PACIFIC NORTHWEST LABORATORY
QUARTERLY REPORT ON
SPACE NUCLEAR SYSTEMS PROGRAM
FOR PERIOD ENDING JUNE 30, 1974

Battelle

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STRONTIUM FLUORIDE COMPATIBILITY PROGRAM
QUARTERLY REPORT FOR PERIOD ENDING JUNE 30, 1974

by

H. T. Fullam

November 1974

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PACIFIC NORTHWEST LABORATORIES
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STRONTIUM FLUORIDE COMPATIBILITY PROGRAM
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H. T. Fullam

SUMMARY

The strontium fluoride compatibility program was reinstated in March 1974 under the technical direction of the Division of Space Nuclear Systems. Work is underway to complete the four series of short-term compatibility tests (up to 6 months duration) which were stopped when the program was terminated at the end of FY73. In the tests nine containment materials are being evaluated with four different grades of SrF_2 at 800, 1000 and 1100°C. The first series of tests with high purity nonradioactive SrF_2 has been completed and couple interaction evaluated. The second series of tests with high purity $^{90}\text{SrF}_2$ has been completed and the couples are being evaluated. The third series of tests with $^{90}\text{SrF}_2$ prepared by the Waste Encapsulation and Storage Facility (WESF) flowsheet (at PNL) has also been completed and the couples are now being evaluated. The fourth series of tests using high purity nonradioactive SrF_2 with controlled levels of added impurities are now underway. The 1500-hr tests have been completed and the 4400-hr tests will be complete in October. Based on the results of the short-term tests, three containment materials will be selected for long-term compatibility testing.

Work has started on a thermodynamic analysis of potential reactions between SrF_2 , its decay product, contained impurities and the nine containment materials under consideration; a computer program has been developed to make the necessary free energy calculations.

Evaluation of the compaction and sintering characteristics of SrF_2 is underway. SrF_2 compacts readily and 25% of theoretical density can be achieved by cold pressing at 10 tons/in.². Sintering characteristics

of SrF_2 at temperatures up to 1100°C are largely determined by the impurities present, especially NaF . At 1100°C SrF_2 powder containing about 2 wt% Na sintered to a hard, dense mass having a bulk density of about 60% of theoretical density.

Work has started on measuring the dissolution rate of SrF_2 in aqueous media. Preliminary results obtained, using a static (nonflow) test system, were unsatisfactory due to analytical limitations. A dynamic (flow) test system is now being used which provides a more reliable measure of the dissolution rate. Initial results with the system indicate a dissolution rate of about $35 \mu\text{g Sr/hr-g SrF}_2$ for high purity nonradioactive SrF_2 fired at 1100°C .

STRONTIUM FLUORIDE COMPATIBILITY PROGRAM

The strontium fluoride compatibility program was reinstated in mid-March 1974. Technical direction for the program was transferred from the Division of Applied Technology to the Isotope Technology Branch - Division of Space Nuclear Systems.

STRONTIUM FLUORIDE COMPATIBILITY TESTS

The original program plan called for a series of short-term compatibility tests (up to 6 months) to be carried out in FY73-74 to select three or four containment materials for long-term (up to 4 years) compatibility testing. The short-term test program was stopped when the program was terminated July 1, 1973 and restarted in March 1974 when the program was reinstated.

In the short-term compatibility tests, nine different metals are being evaluated with four different grades of SrF_2 . The metals are Hastelloy C-276, Hastelloy X, Hastelloy N, Haynes 25, Haynes 188, Inconel 600, Inconel 625, TZM and tungsten. The four grades of SrF_2 are:

1. High purity nonradioactive SrF_2 (<0.1 wt% impurities)
2. High purity $^{90}\text{SrF}_2$ (<2 wt% impurities)^(a)
3. WESF Grade $^{90}\text{SrF}_2$ (<4 wt% impurities)^(b)
4. High-purity, nonradioactive SrF_2 with controlled levels of specific impurities added. (Same SrF_2 as used in No. 1.)

The short-term tests are of 1500- and 4400-hr duration, and the test temperatures are 800, 1000 and 1100°C.

The status of the short-term tests when the program was halted July 1, 1973, was as follows:

-
- (a) Representative ^{90}Sr solution, obtained from ARHCO, was purified by ion exchange and converted to $^{90}\text{SrF}_2$ by the WESF flowsheet in the 325 Building hotcells by PNL.
- (b) Representative ^{90}Sr solution obtained from ARHCO was converted to $^{90}\text{SrF}_2$ by the WESF flowsheet in the 325 Building hotcells by PNL.

