**Quarterly Report on the
Strontium Heat Source
Development Program and the
Beneficial Isotopes Utilization
Program, Division of Nuclear
Research and Applications for
April-June 1977**

H. T. Fullam

July 1977

**Prepared for the Energy Research
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QUARTERLY REPORT ON THE STRONTIUM HEAT
SOURCE DEVELOPMENT PROGRAM AND THE
BENEFICIAL ISOTOPES UTILIZATION PROGRAM,
DIVISION OF NUCLEAR RESEARCH AND
APPLICATIONS FOR APRIL-JUNE 1977

H. T. Fullam

July 1977

BATTELLE
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Richland, Washington 99352

SUMMARY

A draft copy of the Strontium-90 Fluoride Data Sheet has been submitted to DNRA for review and comment. DNRA has also reviewed the topical report on the supplemental screening tests; and the final draft of the report, incorporating DNRA comments, has been prepared and is ready to be issued. A letter report summarizing PNL's recommendations on the $^{90}\text{SrF}_2$ heat source design criteria and qualification test matrix has been reviewed by DNRA, and the final version of the report will be issued in July.

The 12,000 hour compatibility tests with nonradioactive SrF_2 have been completed, and the specimens analyzed. Overall, the specimens exposed to SrF_2 for 12,000 hours suffered no greater attack than those exposed for 6,000 hours. The results are further indication that metal attack is due primarily to fluoride impurities in the SrF_2 , and the impurities are consumed in a few thousand hours.

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STRONTIUM HEAT SOURCE DEVELOPMENT PROGRAM

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H. T. Fullam, Principal Investigator
D. G. Atteridge

At Hanford, strontium is separated from the high-level waste, converted to the fluoride, and doubly encapsulated in small, high-integrity containers for subsequent long-term storage. The fluoride conversion, encapsulation and storage take place in the Waste Encapsulation and Storage Facilities (WESF). The encapsulated strontium fluoride represents an economical source of ^{90}Sr if the WESF capsule can be licensed for heat source applications under anticipated use conditions. The objectives of this program are to obtain the data needed to license $^{90}\text{SrF}_2$ heat sources and specifically the WESF $^{90}\text{SrF}_2$ capsules. The information needed for licensing can be divided into three general task areas:

- Task 1 - Chemical and Physical Properties of $^{90}\text{SrF}_2$*
- Task 2 - $^{90}\text{SrF}_2$ Compatibility Studies*
- Task 3 - Capsule Qualification and Licensing*

Efforts are proceeding concurrently on all three tasks to obtain the required information.

TASK 1 - CHEMICAL AND PHYSICAL PROPERTIES OF $^{90}\text{SrF}_2$ (H. T. Fullam)

The Strontium-90 Fluoride Data Sheet (BNWL-2248) has been prepared and a draft copy sent to DNRA for review and comment. The data sheet is a compilation of available data and appropriate literature references on the properties of $^{90}\text{SrF}_2$ and nonradioactive SrF_2 . It represents an updating of one section of the Strontium-90 Data Sheets (ORNL-4358) prepared by S. J. Rimshaw and E. E. Ketchen of the Oak Ridge National Laboratory. As soon as DNRA comments are received, the report will be revised and issued.

TASK 2 - $^{90}\text{SrF}_2$ COMPATIBILITY STUDIES

Supplemental Screening Tests

A topical report (BNWL-2259) summarizing the results of the supplemental screening tests with nonradioactive SrF_2 has been written, and

the initial draft reviewed by DNRA. The final draft of the report, incorporating DNRA comments, has been completed and will be ready for printing in July.

