**Pacific Northwest Laboratory
Quarterly Report to USERDA
Advanced Nuclear Energy Systems,
Space and Special Purposes Division
for July-September 1975**

October 1975

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PACIFIC NORTHWEST LABORATORY QUARTERLY REPORT
TO USERDA ADVANCED NUCLEAR ENERGY SYSTEMS,
SPACE AND SPECIAL PURPOSES DIVISION
FOR JULY-SEPTEMBER 1975

By
H. T. Fullam and K. M. Harmon

October 1975

Battelle
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Richland, Washington 99352

EXECUTIVE SUMMARY

STRONTIUM HEAT SOURCE DEVELOPMENT PROGRAM

The design of the long-term $^{90}\text{SrF}_2$ compatibility tests has been finalized. The tests will be carried out at 600, 800 and 1000°C for periods up to 30,000 hr. TZM, Haynes Alloy 25 and Hastelloy C-276 will be evaluated in the tests. Tensile and Charpy specimens will be tested to determine the effect of $^{90}\text{SrF}_2$ attack on the mechanical properties of the alloys.

The effect of thermal aging on the impact strength of Hastelloy C-4 is being evaluated using Charpy test specimens. The tests are being run at 600, 800, 900 and 1000°C. After 1000 hr only the specimens aged at 600°C show a measurable decrease in impact strength.

The solubility of SrF_2 in seawater and demineralized water was determined at 23°C. The equilibrium concentration of Sr in demineralized water is greatly influenced by the presence of fluoride impurities in the SrF_2 , whereas in seawater the presence of fluoride impurities has little effect on the Sr concentration.

The effect of impurity fluorides and decay product (ZrF_4) on the melting point of SrF_2 was determined using differential thermal analysis. The lowest melting point observed in the system containing SrF_2 , ZrF_4 and impurity fluorides found in WESF $^{90}\text{SrF}_2$ was $851 \pm 5^\circ\text{C}$.

BENEFICIAL ISOTOPES UTILIZATION PROGRAM

Task I. A report was issued tabulating the probable availability of 20 potentially useful isotopes through the year 2000.

Task II. A report was drafted summarizing the previous year's activities in investigating potential applications of isotopes in the Cold Regions. Heat requirements to keep Cold Regions sewage treatment facilities (septic tanks and sewage lagoons) from freezing were estimated, and it was concluded that the heat from one WESF $^{90}\text{SrF}_2$ capsule would be adequate for a 10,000-gallon septic tank.

Task III. Theoretical work continued on the effect of source geometry on radiation efficiency of $^{137}\text{CsCl}$ capsules. ARHCO evaluated the effect on WESF of converting to a 1-in. diameter capsule, and it was concluded that such a change should not be considered further. Reduction in capsule wall thickness is under review.

Further studies were made of design concepts for using WESF $^{90}\text{SrF}_2$ capsules to proven dynamic electric generators.

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STRONTIUM HEAT SOURCE DEVELOPMENT PROGRAM

H. T. Fullam

At Hanford, strontium will be 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 will 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 areas:

- 1. Long-term SrF_2 compatibility data.*
- 2. Chemical and physical property data on $^{90}\text{SrF}_2$.*
- 3. Capsule property data such as external corrosion resistance, crush strength, etc.*

The current program is designed to provide the required information.

LONG-TERM COMPATIBILITY TESTS

Two meetings were held with the program sponsor at ERDA headquarters to discuss the long-term compatibility tests. At the meetings the design of the long-term tests was finalized. The test conditions selected and materials to be evaluated are summarized in Table 1. The various test couples will be fabricated using procedures similar to those used to encapsulate $^{90}\text{SrF}_2$ at WESF. The effect of fluoride attack on the mechanical properties of the containment materials will be evaluated by testing

TABLE 1. Conditions Selected for the Long-Term
Compatibility Tests

A. Fuel Compositions Tested

1. Fuel-grade $^{90}\text{SrF}_2$ produced at WESF (50 to 55% ^{90}Sr Isotopic content).
2. Nonradioactive SrF_2 simulating WESF $^{90}\text{SrF}_2$ in chemical composition.

