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AN EVALUATION OF STRONTIUM AND CESIUM COMPOUNDS FOR WASTE PACKAGING

by

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AN EVALUATION OF STRONTIUM AND CESIUM COMPOUNDS FOR WASTE PACKAGING

T. S. Soine
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1. INTRODUCTION

As part of the overall waste management program at Hanford, various methods have been evaluated for the long-term disposal of high level cesium and strontium liquid wastes. The method selected involves conversion of the separated ^{90}Sr and ^{137}Cs liquid streams to solid compounds followed by encapsulation of the solids in metal cans. Final disposal of the solid-compact cans would be in - as yet unselected - storage sites.

The Atlantic Richfield Hanford Company has requested the Pacific Northwest Laboratory to complete the development of the technology necessary for design and construction of a waste packaging plant based on the solid-compact concept. The first step in the development program involves: (1) an evaluation of the various strontium and cesium compounds which could be considered for encapsulation, and (2) selection of the two compounds which appear to be most suitable for packaging and long-term storage. This report summarizes the results of the compound evaluations study.

2. SUMMARY

Cesium chloride and strontium fluoride were selected as the primary candidates for packaging and long-term storage. The bases for their selection were three-fold: (1) The shortest development time, prior to starting plant design, could be achieved with CsCl and SrF_2, (2) Considerable compatibility data (up to nine years) is available for the two compounds, and (3) It was felt that CsCl and SrF_2 could be prepared and packaged at the lowest unit cost.

Two secondary candidates, cesium diuranate and strontium pyrophosphate, were elected as possible replacements for the chloride and fluoride if either or both hould be rejected for any reason.

Initial studies indicate that the development of technology for design of a ompacts waste packaging plant be based on the selection of cesium chloride and strontium luoride as the compounds to be packaged. In addition, approximately 20% of the develop- ent program should be devoted to developing the technology of the two secondary andidates.

. BASES FOR COMPOUND SELECTION

In evaluating the various cesium and strontium compounds being considered or packaging and storage, the basic requirement which the compounds must meet is hat they be capable of storage for the required disposal times under consideration >100 years). In other words, the rate of reaction between the compound and its encap- ulating material must be sufficiently slow, at the temperatures involved, that premature adding failure will not occur. Since there is only a very limited amount of compat- ibility data for a few strontium and cesium compounds available, it is difficult to ake an accurate estimate of the storage potential of each compound under considera- on. Therefore, it has been assumed in rating the compounds that by a proper selec- on of encapsulating materials all of the compounds under consideration can be uccessfully stored for the required times. However, those compounds for which com- atibility data are available received much greater consideration than those compounds or which no compatibility data is available.

Two other factors were considered as bases for compound selection: (1) the evelopment time required prior to plant design for each compound, and (2) the elative cost of preparing and packaging each compound.

The time required for development work before plant design can start has an important bearing on compound selection. In order to meet the projected time schedule for the waste packaging program, plant design should start by mid-1969. Therefore, the process development work should be close to completion by that time. This means that these compounds for which little or no development data is available would require a much larger development program to meet the time schedule.

Economics also have an effect on compound selection, and other factors being equal, those compounds which can be prepared and packaged for the lowest unit cost are the logical candidates for storage. It is difficult, if not impossible, to make an accurate cost analysis of each compound under consideration because of the lack of technical data. It is possible, however, to make a rough comparative analysis based on certain factors which affect the overall cost picture. These factors include (in order of importance):

a. <u>The labor costs for compound preparation and encapsulation</u>. A relative estimate of preparative costs can be made based on the complexities of the various flowsheets. Encapsulation costs should be almost directly proportional to the number of containers required for each compound. This in turn will depend on the compound radionuclide density and on the size of the container, which in turn, depends on a number of factors such as compound temperature limitations, thermal conductivity, thermal expansion, etc.

b. <u>Plant construction costs</u>. A detailed analysis of plant construction costs for each compound is impossible, but a rough comparison can be made by evaluating the complexities of the process flowsheets, encapsulation techniques required, and the materials of construction required.