Long-Term Compatibility Tests

It was previously reported that a number of $^{90}\text{SrF}_2$ test couples which were being held in an air atmosphere furnace at 800°C were accidentally destroyed. Both the test couples and their Inconel 600 protective jackets were badly corroded, although the furnace was operating correctly when the damage was discovered. Neither the furnace temperature controller, a separate overtemperature protection circuit, or the strip-chart recorder, used to continuously monitor the temperature, gave any indication that the furnace had overheated. Specimens from the failed Inconel 600 jackets were subjected to metallographic examination. The photomicrographs obtained indicated that the Inconel 600 was badly oxidized, and that it had been heated to a temperature in excess of $1,100^\circ\text{C}$. It has not been determined how the overheating could have occurred without the furnace control system giving an indication of a temperature excursion. To reduce the possibility of similar occurrences in the future, the separate overtemperature circuit of each furnace has been modified to improve its reliability and a second separate overtemperature protection circuit will be installed on each furnace as soon as the parts are received from the vendor.

All of the remaining compatibility tests are proceeding on schedule. No additional problems have been encountered with the furnaces since the overtemperature-protection circuits were modified.

All of the specimens from the 6,000 hour tests with WESF $^{90}\text{SrF}_2$ have been shipped to ORNL for evaluation. ORNL has completed testing of the tensile specimens, including controls, from the 6,000 hour tests as well as the metallographic examination of the TZM specimens. The results will be reported as soon as the photomicrographs and test data are received from ORNL. Work is continuing on the evaluation of Haynes Alloy 25 and Hastelloy C-276 metallographic specimens and the Charpy specimens from the 6,000 hour tests.

The 12,000 hour tests with nonradioactive SrF_2 were completed. Duplicate compatibility couples were tested at each temperature. The test couples were sectioned, and the metal specimens from the various couples subjected to metallographic examination. Photomicrographs were obtained of the areas showing maximum attack. Estimates of metal attack, based on evaluation of the micrographs, are presented in Table 1. The data from the 1,000 and 6,000 hour tests with nonradioactive SrF_2 are also provided for comparison purposes. Electron microprobe analysis of the test specimens is underway in order to identify the reaction mechanisms involved in the metal attack. Preliminary interpretations of the metal attack based on the micrographs are provided in the following sections.

Hastelloy C-276 specimens exposed to the nonradioactive SrF_2 for 12,000 hours suffered only limited chemical attack, but microstructural changes occurred to considerable depths especially at 600 and 1,000°C (see Figure 1). The chemical attack was approximately the same at all three temperatures, but the types of attack varied with the temperature. At 600°C the specimens exhibited a general surface reaction with formation of an adherent reaction layer. Grain boundary attack was limited to a few isolated areas. Microstructural change consisted of a broad surface layer containing extensive grain boundary precipitates. At 800°C the chemical attack consisted primarily of surface dissolution with some grain boundary attack and subsurface void formation. Microstructural changes were limited to a narrow surface zone in which normal alloy precipitates were partially depleted. At 1,000°C the chemical attack consisted of surface dissolution with limited grain boundary attack. What appears to be extensive subsurface voids are actually precipitates. The microstructural changes consisted of: (1) a narrow surface layer containing finely dispersed precipitates and large globular precipitates not found in the main body of the specimen and, (2) a broad inner zone in which normal alloy precipitates were partially depleted.

TABLE 1: Estimated Attack of Metal Specimens Exposed to Nonradioactive SrF_2 (a, b)

Material Tested	Temperature °C	Depth of Metal Affected, (c) mils					
		Chemical Attack			Change in Microstructure		
		1,000 Hr.	6,000 Hr.	12,000 Hr.	1,000 Hr.	6,000 Hr.	12,000 Hr.
Hastelloy C-276	600	<1	3	2	0	4	8
"	600	1	4	2	5	5	7
"	800	<1	2	2	<1	3	3
"	800	1	2	2	3	3	2
"	1,000	2	2	4	4	6	7
"	1,000	3	3	2	4	7	8
Haynes Alloy 25	600	<1	2	2	5	3	2
"	600	<1	1	2	3	2	<1
"	800	<1	3	3	3	6	4
"	800	<1	3	3	1	4	5
"	1,000	2	3	4	1	7	4
"	1,000	2	3	3	1	4	12
TZM	600	0	<1	0	0	0	0
"	600	<1	<1	(d)	0	0	(d)
"	800	<1	<1	<1	0	0	0
"	800	<1	<1	<1	0	0	0
"	1,000	<1	<1	<1	0	0	0
"	1,000	<1	<1	<1	0	0	0

(a) The SrF_2 contained impurities approximating those found in WESF produced $^{90}\text{SrF}_2$.