1. The tests with high-purity, nonradioactive SrF_2 were completed and the couples evaluated; the results were summarized in an earlier report.⁽¹⁾
2. The tests with WESF grade $^{90}\text{SrF}_2$ were completed and sectioning of the capsules was underway; evaluation of the test specimens had just started.
3. The high-purity $^{90}\text{SrF}_2$ capsules had been fabricated, but testing had not started.
4. The hardware had been fabricated for the couples containing high-purity, nonradioactive SrF_2 with controlled levels of impurities, but preparation of the couples had not started.

When the program was stopped, all materials, couples and specimens were retained and the equipment maintained in standby condition. When the program was reinstated the test program was resumed.

Testing of the couples containing high-purity $^{90}\text{SrF}_2$ was started in December 1973 at the first indication the program would be reinstated. The 1500-hr tests were completed in February 1974 and the 4400-hr tests in June 1974. The early start of testing permitted a time saving of about three months in the program schedule. From July to December the couples were retained in the 325 Building hot cell. During that period the couples were at a temperature of 125 to 150°C. Holding the couples at this temperature for 5 months prior to testing could have some affect on the results obtained, but the effect should be minimal because of the low temperatures involved. Sectioning of the couples will begin in July.

Testing of the WESF-grade $^{90}\text{SrF}_2$ couples was completed in FY73. Some sectioning of the couples was accomplished in FY73, but the bulk of the couples was retained in the hot cell until the program was reinstated. During that time the couples were at a temperature of about 100°C. Holding the couples at this temperature for about 9 to 10 months could possibly affect the results obtained, but the temperature is so low that the effects should be minimized.

1. BNWL-B-290, "Pacific Northwest Laboratory Monthly Report on Isotopes Development Programs for June 1973."

Sectioning and evaluation of the WESF grade $^{90}\text{SrF}_2$ capsules is now underway. Considerable delay has been encountered in sectioning the couples because of hot cell filter problems in the 325 Building where the couples are being sectioned with an air-cooled abrasive saw inside the cell. It has been necessary to enclose the saw in a special box, equipped with a blower and absolute filter, to prevent the $^{90}\text{SrF}_2$ from entering the cell filter system. This procedure reduces the rate at which the couples can be sectioned. Removal of the test specimens from the sectioned couples has proved to be quite difficult. At the test temperatures (800, 1000 and 1100°C) the $^{90}\text{SrF}_2$ sinters to a solid mass and adheres strongly to the test specimens, making removal difficult.

The metallography on all of the $^{90}\text{SrF}_2$ test specimens is being done by the radiometallurgical facility operated by HEDL. The work load at the facility has been very heavy during the year and they have been unable to spend much time on our samples. The situation will improve beginning in July when the facility will begin two-shift operation.

To date, six WESF-grade $^{90}\text{SrF}_2$ couples have been examined and the results of these examinations are presented in Table 1; the results obtained with high-purity, nonradioactive SrF_2 under similar test conditions are also presented for comparison purposes. In the case of Hastelloy C-276 the depth of attack is about the same for the two grades of SrF_2 , but the types of attack observed were different. With the nonradioactive SrF_2 there was a slight roughening of the Hastelloy C-276 surface with subsurface void formation and intergranular attack. With the WESF grade $^{90}\text{SrF}_2$ there was a general layered attack of the metal surface with pitting and some subsurface void formation. There was little evidence of intergranular attack.

The Hastelloy X and Haynes 25 showed significantly more attack by the $^{90}\text{SrF}_2$ than the nonradioactive $^{90}\text{SrF}_2$. However, the types of attack observed were similar in each case and consisted of a general layered attack of the surface with some pitting and subsurface void formation.

