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Design and Qualification Testing of a Strontium-90 Fluoride Heat Source

H. T. Fullam

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SUMMARY

The Strontium Heat Source Development Program began at the Pacific Northwest Laboratory (PNL) in 1972 and is scheduled to be completed by the end of FY-1981. The program is currently funded by the U.S. Department of Energy (DOE) By-Product Utilization Program.

The primary objective of the program has been to develop the data and technology required to permit the licensing of power systems for terrestrial applications that utilize $^{90}\text{SrF}_2$ -fueled radioisotope heat sources. A secondary objective of the program has been to design and qualification-test a general purpose $^{90}\text{SrF}_2$ -fueled heat source. This report describes the effort expended in the design and testing of the heat source.

A radioisotope heat source to be used in a licensed power generator system must meet certain qualification-test requirements. Five qualification tests have been defined for the $^{90}\text{SrF}_2$ heat source based on current Federal and international regulations. They are:

- a 9-m drop (impact) test;
- a 1000-bar external hydrostatic pressure test;
- a 7 kg-m percussion test;
- a 800°C thermal test;
- a 800°C to 0°C thermal quench test.

In addition, DOE and PNL have defined additional qualification-test requirements for the heat source to enhance its usefulness and inherent safety. The additional requirements are:

- The heat source must meet all qualification-test requirements in the post-service as well as the as-fabricated condition.
- The heat-source outer capsule must meet the qualification-test requirements without support from the heat-source inner capsule.
- The heat-source outer capsule must have adequate oxidation resistance.
- The heat-source outer capsule must have adequate seawater corrosion resistance.

The American National Standards Institute (ANSI) has defined certain qualification-test requirements for sealed radioactive sources. These requirements do not currently apply to radioisotope heat sources but may be applied in the future. Therefore, certain of the ANSI requirements were applied to the $^{90}\text{SrF}_2$ heat source. They were:

- a 20 kg-m percussion test;
- a 24,500 psi external hydrostatic pressure test;
- a 1 kg-m puncture test.

It must be remembered, however, that failure of the heat source to meet the ANSI qualification tests would not currently preclude its use in a licensable power-generating system.

By DOE directive, the general purpose $^{90}\text{SrF}_2$ heat source is to use as much of the existing Waste Encapsulation and Storage Facility (WESF) $^{90}\text{SrF}_2$ storage capsule as possible. Unfortunately, preliminary tests and calculations show that the WESF $^{90}\text{SrF}_2$ capsule cannot

meet licensing requirements. Therefore, it was decided by DOE and PNL to develop a new heat source that would use the existing WESF Hastelloy C-276 inner capsule and a new outer capsule. The existing Hastelloy C-276 inner capsule would provide the primary containment of the $^{90}\text{SrF}_2$, and the new outer capsule would provide the structural strength needed to meet licensing requirements.

Based on a number of considerations, Hastelloy S was selected as the outer capsule material with Hastelloy C-4 as a backup material in case the Hastelloy S had to be rejected for any reason. The 1000-bar hydrostatic pressure test and the 9-m drop test could most seriously test the integrity of the outer capsule. In designing the new outer capsule, stress analyses focused on the 1000-bar external pressure and the 30-ft drop impact loadings. The capsule wall thickness was determined by consideration of the collapse condition for external pressure. The 30-ft drop condition resulted in maximum damage for an impact on the capsule corner, and, consequently, the corner joint design was dictated by the loading encountered during impact. The design analyses were based on the mechanical properties Hastelloy S would possess after extended aging at anticipated heat source operating temperatures.

The design calculations indicated the need for a thick-wall outer capsule that utilized an interlocking endcap design. Preliminary external pressure and impact tests were conducted on outer capsules fabricated from AISI-1018 low-carbon steel to evaluate the adequacy of the design. This steel was selected because its yield strength was similar to that of thermally aged Hastelloy S. By varying the temperature of the AISI-1018 carbon steel capsules, it was possible to vary the impact energy of the steel and duplicate the impact energy of thermally aged Hastelloy S. Therefore, the impact tests with the steel capsules were carried out at various temperatures to determine the impact energy needed for the capsules to meet the impact test requirement, and to determine if the impact energy of thermally aged Hastelloy S was adequate for the capsule design proposed.

Results obtained with the AISI-1018 carbon steel capsules demonstrated the adequacy of the proposed outer capsule design. The design calls for a nominal inner diameter of 2.375 in. and a nominal wall thickness of 0.500 in. The overall length of the outer capsule was ~21 in. The interlocking joint design was used for the end closures. Using the interlocking design, a weld penetration of only 0.1 in. was required for the closure weld, which can be readily accomplished remotely in a shielded facility.

A total of 16 Hastelloy S and 7 Hastelloy C-4 prototype outer capsules were fabricated for the qualification-testing program. Fabrication and welding of the outer capsules was provided by the Cabot Corporation. Five of the capsules, which were to be subjected to a primary impact test, contained a standard WESF Hastelloy C-276 inner capsule filled with nonradioactive SrF_2 . Radiography of the capsule welds showed that the weld penetration was substantially less than 0.1 in. specified. The capsules to be subjected to the primary impact test, and which contained inner capsules, were rewelded before testing. The other capsules were tested without rewelding.

Each of the outer capsules was subjected to one of the five primary qualification tests listed below:

1. a 9-m impact (drop) test;
2. a 1000-bar hydrostatic pressure test;
3. a 7 kg-m percussion test;
4. an 800°C thermal test;
5. a thermal quench test (800°C to <0°C).

A capsule was assumed to have passed the qualification test if it did not leak at a rate greater than 1×10^{-8} std cm³/s, as determined by a helium leak test. Some of the capsules were qualification tested in the as-fabricated condition; others were tested after aging at 650° or 725°C for 8000 h.

If a capsule passed the primary qualification test, it was then subjected to four secondary qualification tests. The secondary tests were simplified versions of the primary qualification tests. If the capsules passed the secondary qualification tests, they were then subjected to one or more of the following supplemental tests:

- a 20 kg-m percussion test;
- a 24,500 psi hydrostatic pressure test;
- a 1 kg-m puncture test.

After all testing was completed, the capsules were subjected to a final helium leak test.

The as-fabricated and the thermal-aged Hastelloy S prototype outer capsules passed the primary and secondary qualification tests without a problem. They also passed the supplemental percussion and puncture tests without a problem. The Hastelloy S capsules subjected to the supplemental pressure test partially collapsed at a pressure of 24,000 psi; but the partially collapsed capsules passed the helium leak test (leak rate $<1 \times 10^{-8}$ std cm³/s).

The as-fabricated Hastelloy C-4 capsules and the Hastelloy C-4 capsules aged at 725°C passed the primary and secondary qualification tests without a problem. They also passed the supplemental percussion and puncture tests without difficulty. The capsules subjected to the supplemental pressure tests partially collapsed at a pressure of 20,000 to 21,000 psi but passed the helium leak test (leak rate $<1 \times 10^{-8}$ std cm³/s).

The only problem encountered with any of the capsules was the Hastelloy C-4 capsule aged at 650°C for 8000 h. The capsule was subjected to the primary hydrostatic pressure test and then to the secondary impact and percussion tests, which it passed without apparent difficulty. After the capsule was subjected to the secondary thermal test, a leak was detected. Examination of the capsule showed a crack about 1 in. long in the weld zone immediately adjacent to the point where the capsule had been impacted in the impact test. Metallographic examination of the affected area showed the weld penetration was only about 0.04 in. The data indicate that the crack was probably started by stresses produced by the impact test and propagated to the point of failure by stresses resulting from the thermal test. The data also indicate that a full penetration weld would probably eliminate the problem.

The results obtained in the qualification tests show that the outer capsule design proposed for the $^{90}\text{SrF}_2$ heat source is capable of meeting current licensing requirements when Hastelloy S is used as the outer capsule material. The data also indicate that an outer capsule of Hastelloy C-4 would probably also meet licensing requirements, although Hastelloy S is the preferred material. Therefore, based on the results of this study, the general purpose $^{90}\text{SrF}_2$ heat source will consist of a standard WESF Hastelloy C-276 inner capsule filled with $^{90}\text{SrF}_2$ and a Hastelloy S outer capsule having a 2.375-in. inner diameter and 0.500-in. wall thickness. The end closures for this study, the general purpose $^{90}\text{SrF}_2$ heat source will consist of a standard WESF Hastelloy C-276 inner capsule filled with $^{90}\text{SrF}_2$ and a Hastelloy S outer capsule having a 2.375-in. inner diameter and 0.500-in. wall thickness. The end closures for the outer capsule will utilize an interlocking joint design requiring a 0.1-in. penetration closure weld.

CONTENTS

SUMMARY	iii
1.0 INTRODUCTION	1
2.0 OBJECTIVES	3
3.0 WESF STRONTIUM-90 FLUORIDE STORAGE CAPSULE	5
4.0 HEAT SOURCE LICENSING REQUIREMENTS	7
4.1 BASIC LICENSING REQUIREMENTS	7
4.2 HEAT SOURCE QUALIFICATION TEST REQUIREMENTS	7
4.3 DOE-PNL QUALIFICATION TEST REQUIREMENTS	7
4.4 SUPPLEMENTAL QUALIFICATION TESTS	9
5.0 HEAT SOURCE DESIGN	11
6.0 HEAT SOURCE QUALIFICATION TESTS	17
6.1 TESTING REQUIREMENTS	17
6.1.1 Qualification Testing Matrix	17
6.1.2 Mechanical Property Tests	19
6.1.3 Metal-Metal Compatibility Tests	19
6.2 AUXILIARY MEASUREMENTS	19
6.2.1 Physical Measurements	22
6.2.2 Leak Tests	22
6.2.3 Radiography	25
6.3 FABRICATION OF CAPSULES AND TEST SPECIMENS	25
6.3.1 Outer Capsules	25
6.3.2 Inner Capsules	27
6.3.3 Outer Capsule Welding	27
6.3.4 Helium Filling	29
6.3.5 Mechanical Property Specimens	29
6.3.6 Metal-Metal Compatibility Capsules	31
6.4 THERMAL AGING	31
6.5 CAPSULE TESTING PROCEDURE	32
6.6 RESULTS AND DISCUSSION	35
6.6.1 Qualification Tests	35

6.6.2	Effect of Aging on Mechanical Properties	69
6.6.3	Metal-Metal Compatibility	76
7.0	CONCLUSIONS AND RECOMMENDATIONS	89
8.0	APPENDIX	91
8.1	QUALIFICATION TEST PROCEDURES	91
8.1.1	Impact Test	91
8.1.2	Hydrostatic Pressure Test	96
8.1.3	Percussion Test	98
8.1.4	Thermal Test	101
8.1.5	Thermal Quench Test	104
8.2	SUPPLEMENTAL TEST PROCEDURES	106
8.2.1	Supplemental Percussion Test	106
8.2.2	Supplemental Pressure Test	108
8.2.3	Supplemental Puncture Test	108
8.3	QUALIFICATION TEST DATA	110
8.3.1	As-Fabricated Capsule	111
8.3.2	Capsule Aged 8000 h at 650°C	120
8.3.3	Capsule Aged 8000 h at 725°C	131
8.4	OXIDATION TESTS	145
8.4.1	Oxidation Rates	145
8.4.2	Effect of Oxidation on Mechanical Properties	146
8.4.3	Effects of Long-Term Oxidation	149
8.5	SEAWATER CORROSION TESTS	150
9.0	REFERENCES	153
10.0	ACKNOWLEDGMENTS	155

FIGURES

1	Typical Data (At Time of Capsule Loading) for a WESF $^{90}\text{SrF}_2$ Storage Capsule	5
2	Dimensions of the WESF $^{90}\text{SrF}_2$ Hastelloy C-276 Inner Capsule	12
3	Components of the Outer Capsule and a Completed AISI-1018 Low-Carbon Steel Outer Capsule Ready for Testing	13
4	Design Selected for the $^{90}\text{SrF}_2$ Heat-Source Outer Capsule	15
5	Locations Where the Outer Capsule Dimensions were Determined	24
6	Equipment Arrangement for Leak Testing a Helium-Filled Capsule	25
7	Sketch Showing Location of Notch Used to Remove Endcaps from Capsules That were to be Rewelded	29
8	Sketch Showing Hole and Pin Arrangement Used to Fill Outer Capsules with Helium	30
9	Capsule Design Used for the Metal-Metal Compatibility Studies	32
10	Prototype Outer Capsules Before Qualification Testing in the As-Fabricated Condition	36
11	The As-Fabricated Hastelloy C-4 Capsule After Being Subjected to the Initial Drop of the Impact Test--View A	38
12	The As-Fabricated Hastelloy C-4 Capsule After Being Subjected to the Initial Drop of the Impact Test--View B	38
13	Impacted End of Capsule S-12 After the Initial Impact--View A	39
14	Impacted End of Capsule S-12 After the Initial Impact--View B	39
15	Photomicrograph of the Affected Endcap of the As-Fabricated Hastelloy C-4 Capsule Subjected to the Primary Impact Test--Showing the Distortion of the Endcap, Capsule Wall, and Interlocking Joint that Resulted from the Impact Loading	40
16	Photomicrographs of the Weld Zone Adjacent to the Point of Impact of the Endcap from the Hastelloy C-4 Capsule Subjected to the Primary Impact Test	41
17	Photomicrograph of the Affected Endcap of the Hastelloy S Capsule Subjected to the Primary Impact Test--Showing the Distortion of the Endcap and Capsule Wall	41
18	Photomicrograph of the Affected Endcap of an As-Fabricated Hastelloy S Outer Capsule Subjected to a Secondary Impact Test--Without an Inner Capsule Present	42
19	Photomicrographs of the Weld Zone Adjacent to the Point of Impact of the Capsule Endcap Shown in Figure 18	42
20	The Affected Endcap from an As-Fabricated Hastelloy C-4 Capsule Subjected to the Secondary Impact Test--Without an Inner Capsule Present	43
21	Photomicrographs of the Weld Zone of the Capsule Endcap Shown in Figure 20	44

22	As-Fabricated Hastelloy C-4 Capsule No. 7R After Being Subjected to an External Hydrostatic Pressure of 20,750 psi	47
23	An As-Fabricated Hastelloy C-276 Inner Capsule After Being Subjected to an External Hydrostatic Pressure of Approximately 9700 psi	50
24	Photomicrographs of the Weld Zone Adjacent to the Point of Impact for the Hastelloy S Capsule Subjected to the Primary Impact Test After Aging at 650° for 8000 h	52
25	The Hastelloy S Capsule Aged at 650°C for 8000 h After Being Subjected to an External Hydrostatic Pressure of 24,500 psi for 30 Minutes	53
26	Sketch of Failed Capsule Showing Location of Crack	56
27	Section of the Endcap of the Failed Hastelloy C-4 Capsule--180° Removed from the Crack Area	57
28	Micrographs Showing the Weld Zone of the Endcap Section Shown in Figure 27	58
29	Section of the Endcap of the Failed Hastelloy C-4 Capsule--Taken from the Crack Area	59
30	Micrographs Showing the Crack Area of the Endcap from the Failed Hastelloy C-4 Capsule	60
31	Prototype Hastelloy S and Hastelloy C-4 Outer Capsules Aged at 725°C for 8000 h	61
32	Weld Zone Adjacent to the Point of Impact of the Hastelloy S Capsule Subjected to the Primary Impact Test After Being Aged at 725°C for 8000 h	63
33	Photomicrographs of the Unimpacted Endcap from the Hastelloy S Capsule Subjected to the Primary Impact Test After Aging at 725°C for 8000 h	64
34	Micrographs of the Weld Zone Adjacent to the Point of Impact of the Hastelloy C-4 Capsule Subjected to the Primary Impact Test After Being Aged at 725°C for 8000 h	65
35	Impacted Endcap from a Hastelloy C-4 Capsule Subjected to the Secondary Impact Test After Being Aged at 725°C for 8000 h	66
36	The Hastelloy S Capsule Aged at 725°C for 8000 h After Being Subjected to an External Hydrostatic Pressure of 24,250 psi	68
37	The Hastelloy C-4 Capsule Aged at 725°C for 8000 h After Being Subjected to an External Pressure of 20,600 psi	71
38	Impacted Endcap of the Hastelloy C-276 Inner Capsule Subjected to the Secondary Impact Test After Being Aged at 725°C for 8000 h	74
39	The Hastelloy C-276 Inner Capsule Aged at 725°C for 8000 h After Being Subjected to an External Hydrostatic Pressure of 12,600 psi	75
40	Effects of Thermal Aging on the Room-Temperature Tensile Properties of Hastelloy S--3.5-in.-dia Barstock	77
41	Effects of Thermal Aging on the Room-Temperature Tensile Properties of Hastelloy C-4 Specimens--3.5-in.-dia Barstock	78
42	Effect of Aging Temperature on the Room-Temperature Yield Strength of Hastelloy S--Specimens Aged for 8000 h	80

43	Effects of Aging Temperature on the Room-Temperature Yield Strength of Hastelloy C-4 for Specimens Aged for 8000 h	82
44	Effects of Aging on the Charpy Impact Energy of Hastelloy S and Hastelloy C-4--Specimens Machined from 3.5-in.-dia Barstock	83
45	Hastelloy C-276/Hastelloy S Specimens Held in Direct Contact at 800°C for 10,000 h	86
46	Hastelloy C-276/Hastelloy C-4 Specimens Held in Direct Contact at 800°C for 10,000 h	87
47	Sketch of Equipment Used in the Impact Test	92
48	Sketch of Capsule Clip and Correct Impact Angle	93
49	Photograph of a Capsule Just Before Impact	94
50	Autoclave and Auxiliary Equipment Used in the Hydrostatic Pressure Tests	97
51	Equipment Used for the Primary Percussion Test	99
52	Sketch Showing the Five Locations Where the Capsule was Impacted in the Primary Percussion Test	100
53	Orientation of the Capsule and Thermocouples in the Furnace in the Thermal Test	103
54	Sketch of the Equipment Used for the Thermal Quench Test	105
55	Sketch of the Steel Billet Used for the Supplemental Percussion Test	107
56	Sketch Showing Steel Billet Used for the Puncture Test and the Three Locations Where the Capsule was Impacted	109
57	Capsule No. S-14 After Being Subjected to an External Hydrostatic Pressure of 24,500 psi for 30 Minutes	130

TABLES

1	Qualification Tests for the $^{90}\text{SrF}_2$ Heat Source Capsule	8
2	Additional Qualification Test Requirements for the $^{90}\text{SrF}_2$ Heat-Source Capsule as Defined by PNL and DOE	9
3	Supplemental Qualification Tests for the $^{90}\text{SrF}_2$ Heat Source Outer Capsule	10
4	The Testing Sequences for the Hastelloy S Prototype Outer Capsules Used in the Qualification Testing Program	20
5	The Testing Sequences for the Hastelloy C-4 Prototype Outer Capsules Used in the Qualification Testing Program	23
6	Composition and Mechanical Properties of the Hastelloy S and Hastelloy C-4 Used in Fabricating the Outer Capsules and Mechanical Property Specimens	26
7	Composition of the Inert SrF_2 Used to Fill the Inner Capsules	27
8	Weld Penetration of the Seal Welds on the Prototype Outer Capsules as Determined by Radiography	28
9	Composition and Mechanical Properties of the Hastelloy C-276 Used in Fabricating the CVN Specimens	30
10	Results Obtained in the Qualification Testing of the As-Fabricated Hastelloy S and Hastelloy C-4 Prototype Outer Capsules	37
11	The Dimensions of Hastelloy S Capsule No. S-15 Before the Supplemental Pressure Test	45
12	The Dimensions of Hastelloy S Capsule No. S-15 After Being Subjected to an External Hydrostatic Pressure of 24,000 psi	46
13	The Dimensions of Hastelloy C-4 Capsule No. 7R Before the Supplemental Pressure Test	48
14	The Dimensions of Hastelloy C-4 Capsule No. 7R After Being Subjected to an External Hydrostatic Pressure of 20,750 psi	49
15	Results Obtained in the Qualification Testing of Hastelloy S and Hastelloy C-4 Prototype Outer Capsules Aged at 650° for 8000 h	51
16	Dimensions of the Hastelloy S Capsule Aged at 650° for 8000 h Before the Supplemental Pressure Test	54
17	Dimensions of the Hastelloy S Capsule Aged at 650°C for 8000 h After Being Subjected to an External Hydrostatic Pressure of 24,500 psi	55
18	Results Obtained in the Qualification Testing of Hastelloy S and Hastelloy C-4 Prototype Outer Capsules Aged at 725°C for 8000 h	62
19	Dimensions of the Hastelloy S Capsule Aged at 725°C Before the Supplemental Pressure Test	69
20	Dimensions of the Hastelloy S Capsule Aged at 725°C After Being Subjected to an External Hydrostatic Pressure of 24,250 psi	70
21	Dimensions of the Hastelloy C-4 Capsule Aged at 725°C for 8000 h Before Being Subjected to the Supplemental Pressure Test	72

22	Dimensions of the Hastelloy C-4 Capsule Aged at 725°C for 8000 h After Being Subjected to an External Hydrostatic Pressure of 20,600 psi	73
23	Effects of Thermal Aging on the Room-Temperature Tensile Properties of Hastelloy S and Hastelloy C-4	76
24	Room-Temperature Tensile Properties of Aged Hastelloy S and Hastelloy C-4	79
25	The Room-Temperature Tensile Properties of Solution-Heat-Treated Hastelloy S and Hastelloy C-4	81
26	The Effects of Aging on the Room-Temperature Charpy Impact Energy of Hastelloy S and Hastelloy C-4	82
27	Room-Temperature Charpy Impact Energy of Aged Hastelloy S and Hastelloy C-4	84
28	Room-Temperature Charpy Impact Energy of Hastelloy C-276--Subsize Specimens 5 mm Thick	85
29	Parabolic Rate Constants for the Long-Term Oxidation of Hastelloy S and Hastelloy C-4 in Air	146
30	Depth of Metal Affected When Hastelloy S and Hastelloy C-4 Specimens are Oxidized in Air at 600° to 800°C	147
31	Room-Temperature Tensile Data for Hastelloy S Specimens Aged in Air or Vacuum at 600° to 800°C	148
32	Room-Temperature Tensile Data for Hastelloy C-4 Specimens Aged in Air or Vacuum at 600° to 800°C	148
33	The Room-Temperature Charpy Impact Energy of Hastelloy S and Hastelloy C-4 CVN Specimens Heated in Air or Vacuum	150
34	Corrosion of Hastelloy S and Hastelloy C-4 in Seawater at Ambient Temperature	151
35	Room-Temperature Tensile Data for Hastelloy S and Hastelloy C-4 Specimens Exposed to Flowing Seawater at Ambient Temperature (10° to 25°C)	152

1.0 INTRODUCTION

At Hanford, strontium-90, recovered from the fission product stream generated by the reprocessing of spent nuclear fuels, is converted to strontium fluoride. The strontium fluoride is then doubly encapsulated in metal containers and stored in a water basin on the Hanford Reservation. The fluoride preparation, encapsulation, and storage take place in the Waste Encapsulation and Storage Facility (WESF), which is operated for the U.S. Department of Energy (DOE) by Rockwell International (RHO).

The encapsulated $^{90}\text{SrF}_2$ represents an economical source of ^{90}Sr for radioisotope heat-source applications if the $^{90}\text{SrF}_2$ fuel form can be licensed for use in terrestrial applications. A program has been underway for several years at the Pacific Northwest Laboratory (PNL) to develop the technology needed to permit the licensing of $^{90}\text{SrF}_2$ heat sources. The program is currently funded by the By-Products Utilization Program of the DOE. The PNL program is divided into three tasks that are being carried out concurrently:

- Task 1--Chemical and Physical Properties of $^{90}\text{SrF}_2$;
- Task 2-- $^{90}\text{SrF}_2$ Compatibility Studies;
- Task 3--Heat Source Design and Qualification Testing.

Work on the first two tasks has been completed, and the results obtained are summarized in a series of topical reports (Fullam 1976a; Fullam 1976b; Fullam 1977a; Fullam 1977b; Fullam 1980a; Fullam 1981a; Fullam 1981b; Yabusaki 1981). This report summarizes the results of the Task 3 activities to design and qualification-test a $^{90}\text{SrF}_2$ -fueled heat source.

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2.0 OBJECTIVES

The primary objective of Task 3 of the PNL program is to design and qualification-test a $^{90}\text{SrF}_2$ -fueled general-purpose heat source for terrestrial applications. The heat source is to be capable of meeting all licensing requirements currently in effect in the United States. No attempt is to be made by PNL to license the $^{90}\text{SrF}_2$ heat source. However, sufficient qualification testing of the heat source is to be carried out at PNL to permit licensing of the heat source by potential users without the need for additional qualification testing of the bare heat source. By DOE directive, the heat source design is to use as much of the existing WESF $^{90}\text{SrF}_2$ storage capsule as practical and is to provide at least double encapsulation of the $^{90}\text{SrF}_2$.