(b) Each test couple had a metal surface-to- SrF_2 volume ratio of 4.5 cm^{-1} .

(c) Estimated from photomicrographs.

(d) Test couple was accidentally destroyed during testing.

FIGURE 1: Hastelloy C-276 Specimens Exposed to Nonradioactive SrF_2 for 12,000 Hours

The Haynes Alloy 25 specimens exposed to nonradioactive SrF_2 for 12,000 hours suffered some chemical attack, and extensive microstructural changes especially at the higher temperatures (see Figure 2). The chemical attack increased with increasing exposure temperature, and the attack mechanism appeared to vary with temperature. At 600°C the specimens suffered a general surface attack with formation of an adherent reaction layer. Microstructural changes consisted of a narrow surface zone which was partially depleted of normal grain boundary precipitates. At 800°C the chemical attack appeared to consist primarily of the leaching of alloy components leaving free grains of unreacted metal in the reaction zone. The microstructural changes consisted of (1) a narrow surface layer depleted of normal alloy precipitates, and (2) a narrow inner zone containing what appear to be small voids but are actually small discrete globular precipitates. At 1,000°C the chemical attack consisted of general surface dissolution with some large isolated subsurface voids. Microstructure changes consisted of a broad surface layer in which normal intragranular precipitates were partly depleted, especially near grain boundaries, and which contained a few very large discrete globular precipitates not found in the unaffected areas of the specimens.

The TZM specimens exposed to nonradioactive SrF_2 for 12,000 hours suffered some slight chemical attack, but none of the specimens exhibited any microstructural changes due to SrF_2 attack (see Figure 3). The type of chemical attack appeared to be the same at all three temperatures and appeared to involve metal dissolution, primarily at the grain boundaries. The extent of chemical attack increased slightly with increasing exposure temperature.

Based on the data from the three sets of tests with nonradioactive SrF_2 (1,000, 6,000, and 12,000 hours) certain tentative conclusions can be reached regarding the containment of SrF_2 .

- Attack of test specimens exposed to SrF_2 proceeds relatively rapidly during the first few thousand hours of exposure and then remains relatively constant thereafter. Overall, the

FIGURE 2: Haynes Alloy 25 Specimens Exposed to Nonradioactive SrF_2 for 12,000 Hours

FIGURE 3: TZM Specimens Exposed to Nonradioactive SrF_2 for 12,000 Hours

test specimens exposed to nonradioactive SrF_2 for 12,000 hours suffered no more attack than the specimens exposed for 6,000 hours. Figures 4, 5, and 6 show photomicrographs of Hastelloy C-276, Haynes Alloy 25, and TZM specimens exposed to SrF_2 at 800°C for three time periods, and it is apparent from the micrographs that increasing the exposure time above 6,000 hours has not increased the extent of attack significantly. Similar results were observed with the specimens tested at 600 and $1,000^\circ\text{C}$. Indications are that the metal attack is due primarily to fluoride impurities in the SrF_2 , and the impurities are consumed in a relatively short period of time. Electron microprobe data indicate the fluorides of iron and chromium are the principal cause of metal attack. It appears that at $600\text{--}1,000^\circ\text{C}$ diffusion of harmful impurities in the SrF_2 matrix is sufficiently rapid to permit their migration to the metal surface, and subsequent reaction with the metal, within a period of a few thousand hours.