B. Containment Materials Evaluated

1. TZM
2. Haynes Alloy 25
3. Hastelloy C-276

C. Test Temperatures

1. 600°C
2. 800°C
3. 1000°C

D. Test Duration

1. 1,500 hr
2. 7,500 hr
3. 15,000 hr
4. 22,500 hr
5. 30,000 hr

E. Metal Surface to Fuel Volume Ratios (S/V) Tested

1. $S/V = 4.5 \text{ cm}^{-1}$
2. $S/V = 2.5 \text{ cm}^{-1}$
3. $S/V = 0.8 \text{ cm}^{-1}$ (equivalent to WESF $^{90}\text{SrF}_2$ capsule)

F. Test Specimen Design

1. Metallographic
2. Tensile
3. Charpy V-Notch

Total Number of Couples Required

Radioactive = 220

Nonradioactive = 126

Control Specimens = 118

tensile and Charpy specimens with the WESF $^{90}\text{SrF}_2$. A critical variable to be considered in compatibility testing is the metal surface to fuel volume ratio (S/V) of the test couples. The S/V ratio is especially important when impurities in the fuel are involved in the fuel-metal reactions. In the long-term compatibility tests three different S/V ratios will be evaluated with the lowest ratio ($\text{S/V} = 0.8 \text{ cm}^{-1}$) being equivalent to that found in the WESF capsule.

Because of the many variables to be considered and the range of conditions to be studied, it is impossible to test couples for each possible set of conditions. A complete matrix would require several hundred $^{90}\text{SrF}_2$ couples and an equivalent number of nonradioactive SrF_2 couples, and is beyond the scope of the program. It is planned that 220 $^{90}\text{SrF}_2$ couples, 126 nonradioactive couples, and 118 control specimens will be tested to span the range of conditions listed in Table 1.

Preparation of the test couples will start as soon as fuel-grade $^{90}\text{SrF}_2$ is available from WESF. Current indications are that the $^{90}\text{SrF}_2$ will be available in late November or December. A cask suitable for transferring the $^{90}\text{SrF}_2$ from WESF to PNL has been ordered and should be available in October. Approximately 7 kg of fuel-grade $^{90}\text{SrF}_2$ ($\sim 350,000 \text{ Ci}$ of ^{90}Sr) will be transferred to PNL for the compatibility tests. The $^{90}\text{SrF}_2$ will be loose-packed in the standard WESF capsule for the transfer. Each capsule will contain about 1 kg of fluoride, requiring a total of seven capsules for the shipment. Preparation of the operational safety analysis reviews (SAR) required for the transfer is now underway.

In addition to the long-term compatibility tests, it has been decided to initiate additional scouting tests to evaluate potential container materials not covered in the initial short-term scouting tests. The new tests will be carried out at 800°C for 1500 and 4400 hr using nonradioactive SrF_2 . The containment materials to be tested have not been completely identified at the time, but will include various precious metal and refractory metal alloys, Hastelloy C-4, as well as Ni- and Co-base alloys not previously tested.

It is also planned that two full size WESF capsules containing fuel-grade $^{90}\text{SrF}_2$ will be tested at 800°C for 6 and 12 months. After testing, the capsules will be sectioned and specimens from various locations in the inner capsule will be analyzed to determine the extent of fluoride attack.

The initial draft of the topical report summarizing the results of the short-term compatibility tests has been reviewed and approved by DANES. Preparation of final draft is now underway.

An abstract for a paper entitled "Containment of $^{90}\text{SrF}_2$ " by H. T. Fullam has been submitted for presentation at a symposium on "Materials for Heat Sources of Radioisotopic Power Systems" to be held at the 1976 Annual Meeting of the AIME.