- Materials costs. These costs should be a very minor part of the overall packaging costs for any compound, unless the compound under consideration should require unusually expensive materials (i.e., if a compound should require a metal such as platinum or tantalum) for encapsulation.

- Process development costs. Process development costs for the design and construction of the waste packaging plant should be a relatively small part of the overall cost picture. However, they can vary over a wide range depending on the compounds under consideration. For example, a great deal of development data is already available on the preparation and encapsulation of cesium chloride, while only a limited amount of information is available on other cesium compounds.

In the preceding discussion, there was one factor which was not included as basis for evaluation; that is, compound water solubility as it relates to safety d radionuclide containment. In making the selection of the primary candidates for capsulation, it was assumed that water solubility was not a criterion for compari-n, that sufficient protection would be achieved through double encapsulation and ndestructive testing techniques. If safety considerations negate this assumption, en water solubility does play an important part in compound selection and the mpounds chosen should have a low water solubility. The secondary compounds chosen backups to the two primary compounds were picked in a large part because of their w water solubility.

. COMPOUND EVALUATION

As stated previously, it was assumed that all the strontium and cesium ompounds would meet the requirement of being storable for the required disposal imes. However, there was one material which was rejected as not meeting this requireent, namely, strontium metal. It was felt that metal-metal diffusion was sufficiently reat at the temperature involved to preclude the use of strontium metal.

.1 Cesium Compounds

A number of compounds were considered as potential candidates for encapsulation. Table I lists the compounds evaluated and summarizes their pertinent physical properties such as cesium density and melting point. Except for the chloride, the data available on the preparation and encapsulation of most of the cesium compounds are extremely limited. For those compounds where little data are available, process development costs and time requirements have an important bearing on their relative ranking in the selection of the primary candidate for encapsulation.

.1.1 Primary Candidate - Cesium Chloride

Of all the cesium compounds evaluated, the chloride appears to be the logical hoice for encapsulation based on the requirements set forth in Section 3. Examinaion of the literature shows that most of the work that has been done on cesium ackaging has involved the chloride. It is relatively simple to prepare and the reparation technology is fairly well developed. A minimum of process development

TABLE I

PHYSICAL AND CHEMICAL PROPERTIES OF CESIUM COMPOUNDS

Compound	Theoretical Density (g/cm^3)	Approximate Package Density (g/cm^3)	Packaged Cs Density (g/cm^3)	Melting Point (°C)	Phase Transitions	Transition Temperature (°C)	Water Solubility (g/liter)	Hygroscopic
CsCl	3.99	3.79	3.00	646	yes	451(2)	1620	yes
CsF	4.1	~3.80	3.3	682	no		3670	yes
$Cs_2U_2O_7$	5+	5.2	1.62	1000			<.1*	no
$CsNbO_3$	~4.8	4.8	1.84	940			<.1**	no
Cs_2O	4.25	~3.6	3.4	490 (in N_2)			Very Soluble	yes
$CsTaO_3$	6.6	5.5	2.02	1400			<.1**	no
Cs_2SO_4	4.24	~3.6	2.6	Decom. 1010	yes	660	1670	yes
$Cs_2O\cdot(B_2O_3)_x$	~3.4	2.9	2.2	650-700 (Est)			Soluble	
$Cs_2O\cdot Al_2O_3\cdot 2SiO_2$	2.9	~2.5	1.5	1400			<.1**	no
$Cs_xNb_{1-x}O_3$		3.92	1.90	>1000			<.1**	no
$Cs_{0.32}WO_3$	~7	6.15	0.97	950			<.1**	no
Cs_2TiO_3	~4	3.4	2.5	840			Soluble**	no
$2Cs_2O\cdot 2Al_2O_3\cdot 9SiO_2\cdot H_2O$	~3.4	~2.9	1.2	>1000			<.1	no
Cesium Glass		3.0	1.5	875			0.01	no
CsBr	4.4	~3.8	2.4	636			1243	yes
CsI	4.5	~3.8	1.9	621			440	yes
Cs_2CO_3		3.3	2.7	Decom. 610			2600	yes

*Solubility increases with temperature about 6 grams/liter at 65°C.