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3.0 WESF STRONTIUM-90 FLUORIDE STORAGE CAPSULE

The $^{90}\text{SrF}_2$ produced at WESF is loaded into a Hastelloy C-276® inner capsule by pneumatic impaction, which is essentially a cold-pressing operation. The density of the fluoride in the capsule is about 65 to 70% of theoretical. The capsule is closed by tungsten inert gas (TIG)-welding a lid in place. The capsule is leak-checked and decontaminated. The cleaned capsule is then sealed in an outer capsule of 316L stainless steel by TIG welding. The integrity of the outer capsule weld is checked using ultrasonic techniques. After weighing and calorimetry to determine the heat output, the capsule is stored in a water-filled basin.

The strontium-90 isotopic content of the SrF_2 produced at WESF can vary from about 55% to as low as 25 to 30% depending on the age of the fission product strontium. The chemical purity of the SrF_2 is about 94 wt% with metal fluorides making up the bulk of the impurities.

Pertinent data for a typical WESF storage capsule containing strontium fluoride having an initial ^{90}Sr isotopic content of 55% are given in Figure 1. The heat output, power density, and activity of the capsules will vary from the nominal values shown in the figure, depending on the initial ^{90}Sr content, and will decrease with time as the ^{90}Sr decays.

	INNER CAPSULE	OUTER CAPSULE
1. MATERIAL	HASTELLOY C-276	316L STAINLESS STEEL ^(a)
2. INNER DIAMETER	2.01 in.	2.39 in.
3. OUTER DIAMETER	2.25 in.	2.63 in.
4. TOTAL LENGTH	19.05 in.	20.10 in.

STRONTIUM FLUORIDE DATA:

1. AMOUNT ~2.8 kg/CAPSULE
2. CHEMICAL PURITY ~95%
3. ^{90}Sr ISOTOPIC CONTENT ~55%
4. BULK DENSITY ~70% OF THEORETICAL DENSITY
5. ACTIVITY ~140,000 Ci/CAPSULE
6. HEAT LOAD ~960 W/CAPSULE
7. POWER DENSITY ~1.0 W/cm³

^(a) HASTELLOY C-276 WAS USED FOR THE OUTER CAPSULE DURING THE EARLY STAGES OF THE WESF OPERATION

FIGURE 1. Typical Data (At Time of Capsule Loading) for a WESF $^{90}\text{SrF}_2$ Storage Capsule

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4.0 HEAT SOURCE LICENSING REQUIREMENTS

4.1 BASIC LICENSING REQUIREMENTS

A radioisotope heat-source capsule cannot be licensed under existing Federal regulations. A power generator, or similar device, containing a radioisotope heat source can be licensed. The basic licensing requirements for radioisotope-fueled power generation systems to be used for terrestrial applications are defined in the United States Atomic Energy Commission Regulatory Guide No. 6.3 (Anon 1974). The requirements set forth in the guide are based, with certain modifications, on the guidelines set forth in the International Atomic Energy Agency Safety Series No. 33 (Anon 1970). Additional requirements for radioisotope-fueled power generators are defined in 10 CFR, Parts 20 and 71 (Anon 1980).

4.2 HEAT SOURCE QUALIFICATION TEST REQUIREMENTS

Based on the various national and international regulations, there are several qualification tests a radioisotope-fueled heat source must meet in order to be licensed as part of a power generation system for terrestrial applications. These qualification test requirements are defined in Table 1. The thermal test and the thermal quench requirements spelled out in Table 1 assume that the metal clad/ $^{90}\text{SrF}_2$ interface temperature of the heat source will be limited to a maximum of 800°C. This limitation is based on the results of long-term compatibility tests at PNL involving WESF $^{90}\text{SrF}_2$ and Hastelloy C-276 (Fullam 1981b).

IAEA Safety Series No. 33 specified that for certain applications, corrosion, vibration, irradiation, and creep tests may be specified for the heat-source capsule by competent authority. To date, the Nuclear Regulatory Commission (NRC) has not defined specific tests in each area for radioisotope heat sources. The Department of Energy and PNL have specified oxidation and seawater corrosion requirements for the heat-source outer capsule (see Section 4.3). Extensive tests were carried out at PNL to evaluate WESF $^{90}\text{SrF}_2$ compatibility with potential containment materials. No vibration, irradiation, or creep qualification-test requirements were defined for the heat-source. It was decided by DOE and PNL that a vibration test was best applied to the power generator system, and that irradiation and creep tests were not critical to the $^{90}\text{SrF}_2$ heat-source capsule being developed at PNL.

4.3 DOE-PNL QUALIFICATION TEST REQUIREMENTS

In addition to the qualification test requirements listed in Table 1, DOE and PNL have defined additional requirements that the $^{90}\text{SrF}_2$ heat-source capsule must meet. These requirements are based on the fact that the heat-source design will provide at least double encapsulation of the $^{90}\text{SrF}_2$. The additional requirements, which are designed to enhance the usefulness and inherent safety of the $^{90}\text{SrF}_2$ heat source, are given in Table 2. The critical requirement defined by DOE and PNL, based on double encapsulation of the $^{90}\text{SrF}_2$, is that

TABLE 1. Qualification Tests for the $^{90}\text{SrF}_2$ Heat Source Capsule

1. Pressure Test

The heat-source capsule shall not leak at a rate greater than $1 \times 10^{-8} \text{ cm}^3/\text{s}$ after being subjected to an external hydrostatic pressure of 14,500 psi (1000 bar).

2. Percussion Test

The capsule shall not leak at a rate greater than $1 \times 10^{-8} \text{ cm}^3/\text{s}$ after being struck by the flat face of a 2.5-cm-dia (~1-in.) steel billet with an impact energy equivalent to 7 kg-m (~50 ft-lb). The billet edges shall be rounded off to a radius of not less than 3 mm, and the capsule shall rest on a lead sheet equal to or less than 25 mm thick with a Brinell hardness of 3.5 to 4.5. The lead sheet shall cover a greater area than the capsule and shall rest on a smooth, solid surface. A fresh lead sheet shall be used for each test, and the capsule shall be struck in such a manner that the maximum damage will result.

3. Impact Test

The capsule shall not leak at a rate greater than $1 \times 10^{-8} \text{ cm}^3/\text{s}$ when dropped from a height of 9 m (approximately 30 ft) onto a steel plate at least 1.25 cm (~1/2-in.) thick that has been wet-floated onto a concrete block with a mass of at least 10 times that of the capsule and set on firm soil. The steel plate shall be flat and horizontal, and the capsule shall be dropped in such a manner that the maximum possible damage will result.

4. Thermal Test

The capsule shall not leak at a rate greater than $1 \times 10^{-8} \text{ cm}^3/\text{s}$ after being heated to its maximum operating temperature of 800°C and then held at 800°C for 30 min.

5. Thermal Quench Test

The capsule shall not leak at a rate greater than $10^{-8} \text{ cm}^3/\text{s}$ after being heated to its maximum operating temperature of 800°C and then plunged into a water-ethylene glycol solution at 32°F (0°C). The capsule shall remain for 1 h in the circulating solution, which shall at no time exceed 41°F (5°C).

the heat-source outer capsule must be capable of meeting the qualification test requirements given in Table 1 in the post-service as well as as-fabricated condition, without support from the inner capsule. This means that the outer capsule must meet the qualification test requirements despite the degradation in mechanical properties of the outer capsule material that may result from long-term aging reactions at heat-source operating temperatures.

The impact test defined in Table 1 requires that the heat-source capsule must be dropped in such a manner that the maximum possible damage will result. This can only occur if the outer capsule contains an inner capsule filled with strontium fluoride. Therefore, the impact qualification test must be carried out using both the outer and inner capsules. The other qualification tests defined in Table 1 must be carried out, however, using just the outer capsule.

TABLE 2. Additional Qualification Test Requirements for the $^{90}\text{SrF}_2$ Heat-Source Capsule as Defined by PNL and DOE

1. The outer capsule of the $^{90}\text{SrF}_2$ heat source must meet the qualification tests defined in Table 1 in both the as-fabricated and post-service conditions, without support from the inner capsule.
2. The outer capsule of the $^{90}\text{SrF}_2$ heat source must be sufficiently oxidation resistant that air oxidation occurring during the service life of the heat source will not degrade the properties of the capsule to the extent that it cannot meet the qualification test requirements defined in Table 1. The oxidation requirement for the outer capsule is based on the conditions that:
 - a. the outer capsule surface temperature will not exceed 800°C , and
 - b. the service life of the heat source will not exceed 10 years.

The ability of the heat-source outer capsule to meet the oxidation requirement may be demonstrated by the extrapolation of reliable short-term oxidation data using established procedures.

3. The outer capsule of the $^{90}\text{SrF}_2$ heat source must be sufficiently resistant to seawater corrosion so that the accidental exposure of the heat source to seawater for a period of 1 year will not degrade the properties of the capsule to the extent that it cannot meet the qualification test requirements given in Table 1. This requirement is based on the assumption that a heat source that is accidentally exposed to a seawater environment will be retrieved from the environment within a period of 1 year.^(a)

(a) It is anticipated that material chosen for the outer capsule will be very resistant to seawater corrosion and that the capsule will be able to withstand exposure to seawater for periods much in excess of 1 year without suffering serious degradation of properties. If, however, the heat source is to be used in undersea service, additional protection against seawater corrosion may be required.

4.4 SUPPLEMENTAL QUALIFICATION TESTS

The American National Standards Institute (ANSI) has produced a standard for sealed radioactive sources--ANSI-N542 (Anon 1977). This standard does not currently apply to radio-isotope-fueled power generation systems, but there are indications NRC may, in the future, apply the standard to such systems. ANSI-N542 defines certain test requirements for large-size radioactive sources that are more stringent, or which supplement the qualification test requirements listed in Table 1. Based on the ANSI-N542 requirements, PNL developed three supplemental qualification tests to which the $^{90}\text{SrF}_2$ heat-source outer capsule was subjected in both the as-fabricated and post-service conditions. These supplemental qualification tests are presented in Table 3. Since the tests described in Table 3 are not currently part of the NRC requirements for licensing power generator systems, failure of the heat-source capsule to meet the tests would not currently preclude its use in licensable power generation systems. The inherent safety of the $^{90}\text{SrF}_2$ heat source would be increased, however, if it did meet the supplemental test requirements given in Table 3.

TABLE 3. Supplemental Qualification Tests for the $^{90}\text{SrF}_2$ Heat Source Outer Capsule

1. Supplemental Pressure Test

The heat-source outer capsule shall not leak at a rate greater than 1×10^{-8} cm^3/s after being subjected to an external hydrostatic pressure of 24,000 psi (or the pressure at which the capsule collapses).

2. Supplemental Percussion Test

The capsule shall not leak at a rate greater than 1×10^{-8} cm^3/s after being struck by the flat face of a 2.5-cm-dia (~1-in.) steel billet with an impact energy equivalent to 20 kg-m (~145 ft-lb). The billet edges shall be rounded off to a radius of not less than 3 mm, and the capsule shall rest on a lead sheet equal to or less than 25 mm thick with a Brinell hardness of 3.5 to 4.5. The lead sheet shall cover a greater area than the capsule and shall rest on a smooth, solid surface. A fresh lead sheet shall be used for each test, and the capsule shall be struck in such a manner that the maximum possible damage will result.

3. Puncture Test

The capsule shall not leak at a rate greater than 1×10^{-8} cm^3/s after being struck by a 3-mm-dia pin, rigidly attached to a 2.5-cm-dia steel billet, with an impact energy equivalent to 1 kg-m. The pin shall be 6 mm long and machined from one end of the steel billet. The impact end of the pin shall be hemispherical. The capsule shall rest on a steel plate at least 1.25 cm (0.5 in.) thick which covers an area greater than that of the capsule and which rests on a smooth, solid surface. The capsule shall be struck in such a manner that the maximum possible damage will result.

5.0 HEAT SOURCE DESIGN

By sponsor-directive, the general-purpose $^{90}\text{SrF}_2$ heat source being developed at PNL must use as much of the WESF $^{90}\text{SrF}_2$ storage capsule as practical. Unfortunately, preliminary tests and theoretical calculations showed that the WESF storage capsule could not meet the qualification test requirements outlined in Tables 1 and 2. Therefore, it was decided by PNL and the sponsor to design a new $^{90}\text{SrF}_2$ heat source that would utilize the existing inner capsule from the WESF $^{90}\text{SrF}_2$ storage capsule and a new outer capsule. The design philosophy used in developing the new heat source was that the existing inner capsule would provide the primary containment barrier for the $^{90}\text{SrF}_2$, while the new outer capsule would provide the structural strength needed to meet the licensing requirements, without support from the inner capsule. It was further decided that the outer capsule would have to meet the licensing requirements in both the as-fabricated and post-service conditions.

The dimensions of the WESF Hastelloy C-276 inner capsule establish the basic dimensions for the $^{90}\text{SrF}_2$ heat source. Figure 2 shows the specifications for the Hastelloy C-276 inner capsule. Based on these specifications, the new $^{90}\text{SrF}_2$ heat source will have a length-to-diameter (L/D) ratio in the range of six to eight, depending on the dimensions of the outer capsule. The maximum heat output of the heat source would be ~950 watts at the time of fabrication, assuming the $^{90}\text{SrF}_2$ had a strontium-90 isotopic content of 55%.

The heat-source outer capsule was designed to meet the qualification-tests requirements given in Tables 1 and 2 using Hastelloy S® as the capsule material. Hastelloy C-4® was selected as a backup material in case the Hastelloy S had to be rejected for any reason. Hastelloy S was selected for the outer capsule based on its high-temperature mechanical properties, thermal stability, seawater corrosion resistance, oxidation resistance, weldability, availability, and compatibility with the Hastelloy C-276 inner capsule. Other materials considered for use include Hastelloy C-4, Inconel 625,® and Inconel 617.®

The 1000-bar hydrostatic pressure test and the impact test most severely test the integrity of the outer capsule. In designing the new outer capsule, stress analyses focused on the 1000-bar external pressure and the 30-ft-drop impact loadings. The capsule wall thickness was determined by consideration of the collapse condition for external pressure. The 30-ft-drop condition resulted in maximum damage for an impact on the capsule corner, and, consequently, the corner joint design was dictated by the loading encountered during impact. The design analyses were based on the mechanical properties Hastelloy S would possess after extended aging at anticipated heat-source operating temperatures. Complete details of the design calculations are provided in Simonen (1980a; 1980b).

® Cabot Corp., Kokomo, Indiana.
® Cabot Corp., Kokomo, Indiana.
® Huntington Corp., Huntington, West Virginia.
® Huntington Corp., Huntington, West Virginia.

TUBING ECCENTRICITY = $\pm 10\%$ OF NOMINAL WALL THICKNESS

FIGURE 2. Dimensions of the WESF $^{90}\text{SrF}_2$ Hastelloy C-276 Inner Capsule

The design calculations indicated the need for a thick-wall outer capsule that utilized an interlocking-endcap design. Preliminary external pressure and impact tests were conducted on outer capsules fabricated from AISI-1018 low-carbon steel to evaluate the adequacy of the design. Figure 3 shows the steel capsules used for the preliminary tests. The short section of high-pressure steel tubing welded to one endcap of the capsule was used to helium leak-check the capsule before and after testing. The AISI-1018 low-carbon steel was selected because its yield strength is similar to that of the thermally aged Hastelloy S. By varying the temperature of the low-carbon steel capsules, it was possible to vary the impact energy of the steel and duplicate the impact energy of thermally aged Hastelloy S. Therefore, the impact tests with the steel capsules were carried out at various temperatures to determine

FIGURE 3. Components of the Outer Capsule and a Completed AISI-1018 Low-Carbon Steel Outer Capsule Ready for Testing

the impact energy needed for the capsules to meet the impact-test requirement, and to determine if the impact energy of thermally aged Hastelloy S was adequate for the capsule design proposed.

Results obtained with the low-carbon steel capsules demonstrated the adequacy of the proposed outer capsule design. Based on the design calculations and the preliminary test results, the design shown in Figure 4 was chosen for the outer capsule. The design calls for a nominal capsule inner diameter of 2.375 in. and a nominal wall thickness of 0.500 in., which total 3.375 in. for a nominal outer diameter. The capsule inner diameter of 2.375 in. was dictated by the nominal 2.250-in. outer diameter of the Hastelloy C-276 inner capsule and the need for adequate clearance between the two capsules.

The 9-m drop impact requirement was the primary consideration in the design of the interlocking end closure. Maximum damage results when the capsule is dropped on its corner in an orientation such that the capsule's center of gravity is in line with the impact point and the direction of travel. The presence of an inner capsule filled with SrF_2 increases the severity of the impact. Such an impact results in maximum deformation of the closure weld. The interlocking design selected for the end closure reduces to a minimum the plastic deformation at the closure weld location that results from the impact loading. The interlocking design also reduces the extent of penetration required for the closure weld. This is an important consideration when the welding must be done remotely in a shielded facility such as a hot cell. The endcap design selected requires a weld penetration of 0.1 in., which should be achievable in a hot cell using TIG welding without filler metal.

The design shown in Figure 4 utilizes two endcaps for each capsule. The capsule body would be fabricated by core-drilling bar stock. The design could be modified to utilize a single endcap. In the latter case, the capsule body would be fabricated from bar stock by drilling and reaming the required hole. This approach would eliminate one seal weld and one potential problem area. The double endcap design was selected for the prototype capsules used in the qualification tests. Discussion with Cabot Corp., which supplied the test capsules (see Section 6.4.1), indicated, however, that a single endcap design was economically more feasible than the double endcap design used for the prototype capsules. For production of a large number of heat-sources capsules, it would probably be technically and economically advantageous to use a single endcap design.

FIGURE 4. Design Selected for the $^{90}\text{SrF}_2$ Heat-Source Outer Capsule

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6.0 HEAT SOURCE QUALIFICATION TESTING

The qualification testing program was designed to demonstrate that the general-purpose $^{90}\text{SrF}_2$ heat source developed at PNL was capable of meeting the various licensing and design requirements defined in Section 4.0. The program was divided into three phases:

- laboratory-scale studies to evaluate the oxidation resistance of the heat-source outer capsule and to show that the capsule can meet the oxidation requirement defined in Table 2;
- laboratory-scale seawater corrosion studies to evaluate the corrosion resistance of the heat-source outer capsule and to show that the capsule can meet the seawater corrosion requirement defined in Table 2;
- qualification testing of prototype heat-source outer capsules to show that the capsule can meet the requirements defined in Tables 1 and 2 in both the as-fabricated and post-service (thermally aged) condition.

Details of the oxidation and seawater corrosion studies were presented in Fullam (1980b, 1981c) and are summarized in the Appendix (Sections 8.4 and 8.5). Details of the qualification testing of the prototype capsules are described in this section of the report.

6.1 TESTING REQUIREMENTS

6.1.1 Qualification Testing Matrix

The basic licensing requirements that the heat source must meet are given in Table 1. Five primary qualification tests are required to show that the heat source is capable of meeting the licensing requirements given in Table 1. They are:

1. an impact test (9-m drop);
2. a hydrostatic pressure test (1000 bar);
3. a percussion test (7 kg-m impact energy);
4. an 800°C thermal test; and
5. a thermal quench test (800°C to <0°C).

The procedures used in carrying out the primary qualification tests are presented in the Appendix (Section 8.1). The DOE- and PNL-imposed requirements that the heat source must meet (Table 2) specify that the outer capsule of the heat source must meet the licensing requirements given in Table 1 without support from the inner capsule,^(a) and it must meet the requirements in both the as-fabricated and post-service conditions. The latter requirement means that qualification testing must be done on thermally aged as well as as-fabricated outer capsules.

(a) In the primary impact test, the outer capsule must contain an inner capsule filled with SrF_2 because this increases the severity of the test.

When Hastelloy S and Hastelloy C-4 are aged between 450° to 900°C for extended periods of time, each suffers a significant loss of ductility and Charpy impact energy (Matthews 1976; Fullam 1981c). This degradation in properties can affect the ability of an outer capsule fabricated from either material to meet the licensing requirements after it has been in service for an extended period of time. In the qualification testing program, it was impossible to reproduce the conditions the outer capsule would encounter in actual heat-source service. It was decided, therefore, to qualification-test some of the outer capsules after they had been aged at 650°C or 725°C for 8000 h. Selection of the aging conditions was based on the following considerations:

- Compatibility considerations dictate that the Hastelloy C-276/⁹⁰SrF₂ interface temperature should not exceed 800°C during heat-source service (Fullam 1981b). Allowing for a realistic temperature drop between the inner and outer capsules of the heat source, the surface temperature of the outer capsule should not exceed about 725°C. To ensure maximum conversion efficiency for the power generation system, the outer capsule surface temperature should be as high as possible, and it is unlikely that the temperature would be below 650°C in a well-designed system. Therefore, aging temperatures of 650°C and 725°C were selected for the qualification tests. As will be seen later, this is a temperature range where aging reactions have a significant effect on the properties of Hastelloy S and Hastelloy C-4.
- Program time restraints prevented the aging of capsules for more than 8000 h. This time was adequate to ensure that the impact properties and ductilities of the Hastelloy C and Hastelloy C-4 used for the prototype outer capsules were reduced to near minimum values (see Section 8.4).

A number of the outer capsules were also qualification-tested in the as-fabricated condition.

Hastelloy S is the primary candidate material for the heat-source outer capsule. It was decided to subject each Hastelloy S prototype outer capsule to a single primary qualification test. Since there are five primary qualification tests and the capsules must be tested in three different conditions (as-fabricated, aged at 650°C, and aged at 725°C), a total of 15 Hastelloy S outer capsules were required for a complete test matrix.

Hastelloy C-4 was selected as the backup material for the outer capsule. Funding limitations prevented testing a complete set (15) of Hastelloy C-4 capsules, and testing was limited to seven capsules. The Hastelloy C-4 capsules were subjected to only three primary qualification tests: impact, pressure, and percussion.

If a test capsule passed the primary qualification test without significant damage, it was subjected to a series of four secondary qualification tests. These were simplified versions of the primary tests. The procedures used for the secondary qualification tests are described in Section 8.1. This approach allowed a substantial amount of data to be obtained from a relatively small number of capsules.

Several of the prototype capsules, which passed the primary and secondary qualification tests, were subjected to one or more supplemental qualification tests to show that the capsules would meet the requirements defined in Table 3. Three supplemental tests were used:

- an enhanced percussion test (20 kg-m impact energy);
- a puncture test (ANSI-N542);
- A 24,500 psi hydrostatic pressure test.^(a)

The procedures used for the supplemental qualification tests are described in Section 8.2. As discussed in Section 4.0, failure of the outer capsules to meet the supplemental qualification tests would not currently preclude the heat source from being used in a licensable power generation system.

The qualification testing sequences for the 15 Hastelloy S prototype outer capsules are given in Table 4. The testing sequences for the seven Hastelloy C-4 capsules are given in Table 5.

6.1.2 Mechanical Property Tests

To determine the effects of thermal aging on the mechanical properties of Hastelloy S and Hastelloy C-4, tensile and Charpy V-notch (CVN) specimens of each alloy were aged at 650° and 725°C for 2500, 5000 and 8000 h. Two tensile and three CVN specimens were aged for each time period. The test specimens were fabricated from the same heats of metal used in fabricating the prototype outer capsules. After aging, the specimens were tested at room temperature. Several specimens of each alloy were also tested without aging.

Hastelloy C-276 CVN specimens were aged at 750°, 725° and 800°C to determine the effects of aging reactions on the ductility and impact properties of the alloy. The specimens were aged for 2500, 5000 and 8000 h and were impact-tested at room temperature. Specimens of the solution heat-treated alloy were also impact-tested at room temperature.

6.1.3 Metal-Metal Compatibility Tests

The heat-source design calls for an inner capsule of Hastelloy C-276 and an outer capsule of Hastelloy S or Hastelloy C-4. The possibility exists that interactions could occur between the inner and outer capsules at heat-source operating temperatures that could affect the integrity of either or both capsules. To determine if such interaction was possible, tests were carried out to evaluate the compatibility of Hastelloy C-276 with Hastelloy S and Hastelloy C-4 at 700° and 800°C. The tests lasted up to 10,000 h.

6.2 AUXILIARY MEASUREMENTS

Each prototype outer capsule used in the qualification tests was subjected to a number of measurements and auxiliary tests prior to, during, and after the actual qualification tests. These included weight and dimensional measurements, leak checks, and radiography.

(a) If the capsule collapsed below 24,000 psi, the test was stopped.