TABLE 1. Compatibility Data on SrF_2 and WESF-Grade $^{90}\text{SrF}_2$ Couples

Metal	Time, hr	Temperature, °C	Depth of Metal Affected, Mils	
			WESF Grade $^{90}\text{SrF}_2$	High-Purity SrF_2
Hastelloy C-276	1500	800	3	1
Hastelloy C-276	1500	1000	4-5	4
Hastelloy C-276	1500	1100	4-5	6
Hastelloy C-276	4400	800	3-4	4
Hastelloy-X	1500	800	2-3	1
Haynes 25	1500	800	1-2	<<1

The couples containing high-purity SrF_2 with controlled levels of impurities have been fabricated and testing is now underway. The 1500-hr tests were completed in June and the couples are now being sectioned. The 4400-hr tests will be completed in October and couple evaluation should be completed by December. The SrF_2 compositions being tested are listed in Table 2. The ten impurities being evaluated were selected based on analysis of ^{90}Sr solution, obtained from ARHCO, and the $^{90}\text{SrF}_2$ prepared by the WESF flowsheet at PNL. Each composition is being tested with the nine metals under consideration. Because of the large number of couples which would be required to test individual couples for each set of conditions (10 impurities x 9 metals x 2 times x 3 temperatures = 540 couples), it was decided to test the couples at one temperature only - 1000°C. This reduced the number of couples required to 180. Because duplicate tests are being run for those couples containing H_2O and NO_3^- the couples involved total 216.

When the short-term tests are complete and all the couples evaluated, the three best materials will be selected for long-term testing. This decision should be reached by December 1974.

Many of the materials, chemicals and supplies needed for the long-term compatibility tests and other phases of the program have been ordered and most of the ordered materials have been received.

TABLE 2. Compositions of SrF_2 -Impurities Mixtures
Used in Short-Term Compatibility Tests

Composition	wt%
1. Sr^{+2} (as SrF_2)	95
Na^+ (as NaF)	5
2. Sr^{+2} (as SrF_2)	95
Ca^{+2} (as CaF_2)	1.67
Mg^{+2} (as MgF_2)	1.67
Ba^{+2} (as BaF_2)	1.66
3. Sr^{+2} (as SrF_2)	98
Mn^{+2} (as MnF_2)	2
4. Sr^{+2} (as SrF_2)	98
Pb^{+2} (as PbF_2)	2
5. Sr^{+2} (as SrF_2)	98
Cu^{+2} (as CuF_2)	2
6. Sr^{+2} (as SrF_2)	98
Fe^{+3} (as FeF_3)	2
7. Sr^{+2} (as SrF_2)	98
Al^{+3} (as AlF_3)	2
8. Sr^{+2} (as SrF_2)	99
O^{-2} (as SrO)	1
9. Sr^{+2} (as SrF_2)	99.95
NO_3^- [as $\text{Sr}(\text{NO}_3)_2$]	0.05
10. SrF_2	99.95
H_2O	0.05

Mr. J. D. Watrous (D. W. Douglas Laboratories) has been retained on a consulting basis to assist in evaluating the compatibility data. His analysis of the high-purity nonradioactive test couples will be reported in the July Monthly Progress Report. Services of a Battelle metallurgist (K. R. Wheeler) are anticipated at about 1/3 time in FY75 to assist in evaluating the compatibility data and in designing the long-term tests. The long-term compatibility tests will involve testing mechanical property specimens (i.e., tensile specimens) as well as metallographic specimens. The tests will also include couples in which the metal surface-to-SrF₂ volume ratio will be varied over a wide range. An attempt will be made to match the WESF storage capsule surface-volume ratio as closely as equipment limitations will permit.

THERMODYNAMICS OF SrF₂ COMPATIBILITY

Work has started on a thermodynamic analysis of potential reactions between SrF₂, its decay products, contained impurities and the components of the nine metals and alloys being tested. A preliminary analysis of the problem indicates many hundreds of reactions must be considered. Initial efforts have been devoted to obtaining the necessary thermodynamic data from the literature and preparing a computer program for making the necessary calculations. The program is capable of making the required entropy and heat capacity calculations and providing the free energy of reaction and equilibrium constant for a given reaction as a function of temperature in both printout and graphical form. It is estimated that the complete thermodynamic analysis will require approximately 6 to 8 man months effort to complete.

WESF CAPSULE ANALYSIS

A theoretical analysis of the WESF capsule to meet IAEA Series 33 test requirements is being carried out assuming various capsule materials. The analysis will be complete by the end of June and the results will be included in the July monthly report.

SrF₂ COMPACTION AND SINTERING

In the Waste Encapsulation and Storage Facility (WESF) SrF₂ will be prepared, fired at 1100°C for 16 hr, cooled to ambient temperature, crushed to a manageable size and loaded into the storage containers using a pneumatic impactor. The size of the SrF₂ granules will be -1/2 in. while the compacted density of the SrF₂ in the container will be 75 to 80% of theoretical density.