**Qualitative tests.

work would be required for plant design. The chloride has a very high cesium density (3.13 g Cs/cm^3), which would reduce the number of storage containers to a minimum. Various encapsulation techniques have been studied in some detail and only a limited amount of development work would be required for plant design. Compatibility studies of the chloride with various encapsulating materials have been underway for at least nine years.[1] There is no problem of radiolytic decomposition as long as the chloride is anhydrous.

The chloride does have certain disadvantages, however, which keep it from being the ideal compound for packaging.

a. At about 451°C the chloride undergoes a phase transition which results in a 17% increase in volume.[2] This means that if the package is loaded to greater than 80% of theoretical density, the container must be maintained at a temperature below 450°C to prevent a rupture due to chloride expansion.

b. Use of the chloride requires processing equipment and facilities compatible with a chloride system.

c. The chloride is very soluble in water. This is not too serious a factor, however, since all cesium compounds show some degree of water solubility.

While the disadvantages listed are important, they are not enough to outweigh the obvious advantages of the chloride which make it the first choice for encapsulation.

1.2 Secondary Candidates

Although cesium chloride was selected as the primary candidate for ıcapsulation, there are three other compounds which appear promising, and ıich should be considered as a replacement for the chloride if it is rejected ›r any reason. Each of the three is discussed below in order of their relative ınking.

Cesium fluoride - The fluoride has a cesium density about the same as the chloride (3.14 g Cs/cm^3). It has a big advantage over the chloride in that it does not undergo a phase change below the melting point. This could increase the temperature limit from <450°C for the chloride to about 680°C for the fluoride. In addition, the fluoride would not require that the processing equipment and facilities be compatible with a chloride system. Also, the fluoride should not suffer radiolytic decomposition as long as it is anhydrous. The fluoride is, however, very soluble in water and is very hygroscopic (on the same order as calcium chloride). This might require that the preparation and packaging be carried out in a dry or inert atmosphere to prevent moisture pickup. Little or no work has been done on the preparation and encapsulation of the fluoride. Therefore, a great deal of development work would be required prior to plant design. Little is known of the compatibility of the fluoride with encapsulating materials at elevated temperatures so a long-term compatibility program would be needed to select the best cladding material.

Cesium diuranate - The diuranate ($Cs_2U_2O_7$) is easy to prepare since it can be precipitated from an aqueous solution.[3] It is less soluble in water than the

halides, but has a cesium density of only about one-half that of the chloride. Its upper temperature limit would be about 1000°C. Little work has been done on encapsulation of the diuranate, and an extensive compatibility program would be required. Process development requirements for plant design would be moderate.

c. Cesium Niobate - The niobate ($CsNbO_3$) has two advantages over the chloride in that: (1) it is relatively insoluble in water, and (2) its temperature limit would be about 900°C. However, its cesium density is relatively low, and it is quite difficult to prepare, requiring a high temperature fusion (>1100°C) of Cs_2CO_3 and Nb_2O_5.[3] Volatilization of Cs_2O during the fusion step may be a problem. Extensive process development work and compatibility studies would be required prior to plant design.

Both the diuranate and niobate would rate considerable attention if water solubility becomes an important factor in compound selection. However, both compounds are at least slightly soluble so the question is one of degree. If a capsule develops a leak while in an aqueous media, a contamination spread will result regardless of the compound encapsulated. The contamination spread would be less if the encapsulated material is the diuranate or the niobate, but it would still be a problem.