TABLE 4. The Testing Sequences for the Hastelloy S Prototype Outer Capsules Used in the Qualification Testing Program

Capsule No.	Capsule Test Condition	Qualification Testing Sequence	
		Primary Test	Impact
S-12(a)	As-Fabricated	Secondary Tests	Pressure Percussion Thermal Thermal Quench
		Supplemental Test	Percussion
		Primary Test	Thermal Quench
S-13	As-Fabricated	Secondary Tests	Thermal Percussion Pressure Impact
		Supplemental Test	Percussion
		Primary Test	Thermal
S-15	As-Fabricated	Secondary Tests	Thermal Quench Pressure Percussion Impact
		Supplemental Tests	Percussion Pressure
		Primary Test	Thermal
S-17	As-Fabricated	Secondary Tests	Percussion Thermal Thermal Quench Impact
		Supplemental Tests	Percussion Pressure
		Primary Test	Pressure
S-20	As-Fabricated	Secondary Tests	Pressure Thermal Thermal Quench Impact
		Supplemental Test	Percussion
		Primary Test	Percussion
S-8(a)	Aged at 650°C	Secondary Tests	Pressure Percussion Thermal Thermal Quench
		Primary Test	Impact
		Supplemental Test	Percussion
S-14	Aged at 650°C	Primary Test	Thermal

TABLE 4. (contd)

Capsule No.	Capsule Test Condition	Qualification Testing Sequence	
		Secondary Tests	Thermal Quench Pressure Percussion Impact
S-18	Aged at 650°C	Supplemental Test	Pressure
		Primary Test	Thermal Quench
		Secondary Tests	Thermal Pressure Percussion Impact
		Supplemental Tests	Percussion Puncture
S-22	Aged at 650°C	Primary Test	Pressure
		Secondary Tests	Impact Percussion Thermal Thermal Quench
		Supplemental Tests	Pressure
		Primary Test	Percussion
S-23	Aged at 650°C	Secondary Tests	Pressure Thermal Thermal Quench Impact
		Supplemental Test	Percussion
		Primary Test	Impact
		Secondary Tests	Pressure Percussion Thermal Thermal Quench
S-9(a)	Aged at 725°C	Primary Test	Percussion
		Secondary Tests	Pressure Thermal Thermal Quench Impact
		Supplemental Test	Pressure
		Primary Test	Impact
S-11	Aged at 725°C	Secondary Tests	Pressure Thermal Thermal Quench Impact
		Supplemental Test	Pressure
		Primary Test	Thermal Quench
		Secondary Tests	Thermal Pressure Percussion Impact
S-16	Aged at 725°C	Supplemental Test	Percussion

TABLE 4. (contd)

Capsule No.	Capsule Test Condition	Qualification Testing Sequence	
S-19	Aged at 725°C	Primary Test	Pressure
		Secondary Tests	Impact Percussion Thermal Thermal Quench
		Supplemental Test	Puncture
S-21	Aged at 725°C	Primary Test	Thermal
		Secondary Tests	Thermal Quench Pressure Percussion Impact

(a) Capsule contained a Hastelloy C-276 inner capsule filled with SrF_2 .

6.2.1 Physical Measurements

A calibrated Mettler Model PS 15 top-loading balance was used to weigh the capsules that did not contain inner capsules. The balance had a maximum capacity of 15 kg, a precision of ± 0.5 g and a readability of 1 g. A calibrated Sauter Model K-120 balance was used to weigh the outer capsules that contained inner capsules. The balance had a capacity of 120 kg, a precision of ± 5 g and a readability of 10 g.

The dimensions of each outer capsule were determined at the 11 locations shown in Figure 5. The major and minor axes were measured at each location using a micrometer that could be read to 0.001 in. Design specifications call for the capsules to have an outer diameter of 3.370 to 3.375 in. It was found, however, that the capsules were not exactly round, and at a given location the diameter varied up to 0.001 in. Therefore, major and minor axes were determined for each location.

6.2.2 Leak Tests

The licensing requirements state that a capsule must not leak at a rate greater than 1×10^{-8} std cm^3/s after being subjected to the qualification tests. A helium mass spectrometer leak detector was used to leak-test the capsules. The instrument used for the tests was a Varian Model 925-40 leak detector that had a sensitivity of 1×10^{-10} std cm^3/s helium. Figure 6 shows a sketch of the equipment arrangement used for the helium leak test. The helium-filled test capsule was placed in a vacuum chamber that was connected to the fore-vacuum (high pressure) port of the leak detector diffusion pump. A mechanical vacuum pump was connected to the line from the vacuum chamber to the diffusion pump. A calibrated standard leak was connected to the vacuum chamber to be used for internal calibration of the leak detector. Standard leaks having helium leak rates of 1.5×10^{-7} , 3.5×10^{-8} and 6×10^{-9} std cm^3/s were used for the calibration.

TABLE 5. The Testing Sequences for the Hastelloy C-4 Prototype Outer Capsules
Used in the Qualification Testing Program

Capsule No.	Capsule Test Condition	Qualification Testing Sequence	
2(a)	As-Fabricated	Primary Test	Impact
		Secondary Tests	Pressure Percussion Thermal Thermal Quench
		Supplemental Test	Percussion
3	As-Fabricated	Primary Test	Pressure
		Secondary Tests	Percussion Thermal Thermal Quench Impact
		Supplemental Test	Puncture
7R	As-Fabricated	Primary Test	Percussion
		Secondary Tests	Pressure Thermal Thermal Quench Impact
		Supplemental Tests	Percussion Pressure
4	Aged at 650°C	Primary Test	Pressure
		Secondary Tests	Impact Percussion Thermal Thermal Quench
		Supplemental Tests	Pressure Percussion Puncture
1(a)	Aged at 725°C	Primary Test	Impact
		Secondary Tests	Pressure Percussion Thermal Thermal Quench
6	Aged at 725°C	Primary Test	Pressure
		Secondary Tests	Impact Percussion Thermal Thermal Quench
		Supplemental Test	Pressure
7	Aged at 725°C	Primary Test	Percussion
		Secondary Tests	Pressure Thermal Thermal Quench Impact
		Supplemental Tests	Percussion Puncture

CAPSULE NO

FIGURE 5. Locations Where the Outer Capsule Dimensions were Determined

The helium leak detector is not adequate for detecting gross leaks in a capsule ($>1 \times 10^{-4}$ std cm^3/s helium). To detect any gross leak in a capsule, the capsule was subjected to a water leak test before the helium leak test. In the water leak test, the capsule was placed in a vessel fabricated from 6-in.-dia Lucite tubing. The vessel was filled with a dilute soap solution, sealed, and a rough vacuum applied (~ 28 in. Hg vacuum). Bubbles

FIGURE 6. Equipment Arrangement for Leak Testing a Helium-Filled Capsule

emerging from the capsule indicated the presence of a leak. It is estimated that the water leak test could detect leaks as small as 1×10^{-4} std cm^3/s helium.

6.2.3 Radiography

Radiography was used to determine the weld penetration obtained with prototype outer capsule seal welds and to identify any possible defects in the welds. Radiography was provided by the Hanford Engineering Development Laboratory (HEDL), which is operated for DOE by the Westinghouse Electric Corporation. The capsule welds were radiographed before testing and after the primary qualification test was completed. The radiography was carried out using a ^{90}Ci ^{192}Ir source having a 0.100-in. dia.

6.3 FABRICATION OF CAPSULES AND TEST SPECIMENS

6.3.1 Outer Capsules

The prototype outer capsules used in the qualification testing program were fabricated according to the design specifications shown in Figure 4. Each capsule had a 0.5-in. wall thickness and utilized the interlocking endcap design. The capsules were machined from 3.5-in.-dia bar stock by the Stellite Division of the Cabot Corp. Sixteen^(a) Hastelloy S outer

(a) One spare Hastelloy S capsule was fabricated and aged at 650°C but was not qualification-tested.

capsules were fabricated from a single heat of the alloy. The chemical composition and mechanical properties of the Hastelloy S used, based on the certified report of tests supplied by Cabot Corp., is given in Table 6.

The seven Hastelloy C-4 outer capsules used in the testing program were fabricated from a single heat of the alloy. The chemical composition and mechanical properties of the material used are also given in Table 6.

All capsule components were examined by an authorized representative of PNL to ensure that they conformed to the design specifications before the endcaps were welded in place.

TABLE 6. Composition and Mechanical Properties^(a) of the Hastelloy S and Hastelloy C-4 Used in Fabricating the Outer Capsules and Mechanical Property Specimens

Composition, wt%	Hastelloy S 3.5-in.-Dia Bar Stock--Solution Heat Treated at 1950°F and Centerless Ground	Hastelloy C-4 3.5-in.-Dia Bar Stock--Solution Heat Treated at 1950°F and Centerless Ground
Al	0.30	
B	0.009	
C	0.007	0.004
Co	0.05	0.17
Cr	14.91	15.4
Cu	0.05	
Fe	0.97	0.84
La	0.028	
Mn	0.64	0.08
Mo	14.84	15.31
Ni	Balance	Balance
P	0.006	0.011
S	<0.002	0.004
Si	0.43	0.03
Ti		0.27
W	<0.10	
<u>Mechanical Properties</u>		
Ultimate Tensile Strength, MPa	817	754
Yield Strength, MPa	335	328
% Elongation in 4 D	54.0	65.0
Hardness, Rock B	94	

(a) Based on the Certified Reports of Tests supplied by the Cabot Corp.

6.3.2 Inner Capsules

The five Hastelloy C-276 inner capsules used in the qualification tests were obtained from RH0. The capsules were standard production-grade inner capsules that are used in the WESF $^{90}\text{SrF}_2$ operation, and they had the typical dimensions shown in Figure 2.

The inner capsules were filled with nonradioactive strontium fluoride prior to encapsulation in the outer capsules. The strontium fluoride was compacted into the inner capsules to about 70% of theoretical density by cold-step pressing. The composition of the inert SrF_2 was similar to that of the nonradioactive fluoride used in the long-term compatibility tests and is shown in Table 7.

The filled capsules were sealed by tungsten inert gas (TIG)-welding the capsule lids in place. After welding, the capsules were subjected to the water leak check.

6.3.3 Outer Capsule Welding

The prototype capsules were sealed by TIG-welding the endcaps in place without using filler metal. Welding of the outer capsules was provided by the Cabot Corp. Hastelloy C-276 inner capsules, provided by PNL, were inserted in five of the outer capsules before welding. Test welds were performed and examined prior to welding of the capsules, and a representative of PNL was present at the Cabot Corp. facility throughout the welding operations.

When the welding operations were completed, the capsules were shipped to PNL. At PNL the capsules were subjected to a primary water-leak check that had a sensitivity of $\sim 1 \times 10^{-4} \text{ cm}^3/\text{s}$. All of the capsules passed the leak check without difficulty. The capsule welds were then radiographed. The design specifications call for a full penetration weld of 0.10 in. The initial test welds conducted at the Cabot Corp. showed that this degree of penetration could be achieved without filler metal. Radiography of the capsule welds by HEDL showed, however, that the weld penetrations varied from 0.020 to 0.080 in., with the majority of the welds being between 0.04 and 0.07 in. (see Table 8). Metallographic examination of

TABLE 7. Composition of the Inert SrF_2 Used to Fill the Inner Capsules

Element	wt%	Element	wt%
Sr	65.9	Mn	0.01
F	29.0	Na	2.4
Al	0.26	Nd	1.0
Ba	0.70	Ni	0.02
Ca	0.21	Pb	0.06
Cd	0.01	Si	0.02
Cr	0.050	H ₂ O	<0.01
Fe	0.020	NO ₃	<0.01
K	0.01	O ⁻²	<0.01
Mg	0.13		

TABLE 8. Weld Penetration of the Seal Welds on the Prototype Outer Capsules as Determined by Radiography

<u>Material</u>	<u>Capsule No.</u>	<u>Weld Penetration, in.</u>	
		<u>Minimum</u>	<u>Maximum</u>
Hastelloy S	S-8(a)	0.050	0.070
	S-9(a)	0.050	0.065
	S-10	0.040	0.055
	S-11	0.055	0.065
	S-12(a)	0.045	0.060
	S-13	0.020	0.055
	S-14	0.050	0.080
	S-15	0.045	0.076
	S-16	0.045	0.050
	S-17	0.050	0.065
	S-18	0.050	0.065
	S-19	0.055	0.060
	S-20	0.050	0.055
	S-21	0.050	0.065
	S-22	0.060	0.075
	S-23	0.055	0.075
Hastelloy C-4	1(a)	0.040	0.045
	2(a)	0.045	0.045
	3	0.040	0.055
	4	0.040	0.045
	6	0.050	0.055
	7	0.045	0.065
	7R	0.030	0.050

(a) Indicates capsules that were rewelded; weld penetration was essentially 100% (0.10 in.) in rewelded capsules

several of the capsule endcaps, after the qualification tests were completed, showed that the actual weld penetrations agreed very closely with values determined by radiography.

It was decided that the five outer capsules that contained inner capsules, and which were to be subjected to the primary impact test, would be rewelded before testing. The remaining capsules were qualification-tested without rewelding. The rewelding was carried out at PNL. The endcaps were removed from each of the five capsules by machining a narrow V-notch through the weld metal as shown in Figure 7. The capsules were then resealed by TIG-welding the endcaps in place using filler metal (Hastelloy S or Hastelloy C-4 depending on the capsule). Radiography of the rewelded capsules showed the weld penetration to be essentially 100%. This was confirmed by metallography after the capsules were tested.

FIGURE 7. Sketch Showing Location of Notch Used to Remove Endcaps from Capsules That were to be Rewelded

Radiography also indicated the presence of minor defects in some of the welds, but the defects were not serious enough to warrant further welding.

Prior to the rewelding, Hastelloy C-276 spacers were inserted into the capsules to reduce the potential for longitudinal movement of the inner capsules within the outer capsules.

6.3.4 Helium Filling

The helium leak check used in the qualification tests required that the capsules contain some amount of helium. Two methods were used to fill the prototype outer capsules with helium. The first method involved a bombing technique in which the sealed capsule was placed in an autoclave and held under a very high helium pressure for a long period of time. This procedure was used with only a few capsules because of the very long time required for each capsule.

The second method involved drilling a small tapered hole in one endcap of the sealed capsule (see Figure 8). The capsule was then placed in a vacuum chamber that was alternately evacuated and filled with helium several times until the capsule contained essentially pure helium. A close-fitting tapered plug, of the same metal as the capsule, was then inserted in the hole in the endcap and TIG-welded in place to seal the capsule.

6.3.5 Mechanical Property Specimens

The Hastelloy S and Hastelloy C-4 tensile and CVN specimens used in the qualification testing program were fabricated from the same heats of metal used in fabricating the outer capsules. The specimens were machined at PNL from 3.5-in.-dia bar stock, the same size of material used for the outer capsules. The tensile specimens were fabricated in accordance

FIGURE 8. Sketch Showing Hole and Pin Arrangement Used to Fill Outer Capsules with Helium

with ASTM Specification E8-78 (Anon 1978) and had a reduced section diameter of 0.500 in. The CVN specimens were fabricated according to ASTM Specification E23-72 (Anon 1978) and were the standard 10-mm-square specimens.

The Hastelloy C-276 CVN specimens used in the qualification tests were subsize specimens 5 mm thick by 10 mm wide. The specimens were machined from 0.5-in.-dia bar stock. The chemical composition and mechanical property data for the material, based on the Certified Report of Tests obtained from the manufacturer, are given in Table 9.

TABLE 9. Composition and Mechanical Properties of the Hastelloy C-276^(a) Used in Fabricating the CVN Specimens

Element	wt%	Element	wt%
C	0.003	Ni	Balance
Co	2.06	P	0.017
Cr	15.35	S	<0.005
Fe	6.45	Si	0.03
Mn	0.53	V	0.21
Mo	15.17	W	3.40
<u>Mechanical Properties</u>			
Ultimate Tensile Strength, MPa		745	
Yield Strength, MPa		317	
% Elongation in 4 D		67.4	

(a) 1/2-dia barstock.

Some tensile and CVN specimens were also machined from some of the outer capsules after they had been qualification-tested. The tensile specimens had a reduced section diameter of 0.25 in., and the CVN specimens were 10 mm square.

All of the tensile specimens were tension-tested at room temperature, using a strain rate of 0.02 in./in. The CVN specimens were impact-tested at room temperature.

6.3.6 Metal-Metal Compatibility Capsules

In an operating heat source, the Hastelloy C-276 inner capsule will be in contact with the outer capsule. The possibility exists that the Hastelloy C-276 could react with the outer capsule material to the detriment of one or both capsules. A limited study was carried out, therefore, to evaluate potential interactions between Hastelloy C-276 and Hastelloy S or Hastelloy C-4 at elevated temperatures. The capsule design shown in Figure 9 was used for the tests. The capsules were machined from 1-in.-dia Hastelloy C-276 bar stock. One-half-inch-diameter discs of Hastelloy C-276 and Hastelloy S or Hastelloy C-4 were stacked in the capsules, as shown in Figure 9. An interference fit lid was pressed into the capsule, using a hydraulic press, to provide intimate contact between the discs. The capsule was sealed by TIG-welding the lid in place and leak-checked.

The flat faces of the discs were polished to a metallographic finish using 0.3- μ m alumina for the final polish. Some of the polished discs were oxidized in air at 800°C for 24 h before their insertion into the oxide. This was done to determine what effect the oxide coatings would have on metal-metal compatibility. In the heat source, it is very likely that the capsules would have oxidized surfaces in order to improve their emissivities.

The test capsules were sealed in evacuated quartz envelopes and then heated at 700° or 800°C for up to 10,000 h. At the conclusion of a test, a capsule was cooled to room temperature and sectioned, while care was taken not to damage the contained discs. The discs were removed from the capsule and examined visually for potential interaction. The discs were then subjected to metallographic examination.

6.4 THERMAL AGING

Some of the prototype capsules and mechanical property specimens were tested in the as-fabricated (solution heat-treated) condition. The remaining capsules and specimens were tested after aging at an elevated temperature. The outer capsules were aged at 650°C or 725°C for 8000 h. The Hastelloy S and Hastelloy C-4 tensile and CVN specimens were aged at 650° or 725°C for 2500, 5000 and 8000 h. The Hastelloy C-276 CVN specimens were aged at 650°, 725° or 800°C for 2500, 5000 and 8000 h.

The capsules and mechanical property specimens were aged in air-atmosphere muffle furnaces. The mechanical property specimens were sealed in evacuated quartz envelopes to prevent oxidation during the aging operation. Each muffle furnace was equipped with a solid-state proportioning controller and two separate and independent over-temperature protection (OTP) circuits. The OTP circuits were designed to turn off power to the furnaces if the furnace temperature exceeded the control temperature by more than ~20°C. The furnace

NOTES:

- CAPSULE MACHINED FROM 1-in.-DIA BAR STOCK
- DISCS SECTIONED FROM 1/2-in.-DIA BAR STOCK
- FLAT FACES OF DISCS POLISHED TO METALLOGRAPHIC FINISH USING 0.3- μ m ALUMINA FOR FINAL POLISH
- SOME DISCS OXIDIZED IN AIR AT 800°C FOR 24 HOURS TO FORM OXIDE COATING ON SURFACE
- INTERFERENCE FIT LID PRESSED INTO PLACE USING HYDRAULIC PRESS TO PROVIDE INTIMATE CONTACT BETWEEN DISCS

FIGURE 9. Capsule Design Used for the Metal-Metal Compatibility Studies

temperatures were monitored continuously with calibrated Platinel II thermocouples and calibrated strip-chart recorders. The output of the thermocouples was also monitored intermittently with a calibrated digital thermometer.

6.5 CAPSULE TESTING PROCEDURE

The exact procedure used in qualification-testing the prototype capsules varied somewhat, depending on the specific capsule, but the general procedure was the same for all capsules. The basic steps involved in qualification-testing a capsule were as follows (using as an example Hastelloy S capsule S-19 that was to be subjected to the primary pressure test after aging at 725°C).

1. The capsule parts were machined at the Cabot Corp. and examined by a PNL representative to ensure that they met design specifications.
2. The capsule was sealed by TIG-welding the endcaps in place. Welding was performed at the Cabot Corp. and witnessed by a PNL representative. The capsule was marked with an identifying number.
3. The capsule was shipped to PNL.
4. At PNL the capsule was weighed and its external dimensions determined.
5. The capsule was subjected to the water-leak check.
6. The capsule welds were radiographed.
7. The capsule was filled with helium.
8. The capsule was subjected to the helium-leak check.
9. The capsule was placed in the furnace and held at 725°C for 8000 h.
10. The capsule was removed from the furnace, cooled, weighed and its dimensions determined.
11. The capsule was subjected to the water-leak check.
12. The capsule was subjected to the helium-leak check.
13. The capsule was subjected to the primary pressure test:
 - a. The capsule was subjected to an external hydrostatic pressure of 10,000 psi for 30 min.
 - b. The capsule was weighed and its dimensions determined.
 - c. The capsule was subjected to the water-leak check.
 - d. The capsule was subjected to the helium-leak test.
 - e. The capsule was subjected to an external hydrostatic pressure of 12,000 psi.
 - f. The capsule was weighed and its dimensions determined.
 - g. The capsule was subjected to the water-leak check.
 - h. The capsule was subjected to the helium-leak check.
 - i. The capsule was subjected to an external hydrostatic pressure of 13,000 psi.
 - j. The capsule was weighed and its dimensions determined.
 - k. The capsule was subjected to the water-leak check.
 - l. The capsule was subjected to the helium-leak test.
 - m. The capsule was subjected to an external hydrostatic pressure of 14,500 psi (1000 bar).

- n. The capsule was weighed and its dimensions determined.
 - o. The capsule was subjected to the water-leak check.
 - p. The capsule was subjected to the helium-leak check.
14. The capsule welds were radiographed.
15. The capsule was subjected to the secondary impact test.
- a. The capsule was dropped from a height of 9 m and impacted on the steel plate set in concrete.
 - b. The capsule was weighed and its dimensions determined.
 - c. The capsule was subjected to the water-leak check.
16. The capsule was subjected to the secondary percussion test.
- a. The capsule was impacted at the five locations prescribed using a steel billet having an impact energy of 7 kg-m.
 - b. The capsule was weighed and its dimensions determined.
 - c. The capsule was subjected to the water-leak check.
17. The capsule was subjected to the secondary thermal test.
- a. The capsule was placed in the furnace, heated to 800°C, and held at 800°C for 30 min.
 - b. After cooling to room temperature, the capsule was weighed and its dimensions determined.
 - c. The capsule was subjected to the water-leak check.
18. The capsule was subjected to the secondary thermal quench test.
- a. The capsule was placed in the furnace and heated to 800°C.
 - b. The capsule was removed from the furnace and quenched in a water-ethylene glycol bath held at $\leq 0^{\circ}\text{C}$; the capsule was held in the bath for 1 h.
 - c. The capsule was weighed and its dimensions determined.
 - d. The capsule was subjected to the water-leak check.
19. The capsule was subjected to the helium-leak check.
20. The capsule was subjected to the supplemental puncture test.
- a. The capsule was impacted at the three locations prescribed in the procedure using a steel billet having an impact energy of 1 kg-m. The capsule was examined for visible damage after each impact.
 - b. The capsule was subjected to the water-leak check.
 - c. The capsule was subjected to the helium-leak check.

As mentioned earlier, the same general testing procedure, as outlined above, was applied to all of the prototype capsules; but the specific procedures varied according to the primary qualification test to which the capsule was subjected.

6.6 RESULTS AND DISCUSSION

6.6.1 Qualification Tests

A total of 15 Hastelloy S and 7 Hastelloy C-4 prototype outer capsules were subjected to the five primary and secondary qualification tests as well as the three supplemental tests. Some of the capsules were tested in the as-fabricated conditions, while others were tested after aging for 8000 h at 650° or 725°C. All of the capsules passed the primary and secondary qualification tests, except for the Hastelloy C-4 capsule that had been aged at 650°C. The latter capsule developed a leak after the secondary thermal test (see Sections 6.6.1.2). Capsules subjected to the various supplemental tests passed the final helium-leak test, even though some of the capsules partially collapsed when subjected to the supplemental pressure test. Details of the test results are provided in the following sections, while typical test data for three of the capsules are presented in Section 8.3.

6.6.1.1 As-Fabricated Capsules

Five Hastelloy S and three Hastelloy C-4 prototype outer capsules were subjected to the various qualification tests and supplemental tests in the as-fabricated condition. Figure 10 shows three of the as-fabricated capsules before testing. Results of the various qualification tests are summarized in Table 10. Typical test data (for capsule No. S-12) are given in Section 8.3.1. Table 10 shows that the capsules passed the primary and secondary qualification tests without difficulty. The capsules suffered some minor damage from the qualification tests, primarily the impact tests, but all of the capsules passed the helium-leak check after testing. The capsules also passed the supplemental tests, although the capsules subjected to the supplemental pressure test partially collapsed. Even though the capsules had partially collapsed, they still passed the helium leak test.