THERMAL AGING OF HASTELLOY C-4

When considering the use of Ni- and Co-base alloys as a containment material for $^{90}\text{SrF}_2$, the problem of thermal aging of the alloys must be taken into account. For example, Haynes Alloy 25 provides good resistance to attack by WESF-grade $^{90}\text{SrF}_2$ at temperatures of 800 to 1100°C. However, the Haynes 25 undergoes a severe loss of impact strength, toughness and ductility when thermally aged at 650 to 900°C, reducing its usefulness as a containment material for $^{90}\text{SrF}_2$. Most other Ni- and Co-base alloys undergo aging reactions in the same general temperature range that affects their mechanical properties. Hastelloy C-4 is reported to suffer less from deleterious thermal aging reactions than most of the Ni- and Co-base alloys. Therefore, a series of thermal aging tests on Hastelloy C-4 has been initiated to see if the alloy can be considered for use as the strength member of a double-walled container for ^{90}Sr in the range of 600 to 900°C. Charpy V-notch specimens are being aged at 600, 800, 900 and 1000°C for periods of 1000, 5000, 10,000 and 30,000 hr. The test specimens were fabricated from 1/2 in. Hastelloy C-4 plates which had been solution heat treated at 1950°F and rapid quenched. The specimen size corresponded to the ASTM 10 mm square design (E-23).

The 1000-hr tests have been completed and the results are summarized in Table 2. A Baldwin Charpy impact tester with a rated capacity of 240 ft-lb was used to evaluate the test specimens at room temperature. Three specimens were tested at each temperature, as well as three control specimens. The specimens aged at 800, 900 and 1000°C, as well as the control specimens, failed to break in two upon testing. Only at 600°C was there a measurable effect on the impact strength of the alloy, and a room temperature value of 103 ± 4 ft-lb was obtained.

TABLE 2. Charpy Impact Data for Hastelloy C-4 Specimens
Thermally Aged for 1000 hr

Aging Temperature °C	Room Temperature Charpy Impact Strength, ft-lb
"As Received"	240 ^(b)
600 ^(a)	103 ± 4
800	240 ^(b)
900	240 ^(b)
1000	240 ^(b)

(a) Three specimens tested at each temperature.

(b) Test specimens did not break in two.

SOLUBILITY OF STRONTIUM FLUORIDE

The solubility of strontium fluoride in seawater and demineralized water at 23°C was measured. Three grades of strontium fluoride containing different levels of fluoride impurities were used in the tests:

- high-purity SrF_2 containing less than 1000 ppm total impurities with sodium (300 ppm) and calcium (220 ppm) the major impurities.
- commercial-grade SrF_2 containing about 5000 ppm impurities with Na, Ca, Ba and $\text{SO}_4^{=}$ the principal impurities.
- WESF-grade SrF_2 containing ~4.5 wt% fluoride impurities and corresponding in composition to WESF-produced $^{90}\text{SrF}_2$.

In the tests the different grades of SrF_2 were contacted with water, with stirring, for periods up to 6 months at 23°C. The mixtures were sampled periodically, filtered, and the clear solution analyzed for dissolved strontium and fluorine. The Sr and F concentrations reach equilibrium levels (within experimental error) after approximately 30 days exposure. The equilibrium concentrations for the three grades of SrF_2 in the demineralized water and seawater are given in Table 3. The presence of fluoride impurities in the SrF_2 has a marked influence on the equilibrium Sr concentration in demineralized water. In the seawater, however, the Sr concentration appears to be unaffected by the presence of fluoride impurities in the SrF_2 .

TABLE 3. Solubility of Strontium Fluoride at 23°C in Seawater and Demineralized Water

Grade of SrF_2 Used	Concentration, M ^(a)			
	Demineralized Water		Seawater	
	Sr	F	Sr	F
High-Purity SrF_2	0.0010 ±0.00013	0.0019 ±0.00021	0.0011 ±0.00003	0.0026 ±0.00015
Commercial SrF_2	0.0006 ±0.000061	0.0045 ±0.00024	0.0013 ±0.00022	0.0032 ±0.00020
WESF-Grade SrF_2	0.000093 ±0.000017	0.011 ±0.00042	0.0012 ±0.00008	0.0024 ±0.00031

(a) Each value represents an average of at least four analyses obtained over a 4-month period.