4.1.3. Discarded Candidates

A number of other compounds (which have been studied by other investigators previously for use as isotopic heat sources) were also evaluated. They were discarded as candidates for encapsulation for the reasons stated below. Any cesium compounds not listed were eliminated from consideration because they had no apparent advantages over the primary and secondary candidates, and because they were all soluble in water.

a. Cesium borate - The borates ($Cs_2O \cdot (B_2O_3)_x$) all have a low cesium density and a high water solubility. Little information is available on their physical properties. Their stability to radiolytic decomposition is questionable.

b. Cesium aluminum silicate ($Cs_2O \cdot Al_2O_3 \cdot 2SiO_2$) - This compound is soluble in water, has a low cesium density, and requires a high temperature fusion (1000°C) for preparation.[3]

c. Cesium oxide - The oxide (Cs_2O) is soluble in water and hygroscopic to the extent that it would probably have to be handled in a dry or inert atmosphere during the encapsulation process. Studies in this laboratory have shown that formation by calcination of the Cs_2CO_3 or $CsNO_3$ results in large losses due to the volatility of the oxide.[7]

d. Cesium sulfate - The sulfate (Cs_2SO_4) is very soluble in water. Its radiation stability is questionable, and it has a slightly lower cesium density. It undergoes a phase transition at 660°C.

e. Cesium tantalate - The tantalate ($CsTaO_3$) has the same characteristics as the niobate which is discussed in the previous section. Volatilization of Cs_2O during preparation of the tantalate would be a definite problem.[3]

f. Cesium tungsten niobate - This compound ($CsW_xNb_{1-x}O_3$) has the same characteristics as the niobate but it is much more difficult to prepare.[3]

g. Cesium tungsten bronze - This compound ($Cs_{0.32}WO_3$) has a low solubility in water, but is difficult to prepare and the cesium density is quite low.[3]

h. Cesium titanate - The titanate ($CsTiO_3$) is difficult to prepare, requiring a high temperature fusion (1470°C) under carefully controlled conditions. The volatilization of Cs_2O is a problem during the fusion step.[3]

i. Pollucite - This compound ($2Cs_2O \cdot 2Al_2O_3 \cdot 9SiO_2 \cdot H_2O$) is relatively insoluble in water. However, it has a low cesium density and is difficult to prepare. The preparation involves high temperature fusions which require homogeneous slurries of SiO_2, $CsCO_3$ and organoaluminum compounds.[4]

j. Cesium bromide - Cesium bromide (CsBr) has about the same characteristics as cesium chloride except that the cesium density is lower. Its preparation involves the use of HBr rather than HCl.

k. Cesium iodide - Cesium iodide (CsI_2) is in the same category as the chloride and bromide. It has the lowest cesium density of the three.

l. Cesium carbonate - Cesium carbonate (Cs_2CO_3) is very soluble in water, decomposes on heating and is hygroscopic.

m. Cesium glass - Cesium borosilicate glass is reported to have very favorable qualities as far as water solubility and compatibility with container materials are concerned. However, both of the current methods of preparation involve high temperature fusions which rely on intimate mixtures of solid materials. These procedures do not appear to be too applicable to high through put hot cell operations which would be necessary to handle large quantities of cesium waste.[5]

2. <u>Strontium Compounds</u>

As was the case with cesium, a number of strontium compounds were considered as potential candidates for encapsulation. Table II lists their pertinent properties. Unlike the case of cesium, however, a number of strontium compounds