The two capsules (Nos. S-12 and 2) subjected to the primary impact test suffered significant distortion of the endcap at the point of impact and the capsule wall adjacent to the point of impact. Figures 11 and 12 show two views of the impacted end of the Hastelloy C-4 capsule (No. 2) after the impact test. The distortion of the endcap and capsule wall is readily apparent from the photographs. In the primary impact test, each capsule was dropped twice and impacted at locations 180° removed from each other. Figure 11 and 12 show the point of initial impact. The distortion resulting from the second drop was essentially identical to that resulting from the initial drop.

Photographs of the impacted end of the Hastelloy S No. S-12, after the initial drop, are shown in Figures 13 and 14. Again, the distortion resulting from the impact is readily apparent.

After qualification testing was completed, the two capsules subjected to the primary impact test were sectioned and the endcaps examined. The endcaps were sectioned and the

FIGURE 10. Prototype Outer Capsules Before Qualification Testing in the As-Fabricated Condition

TABLE 10. Results Obtained in the Qualification Testing of the As-Fabricated Hastelloy S and Hastelloy C-4 Prototype Outer Capsules

Qualification Tests	Hastelloy S Capsule Number					Hastelloy C-4 Capsule Number		
	S-12 ^(a)	S-13	S-15	S-17	S-20	2 ^(a)	3	7R
A. Primary Qualification Tests								
Impact	Pass	---	---	---	---	Pass	---	---
Pressure	---	---	---	Pass	---	---	Pass	---
Percussion	---	---	---	---	Pass	---	---	Pass
Thermal	---	---	Pass	---	---	---	---	---
Thermal Quench	---	Pass	---	---	---	---	---	---
B. Secondary Qualification Tests								
Impact	---	Pass	Pass	Pass	Pass	---	Pass	Pass
Pressure	Pass	Pass	Pass	---	Pass	Pass	---	Pass
Percussion	Pass	Pass	Pass	Pass	---	Pass	Pass	---
Thermal	Pass	Pass	---	Pass	Pass	Pass	Pass	Pass
Thermal Quench	Pass	---	Pass	Pass	Pass	Pass	Pass	Pass
C. Supplemental Tests								
Pressure	---	---	P-C ^(b)	---	---	---	---	P-C
Percussion	Pass	Pass	Pass	---	Pass	Pass	---	Pass
Puncture	---	Pass	---	---	---	---	Pass	---

(a) Capsule contained a Hastelloy C-276 inner capsule filled with inert SrF₂.

(b) P-C--Some distortion of capsule, but the capsule passed the helium-leak test.

areas affected by the impact test subjected to metallographic examination. Figure 15 shows a composite photomicrograph of the impacted end of the Hastelloy C-4 capsule. The micrograph shows the distortion of the endcap and capsule wall which resulted from the impact. The micrograph also shows that some distortion of the interlocking joint resulted from the impact test. Figure 16 shows higher magnification micrographs of the capsule weld zone adjacent to the point of impact. Figures 15 and 16 show that complete weld penetration was obtained when the capsule was rewelded before testing. Close examination of Figure 16 shows that a small crack has started at the edge of the weld zone. The crack apparently was caused by the distortion of the capsule wall that resulted from the impact test. The crack did not affect the integrity of the capsule.

Similar results were obtained with the as-fabricated Hastelloy S capsule (No. S-12) subjected to the primary impact test. Figure 17 show the impacted end of the capsule. The weld zone again showed a small crack at the edge of the weld zone that resulted from the impact loading, but the crack did not affect capsule integrity.

The capsules subjected to the secondary impact test, without an inner capsule present, also suffered some distortion of the endcap at the point of impact. The distortion that

FIGURE 11. The As-Fabricated Hastelloy C-4 Capsule After Being Subjected to the Initial Drop of the Impact Test--View A

FIGURE 12. The As-Fabricated Hastelloy C-4 Capsule After Being Subjected to the Initial Drop of the Impact Test--View B

FIGURE 13. Impacted End of Capsule S-12 After the Initial Impact--
View A

FIGURE 14. Impacted End of Capsule S-12 After the Initial Impact--
View B

FIGURE 15. Photomicrograph of the Affected Endcap of the As-Fabricated Hastelloy C-4 Capsule Subjected to the Primary Impact Test-- Showing the Distortion of the Endcap, Capsule Wall, and Interlocking Joint that Resulted from the Impact Loading

resulted, however, was much less than when an inner capsule was present. Figure 18 shows a composite photomicrograph of the affected endcap of Hastelloy S capsule No. S-17 that was subjected to a secondary impact test. The micrograph shows significant distortion of the endcap at the point of impact, but only slight distortion of the capsule wall and interlocking joint. Higher magnification micrographs of the weld zone are shown in Figure 19. They show the weld penetration was only about 50%, which confirms the results obtained by radiography. The micrographs also fail to show any evidence of cracking in the weld zone as a result of the impact loading. Similar results were obtained with the other as-fabricated Hastelloy S capsules subjected to the secondary impact test.

Figure 20 shows the endcap of an as-fabricated Hastelloy C-4 capsule subjected to the secondary impact test. The impact loading produced only slight distortion of the capsule

FIGURE 16. Photomicrographs of the Weld Zone Adjacent to the Point of Impact of the Endcap from the Hastelloy C-4 Capsule Subjected to the Primary Impact Test

FIGURE 17. Photomicrograph of the Affected Endcap of the Hastelloy S Capsule Subjected to the Primary Impact Test--Showing the Distortion of the Endcap and Capsule Wall

FIGURE 18. Photomicrograph of the Affected Endcap of an As-Fabricated Hastelloy S Outer Capsule Subjected to a Secondary Impact Test--Without an Inner Capsule Present

FIGURE 19. Photomicrographs of the Weld Zone Adjacent to the Point of Impact of the Capsule Endcap Shown in Figure 18

FIGURE 20. The Affected Endcap from an As-Fabricated Hastelloy C-4 Capsule Subjected to the Secondary Impact Test--Without an Inner Capsule Present

wall and interlocking joint. Weld penetration was about 45%. Higher magnification micrographs of the weld zone (Figure 21) show what appears to be the start of a crack at the inner edge of the weld zone. The crack appears to be the result of the stresses developed in the qualification tests rather than a welding defect.

The primary and secondary external hydrostatic pressure tests had no measurable effect on the as-fabricated prototype outer capsules. Dimensional measurements at the 11 locations specified in the test procedure showed that the capsule dimensions were unchanged when the capsules were subjected to an external hydrostatic pressure of 14,500 psi (1000 bar).

FIGURE 21. Photomicrographs of the Weld Zone of the Capsule Endcap Shown in Figure 20

The primary and secondary percussion tests, utilizing an impact energy of 7 kg-m (50 ft-lb), had no significant effect on the as-fabricated capsules. A very slight indentation was visible in the capsule wall at each point of impact, but the depths of the indentation never exceeded 0.003 in.

The primary and secondary thermal and thermal quench tests had no measurable effects on the as-fabricated capsules, except to produce a slight discoloration of the capsule surface due to formation of an oxide film. No scaling of the oxide film was observed when the capsules were quenched from 800°C to 0°C or less.

The supplemental percussion test, using an impact energy of 20 kg-m (145 ft-lb) had no significant effect on the as-fabricated prototype capsules. A slight indentation of the capsule wall was observed at the points of impact. This amounted to no more than 0.005 in. and had no effect on capsule integrity.

The supplemental puncture test, using an impact energy of 1 kg-m (7.2 ft-lb) had no significant effect on the as-fabricated capsules, except to produce a small indentation in the capsule wall at the points of impact. The depth of the indentation never exceeded 0.002 in., and the indentations had no effect on capsule integrity.

As-fabricated capsules subjected to the supplemental pressure test suffered partial collapse; but the capsules passed the helium-leak test. Hastelloy S capsule No. S-15 was pressurized to 24,000 psi. The dimensions of the capsule before and after pressurization are shown in Tables 11 and 12. The as-fabricated Hastelloy C-4 capsule (No. 7R) subjected to the supplemental pressure test collapsed at a pressure of 20,750 psi. Figure 22 shows the capsule after the supplemental pressure test, and Tables 13 and 14 give the capsule dimensions before and after the supplemental pressure test. Despite the fact that both the Hastelloy S

TABLE 11. The Dimensions of Hastelloy S Capsule No. S-15 Before the Supplemental Pressure Test

MINOR AXIS, in.	MAJOR AXIS, in.		CAPSULE TOP	
<u>3.374</u>	<u>3.375</u>	→	1 in.	21 in.
<u>3.374</u>	<u>3.375</u>	→	2 in.	
<u>3.374</u>	<u>3.374</u>	→	2 in.	
<u>3.374</u>	<u>3.374</u>	→	2 in.	
<u>3.374</u>	<u>3.374</u>	→	2 in.	
<u>3.374</u>	<u>3.375</u>	→	1-½ in.	
<u>3.374</u>	<u>3.374</u>	→	1-½ in.	
<u>3.374</u>	<u>3.375</u>	→	2 in.	
<u>3.374</u>	<u>3.374</u>	→	2 in.	
<u>3.374</u>	<u>3.375</u>	→	2 in.	
<u>3.375</u>	<u>3.375</u>	→	2 in.	
		→	1 in.	

and Hastelloy C-4 as-fabricated capsules partially collapsed when subjected to the supplemental pressure test, neither capsule leaked at a rate greater than 1×10^{-8} std cm³/s.

The Hastelloy C-276 inner capsules, which were contained in the outer capsules used for the primary impact tests (Nos. S-12 and 2) were also subjected to certain qualification tests. After testing of the outer capsules was completed, the capsules were sectioned and the inner capsules removed and examined. The inner capsules showed no evidence of damage resulting from qualification testing of the outer capsules. The two inner capsules were then subjected to the water-leak check, which they passed. Both capsules were then subjected to the secondary percussion test using an impact energy of 7 kg-m. The capsules passed the percussion test without damage except for slight indentations (up to 0.003 in. deep) in the capsule wall at the points of impact.

TABLE 12. The Dimensions of Hastelloy S Capsule No. S-15 After Being Subjected to an External Hydrostatic Pressure of 24,000 psi

MINOR AXIS, in.	MAJOR AXIS, in.		CAPSULE TOP	
3.372	3.377	→	1 in.	21 in.
		→	2 in.	
3.344	3.386	→	2 in.	
		→	2 in.	
3.322	3.402	→	2 in.	
		→	2 in.	
3.308	3.420	→	2 in.	
		→	1-½ in.	
3.305	3.422	→	1-½ in.	
3.302	3.427	→	2 in.	
3.306	3.423	→	2 in.	
		→	2 in.	
3.312	3.415	→	2 in.	
		→	2 in.	
3.333	3.403	→	2 in.	
3.355	3.389	→	2 in.	
		→	2 in.	
3.369	3.379	→	1 in.	

One of the Hastelloy C-276 capsules was then subjected to the secondary impact test. The impact test produced some distortion of the capsule endcap, but the capsule passed the water-leak check. The capsule was then subjected to the secondary hydrostatic pressure test. The capsule partially collapsed at a pressure of about 9700 psi, and the test was halted. Figure 23 shows the capsule after the pressure test. Although the capsule had partially collapsed, it passed the water-leak check.

In summary, the as-fabricated Hastelloy S and Hastelloy C-4 prototype outer capsules passed the primary and secondary qualification tests without leaking at a rate greater than 1×10^{-8} std cm³/s. The primary and secondary impact tests produced some distortion of the capsule endcap, weld and interlocking joint but did not affect capsule integrity. The capsules subjected to the supplemental tests did not leak at a rate greater than 1×10^{-8} std

FIGURE 22. As-Fabricated Hastelloy C-4 Capsule No. 7R
After Being Subjected to an External Hydro-
static Pressure of 20,750 psi

TABLE 13. The Dimensions of Hastelloy C-4 Capsule No. 7R Before the Supplemental Pressure Test

MINOR AXIS, in.	MAJOR AXIS, in.		CAPSULE TOP	
3.375	3.375	→	1 in.	21 in.
3.375	3.375	→	2 in.	
3.375	3.376	→	2 in.	
3.375	3.375	→	2 in.	
3.375	3.375	→	2 in.	
3.375	3.375	→	1-½ in.	
3.374	3.374	→	1-½ in.	
3.375	3.375	→	2 in.	
3.375	3.375	→	2 in.	
3.374	3.375	→	2 in.	
3.375	3.376	→	2 in.	
			1 in.	

cm³/s, even though the capsules subjected to the supplemental pressure test partially collapsed. The Hastelloy C-276 inner capsules, present in the outer capsules used for the primary impact tests, were unaffected by the testing of the outer capsules. When the inner capsules were subjected to secondary impact and percussion tests, they passed the tests without difficulty. When subjected to the secondary hydrostatic pressure test, the Hastelloy C-276 inner capsule collapsed at about 9700 psi but passed the water-leak check.

6.6.1.2 Capsules Aged at 650°C

Five Hastelloy S and one Hastelloy C-4 prototype outer capsules were qualification-tested after aging for 8000 h at 650°C. The aged capsules exhibited a dark gray coloration due to the formation of a thin oxide layer on the capsule surface. The oxide layer was quite

TABLE 14. The Dimensions of Hastelloy C-4 Capsule No. 7R After Being Subjected to an External Hydrostatic Pressure of 20,750 psi

MINOR AXIS, in.	MAJOR AXIS, in.		CAPSULE TOP	
3.366	3.385	→	1 in.	21 in.
			2 in.	
3.294	3.412	→	2 in.	
			2 in.	
3.197	3.474	→	2 in.	
			2 in.	
3.085	3.542	→	2 in.	
			1-½ in.	
2.969	3.607	→	1-½ in.	
2.937	3.628	→	2 in.	
2.957	3.624	→	2 in.	
			2 in.	
3.045	3.583	→	2 in.	
			2 in.	
3.176	3.485	→	2 in.	
			2 in.	
3.314	3.388	→	2 in.	
			2 in.	
3.361	3.383	→	1 in.	

adherent and showed little tendency to scale off during subsequent handling of the capsules. The aging operation had no measurable effect on the weights and dimensions of the capsules.

Results of the qualification testing of the capsules aged at 650°C are summarized in Table 15. Typical test data for a capsule aged at 650° (Hastelloy S capsule No. S-14) are presented in Section 8.3.2. Table 15 shows that the five Hastelloy S capsules passed the primary and secondary qualification tests without difficulty. The aged Hastelloy S capsules also passed the three supplemental tests, although the capsule subjected to the supplemental pressure test partially collapsed at a pressure of 24,500 psi. The partially collapsed capsule passed the helium-leak test.

The Hastelloy C-4 capsule aged at 650°C passed the primary pressure test and secondary pressure and percussion tests. The capsule failed the secondary thermal test due to

FIGURE 23. An As-Fabricated Hastelloy C-276 Inner Capsule
After Being Subjected to an External Hydrostatic
Pressure of Approximately 9700 psi

TABLE 15. Results Obtained in the Qualification Testing of Hastelloy S and Hastelloy C-4 Prototype Outer Capsules Aged at 650°C for 8000 h

<u>Qualification Tests</u>	<u>Hastelloy S Capsule Number</u>					<u>Hastelloy C-4 Capsule No. 4</u>
	<u>S-8^(a)</u>	<u>S-14</u>	<u>S-18</u>	<u>S-22</u>	<u>S-23</u>	
<u>A. Primary Qualification Tests</u>						
Impact	Pass	---	---	---	---	---
Pressure	---	---	---	Pass	---	Pass
Percussion	---	---	---	---	Pass	---
Thermal	---	Pass	---	---	---	---
Thermal Quench	---	---	Pass	---	---	---
<u>B. Secondary Qualification Tests</u>						
Impact	---	Pass	Pass	Pass	Pass	Pass
Pressure	Pass	Pass	Pass	---	Pass	---
Percussion	Pass	Pass	Pass	Pass	---	Pass
Thermal	Pass	---	Pass	Pass	Pass	Fail
Thermal Quench	Pass	Pass	---	Pass	Pass	---
<u>C. Supplemental Tests</u>						
Pressure	---	P-C ^(b)	---	P-C	---	---
Percussion	---	---	Pass	---	Pass	---
Puncture	---	---	Pass	---	---	---

(a) Capsule contained a Hastelloy C-276 inner capsule filled with inert SrF_2 .

(b) P-C--Some distortion of capsule, but the capsule passed the helium-leak check.

development of a crack in the weld zone. As will be discussed later, it appeared that the crack developed from damage that resulted from the secondary impact test.

Results obtained in the qualification testing of the aged Hastelloy S capsules were very similar to the results obtained with the as-fabricated capsules. The aged Hastelloy S capsule, containing a Hastelloy C-276 inner capsule and subjected to the primary impact test, suffered significant distortion of the endcap at the points of impact. Substantial distortion of the capsule wall occurred adjacent to the point of impact together with some distortion of the interlocking joint. Over all, the distortion produced by the impact loading was very similar to that observed with the as-fabricated capsule. Figure 24 shows micrographs of the weld zone adjacent to the initial point of impact. The distortion of the capsule wall as a result of the impact is clearly shown. A small defect is visible near the inner edge of the weld zone. It is impossible to tell if the defect resulted from the impact test or if it is a welding defect, but the latter appears to be the most likely explanation. The defect was not detected by radiography before and after testing. The defect did not affect the capsule integrity, and the impact test did not affect the capsule integrity.

The aged Hastelloy S capsules subjected to the secondary impact test, without an inner capsule present, exhibited some distortion of the endcap at the point of impact. The impact

FIGURE 24. Photomicrographs of the Weld Zone Adjacent to the Point of Impact for the Hastelloy S Capsule Subjected to the Primary Impact Test After Aging at 650°C for 8000 h

produced only slight distortion of the capsule wall and interlocking joint in the area adjacent to the point of impact.

The primary and secondary hydrostatic pressure tests had no measurable effect on the aged Hastelloy S capsules. Dimensional measurements at the 11 locations specified showed that the dimensions of the capsules were unchanged after the capsules were subjected to an external pressure of 14,500 psi.

The primary and secondary percussion tests using an impact energy of 7 kg-m had no significant effect on the aged Hastelloy S capsules. A very slight indentation in the capsule

wall was visible at each point of impact. The indentations were less apparent than with the as-fabricated capsules, and their depth ranged from about 0.001 to 0.002 in.

The primary and secondary thermal and thermal quench tests had no measurable effect on the aged Hastelloy S capsules. No scaling of the oxide layer was observed when the capsules were quenched from 800°C to 0°C or less in ~30 s.

The supplemental percussion test, using an impact energy of 20 kg-m had no significant effect on the aged Hastelloy S capsules. Slight indentations in the capsule wall, up to 0.003 in. deep, were apparent at the points of impact. The indentations had no effect on capsule integrity.

The supplemental puncture test, using an impact energy of 1 kg-m, had no significant effect on the aged Hastelloy S capsule. Slight indentations in the capsule wall, to a depth

FIGURE 25. The Hastelloy S Capsule Aged at 650°C for 8000 h After Being Subjected to an External Hydrostatic Pressure of 24,500 psi for 30 Minutes

TABLE 16. Dimensions of the Hastelloy S Capsule Aged at 650°C for 8000 h Before the Supplemental Pressure Test

MINOR AXIS, in.	MAJOR AXIS, in.		CAPSULE TOP
3.373	3.374	→	1 in.
3.373	3.374	→	2 in.
3.373	3.374	→	2 in.
3.373	3.374	→	2 in.
3.373	3.374	→	2 in.
3.373	3.374	→	1-½ in.
3.373	3.374	→	1-½ in.
3.373	3.374	→	2 in.
3.373	3.374	→	2 in.
3.373	3.374	→	2 in.
3.373	3.376	→	2 in.
			1 in.
			21 in.

of about 0.002 in., were evident at the points of impact, but the indentations had no effect on capsule integrity.

The aged Hastelloy S capsule subjected to the supplemental pressure test partially collapsed at an external pressure of 24,500 psi. The partially collapsed capsule passed the helium-leak test. Figure 25 shows the partially collapsed capsule, and Tables 16 and 17 give the capsule dimensions before and after the test.

The Hastelloy C-4 capsule (No. 4) aged at 650°C for 8000 h suffered no measurable damage when subjected to the primary pressure test. The capsule exhibited no detectable change in dimensions when subjected to an external hydrostatic pressure of 14,500 psi, and the capsule passed the helium-leak test. When the capsule was subjected to the secondary impact test, some distortion of the endcap occurred at the point of impact, but there was only slight

TABLE 17. Dimensions of the Hastelloy S Capsule Aged at 650°C for 8000 h After Being Subjected to an External Hydrostatic Pressure of 24,500 psi

MINOR AXIS, in.	MAJOR AXIS, in.		CAPSULE TOP
3.361	3.386	→	1 in.
3.311	3.412	→	2 in.
3.276	3.443	→	2 in.
3.229	3.472	→	2 in.
3.202	3.482	→	2 in.
3.200	3.485	→	1-½ in.
3.208	3.474	→	1-½ in.
3.231	3.452	→	2 in.
3.269	3.430	→	2 in.
3.320	3.402	→	2 in.
3.376	3.400	→	2 in.
			1 in.
			21 in.

distortion of the capsule wall in the area adjacent to the point of impact. The impacted capsule passed the water-leak check. The aged capsule was then subjected to the secondary percussion test, which it passed without apparent significant damage. Slight indentations in the capsule wall, up to a depth of about 0.003 in., were visible at the points of impact. The capsule passed the water-leak check after the secondary percussion test.

After the aged Hastelloy C-4 capsule was subjected to the secondary thermal test, the water-leak check showed a leak in the capsule. Visual examination of the capsule showed a crack in the weld zone immediately above the point where the capsule had been impacted in the secondary impact test. Figure 26 shows sketches of the affected area of the capsule and the location of the crack. The crack was about 1 in. long and parallel to the weld zone. The capsule was sectioned and the affected endcap from the capsule examined. The endcap was sectioned, and samples from the crack area and a zone 180° removed from the crack area were

FIGURE 26. Sketch of Failed Capsule Showing Location of Crack

subjected to the metallographic examination. Figure 27 shows the section removed from the endcap at a location 180° removed from the crack area. The micrographs show that the weld penetration was only about 0.040 in., which agrees with the results obtained by radiography. The micrographs also show that there was little distortion of the capsule wall and interlocking joint due to the impact loading, which was to be expected. Figure 28 shows higher magnification micrographs of the weld zone. The micrographs show what may be several small cracks at the inner edge of the weld metal in the area adjacent to the gap between the endcap and capsule wall.

Figure 29 shows a section of the endcap of the failed Hastelloy C-4 capsule taken from the area where the crack developed. The micrographs show that the weld penetration was about 0.04 in., which agrees with the results obtained by radiography. The crack through the weld metal occurred at the interface between the endcap and capsule wall. The impact test produced only slight distortion of the capsule wall and interlocking joint. Figure 30 shows higher magnification micrographs of the crack area.

Although the failed capsule passed the secondary impact test, it appears likely that the failure resulted from a crack started by the stresses produced by the impact test. The crack was then propagated to the point of capsule failure by stresses resulting from the thermal test. It is unlikely that the percussion test contributed to the failure because the point of impact for the strike adjacent to the weld zone (see Section 8.1.3) was 90° removed from the area where the crack developed.

FIGURE 27. Section of the Endcap of the Failed Hastelloy C-4 Capsule--180° Removed from the Crack Area

FIGURE 28. Micrographs Showing the Weld Zone of the Endcap Section Shown in Figure 27

It is difficult to explain why the Hastelloy C-4 capsule aged at 650°C for 8000 h failed when none of the other aged or as-fabricated capsules failed. As will be discussed in Section 6.7.2, aging of Hastelloy C-4 at 500 to 800°C results in a loss of Charpy impact energy and ductility. Our results with CVN specimens fabricated from the 3.5-in.-dia barstock show that reduction in impact energy and ductility is somewhat greater when the alloy is aged at 650°C than when it is aged at 725°C. This could explain why the Hastelloy C-4 capsule aged at 650°C failed while the Hastelloy C-4 capsules aged at 725°C did not fail (see Section 7.1.2.3). However, the impact energy and ductility of Hastelloy C-4 aged at 650°C is somewhat greater than the impact energy and ductility of Hastelloy S aged at 650° and 725°C, and none of the aged Hastelloy S capsules failed the qualification tests. Although there is no experimental data to substantiate the claim, it appears very likely that a full penetration seal weld would eliminate the cracking problem and allow Hastelloy C-4 capsules aged at 650°C to meet the qualification test requirements.