EFFECT OF IMPURITIES ON THE MELTING POINT OF SrF_2

The melting point of the fuel is an important consideration in the design of any operating heat source. Pure SrF_2 melts at a temperature in excess of 1400°C. Values ranging from 1400 to 1470°C have been reported by various investigators. Measurements at PNL on high-purity SrF_2 (<1000 ppm total impurities) using differential thermal analysis indicated

a melting point of $1425 \pm 6^\circ\text{C}$. The presence of decay product (ZrF_4) or impurity fluorides, as found in WESF-produced $^{90}\text{SrF}_2$, can result in phases with significantly lower melting points than the pure SrF_2 . For example, the SrF_2 - NaF system exhibits a minimum melting point of 856°C . The effects of ZrF_4 and impurity fluoride additions on the melting point of SrF_2 was investigated using differential thermal analysis. The minimum melting point observed in systems corresponding to WESF $^{90}\text{SrF}_2$ with varying levels of ZrF_4 (up to the equivalent of 2 half-lives) was $851 \pm 5^\circ\text{C}$.

BENEFICIAL ISOTOPES UTILIZATION PROGRAM

K. M. Harmon

The objectives of the program for FY-1976 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 and 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.*

The program is divided into three tasks, as follows:

- Task I - Isotopes Availability*
- Task II - Cold Regions Applications*
- Task III - WESF Product Utilization*

TECHNICAL PROGRESS

Task I - Isotopes Availability

The objectives are to 1) provide an estimate of the quantities of useful isotopes which could be available from commercial nuclear power reactor operations through the year 2000, 2) define chemical flowsheets for the recovery of these isotopes, and 3) estimate capital and operating costs for an isotope recovery plant.

Calculation of Potential Supplies (C. M. Heeb)

A topical report, The Availability of Useful Isotopes from Civilian Nuclear Power Reactors to the Year 2000, by C. M. Heeb (BNWL-B-435) was issued in July. The report presents a number of tables showing the most probable gram weight availability of ^{85}Kr , ^{90}Sr , ^{99}Tc , ^{106}Ru , ^{103}Rh , ^{137}Cs , ^{147}Pm , ^{237}Np , ^{238}Pu , ^{241}Am , ^{242}Cm , ^{214}Cm , and all the isotopes of xenon and palladium, by year from 1972 to 2000.

Task II - Cold Regions Applications

The objective is to identify and scope critical needs for heat and power in the Cold Regions and determine if radioactive isotopes offer unique means of meeting these needs.

Potential Cold Regions Applications (L. D. Perrigo)

A report was prepared, summarizing the previous year's work in meeting with representatives of Federal and Alaska State Agencies and identifying opportunities for the use of isotopes in meeting unique needs for heat and power in Alaska.

Thermal Conditioning with Strontium Fluoride Capsules (W. E. Sande)

The feasibility of using WESF $^{90}\text{SrF}_2$ capsules as heat sources for Cold Regions' waste treatment systems was investigated. Three systems received consideration: an underground septic tank, a sewage lagoon, and an above-ground septic tank. The sizes were based on the needs of a community of 65 people, such as the village of Hughes, Alaska.

It was concluded that a 10,000-gallon septic tank, either buried or above ground, would be adequate for a 65-resident village and could be kept from freezing by the heat from one WESF $^{90}\text{SrF}_2$ capsule (1 kWt). Requirements for tank design, depth of cover soil if buried, and thickness of insulation if above ground would depend on such environmental factors as soil thermal conductivity and average air temperature. Since the response time of internal tank temperatures to changes in air temperature is long, the septic system will not require tank temperature control for daily, weekly, or monthly air temperature variations. Therefore, it should be possible to design a heat transfer system based on the yearly average temperature. A natural thermal convection loop appears to be a viable concept for a heat transfer system. The capsule could be placed either inside or outside the septic tank.