- Hastelloy C-276 approaches Haynes Alloy 25 in its resistance to long-term attack by nonradioactive SrF_2 . After 12,000 hours exposure to SrF_2 , chemical attack of the Hastelloy C-276 specimens appears to be no greater than the chemical attack of the Haynes Alloy 25 specimens. This is the first indication that the Hastelloy C-276 may approach Haynes Alloy 25 in its resistance to SrF_2 attack. In all of the previous compatibility studies with the two alloys and SrF_2 , which only lasted up to 4,400 hours, the Haynes Alloy 25 was far more resistant to attack than the Hastelloy C-276.
- The TZM continues to provide the greatest resistance to fluoride attack of the three materials tested. The TZM specimens tested for 12,000 hours suffered no more attack than those tested for 1,000 hours.

FIGURE 4: Hastelloy C-276 Specimens Exposed to Nonradioactive SrF_2 at 800°C

FIGURE 5: Haynes Alloy 25 specimens Exposed to Nonradioactive SrF_2 at 800°C

FIGURE 6: TZM Specimens Exposed to Nonradioactive SrF_2 at 800°C

It must be remembered that the aforementioned conclusions are tentative and must be confirmed by additional compatibility data. Tests lasting up to 30,000 hours with both nonradioactive SrF_2 and $^{90}\text{SrF}_2$ are now underway and, hopefully, will provide the confirmatory data.

WESF $^{90}\text{SrF}_2$ Capsule Demonstration Tests

The test specimens from the full-size WESF $^{90}\text{SrF}_2$ capsule that was held at 800°C for 5,000 hours have been shipped to ORNL for evaluation. Testing of the second WESF $^{90}\text{SrF}_2$ capsule held at 800°C for one year has been completed. Efforts are now underway to section the capsule. ARHCO has encountered equipment problems, however, and it is not known how soon they will be able to complete the sectioning of the capsule.

TASK 3 - CAPSULE QUALIFICATION AND LICENSING (D. G. Atteridge)

Heat Source Design Criteria and Qualification Requirements

A letter report summarizing PNL recommendations on the $^{90}\text{SrF}_2$ heat source design criteria and qualification test requirements was submitted to DNRA for review. At the request of the sponsor, some revisions were made in the report, and the revised version will be submitted to DNRA in July.

The report recommends that the present WESF inner capsule be retained as the fuel cladding and primary fuel containment barrier of the $^{90}\text{SrF}_2$ heat source. It also recommends that the present WESF outer container be replaced with a new PNL designed outer container (strength member). The new outer container would be required to meet all of the qualification test requirements recommended in the report after thermal aging as well as in the as-fabricated condition.

Capsule Design

No concentrated effort will be made in the capsule design area until the PNL recommendations on the capsule design criteria and qualification test requirements have been approved by DNRA.

Outer Capsule Material Selection

D. G. Atteridge attended a meeting of the Working Group on Superalloys for Terrestrial Isotope Applications at Germantown, Maryland. The consensus of the meeting seemed to be that, based on thermal aging considerations, the new superalloys, Hastelloy S and Inconel 617, would be the best superalloys for general radioisotope container use, with Inconel 625 as a relatively "old" back-up alloy. The recommendations of this committee and the previous work done in this task seem to narrow the $^{90}\text{SrF}_2$ canister outer material selection choices down to the two new alloys, Hastelloy S and Inconel 617, and two relatively proven alloys, Hastelloy C-4 and Inconel 625. An in-depth comparison of such properties of these alloys as cost, availability, high temperature properties, effects of thermal aging, etc., is currently in progress to allow selection of the outer container material by the end of the fiscal year. Final selection of the outer capsule material will depend, however, on DNRA approval of the recommended capsule design criteria and qualification test requirements.

Work has started on the high temperature tensile testing of thermally aged specimens of Hastelloy Alloy C-4 and Hastelloy Alloy S. The test specimens were prepared from material obtained from Cabot Corporation which had been aged for up to 16,000 hours at temperatures between 800 and 1,600°F. The specimens are being tested at 800°C and at their respective aging temperatures. The tests will be completed by the end of the fiscal year.