The Hastelloy C-276 inner capsule, contained in the Hastelloy S outer capsule (No. S-8) aged at 650°C, showed no visible evidence of damage when it was removed from the outer capsule after the qualification tests were completed. The inner capsule passed the water-leak check. The inner capsule was subjected to the secondary impact, percussion and pressure tests. The capsule passed the secondary impact test without significant damage, except for some distortion of the endcap at the point of impact. The capsule passed the secondary percussion test without damage except for slight indentations in the capsule wall at the points of impact. When subjected to the secondary pressure test, the capsule partially collapsed at a pressure of about 10,500 psi. Despite the partial collapse, the capsule passed the water-leak test.

In summary, all of the Hastelloy S capsules aged at 650°C for 8000 h passed the primary and secondary qualification tests without significant damage. The capsules also passed the

FIGURE 29. Section of the Endcap of the Failed Hastelloy C-4 Capsule--Taken from the Crack Area

supplemental percussion and puncture tests without damage. When an aged Hastelloy S capsule was subjected to an external hydrostatic pressure of 24,500 psi, it partially collapsed, although the capsule still passed the helium-leak test. The Hastelloy C-4 capsule aged at 650°C for 8000 h passed the primary pressure test and the secondary impact and percussion tests but failed the secondary thermal test because a crack developed in the weld zone. The crack appeared to have been started by stresses developed during the impact test and propagated to the point of failure by stresses developed during the thermal test.

6.6.1.3 Capsules Aged at 725°C

Five Hastelloy S and three Hastelloy C-4 prototype outer capsules were qualification-tested after aging at 725°C for 8000 h. Figure 31 shows three of the aged capsules. The

FIGURE 30. Micrographs Showing the Crack Area of the Endcap from the Failed Hastelloy C-4 Capsule

FIGURE 31. Prototype Hastelloy S and Hastelloy C-4 Outer Capsules Aged at 725°C for 8000 h

aged capsules exhibited a dark gray coloration due to formation of a thin oxide layer on the capsule surface. The oxide layers that formed at 725°C were very similar to those that formed on the capsules aged at 650°C. The oxide layers were very adherent, and there was little tendency for the oxide to scale off during subsequent handling of the capsules. Visually, it was impossible to differentiate between the aged Hastelloy S and aged Hastelloy C-4 capsules. Aging at 725°C for 8000 h had no measurable effects on the weights and dimensions of the capsules.

Results of the qualification testing of the capsules aged at 725°C are summarized in Table 18. Typical test data for a capsule aged at 725°C (Hastelloy Capsule No. S-19) are

TABLE 18. Results Obtained in the Qualification Testing of Hastelloy S and Hastelloy C-4 Prototype Outer Capsules Aged at 725°C for 8000 h

Qualification Tests	Hastelloy S Capsule Number					Hastelloy C-4 Capsule Number		
	S-9(a)	S-11	S-16	S-19	S-21	1(a)	6	7
A. Primary Qualification Tests								
Impact	Pass	---	---	---	---	Pass	---	---
Pressure	---	---	---	Pass	---	---	Pass	---
Percussion	---	Pass	---	---	---	---	---	Pass
Thermal	---	---	---	---	Pass	---	---	---
Thermal Quench	---	---	Pass	---	---	---	---	---
B. Secondary Qualification Tests								
Impact	---	Pass	Pass	Pass	Pass	---	Pass	Pass
Pressure	Pass	Pass	Pass	---	Pass	Pass	---	Pass
Percussion	Pass	---	Pass	Pass	Pass	Pass	Pass	---
Thermal	Pass	Pass	Pass	Pass	---	Pass	Pass	Pass
Thermal Quench	Pass	Pass	---	Pass	Pass	Pass	Pass	Pass
C. Supplemental Tests								
Pressure	---	P-C(b)	---	---	---	---	P-C	---
Percussion	---	---	Pass	---	---	---	---	Pass
Puncture	---	---	---	Pass	---	---	---	Pass

(a) Capsule contained a Hastelloy C-276 inner capsule filled with inert SrF_2 .

(b) P-C--Some distortion of capsule, but capsule passed the helium-leak check.

presented in Section 8.3.3. All eight of the capsules aged at 725°C passed the primary and secondary qualification tests without difficulty. The capsules also passed the three supplemental tests, although the capsules partially collapsed when subjected to the supplemental pressure test. The Hastelloy S capsule partially collapsed at a pressure of 24,250 psi and the Hastelloy C-4 capsule at a pressure of 20,600 psi. The partially collapsed capsules passed the final helium-leak test with a leak rate of $<1 \times 10^{-8}$ std cm^3/s .

The Hastelloy S capsule subjected to the primary impact test (No. S-9) suffered substantial distortion of the endcap at the point of impact. There was also some distortion of the capsule wall and the interlocking joint in the area adjacent to the point of impact. The distortion was very similar to that observed with the other Hastelloy S capsules subjected to the primary impact test. Figure 32 shows the weld zone adjacent to the point of impact. The distortion of the capsule wall as a result of the impact loading is partially visible in Figure 32A. The micrographs show that a complete penetration weld was obtained when the capsule was rewelded. There was no indication of cracking at the inner edge of the weld zone adjacent to the joint. Cracks are evident, however, in the body of the weld metal, which are welding defects. The defects were detected by radiography before the capsules were aged. The defects did not affect the capsule integrity but could affect the ultimate strength of the capsule.

FIGURE 32. Weld Zone Adjacent to the Point of Impact of the Hastelloy S Capsule Subjected to the Primary Impact Test After Being Aged at 725°C for 8000 h

The unimpacted endcap from Capsule No. S-9 was also sectioned and subjected to metallographic examination. The photomicrographs obtained show that a full penetration seal weld was obtained when the capsule was rewelded (see Figure 33). The micrographs also show two defects in the weld zone that were not detected by radiography before or after qualification testing. One defect, a small pore at the inner edge of the weld metal, should not affect capsule integrity. The second defect, a large U-shaped defect in the weld metal, could affect the structural integrity of the capsule if the endcap was stressed sufficiently. The

FIGURE 33. Photomicrographs of the Unimpacted Endcap from the Hastelloy S Capsule Subjected to the Primary Impact Test After Aging at 725°C for 8000 h

cracks appear to be welding defects rather than the results of stresses developed during qualification testing.

The aged Hastelloy C-4 capsule subjected to the primary impact test exhibited damage very similar to that of the aged Hastelloy S capsules. The impact produced substantial distortion of the endcap at the point of impact and some distortion of the capsule wall and

interlocking joint in the area adjacent to the point of impact. Overall, the distortion was very similar to that observed in the other capsules subjected to the primary impact tests. Figure 34 shows the weld zone adjacent to the point of impact. The distortion of the capsule wall resulting from the impact loading is partially visible in Figure 34A. The micrographs show a full penetration weld. The cracks observed in the weld metal are welding defects detected by radiography before and after testing.

The Hastelloy S capsules aged at 725°C, which were subjected to the secondary impact tests without an inner capsule present, exhibited some distortion of the endcap at the point of impact. There was only slight distortion of the capsule wall and interlocking joint.

FIGURE 34. Micrographs of the Weld Zone Adjacent to the Point of Impact of the Hastelloy C-4 Capsule Subjected to the Primary Impact Test After Being Aged at 725°C for 8000 h

Overall, the distortion was very similar to that observed in the other Hastelloy S capsules subjected to the secondary impact test (see Figure 18).

The Hastelloy C-4 capsules aged at 725°C, which were subjected to the secondary impact test, exhibited slight distortion at the point of impact and very little distortion of the capsule wall or interlocking joint. Figure 35 shows the impacted endcap of one capsule and the weld zone. Weld penetration was about 45%, which was slightly less than the value determined by radiography (50 to 55%). The micrographs show a crack at the inner edge of the weld zone. The location and dimensions of the crack are very similar to the crack found in

FIGURE 35. Impacted Endcap from a Hastelloy C-4 Capsule Subjected to the Secondary Impact Test After Being Aged at 725°C for 8000 h

the as-fabricated Hastelloy C-4 capsule subjected to the secondary impact test (see Figures 20 and 21). The crack appears to be due to stresses developed in the qualification testing rather than a welding defect. The crack did not affect capsule integrity.

The primary and secondary hydrostatic pressure tests had no measurable effects on the capsules aged at 725°C for 8000 h. The dimensions of the capsules were unchanged when they were subjected to an external pressure of 14,500 psi.

The primary and secondary percussion tests had no significant effect on the capsules aged at 725°C. Slight indentation, up to 0.002 in. deep, in the capsule wall were apparent at the points of impact.

The primary and secondary thermal and thermal quench tests had no measurable effect on the capsule aged at 725°C. No scaling of the oxide layer was observed when the capsules were quenched from 800°C to 0°C or lower in ~30 s.

The supplemental percussion test, using an impact energy of 20 kg-m, and the supplemental puncture test had no significant effects on the aged capsules. The percussion test produced slight indentations, up to about 0.004 in. deep, in the capsule walls at the points of impact, while the puncture test produced indentations, up to about 0.002 in. deep, in the walls of the capsules. The indentations had no effect on the integrity of the capsules.

The aged Hastelloy S capsule (No. S-11) subjected to the supplemental pressure test partially collapsed at a pressure of 24,250 psi but still passed the helium-leak test. Figure 36 shows the partially collapsed capsule, and Tables 19 and 20 give the capsule dimensions before and after the supplemental pressure test. Collapse of the capsule aged at 725°C was significantly greater than the collapse of the as-fabricated Hastelloy S capsule or the Hastelloy S capsule aged at 650°C when they were subjected to the supplemental pressure test.

The aged Hastelloy C-4 capsule subjected to the supplemental pressure test partially collapsed at a pressure of 20,600 psi, but the capsule passed the helium leak test. Figure 37 shows the capsule after being pressurized to 20,600 psi and Tables 21 and 22 show the capsule dimensions before and after the test.

When qualification testing of the two capsules aged at 725°C, which contained inner capsules, was completed, the capsules were sectioned and the inner capsules removed. The two Hastelloy C-276 inner capsules exhibited no visible evidence of damage resulting from the qualification tests, and the capsules passed the water-leak check. The two capsules were then subjected to the secondary impact and percussion tests, which they passed without difficulty. The impact test produced some distortion of the endcaps at the points of impact but little distortion of the capsule walls (see Figure 38). The percussion test produced very slight indentations in the capsule walls at the points of impact. One of the Hastelloy C-276 capsules was subjected to the secondary pressure test. The capsule partially collapsed at a pressure of 12,600 psi. Figure 39 shows the capsule after the pressure test. Even though it had partially collapsed, the capsule passed the water-leak check.

FIGURE 36. The Hastelloy S Capsule Aged at 725°C for 8000 h
After Being Subjected to an External Hydrostatic
Pressure of 24,250 psi

In summary, the Hastelloy S and Hastelloy C-4 prototype outer capsules aged at 725°C for 8000 h passed the primary and secondary qualification tests without significant damage. The aged capsules subjected to the primary impact test suffered substantial distortion of the endcaps at the point of impact and distortion of the capsule walls and interlocking joints in the areas adjacent to the points of impact. The distortion resulting from the impact loading

TABLE 19. Dimensions of the Hastelloy S Capsule Aged at 725°C Before the Supplemental Pressure Test

MINOR AXIS, in.	MAJOR AXIS, in.		CAPSULE TOP
3.372	3.373	→	1 in.
3.372	3.373	→	2 in.
3.372	3.373	→	2 in.
3.372	3.373	→	2 in.
3.373	3.373	→	2 in.
3.373	3.373	→	1-½ in.
3.373	3.373	→	1-½ in.
3.373	3.373	→	2 in.
3.373	3.373	→	2 in.
3.373	3.373	→	2 in.
3.373	3.375	→	2 in.
			1 in.
			21 in.

did not affect the integrity of the capsules. The aged capsules passed the supplemental percussion and puncture tests without significant damage. The aged capsules partially collapsed when subjected to the supplemental pressure test, the Hastelloy S capsule at a pressure of 24,250 psi, and the Hastelloy C-4 capsule at a pressure of 20,600 psi. Both of the partially collapsed capsules passed the helium-leak test.

6.6.2 Effect of Aging on Mechanical Properties

6.6.2.1 Tensile Properties

The thermally aged and the control tensile specimens, fabricated from 3.5-in.-dia Hastelloy S and Hastelloy C-4 barstock, were tension-tested at room temperature. The results obtained with the two alloys are summarized in Table 23 and Figures 40 and 41. The data show that, overall, aging of the two alloys at 650° and 725°C for up to 8000 h produced only

TABLE 20. Dimensions of the Hastelloy S Capsule Aged at 725°C After Being Subjected to an External Hydrostatic Pressure of 24,250 psi

MINOR AXIS, in.	MAJOR AXIS, in.		CAPSULE TOP
3.363	3.387	→	1 in.
3.254	3.447	→	2 in.
3.113	3.552	→	2 in.
2.917	3.664	→	2 in.
2.809	3.727	→	2 in.
2.786	3.731	→	1-½ in.
2.818	3.701	→	1-½ in.
2.956	3.613	→	2 in.
3.124	3.508	→	2 in.
3.234	3.414	→	2 in.
3.360	3.397	→	2 in.
			1 in.
			21 in.

slight changes in the tensile properties. The maximum changes were observed with the Hastelloy C-4 aged at 650°C in which the aging resulted in about a 10% increase in ultimate tensile strength and yield strength and a 20% decrease in ductility. From the limited number of specimens of each alloy tested, it is difficult to determine if the tensile properties were continuing to change with exposure time.

Matthews at Cabot Corp. (Matthews 1976, 1977) carried out an extensive study of the effects of thermal aging on the mechanical properties of nickel- and cobalt-base alloys including Hastelloy S and Hastelloy C-4. Tensile and CVN specimens, fabricated from 0.5-in. plate, were aged at 800°F to 1600°F for up to 16,000 h and then tested at room temperature. The results Matthews obtained with the aged Hastelloy S and Hastelloy C-4 tensile specimens are presented in Table 24.

FIGURE 37. The Hastelloy C-4 Capsule Aged at 725°C for 8000 h
After Being Subjected to an External Pressure of
20,600 psi

Comparison of the two sets of data show that there is generally good agreement in the results obtained with aged Hastelloy S. The only significant difference is that PNL obtained a significantly higher yield strength for both the solution heat-treated and thermally aged Hastelloy S. It should be noted that the yield strength reported by Cabot for the 3.5-in.-

TABLE 21. Dimensions of the Hastelloy C-4 Capsule Aged at 725°C for 8000 h Before Being Subjected to the Supplemental Pressure Test

MINOR AXIS, in.	MAJOR AXIS, in.		CAPSULE TOP	
3.371	3.372	→	1 in.	21 in.
3.371	3.372	→	2 in.	
3.371	3.372	→	2 in.	
3.371	3.372	→	2 in.	
3.371	3.372	→	2 in.	
3.371	3.372	→	1-½ in.	
3.371	3.372	→	1-½ in.	
3.371	3.372	→	2 in.	
3.371	3.372	→	2 in.	
3.372	3.372	→	2 in.	
3.372	3.372	→	2 in.	
		→	1 in.	

dia barstock in the Certified Report of Tests (see Table 6) was only 335 MPa, whereas a value of 440 MPa was obtained at PNL for the control specimen machined from the barstock. No reasons for the difference have been identified. Tensile specimens fabricated from the as-fabricated capsules, after qualification testing was completed, exhibited room-temperature tensile properties similar to the control-specimens. Table 25 gives room temperature tensile data reported for a variety of forms of solution-heat-treated Hastelloy S and Hastelloy C-4. The data show a considerable spread in the room temperature properties of Hastelloy S, especially the yield strength and elongation, depending on the form of the material and the source of the data.

One important conclusion can be reached from the Matthews data. Aging of Hastelloy S at 649° to 760°C for times in excess of 8000 h has little effect on the tensile properties of the alloy.

TABLE 22. Dimension of the Hastelloy C-4 Capsule Aged at 725°C for 8000 h After Being Subjected to an External Hydrostatic Pressure of 20,600 psi

MINOR AXIS, in.	MAJOR AXIS, in.		CAPSULE TOP	
3.357	3.384	→	1 in.	
			2 in.	
3.261	3.433	→	2 in.	
			2 in.	
3.175	3.495	→	2 in.	
			2 in.	
3.075	3.555	→	2 in.	
			2 in.	
3.024	3.583	→	1-½ in.	
3.022	3.586	→	1-½ in.	
3.046	3.571	→	2 in.	
			2 in.	
3.118	3.523	→	2 in.	
			2 in.	
3.220	3.470	→	2 in.	
			2 in.	
3.309	3.404	→	2 in.	
			2 in.	
3.361	3.385	→	1 in.	
				21 in.

In the case of Hastelloy C-4, there were significant differences in the tensile property data obtained at PNL and the values reported by Matthews for both the solution-heat-treated alloy and the specimens aged at 649° to 760°C. In general, the PNL specimens exhibited lower UTS and yield strength and higher ductility than the corresponding specimens tested by Matthews. Also, the Matthews data show that aging at 649° to 760°C produced significantly greater changes in the tensile properties of the Hastelloy C-4 than did the PNL data. Reasons for the marked differences in the results obtained with Hastelloy C-4 have not been determined.

When referring to the Matthews data in Table 24, it is apparent that the maximum change in tensile properties of both Hastelloy S and Hastelloy C-4 occurs when the specimens are aged at 1000° to 1100°C. Figure 42, for example, shows how the yield strength of Hastelloy S varies as a function of aging temperature for specimens aged for 7500 or 8000 h. Similarly

FIGURE 38. Impacted Endcap of the Hastelloy C-276 Inner Capsule Subjected to the Secondary Impact Test After Being Aged at 725°C for 8000 h

shaped curves were obtained for the UTS and elongation. The data show that aging Hastelloy S at temperatures above about 650°C has little effect on its room-temperature tensile properties.

If one plots a similar curve for the yield strength of Hastelloy C-4 as a function of aging temperature, the results obtained in Figure 43 are obtained. The plot shows the marked differences in the PNL and Cabot data for aging temperatures between 650° and 800°C. Curves for the UTS and elongation show similar differences between the two sets of data.

6.6.2.2 Impact Properties

The thermally aged and the control CVN specimens, fabricated from 3.5-in.-dia Hastelloy S or Hastelloy C-4 barstock, were impact-tested at room temperature. The results obtained with the two alloys are presented in Table 26 and Figure 44. Aging of the two alloys at 650° or 725°C resulted in marked decreases in their impact energies. From the limited number of specimens tested it is impossible to determine how the impact energies of the alloys would change with exposure times greater than 8000 h.

Matthews studied the effects of aging on the Charpy impact energy of Hastelloy S and Hastelloy C-4 over a temperature range of 800° to 1600°F and exposure times up to 16,000 h, using specimens machined from 0.5-in.-plate (Matthews 1976, 1977). The results obtained are presented in Table 27. The data show that the impact energies of the two alloys decreased slightly between 8000 h and 16,000 h. The data also show that for aging temperatures of 1000°F to 1600°F and aging times of 8000 h or longer, the impact energy of aged Hastelloy C-4 is generally less than that of Hastelloy S.

FIGURE 39. The Hastelloy C-276 Inner Capsule Aged at 725°C for 8000 h After Being Subjected to an External Hydrostatic Pressure of 12,600 psi

In evaluating the two sets of impact data, one point becomes obvious. For comparable specimens, significantly higher impact energies were obtained in the PNL work compared to the results reported by Matthews. Reasons for these differences have not been identified.

TABLE 23. Effects of Thermal Aging on the Room-Temperature Tensile Properties of Hastelloy S and Hastelloy C-4^(a)

Material	Aging Temperature, °C	Aging Time, h	Stress		Elongation, %
			Ultimate, MPa	Yield, MPa	
Hastelloy S	As-Rec. ^(b)	0	858	440	54.5
	650	2500	873	432	51.1
		5000	885	448	47.9
		8000	884	425	49.9
	725	2500	884	430	49.5
		5000	881	410	51.5
		8000	917	443	46.6
	725	2500	775	351	64.3
		5000	775	343	64.7
		8000	781	340	62.1
Hastelloy C-4	As-Rec. ^(b)	0	764	362	66.4
	650	2500	846	412	54.4
		5000	819	391	56.8
		8000	837	406	51.1
	725	2500	775	351	64.3
		5000	775	343	64.7
		8000	781	340	62.1
	725	2500	775	351	64.3
		5000	775	343	64.7
		8000	781	340	62.1

(a) Specimen fabricated from 3.5-in.-dia bar stock.

(b) Solution-heat-treated alloy.

Subsize Hastelloy C-276 CVN specimens, machined from 0.5-in. barstock, were aged at 650°, 725°, or 800°C for up to 8000 h. The aged specimens, which were 5 mm thick, were impact-tested at room temperature. The results obtained are presented in Table 28. The data show that aging Hastelloy C-276 at 650° to 800°C results in a very sharp decrease in impact energy, especially at 725°C and higher. The impact energy of the specimens aged at 800°C were too low for the instrument to measure reliably (<1 J).

In the qualification tests, Hastelloy C-276 inner capsules were aged at 725°C for 8000 h. The impact data presented in Table 28 indicate that the impact strength of the aged capsules should have been very low. It is surprising, therefore, that the bare capsule was able to pass the impact test and percussion tests without failing.

6.6.3 Metal-Metal Compatibility

The Hastelloy C-276 and Hastelloy S or Hastelloy C-4 specimens held in direct contact at 700°C for up to 10,000 h exhibited very little interaction, even between the unoxidized specimens. Metallographic examination of the unoxidized specimens indicated that very slight bonding had occurred between the Hastelloy C-276 and Hastelloy C-4 specimens, which produced some roughening of the specimens' surfaces when the specimens were separated. No interaction was observed between the unoxidized Hastelloy C-276 and Hastelloy S specimens or between the oxidized Hastelloy C-276 and Hastelloy S or Hastelloy C-4 specimens.

Significant interaction was observed between the specimens held at 800°C for up to 10,000 h. The unoxidized specimens exhibited greater interaction than the oxidized

FIGURE 40. Effects of Thermal Aging on the Room-Temperature Tensile Properties of Hastelloy S--3.5-in.-dia Barstock

specimens. Metallographic examination of the Hastelloy C-276/Hastelloy S pairs showed only slight interaction between the oxidized specimens, which produced a slight roughening of the specimens' surfaces when they were separated (see Figure 45). The unoxidized specimens showed evidence of bonding that resulted in the adherence of some Hastelloy S to the Hastelloy C-276 surface when the specimens were separated (see Figure 45). Pits up to 5 μ m deep were observed in the Hastelloy S surface where pieces of metal had been pulled away when the specimens were separated. The oxide films formed on the specimens before testing was started, which was too thin to be detected by metallography at a magnification of 500X, appeared to be adequate to prevent most of the interaction between the two alloys at 800°C.

FIGURE 41. Effects of Thermal Aging on the Room-Temperature Tensile Hastelloy C-4 Specimens--3.5-in.-dia Barstock

In the case of Hastelloy C-276 and Hastelloy C-4, the interaction between oxidized specimens at 800°C was very slight, similar to that observed with the Hastelloy C-276/Hastelloy S pairs, as shown in Figure 46. Much greater interaction was observed between the unoxidized Hastelloy C-276/Hastelloy C-4 specimens (see Figure 46). The Hastelloy C-276 bonded to the Hastelloy C-4 surface, and when the specimens were separated, large fragments of the Hastelloy C-276 were pulled free from the specimen's surface. Pits up to 100 μm deep were observed in the Hastelloy C-276 surface where fragments of metal had been removed. The bonding appeared to involve interdiffusion of alloy components, and separation of the Hastelloy C-276 fragments from the body of the specimen appeared to occur along grain boundaries. As was the case with the Hastelloy C-276/Hastelloy S pairs, the very thin oxide films formed on the Hastelloy C-276 and Hastelloy C-4 specimens before testing was quite effective in preventing interaction at 800°C.