Even though the WESF flowsheet is set, ⁹⁰SrF₂ heat source requirements may make it desirable to be able to produce ⁹⁰SrF₂ compacts of controlled density. Therefore, a study was undertaken to evaluate the sintering and compaction characteristics of SrF₂. It was found that the purity of SrF₂ has a pronounced effect on its sintering characteristics. High purity non-radioactive SrF₂ powder (<0.1 wt% total impurities) showed little tendency to sinter below 1100°C. When this material (which was used in short-term compatibility tests) was held at 1100°C for 16 hr some sintering occurred, but the material was easily crushed between the fingers to a fine powder. The bulk density of the sintered material was about 40 to 45% of theoretical density.

SrF₂ obtained commercially (0.5% impurities) showed a somewhat greater tendency to sinter and after 16 hours at 1100°C had sintered to a fairly dense mass which was moderately difficult to crush. Bulk density of the sintered material was about 50 to 55% of theoretical density. SrF₂ of the approximate composition of the expected WESF ⁹⁰SrF₂ (~96% purity) sintered very readily. After 16 hours at 1100°C the material had sintered to a hard, dense mass. The material was easy to break up into large granules, but was difficult to crush to a fine powder. The bulk density of the sintered mass was about 60% of the theoretical density. The NaF (~2%) in the material appeared to be the cause of the increased agglomeration due to the formation of a low melting phase.

Samples of the three grades of SrF_2 (described above) were fired at various temperatures for varying periods of time and photomicrographs are now being obtained of the microstructure. This will permit a more quantitative evaluation of the sintering characteristics of the SrF_2 . The results will be reported as soon as the tests are completed and the photomicrographs obtained.

Tests evaluating the compaction characteristics of SrF_2 are being carried out by:

1. cold pressing SrF_2 pellets in a split sleeve die, and
2. step-pressing SrF_2 into a metal can contained in a steel die.

The initial results indicate that the temperature at which the SrF_2 is fired (degree of sintering) prior to pressing and the applied pressure have the greatest affect on the compact density obtained. Impurities in the SrF_2 and the particle size distribution appear to have little affect on the compaction characteristics of the SrF_2 . Figure 1 shows compaction characteristics data of the SrF_2 obtained with high-purity, commercial, and WESF-grade SrF_2 which had been fired at 1100°C for 16 hr and crushed to -20 mesh. There appears to be very little difference in the compaction characteristics of the three grades of fluoride. Both methods of compaction were used in the tests and similar results were obtained with each method. Samples of commercial SrF_2 were fired at various temperatures for 84 hr and then crushed to -20 mesh. Each sample was then compacted in a metal can at various pressures and the results are shown in Figure 2. It can be seen from the data that increasing the firing temperature increases the density obtainable with a given pressure. Similar results were obtained with high-purity SrF_2 .

Commercial SrF_2 , which had been fired at 930 and 1100°C for 84 hours, was ball milled and then screened. Compaction tests were run using various size fractions and the results are shown in Table 3. The density obtained with a given pressure appears to decrease slightly as the particle size of the fluoride decreases, but the change is quite small. Similar results were obtained with high-purity SrF_2 .

FIGURE 1. Density as a Function of Pressure for Various Grades of SrF_2 Fired at 1100°C for 16 Hours and Crushed to -20 Mesh.

FIGURE 2. The Effect of Firing Temperature on the Compaction of Commercial SrF_2 .

TABLE 3. Effect of Particle Size on the Compaction of SrF_2 Fired at 1100°C

Size Fraction	Density, % of Theoretical	
	11.3 TSI	34.0 TSI
- 70 +100	78	89
-100 +140	80	89
-140 +200	78	87
-200 +230	75	87
-230 +325	76	86
-325		
Combined Fractions	78	87

SrF_2 DISSOLUTION RATES IN AQUEOUS MEDIA

An important factor to be considered in a safety analysis of $^{90}\text{SrF}_2$ heat sources is the solubility and dissolution rate of the fluoride in aqueous media. The Martin Company measured the dissolution rate of SrF_2 in various solutions several years ago. Unfortunately, they used non-radioactive SrF_2 spiked with ^{85}Sr tracer. Since work with strontium titanate shows a much higher dissolution rate for the radioactive compound compared to the nonradioactive compound, one would expect a similar increase in the dissolution rate for the radioactive fluoride. Therefore, a program has started to measure the dissolution rate of $^{90}\text{SrF}_2$ in aqueous media. The tests will be carried out in a demineralized water (2 megohm resistivity) tap water, synthetic seawater and natural seawater, using both static and dynamic systems. Each system is being checked out using nonradioactive SrF_2 before the tests with $^{90}\text{SrF}_2$ are started. Two analytical methods are being used to determine the dissolved strontium in the tests with nonradioactive SrF_2 : atomic adsorption and gamma counting using ^{85}Sr tracer. In the latter case the SrF_2 is precipitated from a strontium solution spiked with ^{85}Sr .