TABLE II

PHYSICAL AND CHEMICAL PROPERTIES OF STRONTIUM COMPOUNDS

Compound	Theoretical Density (g/cm^3)	Estimated Package Density (g/cm^3)	Packaged Sr Density (g/cm^3)	Melting Point (°C)	Phase Transitions	Transition Temperature (°C)	Water Solubility (g/liter)	Hygroscopic
SrF_2	4.2	3.8	2.7	1400	no		0.011***	no
$Sr_2P_2O_7$	3.4	2.9	1.5	1100	no		nil	no
$SrTiO_3$	5.03	3.8	1.9	1910	no		1 $mg/cm^2/day$	no
SrO	4.63	4.2	3.7	2430	no		0.69****	yes
Sr_2TiO_4	4.93	3.60	2.3	1860	no		10	no
$SrZrO_3$	5.48	4.57	1.8	>1100			.04****	no
$SrCO_3$	3.7	2.9	1.7	>1700	yes	920-929	.001****	no
$Sr(NbO_3)_2$	4.8	4.1*	0.98	>1400				no
$SrSiO_3$	3.54	3.0*	1.6	1580			nil	no
$3SrO \cdot Al_2O_3$	3.3	2.8*	1.2				nil	yes
$SrHPO_4$	3.5	3.0*	1.4	d**			pK = 8.1	no
$SrSO_4$	3.96	3.4*	1.6	1605	yes	1152	.11	no
SrB_6	3.3	2.8*	1.6	2235			Slight Sol.	no
SrS	3.70	3.14*	2.3	>2000			d	no
$SrMoO_4$	4.1	3.5*	1.3	d			.1	no
$SrBr_2$	4.2	3.6*	3.0	643			85.2***	yes
SrC_2	3.9	3.3*	2.9	>1500			Decomposes	yes
$SrCl_2$	3.1	2.6*	1.5	873			53.8****	yes
SrI_2	4.6	3.9*	1.6	402			165***	yes
$SrWO_4$	6.2	5.3*	1.6	Decomposes			0.14****	no

*Estimated at 85% of theoretical density.
**Converts to $Sr_2P_2O_7$ on heating.
***Measured at 0°C.
****Measured at 20°C.

have been studied quite extensively for use as isotopic heat sources, including the oxide, fluoride and titanates. This makes it easier to compare the various compounds, and means that development costs have a lesser effect on compound selection.

4.2.1. Primary Candidate - Strontium Fluoride

Based on the requirement set forth in Section 3 above, strontium fluoride was selected as the most suitable strontium compound for encapsulation. The fluoride has a number of advantages which influenced this decision. It is easy to prepare and can be precipitated from an aqueous solution. The method of preparation is fairly well developed and a minimum of process development time and money would be required for plant design. The fluoride has a high strontium density (2.94 g Sr/cm^3) and a high melting point (1400°C) which would reduce the number of packages required to a minimum. There are no phase transitions which would present an expansion problem and the thermal conductivity is average. The fluoride is very stable and radiolytic decomposition is not a problem. The fluoride is not hygroscopic so the encapsulation would not have to be done in a dry or inert atmosphere. Some encapsulation work has been done and only a moderate development effort would be required in this area. Some container compatibility studies have been made[1,6] which would reduce the development time required. Strontium fluoride does have one disadvantage compared to some other strontium compounds in that it is slightly soluble in water. As in the case of cesium chloride, it is felt that this problem should not preclude its use.

4.2.2. Secondary Candidates

There are three other strontium compounds which have many desirable features and which should be considered as possible replacements for the fluoride in case its use is rejected for any reason. The three are discussed below in order of their relative ranking.

a. Strontium pyrophosphate - The pyrophosphate ($Sr_2P_2O_7$) was rated best of the three secondary candidates. It is easy to prepare and can be precipitated from an aqueous solution. It has a high melting point ($\sim 1200^{\circ}C$). It is relatively insoluble in water.[7] Its major disadvantages are that little development work has been done on the compound, it has a low strontium density, and its radiation stability is unknown.

b. Strontium metatitanate - A considerable amount of work has been done on strontium titanate ($SrTiO_3$) for use as isotopic meta-heat sources. It has a lower water solubility than fluoride and has a high melting point. The preparative methods are well worked out, but they are complex and involve high temperature fusions.[8] It has a low strontium density and this, combined with its difficult preparation, makes it less attractive than either the fluoride or the pyrophosphate.

c. Strontium oxide - The oxide is a borderline secondary candidate. Besides its high strontium density, the main fact. in its favor is that it can be prepared by the direct calcination of strontium nitrate.[7] This is a relatively simple process and could be easily adapted to a hot cell operation. However, the oxide is quite hygroscopic which would require that it be handled in a dry or inert atmosphere.