The study showed that keeping an uncovered 0.65-acre sewage lagoon completely unfrozen is not practical, requiring from 350 to 1700 kWt at an air temperature of -20°F with winds between 0 and 20 mph. However, a cover providing a foot of stagnant air could reduce the heat requirement to 17 kW. Other alternative concepts which may aid sewage treatment are 1) natural convection aerators/mixers, 2) lagoons in series (one heated and several for settling/storage), and 3) cesium chloride capsules providing sludge sterilization by radiation in conjunction with heat from strontium fluoride capsules.

Since the use of strontium fluoride capsules appears to be technically feasible, work in a cost/benefit comparison with other fuel systems has been started. A brief analysis of using a system which would burn the off-gas from anaerobic decomposition of sludge indicates that the sludge from 65 people could yield 0.5 kWt of usable heat (possible 1 kW if community garbage were also fed to the system). This would require a sludge retention time of 30 days, hence would necessitate a method of sludge separation. A candidate method would be an Imhoff tank, but efficient operation would likely require relatively close maintenance. The use of a fuel oil system is also being considered. Examples of questions which must be answered are: 1) If the community already has a 100 kW diesel generator, what is the incremental cost of 282 gal/yr of oil (1 kW) for a furnace or approximately 940 gal/yr for 1 kW electrical? 2) Could a septic system use the waste heat from the generator? 3) If each home is eventually going to be supplied with hot water, would it be practical to insulate the septic system very well and discharge slightly hotter water than normal?

Task III - WESF Product Utilization

The objective is to foster the beneficial use of WESF $^{137}\text{CsCl}$ and $^{90}\text{SrF}_2$ by investigation of concepts for potential applications and by coordination of efforts to optimize capsule design.

Optimization of the $^{137}\text{CsCl}$ Capsule for Use as an Irradiation Source

Radiation Efficiency (R. A. Libby). PNL calculations of radiation efficiency as a function of $^{137}\text{CsCl}$ capsule design were in agreement with

similar calculations made by Marvin Morris of Sandia Laboratories. Expressing the results in terms of the gamma rays reaching the outside of the capsule without collision (as a percentage of the total gammas generated), the PNL results are as follows:

<u>Capsule Parameters (Solid Cylinders, SS Cladding)</u>	<u>Radiation Efficiency</u>
<u>Current WESF Design</u>	30%
Capsule I.D. = 2 in. Total Cladding Thickness = 0.2 in.	
<u>"Optimum" Design</u>	50%
Capsule I.D. = 1 in. Total Cladding Thickness = 0.1 in.	
<u>Intermediate Design</u>	39%
Capsule I.D. = 2 in. Total Cladding Thickness = 0.1 in.	

Impact of Design Changes on WESF Cost and Schedule (L. I. Brecke, ARHCO). ARHCO was asked to evaluate the impact on WESF costs and schedules of converting to a 1-in. diameter $^{137}\text{CsCl}$ capsule. Their response may be summarized as follows:

- Fabrication of 1-in. capsules appears technically feasible. Incremental costs for converting WESF to a new design are estimated at \$250,000.
- WESF design capacity for CsCl encapsulation is 500 capsules per year. It would not change significantly with the production of smaller capsules. Since a 1-in. capsule will hold only one-fourth as much CsCl as the standard 2-in. capsule, the operating cost per curie would increase by a factor of nearly 4 with conversion to the 1-in. design.
- Fabrication of 1-in. and 2-in. capsules would have to be done in successive campaigns, not concurrently. Conversion from one size to the other would require 1 to 2 months, with resultant loss in production.

Recommendations

PNL and Sandia are joining in the recommendation to ERDA/DANES that no further consideration be given to the 1-in. diameter $^{137}\text{CsCl}$ capsule concept for WESF production. We are recommending, further, that the conversion to a thin-wall (0.1 total cladding thickness) 2-in. capsule be evaluated. The evaluation needs to consider safety and licensing requirements and possible WESF operating problems. PNL will submit a proposal for adding to the scope of the Isotopes Utilization Program to include this study if DANES so directs.

