEMP.-°F.	TIME-HRS.	Elong.				
						24,000	1500	24+	67.1%				

REMARKS:

I CERTIFY THAT THIS IS A TRUE AND CORRECT COPY OF THE TESTS SHOWN ON OUR LABORATORY RECORDS.

BY [Signature]
AUTHORIZED SIGNATURE

TEST REPORT

MI-924

TABLE A-3. *Fueled Capsule Pellet Test Results*

<u>Type of Inspection*</u>	<u>Powder Preparation</u>	<u>Pellet As Pressed</u>	<u>Pellet As Sintered</u>
1. Dimension Measurements	Not applicable	1.580 in. diam 1.588** in. long	1.463 in. diam 1.438 in. long
3. Impurity Analysis	Radiochemical analysis Page 31	Not applicable	Not applicable
4. Density Determination	Tap density approximately 30%	70 to 75% of theoretical	90 to 95% of theoretical
7. Isotopic Analysis	Emission spectrography Page 31	Not applicable	Not applicable
8. X-ray Diffraction	Monoclinic Pm_2O_3 (B-type)	Not applicable	Not applicable

* *For Inspection Number Identification and Process Location,
see Figure 13, Encapsulation Process*

** *Denotes 3-piece assembled length*

TABLE A-4. *Fueled Capsule Assembly Test Results*

<u>Type of Inspection</u>	<u>Inner Capsule Assembly</u>	<u>Outer Clad Assembly</u>
5. He Leak*1 Check	Background	Background
6. Metallography	Test weld Figure 24	Test weld Figure 26
10. Ultrasonic*2	Minimum weld 50% or greater	73 to 93% weld profile Figure 25
11. Decontamination Smear Check*3	Nonsmearable	Nonsmearable
12. Radiation*4 Mapping	1 m 7.4 mr/hr	1 m 6.8 to 7.2 mr/hr

---

*1 *CEC Helium Leak Detector*

1. *Chamber with capsule evacuated to 0.05 Torr (30 min)*
2. *Backfilled with He to 15 psi (5 min)*
3. *Sensitivity check before and after capsule leak test*
4. *Minimum detectable leak--less than 7×10^{-7} std.
cm²/sec*

*2 *Broadband Ultrasonic Tester - 15 MHz lithium sulphate;
spherically focused transducers used to establish normal
incidence and shear wave test techniques.*

*3 *Contamination Smear Test--mica window G-M calibrated
by beta-gamma source.*

*4 *CP Type ION Chamber--calibrated by NBS standards*

TABLE A-5. *AMSA Fueled Capsule Final Test Results*

Type of Inspection	Test Results
1. Dimension Measurements	$\frac{2.004 \text{ in.}}{2.005 \text{ in.}} \text{ diam} \times \frac{1.999 \text{ in.}}{2.000 \text{ in.}}$ length*1
5. He Leak Check	No indication either before or after final weld bead removal
9. Radiography*2	Both inner and outer cladding revealed areas of incomplete weld penetration-previously noted on ultrasonic test
10. Ultrasonic Test	In addition to previous ultrasonic test, side walls were inspected for laminar type flaws with normal incidence test. No defect found.
11. Decontamination Smear Check	Nonsmearable both before and after final weld bead removal
12. Radiation Mapping	$\frac{6.8 \text{ mr/hr}}{7.2 \text{ mr/hr}}$ at 1 m. Profile as shipped. Page 53
13. Gamma Spectrometry	See gamma energy plot Page A-7
14. Calorimetry	60.3 $\pm$ 0.2 W 10/1/67 See additional information Page A-8

---

*1 *This dimensional test was made after capsule was placed on ceramic brick for a period of 2 hr and temperature was recorded at 480 °F. Ring gage, (GO gage) 2.005 in. ID, furnished by AC Electronics per Sketch SK-51222, passed over the capsule when fitted cold to the "hot" capsule.*

*2 *Radiographic Source: Iridium 192 Isotope Radiographic Film. Kodak Type "M" Source-Film Distance: 20 in. Exposure Time: 13 min. Eight exposures were made (A through H) in 22-1/2° increments. One exposure was made through end of capsule.*

FIGURE A-1. Gamma Spectrum of 60 W, $^{147}\text{Pm}_2\text{O}_3$ AMSA Capsule

TABLE A-6. AMSA Capsule Calorimeter Data

Calorimeter Factor (for conditions of this test): 1.0825 W/mV
($\pm 0.10\%$ at 95% confidence)