The initial tests were carried out using a static (nonflow) system in which a weighed amount of SrF_2 was placed in a known volume of water

and the strontium content of the solution monitored as a function of time. In each test it was found that the SrF_2 dissolved at a fairly rapid rate initially and at a decreasing rate as time progressed. As the concentration of strontium in solution increased it was difficult to determine the dissolution rate with any degree of accuracy. It was found that at the higher strontium concentration levels the precision of the two analytical procedures approximated the daily increase in strontium concentration. This made it impossible to calculate a differential dissolution rate. It was possible to estimate average dissolution rates for extended time periods and the results of various tests are shown in Table 4. As a result of the poor data obtained with the static system it was decided to concentrate on use of the dynamic test system.

TABLE 4. Average Dissolution Rates for SrF_2 in Demineralized Water Obtained with a Static System

Run No.	SrF_2 Used	Time Period, hr	Analytical Procedure Used	Average Dissolution Rate, mg Sr/hr - g SrF_2
1	High-purity SrF_2 fired at 1000°C for 16 hr	0 - 24	^{85}Sr tracer	2.32
		24 - 264	"	0.10
2	High-purity SrF_2 fired at 1000°C for 16 hr	0 - 24	"	2.53
		24 - 264	"	0.19
3	Commercial SrF_2 fired at 930°C for 86 hr	0 - 24	Atomic Adsorption	0.72
		24 - 264	"	0.025
4	Commercial SrF_2 fired at 1100°C for 86 hr	0 - 24	"	1.26
		24 - 264	"	0.010

One test has been carried out with the dynamic system. In the test demineralized water was fed at a constant rate into a reaction vessel of known volume and containing a weighed amount of SrF_2 . The overflow was analyzed for strontium as a function of time. Figure 3 shows the results obtained to date with a sample of high-purity SrF_2 which had been fired at 1100°C for 20 hr.

The fired material was crushed to -4 mesh prior to use. The initial dissolution rate was quite high, but the rate decreased rapidly with time

until a relatively constant rate was attained after about 50 hr. Table 5 shows a comparison of the results obtained in this test with results obtained by the Martin Co. with SrF_2 , SrTiO_3 and Sr_2TiO_4 using a dynamic system.

It is normal practice to calculate dissolution rates based on the surface area of the material being dissolved. In the case of SrF_2 the problem is complicated by the fact that the SrF_2 sinters in the temperature regime of interest in heat source applications (800 to 1000°C). As a result the surface area of the SrF_2 changes with time and temperature when the source is in use. An additional complicating factor is that at WESF the SrF_2 will be impacted into the container instead of being pressed into pellets as is done with SrTiO_3 and Sr_2TiO_4 . This makes it impossible to calculate SrF_2 dissolution rates based on pellet geometric surface area as is done with the titanates. For these reasons it was decided to calculate and report SrF_2 dissolution rates based on SrF_2 weight rather than surface area. The surface area of SrF_2 , prepared under various conditions,

FIGURE 3. Dissolution Rate of SrF_2 in Demineralized Water - Obtained with a Dynamic System.

will be measured to help characterize the material but the surface area will not be used as the basis for the dissolution rate calculations.

Work is continuing with the dynamic system where the dissolution rates in various solutions of different grades of SrF_2 , which have been fired at various temperatures, will be determined. Once these tests have been completed the tests with $^{90}\text{SrF}_2$ will be started.

TABLE 5. Dissolution Rates of SrF_2 , SrTiO_3 and Sr_2TiO_4 in Demineralized Water - Obtained Using a Dynamic System.

<u>Material</u>	<u>Dissolution Rate ($\mu\text{gSr/hr-gSr Compound}$)</u>	<u>Source</u>
SrF_2	35	PNL
SrF_2	350	Martin*
SrTiO_3	20	Martin*
Sr_2TiO_4	1170	Martin*

*J. H. Gray "Solubility Study of Strontium Fuel Compounds," MND-3062-29, Martin Co., Baltimore, MD (June, 1966).

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