TABLE 24. Room-Temperature Tensile Properties of Aged Hastelloy S and Hastelloy C-4(a)
(Matthews 1976, 1977)

Material	Aging Temperature, °F (°C)	Aging Time, h	Stress		Elongation, %
			Ultimate, MPa	Yield, MPa	
Hastelloy S	SHT(b)	0	864	365	54
	800 (427)	1,000	867	385	60
		4,000	875	383	60
		8,000	874	390	55
		16,000	883	390	56
	1,000 (538)	1,000	996	493	46
		4,000	1,207	707	44
		8,000	1,247	746	38
		16,000	1,258	751	39
	1,200 (649)	1,000	863	390	57
		4,000	867	389	54
		8,000	877	393	50
		16,000	882	409	49
	1,400 (760)	1,000	869	370	55
		4,000	879	373	52
		8,000	870	369	46
		16,000	879	372	47
	1,600 (871)	1,000	867	350	58
		4,000	863	350	56
		8,000	852	354	53
		16,000	850	348	56
Hastelloy C-4	SHT(b)	0	851	380	54.5
	800 (427)	1,000	872	410	63.2
		4,000	875	393	62.7
		8,000	888	414	59.0
		16,000	910	424	62.5
	1,000 (538)	1,000	1,323	784	42.1
		4,000	1,342	779	39.8
		8,000	1,358	801	35.2
		16,000	1,364	774	37.7
	1,200 (649)	1,000	1,076	572	44.6
		4,000	1,011	494	48.5
		8,000	1,107	593	42.0
		16,000	1,144	635	37.5
	1,400 (760)	1,000	892	412	53.4
		4,000	927	397	46.7
		8,000	910	414	41.7
		16,000	919	415	42.5
	1,600 (871)	1,000	863	374	61.6
		4,000	855	374	58.7
		8,000	841	384	54.9
		16,000	826	359	58.7

(a) Specimens machined from 0.5-in. plate.

(b) SHT--Solution heat-treated (not aged).

FIGURE 42. Effect of Aging Temperature on the Room-Temperature Yield Strength of Hastelloy S--Specimens Aged for 8000 h

The results obtained indicated that some interaction will occur between Hastelloy C-276 and Hastelloy S or Hastelloy C-4 when they are held in direct contact at 800°C for long periods of time. This could be a significant factor in an operating heat source that utilized double encapsulation of the $^{90}\text{SrF}_2$ with Hastelloy C-276 as the inner capsule and Hastelloy S or Hastelloy C-4 as the outer capsule. Potential interactions can be reduced to a marked degree by oxidizing the surfaces of the capsules. This would provide the added advantages of increasing the emissivities of the capsule surfaces, improving the heat transfer between capsules, and reducing the temperature drop between capsules. This could be an important factor in the design of an efficient power generation system where the $^{90}\text{SrF}_2$ /Hastelloy C-276 interface temperature is limited to a maximum of 800°C.

TABLE 25. The Room-Temperature Tensile Properties of Solution-Heat-Treated Hastelloy S and Hastelloy C-4

Material	Form	Source of Data	Ultimate, MPa	Yield, MPa	Elongation, %
Hastelloy S	3.5-in.-dia Barstock	PNL	858	440	54.5
	As-Fabricated Capsule	PNL	866	428	49.1
	0.5-in.-dia Barstock	PNL ^(a)	880	469	60.8
	3.5-in.-dia Barstock (Certified Report of Tests)	Cabot ^(b)	817	335	54.0
	0.5-in. Plate	Cabot ^(b)	864	365	54.0
	0.057-in. Sheet	Cabot ^(b)	887	434	58.3
	0.55-in. Plate	ORNL ^(c)	870	395	60.0
Hastelloy C-4	3.5-in.-dia Barstock	PNL	764	362	66.4
	As-Fabricated Capsule	PNL	769	362	58.9
	0.5-in.-dia Barstock	PNL ^(a)	834	379	71.5
	3.5-in.-dia Barstock (Certified Report of Tests)	Cabot ^(b)	745	328	65.0
	0.5-in. Plate	Cabot ^(b)	851	380	54.5

(a) Fullam 1980b (Appendix, Section 8.4).

(b) Matthews (1976).

(c) Data from J. L. Hammond of ORNL.

FIGURE 43. Effects of Aging Temperature on the Room-Temperature Yield Strength of Hastelloy C-4 for Specimens Aged for 8000 h

TABLE 26. The Effects of Aging on the Room-Temperature Charpy Impact Energy of Hastelloy S and Hastelloy C-4^(a)

Aging Temperature, °C	Aging Time, h	Charpy Impact Energy, J	
		Hastelloy S	Hastelloy C-4
SHT ^(b)	0	220	420
650	2500	141	247
	5000	126	160
	8000	136	199
725	2500	132	298
	5000	157	302
	8000	118	254

(a) Specimen machined from 3.5-in.-dia barstock.
 (b) SHT--Solution-heat-treated alloy.

FIGURE 44. Effects of Aging on the Charpy Impact Energy of Hastelloy S and Hastelloy C-4--Specimens Machined from 3.5-in.-dia Barstock

TABLE 27. Room-Temperature Charpy Impact Energy of Aged Hastelloy S and Hastelloy C-4^(a) (Matthews 1976, 1977)

Temperature, °F (°C)	Aging Time, h	Charpy Impact Energy, J	
		Hastelloy S	Hastelloy C-4
SHT ^(a)	0	190	302
800 (427)	1,000	199 ^(c)	276 ^(c)
	4,000	199 ^(c)	283 ^(c)
	8,000	199 ^(c)	266
	16,000	176	274
1,000 (538)	1,000	155	83
	4,000	104	68
	8,000	90	46
	16,000	64	28
1,200 (649)	1,000	115	126
	4,000	91	114
	8,000	73	75
	16,000	66	63
1,400 (760)	1,000	107	99
	4,000	71	56
	8,000	65	43
	16,000	53	35
1,600 (871)	1,000	145	164
	4,000	148	132
	8,000	142	170
	16,000	148	142

(a) Specimens machined from 0.5-in. plate.

(b) SHT--Solution heat-treated alloy (not aged).

(c) Incomplete fracture of specimen.

TABLE 28. Room-Temperature Charpy Impact Energy of
Hastelloy C-276^(a)--Subsize Specimens
5 mm Thick

<u>Aging Temp., °C</u>	<u>Aging Time, h</u>	<u>Charpy Impact Energy, J</u>
SHT ^(b)	0	50.9
650	2,500	13.3
	5,000	21.1
	8,000	15.9
725	2,500	3.0
	5,000	2.8
	8,000	1.9
800	2,500	0.7
	5,000	0.9
	8,000	0.9

(a) Specimens machined from 0.5-in.-
dia barstock.

(b) SHT--Solution-heat-treated alloy.

FIGURE 45. Hastelloy C-276/Hastelloy S Specimens Held in Direct Contact at 800°C for 10,000 h

FIGURE 46. Hastelloy C-276/Hastelloy C-4 Specimens Held in Direct Contact at 800°C for 10,000 h

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7.0 CONCLUSIONS AND RECOMMENDATIONS

Based on the results obtained in the qualification tests and auxiliary studies discussed in this report, the following conclusions can be reached regarding the proposed design for a general purpose $^{90}\text{SrF}_2$ heat source:

- The design proposed for the outer capsule of the heat source is capable of meeting current licensing requirements when Hastelloy S is used for the outer capsule material.
- As-fabricated Hastelloy S prototype outer capsules are capable of meeting current licensing requirements without support from the inner capsules. The capsules are also able to meet the supplemental ANSI requirements without leaking.
- The Hastelloy S outer capsules aged at 650° or 725°C for 8000 h are capable of meeting current licensing requirements without support from the inner capsule. The capsules are also able to meet the supplemental ANSI requirements without leaking. Studies to evaluate the effects of aging on the mechanical properties of Hastelloy S indicate that Hastelloy S outer capsules aged at 750° or 725°C for periods in excess of 8000 h would also be able to meet current licensing requirements without support from the inner capsule.
- The as-fabricated Hastelloy C-4 prototype outer capsule and the capsules aged at 725°C for 8000 h are capable of meeting current licensing requirements without support from the inner capsule. The Hastelloy C-4 capsules are also able to meet the supplemental ANSI requirements without leaking. Studies indicate that aging the capsules at 725°C for periods in excess of 8000 h would not seriously affect the capsule's ability to meet current licensing requirements.
- The single Hastelloy C-4 capsule aged at 650°C for 8000 h failed to meet current licensing requirements because of a leak that developed during the thermal test. The results indicate that the leak was the result of incomplete penetration of the closure weld, and that a complete penetration weld would eliminate the problem. However, this conclusion was not demonstrated experimentally.
- Oxidation studies with Hastelloy S and Hastelloy C-4 show that external oxidation that would occur to the outer capsule during the nominal 10-year service life of a $^{90}\text{SrF}_2$ heat source would not significantly affect the ability of a capsule fabricated from either material to meet current licensing requirements.
- Corrosion tests with Hastelloy S and Hastelloy C-4 show that the accidental exposure of a $^{90}\text{SrF}_2$ heat source to seawater for a period of 1 year would not adversely affect the ability of an outer capsule fabricated from either material to meet current licensing requirements. The corrosion rate of Hastelloy S in seawater is so low that a Hastelloy S outer capsule could be exposed to seawater for periods far in excess of 1 year without significantly degrading the structural strength of the capsule.

- The Hastelloy C-276 inner capsules, removed from the as-fabricated and thermally aged outer capsules after qualification testing was completed, showed no evidence of damage as a result of the qualification tests. The capsules passed secondary percussion and impact tests without difficulty. The capsules partially collapsed at 9,700 to 12,600 psi when subjected to the secondary pressure test, but the partially collapsed capsules passed the water-leak test (leak rate $<1 \times 10^{-4}$ std cm³/s). It is very significant that the Hastelloy C-276 inner capsules aged at 725°C passed the various tests since aging Hastelloy C-276 at 725°C reduces the Charpy impact energy of the alloy to very low levels.

Hastelloy S is the preferred material for the heat-source outer capsule. A Hastelloy S outer capsule, fabricated in accordance with the proposed design, is capable of meeting all of the qualification test requirements defined in Tables 1, 2 and 3. A Hastelloy C-4 outer capsule, fabricated in accordance with the proposed design, should also be able to meet the qualification test requirements defined in Tables 1, 2 and 3. Hastelloy C-4 cannot be recommended for use as the outer capsule material, however, because of the problems encountered with the prototype capsule aged at 650°C.

8.0 APPENDIX

8.1 QUALIFICATION TEST PROCEDURES

The procedures used in qualification testing the prototype outer capsules are described in the following sections. Before qualification testing, each capsule was subjected to certain preliminary tests and measurements (as described in Section 6.2):

1. The capsule was weighed and its dimensions determined at the 11 prescribed locations.
2. The capsule was subjected to the water-leak check.
3. The capsule welds were radiographed.
4. The capsule was filled with helium (see Section 6.4.4).
5. The capsule was helium-leak checked to ensure it did not leak at a rate greater than 1×10^{-8} std cm³/s.
6. If required, the capsule was aged for 8000 h at 650° or 725°C.
7. The aged capsules were subjected to Steps 1, 2, 4 and 5 before qualification testing.

The capsules were also subjected to several of the auxiliary measurements and tests during and after-qualification testing as prescribed by the qualification test procedures.

Each prototype outer capsule was subjected to one primary qualification test and four secondary qualification tests. The secondary tests were simplified versions of the primary tests.

8.1.1 Impact Test

Each prototype outer capsule was subjected to a primary impact test or a secondary impact test. Capsules subjected to the primary impact test contained a Hastelloy C-276 inner capsule filled with nonradioactive SrF₂.

8.1.1.1 Primary Impact Test

Test Requirement. The capsule must not leak at a rate greater than 1×10^{-8} std cm³/s after being dropped from a height of 9 m (29.5 ft) onto a steel plate at least 1.25 cm (~1/2 in.) thick that has been wet-floated onto a concrete block having a mass at least 10 times that of the capsule and set on firm soil. The steel plate shall be flat and horizontal, and the capsule shall be dropped in such a manner that the maximum possible damage will result to the capsule.

To insure that the capsule suffers maximum damage, it must impact the steel plate at a specific angle. The critical impact angle is defined in the following section. Additionally, to insure maximum damage, the capsule must contain a standard WESF Hastelloy C-276 inner capsule filled with inert strontium fluoride.

Equipment Required. The impact test required that the test capsule be dropped from a height of 9 m onto the steel plate set in concrete. Figure 47 shows a sketch of the equipment used for the test. The tests were conducted outdoors. The 1.25-cm-thick steel plate used for the test was 0.45 m wide and 0.62 m long. It had been wet-floated into a concrete pad that was set in firm soil. This pad was 1.2 m wide by 1.8 m long and 0.15 m thick. The capsule was dropped from a platform attached to an existing building. The capsule release mechanism was attached to the guard rail of the platform. The release mechanism was movable so that its position could be varied to prevent the capsules from striking the steel plate at the same location.

FIGURE 47. Sketch of Equipment Used in the Impact Test

To ensure that the capsule suffered the maximum possible damage from the impact test, it had to strike the steel plate at the edge at such an angle that the capsule center of gravity is in line with the impact point and the direction of travel (see Figure 48). The critical angle for an outer capsule containing a Hastelloy C-276 inner capsule filled with SrF_2 is $\sim 9^\circ 5'$. To facilitate release of the capsule and ensure that it impacted the plate at the proper angle, a small clip was welded to the top of the capsules (see Figure 48). The

FIGURE 48. Sketch of Capsule Clip and Correct Impact Angle

capsule was supported in the release mechanism using the small holes drilled in the clip. The release mechanism was designed so that the capsule could be dropped without imparting a rotational motion to the capsule upon release. The test had to be carried out on a windless day and the capsule had to be motionless at the time of release to ensure that the capsule impacted correctly.

A high-speed motion picture camera and crosshatched backdrop were used to record the drop event and determine the position of the capsule at impact. The camera was positioned so that its line of sight was perpendicular to the backdrop, and the capsule fell directly between the camera and the backdrop. A number of impact tests using dummy capsules showed that they impacted at the proper angle, and without rotational motion as long as the test was carried out as outlined above. Figure 49 shows a photograph of a capsule just before the moment of impact. In the dummy tests, the impact angle was $9^\circ \pm 0.25^\circ$, which was considered to be adequate for the tests. In some of the actual tests, two motion picture cameras and two backdrops were used to record the drop events and obtain a more precise measure of the impact angle.

Test Procedure. After the preliminary measurements and tests on the capsule had been performed, the impact test was carried out in the following manner:

FIGURE 49. Photograph of a Capsule Just Before Impact

1. The release clip was spot-welded to the top endcap of the capsule (the endcap containing the helium fill-hole and tapered plug) in the appropriate location (see Figure 48) and the capsule was weighed.
2. A windless day was selected for the impact test.
3. The motion picture camera(s) was set up in the proper location.
4. The capsule was raised into position and attached to the release mechanism using the release clip. The capsule was allowed to stabilize until motionless.
5. The motion picture camera(s) was started. The capsule was released and the entire drop event photographed.
6. After the drop was completed, the capsule was examined visually for damage and to determine if the capsule had rotated during the free fall.
7. The capsule was weighed and its dimensions determined at the 11 locations specified.
8. The capsule was subjected to the water-leak check.
9. The capsule was subjected to the helium-leak check.
10. The capsule welds were radiographed to determine if damage had resulted from the impact test.
- 11a. If the capsule leaked, or radiography showed serious damage to the seal weld or inner capsule, further testing of the capsule was stopped.
- 11b. If the capsule did not leak at a rate greater than 1×10^{-8} std cm³/s and radiography did not indicate serious damage, the release clip was removed from the capsule endcap and rewelded to the capsule endcap at a point 180° from the initial location.
12. Steps 2 through 10 were repeated, and the capsule was impacted at a point on the endcap 180° removed from the initial point of impact.
13. The clip was removed and the capsule was weighed.

If the capsule passed the primary impact test, it was then subjected to the secondary qualification tests.

8.1.1.2 Secondary Impact Test

Each capsule that had been subjected to a primary qualification test other than the primary impact test was subjected to a secondary impact test. The secondary impact test was carried out without the capsule containing a Hastelloy C-276 inner capsule and was a simplified version of the primary impact test. The equipment used for the secondary impact test was the same as that used for the primary impact test. The test procedure was as follows:

1. The release clip was welded to the capsule endcap in the appropriate location.

2. The capsule was weighed.
3. A windless day was selected for the impact test.
4. The motion picture camera was set up in the proper location.
5. The capsule was raised into position and attached to the release mechanism. The capsule was allowed to stabilize until motionless.
6. The camera was started, the capsule was released, and the entire drop event photographed.
7. After the drop, the capsule was examined visually for damage and to determine if the capsule had rotated during the free fall.
8. The capsule was weighed and its dimensions determined at the 11 locations specified.
9. The capsule was subjected to the water-leak check.
10. The clip was removed and the capsule was weighed.

If the capsule passed the secondary impact test, it was then subjected to any other secondary qualification tests required. If the impact test was the last of the secondary qualification tests, the capsule was subjected to the helium-leak check after the impact test was completed.

8.1.2 Hydrostatic Pressure Test

Each prototype outer capsule was subjected to a primary hydrostatic pressure test or a secondary hydrostatic pressure test. Capsules subjected to the primary hydrostatic pressure tests, and most of the capsules subjected to the secondary test, were tested without an inner capsule present. Only those capsule subjected to the primary impact test contained an inner capsule when subjected to the secondary pressure test.

8.1.2.1 Primary Hydrostatic Pressure Test

Test Requirement. The capsule must not leak at a rate greater than 1×10^{-8} std cm^3/s after being subjected to an external hydrostatic pressure of 1,000 bars (14,500 psi).

Test Equipment. The hydrostatic pressure test was carried out in a high-pressure autoclave having a maximum pressure capability of 24,600 psi. Water was supplied to the autoclave using a pump having a rated capability of 25,000 psi. The pressure in the autoclave was measured using a calibrated Heisey pressure gauge. Figure 50 shows the autoclave and auxiliary equipment.

Test Procedure. After the preliminary measurements and tests on the capsule had been completed, the primary hydrostatic pressure test was carried out in the following manner:

1. The capsule was placed in the autoclave. The autoclave was sealed, and the water pressure in the autoclave was increased to 10,000 psi and held for at least 30 min.

FIGURE 50. Autoclave and Auxiliary Equipment Used in the Hydrostatic Pressure Tests

2. The autoclave pressure was released, the autoclave was opened, and the capsule removed.
3. The capsule was weighed and its dimensions determined at the 11 locations previously specified.
4. The capsule was subjected to the water-leak check.
5. The capsule was subjected to the helium-leak check.
- 6a. If the capsule leaked at a rate greater than 1×10^{-8} std cm^3/s or had collapsed,^(a) further testing was stopped.
- 6b. If the capsule did not leak and had not collapsed, Steps 1 through 5 were repeated using a pressure of 12,000 psi.

(a) For testing purposes, capsule collapse was arbitrarily assumed to have occurred if the capsule had flattened to the extent that the major axis was 15% greater than the minor axis.

7. If the capsule passed the 12,000 psi test, Steps 1 through 5 were repeated using a pressure of 13,000 psi.
8. If the capsule passed the 13,000 psi test, Steps 1 through 5 were repeated using a pressure of 14,500 psi (1,000 bars).
9. After the 14,500 psi test was completed, the capsule welds were radiographed to determine if any damage had resulted from the hydrostatic pressure test.

If the capsule passed the primary hydrostatic pressure test without collapsing or without leaking at a rate greater than 1×10^{-8} std cm³/s, it was subjected to the secondary qualification tests.

8.1.2.1 Secondary Hydrostatic Pressure Test

Each capsule, which was subjected to a primary qualification test other than the primary hydrostatic pressure test, was subjected to a secondary pressure test. The secondary hydrostatic pressure test was carried out using the same equipment that was used for the primary pressure test. The procedure used was as follows:

1. The capsule was placed in the autoclave, the autoclave was sealed, and the water pressure in the autoclave was raised to 14,500 psi and held for at least 30 min.
2. The autoclave pressure was released, the autoclave opened, and the capsule removed.
3. The capsule was weighed and its dimensions determined at the 11 locations previously specified.
4. The capsule was subjected to the water-leak check.

If the capsule passed the water-leak check and had not collapsed, it was subjected to the remaining secondary qualification tests. If the secondary pressure test was the last of the planned secondary qualification tests, the capsule was subjected to the helium-leak check to ensure that it did not leak at a rate greater than 1×10^{-8} std cm³/s.

8.1.3 Percussion Test

Each prototype outer capsule was subjected to a primary percussion test or a secondary percussion test. Capsules subjected to the primary percussion test, and most of the capsules subjected to the secondary percussion test, were tested without an inner capsule present. Only those capsules subjected to the primary impact test contained an inner capsule when subjected to the secondary percussion test.

8.1.3.1 Primary Percussion Test

Test Requirement. The capsule shall not leak at a rate greater than 1×10^{-8} std cm³/s after being struck by the flat face of a 2.5-cm-dia (~1-in.) steel billet with an impact energy equivalent to 7 kg-m (~50 ft-lb). The billet edge shall be rounded off to a radius of not less than 3 mm, and the capsule shall rest on a lead sheet equal to or less

than 25 mm thick with a Brinell hardness of 3.5 to 4.5. The lead sheet shall cover a greater area than the capsule and shall rest on a smooth, solid surface. A fresh lead surface shall be used for each test, and the capsule shall be struck in such a manner that the maximum possible damage will result.

Test Equipment. The equipment required for the percussion test consisted of the steel billet, the billet guide mechanism and a section of lead sheet. The billet used for the test was a piece of 1-in.-dia steel rod that was 27 in. long and weighed 6.20 lb (2.81 kg). The lead sheet was 12 in. wide by 24 in. long x 0.5 in. thick and had a Brinell hardness of 4.2. The billet guide mechanism consisted of a section of 1-in.-dia Sch. 10 steel pipe mounted in a vertical direction on a support stand. The support stand was equipped with three leveling screws to ensure that the guide mechanism was absolutely vertical. Figure 51 is a sketch of the equipment. Since the test required an impact energy of 7 kg-m and the steel billet weighed 2810 g, the billet had to be dropped from a height of

FIGURE 51. Equipment Used for the Primary Percussion Test

2.5 m (98.5 in.). Once the pipe had been properly aligned, its inner diameter was sufficient to allow free fall of the steel billet.

In carrying out a test, the capsule was struck with the steel billet at each of the five locations shown in Figure 52. The capsule was repositioned to a new portion of the lead sheet surface after each of the five strikes. The lead sheet was replaced when the available surface area had been used.

Test Procedure. After the preliminary measurements and tests on the capsule had been completed, the primary percussion test was carried out in the following manner:

1. The capsule was positioned on a fresh surface of the lead sheet. The billet guide mechanism was positioned and aligned to allow the billet to strike the capsule at location #1 shown in Figure 52.

FIGURE 52. Sketch Showing the Five Locations Where the Capsule was Impacted in the Primary Percussion Test

2. The billet was placed in position in the guide mechanism. The bottom end of the billet was 2.5 m above the point of impact on the capsule surface.
3. The billet was dropped, which allowed it to strike the capsule at the designated location with an energy of 7 kg-m. The capsule was examined for visible damage.
4. The capsule was repositioned on a fresh surface of the lead sheet. The billet guide mechanism was repositioned to allow the billet to strike the capsule at location #2. The billet was raised into position and dropped. The capsule was again examined for visible damage.
5. Step 4 was repeated, allowing the billet to strike the capsule at location #3.
6. Step 4 was repeated, allowing the billet to strike the capsule at location #4.
7. Step 4 was repeated, allowing the billet to strike the capsule at location #5.
8. The capsule was subjected to the water-leak check.
9. The capsule was weighed and its dimensions determined at the 11 locations previously specified.
10. The capsule was subjected to the helium-leak check.
11. The capsule welds were radiographed to determine if damage had occurred.

If the capsule passed the primary percussion test without damage, it was subjected to the secondary qualification tests.

8.1.3.2 Secondary Percussion Test

Each capsule subjected to a primary qualification test other than the primary percussion test was subjected to a secondary percussion test. The secondary percussion test was carried out using the same equipment used for the primary percussion test. The procedure used for the secondary percussion test was identical to that used for the primary percussion test except that the capsule was not subjected to the final helium-leak check (Step 10) and the welds were not radiographed (Step 11).

If the capsule passed the secondary percussion test, it was then subjected to the remaining secondary qualification tests. If the percussion test was the last of the secondary qualification tests, the capsule was subjected to the helium-leak check to ensure it did not leak at a rate greater than 1×10^{-8} std cm³/s.

8.1.4 Thermal Test

Each prototype outer capsule was subjected to a primary thermal test or a secondary thermal test. Capsules subjected to the primary thermal test and most of the capsules

subjected to the secondary thermal test did not contain an inner capsule. Only those capsules subjected to the primary impact test contained an inner capsule when subjected to the secondary thermal test.

8.1.4.1 Primary Thermal Test

Test Requirement. The capsule shall not leak at a rate greater than 1×10^{-8} std cm^3/s after heated to its maximum operating temperature of 800°C and held at 800°C for at least 30 min.