Utilization of WESF $^{90}\text{SrF}_2$ Capsules

Concept Development: $^{90}\text{SrF}_2$ -Powered Generating Systems (D. H. Lester)

This work is a continuation of earlier studies undertaken to determine if established coolant conditions (temperature and flow rate) for organic Rankine and Stirling engines were compatible with WESF capsule dimensions and with an 800°C (max) constraint on the SrF_2 -liner interface, imposed as a result of preliminary SrF_2 compatibility studies. During this quarter, further evaluation of the capsule for use in heat engine powered generating systems was carried out. The design concept for study has continued to be the multiple capsule heat block.

Heat transfer studies early in the quarter were directed toward the use of emissivity enhancement as a means of reducing temperature gradients from the working fluid to the fuel-metal interface. No credit was taken for conductive mechanisms in the gas gaps since an air atmosphere was assumed. The studies showed that if the emissivity of all gap surfaces can be increased to 0.9, the heat block arrangement shown in Figures 1 and 2 will provide working fluid temperatures adequate for Stirling engine use without exceeding the 800°C inner liner temperature limit.

Further modifications of the computer model were made to determine benefits and problems associated with other modifications such as: 1) filling all gaps with helium, 2) filling the gaps with powdered metals,

3) adding a third can to satisfy strength requirements and 4) increasing the thickness of the existing outer can. Addition of helium to the gaps produced temperature profiles which allowed decrease in the size of the unit cell per capsule and fewer coolant channels/capsule. The unit cell per capsule for this concept is shown in Figure 3 and the overall heat block in Figure 4. The heat block shown would have 2.6 kWe output assuming 20% overall efficiency. The output would decay to 2 kWe in 11 years. All of the conditions for this part of the study correspond to those specified for a Phillips Stirling Engine.⁽¹⁾

Calculations of interface temperature versus emissivity were made for two alternate capsule configurations: the standard two-jacket capsule with the outer jacket thickened to 0.6 in.;⁽²⁾ and the standard two-jacket capsule enclosed in a third liner, 0.6 in. thick. The results, summarized in Figures 5 and 6, show that: 1) with helium in the gaps, the two-jacket capsule with the thickened outer wall is usable without enhancement of emissivity and 2) if the thick can is added as a third can, enhancement of emissivities to at least 0.75 is essential. In the two-can concept, a full range of powdered nickel particle sizes serves as a good substitute for emissivity enhancement, but the powdered metal would have to be also placed in gaps between the capsules and the block. In the three-can concept, the powdered nickel must have helium in its void spaces.

The results to date indicate that use of powdered metal or addition of a third can is feasible but the incremental benefits are dubious.

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1. H. A. Jaspers and F. K. duPres, Paper No. 739035, S.A.E. Conf., Chicago, June 1973, p. 588, 1973.
2. Recommended by Teledyne Isotopes, personal communication.

FIGURE 1. Unit Cell Array of Coolant Channels for Helium Stirling Engine

**WESF CAPSULE
HEAT BLOCK FOR HELIUM STIRLING ENGINE**

13 CAPSULES (13 kW THERMAL)
EFFECTIVE CHANNELS PER CAPSULE = 21
EMISSIONS OF CAPSULE SURFACES = 0.9
WEIGHT = 24,000 LBS
DOSE RATE = 200 mR/hr
AT 1 METER

TEMPERATURES (°C)

COOLANT = 650
OUTER CAPSULE SURFACE = 730
FUEL-CAPSULE INTERFACE = 790
CAPSULE CENTER LINE = 1220

FIGURE 2. WESF Capsule Heat Block for Helium Stirling Engine

FULL SIZE SKETCH OF HEAT BLOCK UNIT CELL

FIGURE 3. Full Size Sketch of Heat Block Unit Cell (Top View)

13 CAPSULE HEAT BLOCK SHOWING
ARRANGEMENT OF HEXAGONAL UNIT CELLS

FIGURE 4. 13 Capsule Heat Block Showing Arrangement of Hexagonal Unit Cells

FIGURE 5. WESF SrF_2 Capsule with Overpack 0.6 in. Thick

FIGURE 6. WESF SrF_2 Capsule with Third 0.6 in. Can

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