Effects of Thermal Aging on Alloy Properties (H. T. Fullam)

Studies are underway to determine the effects of thermal aging on the oxidation and seawater corrosion of Hastelloy C-4 and Hastelloy S. When the solution heat-treated forms of the two alloys are held at temperatures between approximately 500 and 900°C, they undergo long-range ordering reactions and precipitate formation which affect their mechanical properties. These reactions proceed relatively slowly and can occur over a span of many thousands of hours when the alloys are heated. The two alloys are being considered for use as the outer capsule

of the $^{90}\text{SrF}_2$ heat source which must be resistant to oxidation and seawater corrosion. The solution heat-treated forms of the two alloys provide sufficient corrosion resistance; but the effects of microstructural changes, due to thermal aging, on the corrosion resistance of the alloys must be determined.

Samples of Hastelloy C-4 and Hastelloy S are being aged at temperatures of 600, 700, 800, and 900°C for times up to 10,000 hours. The oxidation resistance and seawater corrosion resistance of the aged materials are determined using procedures described in earlier reports.

Seawater Corrosion

The corrosion resistance of Hastelloy C-4 and Hastelloy S specimens, which had been thermally aged for 5,000 hours at 600-900°C, was measured in natural seawater at $25 \pm 1^\circ\text{C}$. The laboratory-scale tests lasted for 1,000 hours. Visual and microscopic examination of the test specimens gave no indication of attack by the seawater. The maximum weight change observed with any specimen, after 1,000 hours exposure, was $+0.02 \text{ mg/cm}^2$. The results obtained confirm those previously obtained with the solution heat-treated alloys and specimens thermally aged for 1,000 hours. The results show that the two alloys are quite resistant to seawater corrosion, and microstructural changes due to thermal aging do not decrease the resistance to seawater attack.

Oxidation

The oxidation resistance of Hastelloy C-4 and Hastelloy S specimens, which had been thermally aged for 5,000 hours at 600-900°C, was measured over a range of 700-1,100°C. The tests lasted for 1,032 hours. At oxidation temperatures of 900°C or less, all specimens exhibited a continuous weight increase with formation of an adherent oxide scale. At 1,000°C and higher the specimens exhibited an initial weight increase followed by a continuous weight loss due to spalling of the oxide scale. Based on the limited data available, thermal aging of the two alloys appears to have minimal effect on their rates of oxidation, although it does appear to lower the temperature at which scaling of the oxide scale occurs

(from 1,100°C to 1,000°C). Slight increases in oxidation rates at 700-900°C were observed with the thermally aged specimens, as compared to the solution heat treated alloys, but the increases were less than the estimated precision of the experimental procedure used.

It was previously reported that thermal aging of Hastelloy C-4 and Hastelloy S at various temperatures for 1,000 hours produced marked differences in their oxidation rates at 1,100°C. The rates varied with the aging temperature and were significantly different from those of the solution heat-treated alloys. These differences were not observed with specimens aged for 5,000 hours. The oxidation rates at 1,100°C of the specimens aged for 5,000 hours agreed, within experimental error, with the oxidation rates obtained with the solution heat-treated alloys. The oxidation tests with the specimens aged for 1,000 hours are being repeated to see if the original results can be confirmed.

A detailed evaluation of the effects of thermal aging on the oxidation resistance of Hastelloy C-4 and Hastelloy S will be provided when the tests with specimens aged for 10,000 hours are completed.

BENEFICIAL ISOTOPES UTILIZATION PROGRAM

H. H. Van Tuyl, Program Manager

The objectives of the program are to identify and develop beneficial uses of nuclear reactor by-products through: (1) estimation of long-term availability and cost of useful isotopes from commercial suppliers; (2) identification and development of beneficial applications of isotopes, including their use in remote regions of the world; (3) identification and evaluation of the actions required to optimize the $^{90}\text{SrF}_2$ and $^{137}\text{CsCl}$ products from the Hanford Waste Encapsulation and Storage Facility (WESF) for beneficial use; and (4) review and evaluation of the use of radioisotopes for fueling U. S. Army thermal systems.