Test Equipment. A three-zone 5-in.-dia hinged tube furnace (Lindberg Model No. 54676) was used for the thermal test. The furnace was equipped with a three-zone solid state proportioning control console (Lindberg Model 59754-A). A split section of 4-in.-dia 304L stainless steel tubing was used to support the capsule in the furnace and prevent contact of the capsule with the heating elements. Plugs of mineral wool insulation were inserted in each end of the furnace tube to reduce the temperature gradient across the length of the furnace (see Figure 53A). Four calibrated sheathed chromel-alumel thermocouples were used to monitor the capsule temperature. The tips of the thermocouple sheaths were spot-welded to the capsule surface at the locations shown in Figure 53B. Outputs of the thermocouples were monitored using a calibrated multipoint digital thermometer (Omega Model No. 2166A). Orientation of the capsule in the furnace is shown in Figure 53C. The furnace required ~ 1 h to reach the test temperature of 800°C . Using the three-zone furnace and the Fibrefrax end plugs, it was possible to reduce the temperature gradient along the length of the capsule to less than 5°C . Tests showed that once the controller settings required to give a capsule temperature of 800°C had been determined, repeated tests could be carried out without changing the settings.

Test Procedure. After the preliminary measurements and tests on the capsules had been completed, the primary thermal test was carried out in the following manner:

1. The four thermocouples were spot-welded to the capsule surface at the locations shown in Figure 53B.
2. The capsule was placed in the tube furnace and the Fibrefrax plugs inserted.
3. The power to the furnace was turned on and the temperature increased until the minimum temperature of the capsule surface at any of the four locations monitored was 800°C . The three controllers were adjusted to bring the temperatures at the four locations to 800°C . The surface temperatures at the four locations and the controller settings required to maintain the temperatures were recorded.
4. The capsule was maintained at the test temperature for at least 30 min.
5. Power to the furnace was turned off and the capsule was allowed to cool to room temperature inside the furnace.

A. FURNACE AND CONTROLLER

B. LOCATIONS OF THERMOCOUPLES FOR THE THERMAL TEST

C. ORIENTATION OF THE CAPSULE IN THE FURNACE

FIGURE 53. Orientation of the Capsule and Thermocouples in the Furnace in the Thermal Test

6. The capsule was removed from the furnace and the thermocouples detached from the capsule surface. Any excess metal remaining on the capsule surface from the spot welds was also removed using a fine file.
7. The capsule was subjected to the water-leak check.
8. The capsule was weighed and its dimensions determined at the 11 locations previously specified.
9. The capsule was subjected to the helium-leak check.
10. The capsule welds were radiographed to determine if damage had occurred.

If the capsule passed the primary thermal test, it was subjected to the secondary qualification tests.

8.1.4.2 Secondary Thermal Test

Each capsule subjected to a primary qualification test other than the primary thermal test was subjected to a secondary thermal test. The secondary thermal test was carried out using the equipment used for the primary thermal test. The procedure used for the secondary thermal test was as follows:

1. The capsule was placed in the furnace and the Fibrefrax plugs inserted.
2. The capsule was heated to 800°C and held at 800°C for at least 30 min. The controller settings identified in the primary thermal test were used for the secondary test, and a single calibrated thermocouple placed in the furnace was used to monitor the temperature and ensure that a test temperature of 800°C was obtained.
3. After cooling in the furnace, the capsule was removed and subjected to the water-leak check.
4. The capsule was weighed and its dimensions determined at the 11 locations previously specified.

If the capsule passed the secondary thermal test, it was subjected to the remaining secondary qualification tests. If the secondary thermal test was the last of the secondary qualification tests, the capsule was subjected to a final helium-leak test to ensure that it did not leak at a rate greater than 1×10^{-8} std cm³/s.

8.1.5 Thermal Quench Test

Each prototype outer capsule was subjected to a primary thermal quench test or a secondary thermal quench test. Capsules subjected to the primary thermal quench test and some of the capsules subjected to the secondary thermal quench test did not contain an inner capsule. Only those capsules subjected to the primary impact test contained an inner capsule when subjected to the secondary thermal quench test.

8.1.5.1 Primary Thermal Quench Test

Test Requirement. The capsule shall not leak at a rate greater than 1×10^{-8} std cm³/s after heated to 800°C and then rapid-quenched in a water-ethylene glycol solution held at 0°C or lower. The capsule shall remain in the circulating solution for at least 1 h, and the temperature of the solution shall not exceed 5°C.

Test Equipment. The three-zone tube furnace used for the thermal (800°C) test was also used for the thermal quench test. The quench tank had a diameter of 28-3/4 in. and a height of 40 in. It could hold up to 100 gal of solution. The solution was circulated in the tank using a Lightnin mixture. Figure 54 shows a sketch of the equipment used for the test. The capsule was transferred from the furnace to the quench tank using a sling and overhead crane. The sling was fabricated from cable and a split section of 5-in. stainless steel tubing. A metal rod was used to push the capsule out of the furnace and into the

FIGURE 54. Sketch of the Equipment Used for the Thermal Quench Test

sling. The sling was then lowered into the quench tank using the crane. The entire transfer operation required ~30 s. The quench solution consisted of a water -10% ethylene glycol mixture, which was cooled to 0°C or lower using dry ice. A calibrated Cr-Al thermocouple and digital thermometer were used to monitor the solution temperature.

Test Procedure. After the preliminary measurements and tests on the capsule had been completed, the primary thermal quench test was carried out in the following manner:

1. The capsule was placed in the three-zone tube furnace, the Fibrefrax plugs inserted, and the capsule heated to 800°C. The controller settings identified in the primary thermal test were used to bring the capsule to the test temperature (periodic checks using a capsule with thermocouples welded to the surface were carried out to ensure that the controller setting gave a capsule temperature of 800°C).
2. The capsule was held at 800°C for at least 30 min.
3. The temperature of the water-ethylene glycol mixture was cooled to 0°C or lower by adding dry ice to the solution. The solution was stirred using the Lightnin mixer.

4. The capsule was transferred from the furnace to the quench tank and left in the tank for at least 1 h. The temperature of the solution in the tank was monitored throughout the test.
5. The capsule was removed from the quench tank and subjected to the water-leak check.
6. The capsule was weighed and its dimensions determined at the 11 locations previously specified.
7. The capsule was subjected to the helium-leak check.
8. The capsule welds were radiographed to determine if damage had occurred.

If the capsule passed the primary thermal quench test, it was then subjected to the secondary qualification tests.

8.1.5.2 Secondary Thermal Quench Test

Each capsule that was subjected to a primary qualification test other than the primary thermal quench test was subjected to a secondary thermal quench test. The same equipment was used for the secondary thermal quench test as was used for the primary thermal quench test. The procedure was the same for the secondary test as for the primary test except that the helium leak check (Step 8) and radiography of the welds (Step 9) were omitted.

If the capsule passed the secondary thermal quench test, it was subjected to any remaining secondary tests. If the secondary thermal quench test was the last of the secondary tests, the capsule was subjected to a final helium-leak check to ensure that it did not leak at a rate greater than 1×10^{-8} std cm³/s.

8.2 SUPPLEMENTAL TESTS PROCEDURES

Several prototype outer capsules were subjected to supplemental tests to show that the capsule could meet the requirements defined in Table 3. Three supplemental tests were used:

- percussion test (20 kg-m impact energy);
- hydrostatic pressure test (24,500 psi);
- puncture test.

The capsules were subjected to the supplemental tests after the primary and secondary qualification tests were completed.

8.2.1 Supplemental Percussion Test

The supplemental percussion test was carried out in the same manner as the primary percussion test described in Section 8.1.3. The impact energy of the billet striking the capsule was 20 kg-m (~145 ft-lb) for the supplemental test versus 7 kg-m (~50 ft-lb) for the primary percussion test. The capsule was impacted at the five locations shown in Figure 52. The steel billet used for the test was 2 in. (5.1 cm) in dia by 21 in. (53.3 cm) long and weighed 20.7 lb (9370 g). The end of the billet striking the capsule was machined as shown in Figure 55. A 2-in.-dia Sch. 40 steel pipe was used as a guide mechanism for the steel

FIGURE 55. Sketch of the Steel Billet Used for the Supplemental Percussion Test

billet. The support stand used for the primary percussion test was used to hold the guide mechanism. The supplemental percussion test was carried out in the following manner:

1. The capsule was placed on a fresh surface of 1/2-in.-thick lead sheet.
2. The guide mechanism was aligned to allow the steel billet to strike the capsule at location #1 as shown in Figure 52.
3. The steel billet was raised into position. The lower (impact) end of the billet was 2.13 m above the capsule surface. The billet was dropped, striking the capsule at location #1 with an impact energy of 20 kg-m.
4. The capsule was examined visually for damage.
5. Steps 1 through 4 were repeated in which the capsule was struck at location #2 (Figure 52).
6. Steps 1 through 4 were repeated in which the capsule was struck at location #3 (Figure 52).
7. Steps 1 through 4 were repeated in which the capsule was struck at location #4 (Figure 52).

8. Steps 1 through 4 were repeated in which the capsule was struck at location #5 (Figure 52).
9. The capsule was subjected to the water-leak check.
10. The capsule was weighed and its dimensions determined at the 11 locations previously specified.
11. The capsule was subjected to the helium-leak check.

The capsule passed the supplemental percussion test if it did not leak at a rate greater than 1×10^{-8} std cm³/s after the test, as determined by the helium-leak check.

8.2.2 Supplemental Pressure Test

The supplemental pressure test was carried out using the same autoclave and auxiliary equipment used for the primary and secondary hydrostatic pressure tests (see Section 8.1.3.1). In the supplemental pressure test, the capsule was pressurized in the autoclave until collapse of the capsule occurred, or to the maximum pressure capability of the equipment, which was 24,600 psi. The procedure used for the supplemental pressure test was as follows:

1. The capsule was placed in the autoclave, the autoclave sealed, and a hydrostatic pressure applied to the capsule.
2. The pressure was increased until the capsule collapsed or a pressure of 24,600 psi was reached. Capsule collapse could be detected from the pump action and the pressure at collapse determined from the monitoring equipment. The capsule was held at pressure for 30 min.
3. The pressure was released, the autoclave opened, and the capsule removed.
4. The capsule was subjected to the water-leak check.
5. The capsule was weighed and its dimensions determined at the 11 locations previously specified.
6. The capsule was subjected to the helium leak check.

The capsule passed the supplemental pressure test if it did not leak at a rate greater than 1×10^{-8} std cm³/s after the test, as determined by the helium-leak check. If the capsule collapsed, it was still assumed to have passed the test if it passed the helium-leak check.

8.2.3 Supplemental Puncture Test

The supplemental puncture test was carried out using much of the equipment used for the primary and secondary percussion tests. In the puncture test, the capsule was struck with a steel billet having an impact energy of 1 kg-m. The end of the billet striking the capsule was machined as shown in Figure 56. The tip of the billet was 3.2 mm in dia and was hemispherical in shape. The billet was 2.54 cm in dia by 15 cm long and weighed 600 g. The capsule was impacted with the billet at the three locations shown Figure 56. The capsule was placed on a section of steel plate that was placed on a solid concrete foundation. The steel

FIGURE 56. Sketch Showing Steel Billet Used for the Puncture Test and the Three Locations Where the Capsule was Impacted

plate was 1/2 in. thick, 12 in. wide and 24 in. long. The guide mechanism and support stand used for the primary percussion test was used to guide the steel billet in the puncture test. The procedure used was as follows:

1. The capsule was placed on the steel plate.
2. The billet guide mechanism was positioned and aligned to allow the billet to strike the capsule at location #1 as shown in Figure 56.

3. The billet was placed in position in the guide mechanism. The bottom impact end of the billet was 1.67 m above the point of impact on the capsule surface.
4. The billet was dropped, which allowed it to strike the capsule at the designated location with an impact energy of 1 kg-m. The capsule was examined for visible damage.
5. The capsule guide mechanism was repositioned to allow the billet to strike the capsule at location #2. The billet was raised into position and dropped. The capsule was again examined for visible damage.
6. Step 5 was repeated, allowing the billet to strike the capsule at location #3.
7. The capsule was subjected to the water-leak check.
8. The capsule was weighed and its dimensions determined at the 11 locations previously specified.
9. The capsule was subjected to the helium-leak check.

The capsule passed the puncture test if it did not leak at a rate greater than 1×10^{-8} std cm^3/s , as determined by the helium-leak check.

8.3 QUALIFICATION TEST DATA

Test results obtained with three of the prototype outer capsules are presented in this section. The data are typical of the results obtained with all of the prototype outer capsules.

8.3.1 As-Fabricated Capsule

CAPSULE NO. S-12(a)

Material	Hastelloy S
Primary Qualification Test	Impact
Secondary Qualification Tests	Pressure Percussion Thermal Thermal Quench
Supplemental Tests	Percussion
Seal Weld Penetration	100% (after rewelding)
Weld Defects (as determined by radiography)	0.025-in. pore 0.10 in. from weld joint at top end of capsule
Initial Capsule Weight	22,210 g
Initial Capsule Dimensions	See page 111
Initial Water-Leak Check	Leak Rate $<1 \times 10^{-4}$ std cm ³ /s
Initial Helium-Leak Check	Leak Rate $<1 \times 10^{-8}$ std cm ³ /s

CAPSULE NO. S-12. Initial Capsule Dimensions at Locations Shown

MINOR AXIS, in.	MAJOR AXIS, in.		CAPSULE TOP
3.374	3.375	→	1 in.
3.375	3.375	→	2 in.
3.375	3.375	→	2 in.
3.375	3.375	→	2 in.
3.375	3.375	→	2 in.
3.375	3.375	→	1-½ in.
3.375	3.375	→	1-½ in.
3.375	3.375	→	2 in.
3.375	3.375	→	2 in.
3.375	3.375	→	2 in.
3.375	3.375	→	2 in.
3.375	3.375	→	1 in.
			21 in.

(a) Capsule contained a Hastelloy C-276 inner capsule filled with SrF₂.

CAPSULE NO. S-12--PRIMARY QUALIFICATION TEST

Type of Test	Impact	Second Capsule Drop ^(b)	August 7, 1980
Initial Capsule Drop	July 9, 1980	Initial Capsule Weight	22,210 g
Final Capsule Weight	22,210 g	Final Capsule Weight	22,210 g
Impact Angle ^(a)	9°5'	Impact Angle	9°
Water-Leak Check	No Leak Detected	Water-Leak Check	No Leak Detected
Helium-Leak Check	Leak Rate $<1 \times 10^{-8}$ std cm ³ /s	Helium-Leak Check	Leak Rate $<1 \times 10^{-8}$ std/cm ³ /s
Radiography	No Change in Weld Zone	Capsule Dimensions	See Page 113
Capsule Dimensions	See Page 112		

The impact test produced some distortion of of the capsule wall near the weld zone adjacent to the point of impact.

(a) Determined from high-speed photographs.

(b) Clip removed and rewelded at location 180° from initial location.

CAPSULE NO. S-12. Capsule Dimensions at the Locations Shown After the Initial Capsule Drop

CAPSULE NO. S-12. Capsule Dimensions at the Locations Shown After the Second Capsule Drop

CAPSULE NO. S-12--SECONDARY QUALIFICATION TESTS

Type of Test	Hydrostatic Pressure (1000 Bar)
Date of Test	August 11, 1980
Test Pressure	14,500 psi
Initial Capsule Weight	22,210 g
Final Capsule Weight	22,210 g
Water Leak Check	No Leak Detected
Capsule Dimension After Test	See Page 115
No Detectable Damage	

Type of Test	Percussion
Date of Test	August 13, 1980
Impact Energy	7 kg-m (50 ft-lb)
Initial Capsule Weight	22,210 g
Final Capsule Weight	22,210 g
Water-Leak Check	No Leak Detected
Capsule Dimensions After Test	See Page 116
No Detectable Damage	

Type of Test	Thermal (800°C)
Date of Test	August 14, 1980
Test Temperature	800°C
Time at Temperature	30 min
Initial Capsule Weight	22,210 g
Final Capsule Weight	22,210 g
Water-Leak Check	No Leak Detected
Capsule Dimensions After Test	See Page 117
No Detectable Damage	

Type of Test	Thermal Quench
Date of Test	August 15, 1980
Capsule Temperature	802°C
Time at Temperature	30 min
Transfer Time	~30 s
Bath Temperature	-4°C to -1°C
Initial Capsule Weight	22,210 g
Final Capsule Weight	22,210 g
Water-Leak Check	No Leak Detected
Capsule Dimension After Test	See Page 118
No Detectable Damage	
Helium-Leak Check	Leak Rate $< 1 \times 10^{-8}$ std /cm ³ /s

CAPSULE NO. S-12.

Capsule Dimensions at Locations Shown After the 1000-Bar Pressure Test

CAPSULE NO. S-12. Capsule Dimensions at the Locations Shown After the Percussion Test

CAPSULE NO. S-12. Capsule Dimensions at Locations Shown After the Thermal (800°C)
Test

CAPSULE NO. S-12.

Capsule Dimensions at the Locations Shown After the Thermal Quench Test

CAPSULE NO. S-12--SUPPLEMENTAL TEST

Type of Test	Percussion
Date of Test	August 21, 1980
Impact Energy	20 kg-m (145 ft-lb)
Initial Capsule Weight	22,210 g
Final Capsule Weight	22,210 g
Water-Leak Check	No Leak Detected
Capsule Dimensions After Test	See Page 119
No Detectable Damage	
Final Helium-Leak Check	Leak Rate $< 1 \times 10^{-8}$ std cm ³ /s

CAPSULE NO. S-12. Capsule Dimensions at Locations Shown After the Supplemental Percussion Test

Final Capsule Disposition. After the final test, the capsule was sectioned. The endcap from the impacted end of the capsule was sectioned and the portion of the endcap affected by the impact was subjected to metallographic examination.

The Hastelloy C-276 inner capsule, after removal from the outer capsule, was examined for damage and then subjected to the secondary percussion test (7 kg-m impact energy). The capsule passed the secondary percussion test without damage and the leak rate was $<1 \times 10^{-4}$ std cm³/s.

Summary of Results. The capsule passed all of the tests without difficulty. The impact test produced some distortion of the capsule wall near the weld zone adjacent to the point of impact. None of the other tests had any visible or measurable effect on the capsule.

8.3.2 Capsule Aged 8000 h at 650°C

CAPSULE NO. S-14

Material	Hastelloy S
Primary Qualification Test	Thermal
Secondary Qualification Tests	Thermal Quench Pressure Percussion Impact
Supplemental Test	Pressure
Seal Weld Penetration	0.050 in. minimum 0.080 in. maximum
Weld Defects	None Apparent
Initial Capsule Weight	14,836 g
Initial Capsule Dimensions	See Page 121
Initial Water-Leak Check	No Leak Detected
Initial Helium-Leak Check	Leak Rate $<1 \times 10^{-8}$ std cm ³ /s
Capsule in Furnace	January 23, 1980
Furnace Temperature	650°C
Capsule out of Furnace	December 22, 1980
Time in Furnace	8004 h
Capsule Weight	14,836
Capsule Dimensions After Aging	See Page 122
Water-Leak Check	No Leak Detected
Helium-Leak Check	Leak Rate $<1 \times 10^{-8}$ std cm ³ /s

CAPSULE NO. S-14. Initial Capsule Dimensions

CAPSULE NO. S-14. Capsule Dimensions After Aging 8000 h at 650°C

CAPSULE NO. S-14--PRIMARY QUALIFICATION TEST

Type of Test	Thermal	Capsule Time at Temperature	30 min
Initial Capsule Weight	14,836 g	Capsule Weight After Test	14,834 g
Date of Test	January 29, 1981	Capsule Dimensions After Test	See Page 123
Furnace On	10:00 am		
Furnace at Temperature	12:10 pm	Water-Leak Check	No Leak Detected
Furnace Off	12:40 pm	Helium-Leak Check	Leak Rate $< 1 \times 10^{-8}$ std cm ³ /s
Furnace Controller Temperature			
Zone 1	833°C	Radiography	No Detectable Change in Weld Zone
Zone 2	824°C		
Zone 3	834°C		
Capsule Temperature			
Thermocouple 1	800°C		
Thermocouple 2	806°C		
Thermocouple 3	806°C		
Thermocouple 4	801°C		

The primary thermal test produced no visible or detectable damage.

CAPSULE NO. S-14. Capsule Dimensions After Primary Thermal Test

CAPSULE NO. S-14--SECONDARY QUALIFICATION TESTS

Type of Test	Thermal Quench
Date of Test	March 12, 1981
Capsule Temperature	806°C
Time at Temperature	33 min
Transfer Time	~30 s
Bath Temperature	-3°C to -2°C
Initial Capsule Weight	14,834 g
Final Capsule Weight	14,833 g
Water-Leak Check	No Leak Detected
Capsule Dimensions After Test	See Page 125
No Detectable Damage	
Type of Test	Hydrostatic Pressure (1000 Bar)
Date of Test	March 17, 1981
Test Pressure	14,550 psi
Initial Capsule Weight	14,833 g
Final Capsule Weight	14,834 g
Water-Leak Check	No Leak Detected
Capsule Dimensions After Test	See Page 126
No Detectable Damage	
Type of Test	Percussion
Date of Test	March 19, 1981
Impact Energy	7 kq-m (50 ft-lb)
Initial Capsule Weight	14,834 g
Final Capsule Weight	14,836 g
Water-Leak Check	No Leak Detected
Capsule Dimensions After Test	See Page 127
No Detectable Damage	
Type of Test	Impact
Date of Test	March 20, 1981
Drop Distance	9 m
Impact Angle	9° 30'
Initial Capsule Weight	14,836 g
Final Capsule Weight	14,838 g
Water-Leak Check	No Leak Detected
Capsule Dimensions After Test	See Page 128
No Significant Damage	Slight Distortion of the Endcap at Point of Impact
Helium-Leak Check	Leak Rate $< 1 \times 10^{-8}$ std cm ³ /s

CAPSULE NO. S-14. Capsule Dimensions at the Locations Shown After the Secondary Thermal Quench Test

CAPSULE NO. S-14. Capsule Dimensions at the Locations Shown After the Secondary Pressure Test

CAPSULE NO. S-14. Capsule Dimensions at the Locations Shown After the Secondary Percussion Test

CAPSULE NO. S-14. Capsule Dimensions at the Locations Shown After the Secondary Impact Test

CAPSULE NO. S-14--SUPPLEMENTAL TESTS

Type of Test	Hydrostatic Pressure
Date of Test	March 30, 1981
Test Pressure	24,500 psi
Initial Capsule Weight	14,838 g
Final Capsule Weight	14,836 g
Water-Leak Check	No Leak Detected
Capsule Dimensions After Test	See Page 129
Helium-Leak Check	Leak Rate $< 1 \times 10^{-8}$ std cm ³ /s

Capsule partially collapsed, but did not leak. Figure 57 shows a photograph of the capsule after the supplemental pressure (24,500 psi) test. The distortion of the capsule is apparent from the photograph.

CAPSULE NO. S-14. Capsule Dimensions at the Locations Shown After the Supplemental Pressure (24,500 psi) Test

FIGURE 57. Capsule No. S-14 After Being Subjected to an External Hydrostatic Pressure of 24,500 psi for 30 Minutes

Final Capsule Disposition. The capsule was retained intact after all of the tests were completed.

Summary of Results. Capsule No. S-14, after being aged at 650°C for 8000 h, passed the primary and secondary qualification tests without difficulty. The capsule showed no

visual evidence of damage except for some distortion of the endcap at the point of impact, which resulted from the impact test. In the supplemental pressure test, the capsule partially collapsed when subjected to an external hydrostatic pressure of 24,500 psi. Even though it had partially collapsed, the capsule passed a final helium-leak check.