Decay Factor: $\lambda = \ln 1/2 / (957 \times 24) = 3.01788 \times 10^{-5} \text{ hr}^{-1}$

Date	Hour	Time, hr	Measured E^* mV	Calculated E_o^{**} mV	$E_o - \bar{E}_o$ μV
10/6	1200	0	55.524	55.524	+2
	1400	2	55.525	55.528	+6
	1600	4	55.523	55.530	+8
	1800	6	55.510	55.520	-2
	2000	8	55.508	55.521	-1
	2200	10	55.506	55.523	+1
	2400	12	55.498	55.518	-4
10/7	0200	14	55.495	55.518	-4
	0400	16	55.490	55.517	-5
	0600	18	55.485	55.515	-7
	0800	20	55.488	55.522	0
	1000	22	55.484	55.521	-1
	1200	24	55.480	55.520	-2
	1400	26	55.490	55.534	+12
	1600	28	55.485	55.532	+10
	1800	30	55.488	55.538	+16
	2000	32	55.478	55.531	+9
	2200	34	55.470	55.527	+5
	2400	36	55.452	55.512	-10
	0200	38	55.465	55.528	+6
	0400	40	55.445	55.511	-11
10/8	0600	42	55.435	55.505	-17
	0800	44	55.435	55.508	-14
	1000	46	55.445	55.522	0
	1200	48	55.455	55.535	+13

$$[\Sigma] = 13.060 \quad [\Sigma] = +14$$

$$\bar{E}_o = 55.522 \text{ mV} \quad e = +0.6 \text{ } \mu\text{V}$$

$$[\Sigma (E_o - \bar{E}_o)^2] = 1738$$

Standard deviation of mean (σ) = $\pm 0.0085 \text{ mV} = \pm 0.015\%$

95% Confidence error = $\pm 0.03 \text{ mV} = \pm 0.06\%$

Capsule power at 1200 October 6 (t_o) = 1.0825 (55.522)
= 60.10 ± 0.07

Capsule power at 1200 October 1 = $60.10 e^{120\lambda} = 60.32 \pm 0.07$

*Two-hour period average, taken from chart record.

**Measured average, corrected for decay ($E_o = E e^{\lambda t}$).

DISTRIBUTIONNo. of
CopiesOFFSITE

1	<u>AC Electronics</u> Division of General Motors Route 128 Wakefield, Massachusetts 01881 W. W. Burroughs, Jr.
1	<u>AEC Chicago Patent Group</u> G. H. Lee
1	<u>AEC Division of Space Nuclear Systems</u> G. P. Dix
260	<u>AEC Division of Technical Information Extension</u>
2	<u>AEC Library, Washington</u> J. N. Maddox R. W. Shivers
3	<u>Air Force Aero Propulsion Laboratory</u> Mr. Lintz Capt. D. C. DePree Maj. J. S. Pizzuto
1	<u>Andrews Air Force Base</u> Washington, D. C. 20331 Maj. G. R. Crane
1	<u>Kirtland Air Force Base</u> Albuquerque, N.M. Capt. B. E. Watlington
1	<u>Sandia Corporation, Albuquerque (AEC)</u> V. E. Blake

ONSITE-HANFORD

1	<u>AEC Chicago Patent Group</u> R. K. Sharp (Richland)

No. of
Copies

1	<u>AEC Richland Operation Office</u> C. L. Robinson
3	<u>Battelle Memorial Institute</u>
66	<u>Battelle-Northwest</u> F. W. Albaugh R. J. Baker A. L. Bement E. A. Berreth R. F. Boolean D. W. Brite (10) T. D. Chikalla G. M. Dalen N. C. Davis (10) D. R. de Halas K. Drumheller E. A. Evans P. L. Farnsworth H. T. Fullam R. L. Gibby S. Goldsmith W. L. Hampson J. E. Hansen J. H. Jarrett J. N. Judy R. L. Knecht C. E. Leach D. C. Lehfeldt L. G. Merker R. L. Moore M. B. Nelson R. E. Nightingale A. M. Platt F. P. Roberts R. K. Robinson C. A. Rohrmann C. E. Saari R. C. Smith R. W. Steffen K. R. Sump U. L. Upson H. H. Van Tuyl C. E. Vogel E. E. Voiland R. G. Wheeler E. J. Wheelwright Technical Information Files (5) Technical Publications (2)