The program is divided into four tasks:

<i>Task I</i>	<i>-</i>	<i>Isotopes Availability</i>
<i>Task II</i>	<i>-</i>	<i>Cold Regions Applications</i>
<i>Task III</i>	<i>-</i>	<i>WESF Product Utilization</i>
<i>Task IV</i>	<i>-</i>	<i>General Application of Radioisotopes
in Army Thermal Systems</i>

TECHNICAL PROGRESS

TASK 1 - ISOTOPE AVAILABILITY

Flowsheet Calculations (D. K. Davis)

A flowsheet for the recovery of several beneficial isotopes (cesium, strontium, neptunium, promethium, curium, and americium) has been developed and commented on internally. The comments are being used to develop a revised flowsheet. New information on the various ion exchange processes in the flowsheet is being used in making the revisions. The final flowsheet will use tributyl phosphate (TBP) in all proposed solvent extraction operations. Fractional extraction, a solvent extraction technique, will be used in making a preliminary separation between the rare earths and actinides. This technique will allow the use of TBP, which will simplify the fuel reprocessing plant waste treatment operations. Rare earth-actinide separations are normally accomplished by the use of chelating compounds, which complicate overall plant waste management operations.

WESF Strontium and Cesium Availability (W. E. Sande)

The availability of strontium and cesium from the Hanford Waste Encapsulation and Storage Facility (WESF) has been updated with assistance by W. C. Johnson, ERDA-RL, and by staff from United Nuclear Industries, Inc., which operates N-Reactor for ERDA. The update, as of June 1, 1977, is presented in Table 2.

TABLE 2. Projection of Strontium and Cesium
Availability from WESF

	<u>Strontium-90</u>	<u>Cesium-137</u>
Current WESF campaign	69 MCi	145 MCi
Addition from Purex FY 1978 to 1983	21 MCi	24 MCi
Each additional N-Reactor year of operation - beyond FY 1983	3.5 MCi	4.0 MCi

TASK II - COLD REGIONS APPLICATIONS

Isotope Heaters (W. E. Sande)

The cost analysis sections of the draft report on Cold Regions heaters fueled with WESF SrF_2 capsules have been revised. The results are summarized in Table 3 for a 1 kW_t and 12 kW_t heater; no development costs are included.

TABLE 3. Annual Isotope Heater System Costs

	<u>Cost, in Equivalent \$/gal of Fuel Oil^(a)</u>	
	For a 1 kW Heater:	For a 12 kW Heater:
Transport	\$ 1.35	\$ 1.30-2.50
Capital	3.50	1.20-1.60
Isotopes	4.10	9.20
Fuel Oil Capital	-1.35	-0.50
Fuel Oil Operating	<u>-1.70</u>	<u>-2.20</u>
Total	\$ 5.90	\$ 9.00-10.60

TASK III - WESF PRODUCT UTILIZATION

Sandia Support - Study of Sludge Handling Alternatives (H. E. McGuire, Jr. and G. W. Dawson)

PNL has prepared the following informal reports:

- Technical Descriptions of Municipal Sludge Treatment Alternatives
- Description of Sewage Sludge Treatment Schemes for Representative U. S. Studies
- Estimated Capital, Operating and Maintenance Costs for Various Sludge Treatment Processes

We are currently preparing updated detailed designs and cost estimates for pasteurization and irradiation.

The project team will be visiting several representative U. S. cities to obtain a cross section on typical sludge handling processes and to assess the feasibility of sludge irradiation. Cities to be visited include:

Denver, Colorado
 Nashville, Tennessee
 Washington, D. C.
 Boston, Massachusetts
 Milwaukee, Wisconsin
 Los Angeles County, California
 Orange County, California

The site visits should be concluded in late July.

TASK IV - GENERAL APPLICATION OF RADIOISOTOPES IN U. S. ARMY THERMAL SYSTEMS
(W. E. Sande)

The final draft, entitled Cost Analysis for Application of Solidified Waste Fission Product Canisters in U. S. Army Steam Plants, was edited based on internal comments and U. S. Army/ERDA-NRA comments, and has been forwarded for final review to the latter two groups. The final document is expected to be issued in August.

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