8.3.3 Capsule Aged 8000 h at 725°C

CAPSULE NO. S-19

Material	Hastelloy S
Primary Qualification Test	Hydrostatic Pressure (1000 Bar)
Secondary Qualification Tests	Impact Percussion Thermal Thermal Quench
Supplemental Test	Puncture
Seal Weld Penetration	0.055 in. minimum 0.060 in. maximum
Weld Defects (as Determined by Radiography)	None Apparent
Initial Capsule Weight	14,874 g
Initial Capsule Dimensions	See Page 132
Initial Water-Leak Check	No Leak Detected
Initial Helium-Leak Check	Leak Rate $< 1 \times 10^{-8}$ std cm ³ /s
Capsule in Furnace	January 23, 1980
Furnace Temperature	725°C
Capsule Out of Furnace	December 22, 1980
Time in Furnace	8004 h
Capsule Weight	14,874 g
Capsule Dimensions After Aging	See Page 133
Water-Leak Check	No Leak Detected
Helium-Leak Check	Leak Rate $< 1 \times 10^{-8}$ std cm ³ /s

CAPSULE NO. S-19. Initial Capsule Dimensions at the Locations Shown

CAPSULE NO. S-19. Capsule Dimensions at the Locations Shown After Aging for 8000 h at 725°C

CAPSULE NO. S-19--PRIMARY QUALIFICATION TEST

Type of Test	Hydrostatic Pressure
Date of Test	February 2, 1981
Test Pressure	10,000 psi
Initial Capsule Weight	14,874 g
Final Capsule Weight	14,875 g
Capsule Dimensions After Test	See Page 135
Water-Leak Check	No Leak Detected
Helium-Leak Check	Leak Rate $<1 \times 10^{-8}$ std cm ³ /s
Date of Test	February 5, 1981
Test Pressure	12,000 psi
Final Capsule Weight	14,874
Capsule Dimensions After Test	See Page 136
Water-Leak Check	No Leak Detected
Helium-Leak Check	Leak Rate $<1 \times 10^{-8}$ std cm ³ /s
Date of Test	February 9, 1981
Test Pressure	13,000 psi
Final Capsule Weight	14,873 g
Capsule Dimensions After Test	See Page 137
Water-Leak Check	No Leak Detected
Helium-Leak Check	Leak Rate $<1 \times 10^{-8}$ std cm ³ /s
Date of Test	February 11, 1981
Test Pressure	14,500 psi
Final Capsule Weight	14,873 g
Capsule Dimensions After Test	See Page 138
Water-Leak Check	No Leak Detected
Helium-Leak Check	Leak Rate $<1 \times 10^{-8}$ std cm ³ /s
Radiography	No Detectable Change in the Weld Zones

The primary hydrostatic pressure test produced no detectable change in the capsule integrity or visible sign of damage.

CAPSULE NO. S-19. Capsule Dimensions at the Locations Shown After Being Pressurized at 10,000 psi for 30 Minutes

CAPSULE NO. S-19. Capsule Dimensions at the Locations Shown After Being Pressurized to 12,000 psi for 30 Minutes

CAPSULE NO. S-19. Capsule Dimensions at the Locations Shown After Being Pressurized to 13,000 psi for 30 Minutes

CAPSULE NO. S-19. Capsule Dimensions at the Locations Shown After Being Pressurized to 14,500 psi for 30 Minutes

CAPSULE NO. S-19--SECONDARY QUALIFICATION TESTS

Type of Test	Impact
Date of Test	March 6, 1981
Drop Distance	9 m
Impact Angle	9° 0'
Initial Capsule Weight	14,873 g
Final Capsule Weight	14,877 g
Capsule Dimensions After Test	See Page 140
Water-Leak Check	No Leak Detected
No Significant Damage	Some Slight Distortion of the Endcap at the Point of Impact
Type of Test	Percussion
Date of Test	March 10, 1981
Impact Energy	7 kg-m (50 ft-lb)
Final Capsule Weight	14,875 g
Capsule Dimension After Test	See Page 141
Water-Leak Check	No Leak Detected
No Detectable Damage	
Type of Test	Thermal (800°C)
Date of Test	March 19, 1981
Test Temperature	800°C
Time at Temperature	30 min
Final Capsule Weight	14,875 g
Capsule Dimensions After Test	See Page 142
Water-Leak Check	No Leak Detected
No Detectable Damage	
Type of Test	Thermal Quench
Date of Test	March 25, 1981
Capsule Temperature	807°C
Time at Temperature	30 min
Transfer Time	~30 s
Bath Temperature	-3°C to 0°C
Final Capsule Weight	14,875 g
Capsule Dimensions After Test	See Page 143
Water-Leak Check	No Leak Detected
Helium-Leak Check	Leak Rate $<1 \times 10^{-8}$ std cm ³ /s
No Detectable Damage	

CAPSULE NO. S-19. Capsule Dimensions at the Locations Shown After the Secondary Impact Test

CAPSULE NO. S-19. Capsule Dimensions at the Locations Shown After the Secondary Percussion Test

CAPSULE NO. S-19. Capsule Dimensions at the Locations Shown After the Secondary Thermal Test

CAPSULE NO. S-19. Capsule Dimensions at the Locations Shown After the Secondary Thermal Quench Test

CAPSULE NO. S-19--SUPPLEMENTAL TESTS

Type of Test	Puncture
Date of Test	June 15, 1981
Impact Energy	1 kg-m (7 ft-lb)
Final Capsule Weight	14,875
Capsule Dimensions After Test	See Page 144
Water-Leak Check	No Leak Detected
Helium-Leak Check	Leak Rate $< 1 \times 10^{-8}$ std cm ³ /s
No Detectable Damage	Slight Indentations in Capsule Wall at Point of Impact

CAPSULE NO. S-19. Capsule Dimensions at the Locations Shown After the Supplemental Puncture Test

Final Capsule Disposition. The capsule was retained intact after all of the tests were completed.

Summary Results. Capsule No. S-19, after being aged at 725°C for 8000 h, passed all of the primary and secondary qualification tests without significant damage. The capsule also passed the supplemental puncture test without significant damage. The impact test produced some slight distortion of the endcap at the point of impact, and the percussion and puncture tests produced some slight indentations in the capsule wall at the points of impact.

8.4 OXIDATION TESTS

The outer capsule of the $^{90}\text{SrF}_2$ heat source must be sufficiently resistant to air oxidation so that oxidation that occurs during the normal service life of the source will not degrade the mechanical properties of the outer capsule to the point that it cannot meet the qualification test requirements. This assumes the service life of the heat source is 10 years and the maximum surface temperatures of the outer capsule is 800°C.

Tests were carried out at 600° to 800°C to determine the oxidation rates of Hastelloy S and Hastelloy C-4 in air, and to determine the effects of the oxidation on the mechanical properties of the two alloys. Details of the oxidation studies are presented in Fullam (1980b, 1981c) and summarized below.

8.4.1 Oxidation Rates

The oxidation of Hastelloy S and Hastelloy C-4 was studied at temperatures of 600°, 700° and 800°C for periods up to 10,000 h. Oxidation rates for the two alloys were determined using a discontinuous gravimetric procedure. This involved heating a large number of metal specimens in air for various times at each test temperature and determining the weight change of each specimen due to oxidation. Tensile and CVN specimens of each alloy were oxidized under similar conditions and then mechanically tested at room temperature. Other tensile and CVN specimens were heated in vacuum at each temperature to serve as controls.

The Hastelloy S and Hastelloy C-4 form adherent oxide layers when heated in air at 600° to 800°C. The oxide layers serve to protect the underlying metal. Overall, the oxidation rates for the two alloys are very slow at 600° to 800°C. The test data show that the oxidation of Hastelloy S and Hastelloy C-4 in air follows parabolic rate relationships, except for initial deviations, for exposures up to 5000 h. Longer exposures lead to negative deviations from the parabolic rate relationships. There was no evidence of breakaway spalling or cracking of the oxide layer during oxidation, but some spalling did occur when the test specimens were cooled.

The parabolic rate relationship is given by the equation

$$\Delta w^2 = k_p T + C$$

where

Δw = weight gain per unit area of surface, mg/cm^2

T = time, h

k_p = rate constant, $\text{mg}^2/\text{cm}^4\text{-h}$

C = constant, mg^2/cm^4 .

When Δw^2 is plotted against time, a straight line indicates adherence to the parabolic relationship, with the rate-constant k_p given by the slope of the line and C by the intercept with the y axis. The parabolic rate constants were determined for the oxidation of Hastelloy S and Hastelloy C-4 in air at 600°, 700° and 800°C, and the values obtained are shown in Table 29. The values for the rate constants are lower than the values published for most oxidation-resistant nickel-base alloys and indicate the good oxidation resistance of the Hastelloy S and Hastelloy C-4 at 600° to 800°C. Since exposures greater than 5000 h lead to negative deviations from the parabolic relationships, the parabolic rate equations can be used to obtain conservative estimates of the long-term (>5000 h) oxidation of the two alloys in air at 600° to 800°C.

The test specimens oxidized in air at 600° to 800°C were subjected to metallographic examination. The photomicrographs obtained show that the depth of metal affected by the oxidation reactions is greater than one would predict based on the weight gain measurements. Table 30 shows estimates of the depth of metal affected by oxidation as a function of time for each alloy. It is difficult to obtain reliable estimates of the depth of metal affected from the photomicrographs, and the values given in Table 30 should only be regarded as approximations. The data presented in Table 30 are quite erratic, and it is impossible to determine what type of rate equation applies for each alloy at the different temperatures. The best estimate that can be made is that the rate is much less than linear and is probably closer to parabolic or exponential.

8.4.2 Effect of Oxidation on Mechanical Properties

The tensile specimens used in the oxidation tests were subsize specimens having a reduced section diameter of 0.113 in. (2870 μm). The tensile specimens that had been heated in air or vacuum at 600°, 700° and 800°C for up to 10,000 h were tension-tested at room

TABLE 29. Parabolic Rate Constants for the Long-Term Oxidation of Hastelloy S and Hastelloy C-4 in Air

Material	Temperature, °C	k_p , $\text{mg}^2/\text{cm}^4\text{-h}$	C , mg^2/cm^4
Hastelloy S	600	1.64×10^{-6}	0.00498
	700	2.26×10^{-5}	0.00767
	800	5.73×10^{-5}	0.0277
Hastelloy C-4	600	1.92×10^{-5}	0.154
	700	3.34×10^{-5}	0.221
	800	8.19×10^{-5}	0.271

TABLE 30. Depth of Metal Affected When Hastelloy S and Hastelloy C-4 Specimens are Oxidized in Air at 600° to 800°C

Temperature, °C	Time, h	Depth of Metal Affected, μm ^(a)	
		Hastelloy S	Hastelloy C-4
600	140	13	8
	300	15	12
	528	17	15
	994	14	10
	2,500	18	16
	5,000	20	18
	7,500	26	23
	10,000	30	30
700	140	7	6
	300	8	8
	528	10	13
	994	10	8
	2,500	18	18
	5,000	15	20
	7,500	20	20
	10,000	18	25
800	140	14	10
	300	16	12
	528	20	13
	994	16	16
	2,500	23	23
	5,000	30	15
	7,500	42	36
	10,000	50	40

(a) Estimated from photomicrographs.

temperature using a strain rate of 0.02 in./in. The results obtained with Hastelloy S are given in Table 31, while the Hastelloy C-4 results are given in Table 32. A statistical evaluation of the test data showed that there was no significant differences in tensile properties existed between the specimens heated in air and the control specimens heated in vacuum. Metallographic examination of the tensile specimens showed that the oxidation reactions affected the underlying metal to depths up to 0.002 in. (51 μm). For a 0.113-in.-dia tensile specimen, this means that up to 7% of the cross-sectional area of the specimens was affected by oxidation. Even this degree of attack did not affect the tensile properties of the specimens significantly.

TABLE 31. Room-Temperature Tensile Data^(a) for Hastelloy S Specimens Aged in Air or Vacuum at 600° to 800°C

Temperature, °C	Time, h	Ultimate Tensile Strength, MPa		Yield Strength, MPa		Elongation, %			
		Vacuum		Air		Uniform		Total	
		Vacuum	Air	Vacuum	Air	Vacuum	Air	Vacuum	Air
(As Rec) ^(b)	0	(880)		(469)		(51.5)		(60.8)	
600	994	938	903	504	541	50.4	50.8	56.0	56.1
	2,500	1,019	1,042	617	598	44.3	44.4	48.8	48.3
	5,000	1,252	1,176	772	748	33.6	36.5	37.1	41.4
	7,500	1,180	1,166	723	701	35.8	37.8	39.8	41.0
	10,000	1,194	1,172	747	723	38.5	38.2	39.5	41.2
700	994	907	879	462	442	53.8	55.4	60.0	61.8
	2,500	882	889	424	457	55.5	47.9	62.6	54.1
	5,000	927	911	479	470	47.6	48.1	53.4	53.7
	7,500	900	895	453	434	50.6	50.0	57.2	57.0
	10,000	957	897	470	458	51.2	50.5	56.5	55.8
800	994	912	892	441	421	53.2	45.9	59.5	63.6
	2,500	876	870	408	408	54.9	54.4	62.7	62.1
	5,000	902	885	420	422	50.9	49.3	59.1	58.2
	7,500	923	864	457	394	47.7	54.0	53.2	62.0
	10,000	903	878	395	410	56.9	57.8	62.5	62.9

(a) Average values for four replicate specimens aged in air and duplicate specimens aged in vacuum. Specimen dia = 0.113 in.

(b) Solution-heat-treated alloy as received from the vendor.

TABLE 32. Room-Temperature Tensile Data^(a) for Hastelloy C-4 Specimens Aged in Air or Vacuum at 600° to 800°C

Temperature, °C	Time, h	Ultimate Tensile Strength, MPa		Yield Strength, MPa		Elongation, %			
		Vacuum		Air		Uniform		Total	
		Vacuum	Air	Vacuum	Air	Vacuum	Air	Vacuum	Air
(As Rec) ^(b)	0	(880)		(469)		(51.5)		(60.6)	
600	2,500	1,257	1,236	714	683	38.7	42.0	43.6	48.6
	5,000	1,240	1,243	704	736	39.3	41.2	46.5	46.2
	7,500	1,229	1,230	719	701	41.2	39.5	47.4	45.1
	10,000	1,210	1,219	692	711	43.8	41.0	50.8	44.8
700	2,500	847	820	419	372	55.3	63.9	61.6	64.1
	5,000	850	843	384	853	58.7	63.3	65.6	72.7
	7,500	867	874	400	396	58.9	58.3	66.3	66.2
	10,000	861	872	397	411	56.3	56.9	63.0	63.6
800	2,500	794	801	334	343	63.8	63.2	69.1	72.3
	5,000	829	818	358	348	60.1	59.6	69.5	67.4
	7,500	829	823	343	347	62.9	60.7	71.7	68.7
	10,000	846	851	325	339	70.3	64.5	79.2	73.4

(a) Average values for duplicate specimens. Specimen dia = 0.113 in.

(b) Solution-heat-treated alloy as received from the vendor.

The CVN specimens used in the long-term tests were subsize specimens 0.098 in. thick by 0.394 in. wide. All specimens were cut from alloy sheet with the specimen notch oriented parallel to the rolling direction and perpendicular to the surface of the sheet. The CVN specimens heated in air or vacuum at 600°, 700°, and 800°C for up to 10,000 h were impact-tested at room temperature. The results obtained with Hastelloy S and Hastelloy C-4 are given in Table 33. A statistical analysis of the test results showed that no significant difference existed in the Charpy impact energy of the oxidized specimens heated in air and the control specimens heated in vacuum.

The oxidation reactions affected the CVN specimens to depths of up to 0.002 in. (51 μm). For the subsize CVN specimens, this means that up to ~6% of the cross-sectional area below the specimen notch was affected by the oxidation reactions. Even this degree of attack did not affect the impact energy of the specimens significantly.

8.4.3 Effects of Long-Term Oxidation

The oxidation requirement necessitates that the outer capsule of the heat source must be resistant enough to withstand 10 years of oxidation in air at temperatures of up to 800°C without suffering a significant degradation in mechanical properties. Conservative estimates of the long-term oxidation of Hastelloy S and Hastelloy C-4 in air can be made using the parabolic rate constants given in Table 29. Such estimates show that even after 20 years of oxidation at 800°C, the maximum weight gain exhibited by the two alloys would be less than 4 mg/cm². The estimates should be valid as long as breakaway spalling is avoided and cracking of the oxide layer and resulting exposure of the metal substrate does not occur during oxidation. In the oxidation tests lasting up to 10,000 h, there was no indication of breakaway spalling or cracking of the protective oxide film. Data reported in the literature on the oxidation of nickel-base alloys indicate that fracture and spalling of the oxide layer generally does not occur until the thickness of the oxide is much greater than the thickness of the oxide that would form on Hastelloy S or Hastelloy C-4 after 20 years at 600° to 800°C.

It is more difficult to make reliable estimates of the depth of metal that would be affected by the long-term oxidation of the two alloys. This arises from the fact that the metal attack observed in the oxidation tests did not adhere to any of the normal rate equations. A very conservative estimate of long-term attack can be made if one assumes the rate is linear with time. This was done for an oxidation temperature of 800°C using the data shown in Table 30. The estimates show that after 10 years at 800°C the depth of metal affected by oxidation reactions would be about 350 μm (0.014 in.) for both the Hastelloy S and the Hastelloy C-4. The heat-source outer capsule has an outer diameter of 3.375 in. and a 0.500-in. wall thickness. Therefore, the metal affected by the oxidation reactions after 10 years at 800°C would amount to ~3.3% of the capsule's cross-sectional area. Based on the results obtained in the oxidation studies, this level of attack should not affect the mechanical properties of the outer capsules to any significant degree.

TABLE 33. The Room-Temperature Charpy Impact Energy of Hastelloy S and Hastelloy C-4 CVN Specimens Heated in Air or Vacuum

Temperature, °C	Time, h	Charpy Impact Energy, (a) ft-lb			
		Hastelloy S		Hastelloy C-4	
		Vacuum	Air	Vacuum	Air
As-Received ^(b)		(14.0)		(29.5)	
600	1,000	10.1	9.8		
	2,500	8.7	8.7	8.7	8.0
	5,000	7.6	7.2	8.4	8.0
	7,500	6.5	7.2	7.6	7.2
	10,000	5.8	6.2	8.0	7.2
700	1,000	9.4	10.9		
	2,500	10.5	11.6	23.9	23.9
	5,000	11.6	10.9	19.5	22.5
	7,500	8.7	10.5	23.3	24.0
	10,000	10.1	10.1	23.8	25.0
800	1,000	15.5	16.3		
	2,500	15.5	16.5	25.8	32.8
	5,000	16.3	16.5	29.0	28.5
	7,500	15.5	16.3	27.5	28.3
	10,000	16.5	17.0	26.8	28.9

(a) Average value for duplicate specimens, except for as-received values where six Hastelloy S and five Hastelloy C-4 specimens were tested.

(b) Solution heat-treated alloy as received from the manufacturer.

8.5 SEAWATER CORROSION TESTS

The heat-source outer capsule must be sufficiently reactant to seawater corrosion that it can meet the qualification test requirements after exposure to seawater for a period of 1 year. Experimental tests were carried out to measure the corrosion rates of Hastelloy S and Hastelloy C-4 in seawater and to determine what effect the attack would have on the tensile properties of the two alloys. Details of the tests are provided in Fullam (1980b) and summarized below.

The seawater corrosion studies were conducted at PNL's facility at Sequim, Washington. The tests were carried out in a small, covered plastic tank, using unfiltered seawater taken from Sequim Bay. The tank was located outdoors, and the test temperature varied between ~10° and 25°C. The seawater flowed through the tank at a rate of 4 L/h. Tensile specimens and prestressed specimens were tested in addition to corrosion coupons. The test specimens were completely immersed in the flowing seawater. The tests lasted up to 10,000 h. Average corrosion rates were determined by weighing the coupons before and after testing. Localized attack was determined by subjecting the coupons to metallographic examination after testing.

The results obtained show that the corrosion of Hastelloy S and Hastelloy C-4 in seawater at 10° to 25°C is extremely low. All of the test specimens exposed to the seawater exhibited some surface discoloration, which appeared to result from a combination of marine fouling and nonadherent corrosion products. The discoloration was easily removed when the coupons were cleaned with detergent solution and a soft bristle brush. Visual examination of the specimens after cleaning showed no indication of corrosion. Weight loss data for the two alloys are given in Table 34. The data show that corrosion of the Hastelloy C-4 was several times greater than that of Hastelloy S but was still very low.

Metallographic examination of the test specimens showed that localized attack of the two alloys was very slight. None of the Hastelloy S specimens tested for 5,000 h or less exhibited any evidence of localized attack. Some isolated pitting to a depth of 2 μm (~ 0.00008 in.) was observed with specimen tested for 10,000 h. Some isolated pitting was observed with Hastelloy C-4 specimens tested for 1,000 h and longer. The maximum depth of the pitting was about 3 μm (0.00012 in.) for specimens tested for 10,000 h.

Microscopic examination of the prestressed specimens showed no evidence of stress-corrosion cracking of either the Hastelloy S or Hastelloy C-4.

The tensile specimens used in the corrosion tests were subsize specimens having a reduced section diameter of 0.113 in. (2870 μm). The specimens were the same size as those used in the oxidation tests. After exposure to seawater, the tensile specimens were tension-tested at room temperature, using a strain rate of 0.02 in./in. The results obtained are given in Table 35. Evaluation of the data shows that exposure of the Hastelloy S and Hastelloy C-4 to seawater for periods up to 7500 h had no significant effect on the tensile properties of the specimens. Significant changes in tensile properties were observed, however, with the specimens exposed to 10,000 h. The Hastelloy S specimens exhibited marked increases

TABLE 34. Corrosion of Hastelloy S and Hastelloy C-4 in Seawater at Ambient Temperature^(a)

Exposure, h	Coupon Weight Change, mg/cm ²	
	Hastelloy S ^(b)	Hastelloy C-4 ^(c)
250	-0.0038 $\pm$ 0.0013	-0.070 $\pm$ 0.0097
500	-0.0066 $\pm$ 0.0016	-0.047 $\pm$ 0.014
1,000	+0.0034 $\pm$ 0.0004	-0.089 $\pm$ 0.029
2,500	-0.0021 $\pm$ 0.0010	-0.125 $\pm$ 0.008
5,000	-0.0038 $\pm$ 0.0010	-0.180 $\pm$ 0.046
7,500	-0.0057 $\pm$ 0.0012	-0.156 $\pm$ 0.052
10,000	-0.0074 $\pm$ 0.0021	-0.174 $\pm$ 0.026

(a) Temperature varied from 10° to 25°C with atmospheric conditions.

(b) Average value for four test coupons.

(c) Average value for three test coupons.

in ultimate tensile strength and elongation and a decrease in yield strength. The Hastelloy C-4 specimens exhibited marked increases in yield strengths and ultimate tensile strength and a decrease in elongation. No explanation for differences observed with the 10,000-h specimens has been found. Metallographic examination of the 10,000-h specimens provided no indication of microstructural changes that could account for the differences observed.

In summary, all of the test results show that the corrosion of Hastelloy S and Hastelloy C-4 in seawater is very low. Some very slight localized attack (in the form of isolated pitting) was observed, but did not present a serious problem. Neither alloy exhibited a susceptibility to stress-corrosion cracking. Evaluation of the test data shows that a $^{90}\text{SrF}_2$ heat source, utilizing an outer capsule of Hastelloy S or Hastelloy C-4, could easily withstand an exposure to seawater of 1 year without having the structural strength of its outer capsule significantly degraded. Even allowing for the adverse effects of a radiation field and increased marine fouling and erosion, the corrosion should not be a problem. Therefore, a $^{90}\text{SrF}_2$ heat source using a Hastelloy S or Hastelloy C-4 outer capsule would be able to meet the seawater corrosion requirement. Since the Hastelloy S exhibits a lower corrosion rate in seawater than does Hastelloy C-4, Hastelloy S is the preferred material for the outer capsule; nevertheless, Hastelloy C-4 could be used if other considerations so dictated.

TABLE 35. Room-Temperature Tensile Data for Hastelloy S and Hastelloy C-4 Specimens Exposed to Flowing Seawater at Ambient Temperature (10° to 25°C)

Material	Exposure, h	Ultimate Tensile Strength, MPa	Yield Strength, MPa	Elongation, %	
				Uniform	Total
Hastelloy S	(As Rec) ^(a)	(880 ± 20)	(469 ± 31)	(51.5 ± 2.3)	(60.8 ± 2.9)
	1,000	889 ± 18	487 ± 33	53.3 ± 1.7	61.7 ± 2.0
	2,500	872 ± 8	479 ± 39	53.2 ± 3.5	62.1 ± 1.4
	5,000	878 ± 22	469 ± 19	51.3 ± 3.1	61.1 ± 3.2
	7,500	870 ± 22	470 ± 8	51.9 ± 4.1	60.8 ± 5.3
	10,000	942 ± 4	434 ± 21	49.3 ± 1.4	68.0 ± 1.9
Hastelloy C-4	(As Rec) ^(a)	(834 ± 10)	(379 ± 12)	(60.9 ± 3.2)	(71.5 ± 5.3)
	1,000 ^(b)	852 ± 11	390 ± 20	59.7 ± 3.8	69.7 ± 4.4
	2,500	831 ± 9	366 ± 6	58.9 ± 0.9	69.1 ± 0.6
	5,000	833 ± 8	365 ± 8	59.3 ± 1.0	69.5 ± 1.4
	7,500	840 ± 8	376 ± 12	57.5 ± 1.8	66.8 ± 2.1
	10,000	924 ± 66	492 ± 53	52.5 ± 2.3	62.4 ± 3.5

(a) Solution-heat-treated alloy as received from the vendor.

(b) Four tensile specimens tested to each time period.

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