4.2.3. Discarded Candidates

The following compounds were considered and discarded for the reasons stated. Any compounds not listed were discarded because of their known water solubility and lack of advantages over the primary and secondary candidates.

a. Strontium silicate - The silicate ($SrSiO_3$) has a low thermal conductivity and a low strontium density. It is soluble in hot water.[8] Could be a possible secondary candidate

b. Strontium zirconate - The zirconate ($SrZrO_3$) is soluble in water and its preparation requires a high temperature fusion of $SrCO_3$ and ZrO_2. Its strontium density is low.[9,10]

c. Strontium orthotitanate - The orthotitanate (Sr_2TiO_4) is comparable to the metatitanate discussed above except that it is more soluble in water.

d. Strontium carbonate - The carbonate gives off CO_2 when heated to >1000°C and its radiation stability is questionable.

e. Strontium niobate - The niobate $Sr(NbO_3)_2$ is relatively insoluble in water, but has a very low strontium density and its preparation involves a high temperature fusion.

f. Strontium aluminate - The aluminate ($3SrO \cdot Al_2O_3$) has a low thermal conductivity, is difficult to prepare, and is hygroscopic. In addition, compact densities are not reproducible.[8]

g. Strontium hydrogen phosphate - This compound ($SrHPO_4$) has a low Sr density and is susceptible to radiolytic decomposition.[8]

h. Strontium sulfate - The sulfate decomposes at elevated temperatures and is susceptible to radiolytic decomposition.[8]

i. Strontium molybdate - The molybdate ($SrMoO_4$) is soluble in water and decomposes on heating.

j. Strontium sulfide - The sulfide (SrS) decomposes in water and is difficult to prepare, requiring a high temperature gas-solid reaction.

k. Strontium boride - The boride (SrB_6) is difficult to prepare, soluble in water and gives an incomplete tie-up of the strontium.[9,10]

l. Strontium bromide - Strontium bromide ($SrBr_2$) has a relatively high strontium density. However, compared to strontium fluoride, it is more soluble in water, has a lower melting point, volatilizes at 770°C, is hygroscopic and is more difficult to prepare (requires evaporation of $SrCO_3$ with HBr).

m. Strontium carbide - Strontium carbide (SrC_2) requires the fusion of strontium oxide and charcoal. It reacts with water in the same manner as calcium carbide to produce acetylene.

n. Strontium chloride - Strontium chloride ($SrCl_2$) has a low strontium density, low melting point, is soluble in water and is hygroscopic.

o. Strontium iodide - Strontium iodide (SrI_2) has a low melting point, is extremely soluble in water, and is hygroscopic forming iodine and strontium hydroxide on standing in moist air.

p. Strontium tungstate - Strontium tungstate ($SrWO_4$) decomposes when heated and had a low strontium weight for the total weight of the material.

RECOMMENDATIONS

As described in the section above, cesium chloride and strontium fluoride were chosen as the most suitable compounds for encapsulation and long-term storage. The bases for selection of these compounds was the availability of process technology and compatibility data for the two compounds and economics. However, if safety considerations become an overriding factor, then water solubility becomes a controlling factor in compound selection. In this case, strontium pyrophosphate and cesium diuranate appear to be the most logical compounds for encapsulation.

Since the importance of water solubility has not been well defined, it is proposed that the following course of action be taken with regard to the future development program

:rontium bromide - Strontium bromide ($SrBr_2$) has a relatively high strontium :nsity. However, compared to strontium fluoride, it is more soluble in ıter, has a lower melting point, volatilizes at 770°C, is hygroscopic and ı more difficult to prepare (requires evaporation of $SrCO_3$ with HBr).

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1. Development of the technology for design of a compacts waste packaging plant will be based on the selection of cesium chloride and strontium fluoride as the two compounds which will be encapsulated.

2. In addition, a limited amount of development work will be carried forward on the two alternate compounds, cesium diuranate and strontium pyrophosphate, concurrently with the work on the two primary compounds. This would serve as a backup effort in case unexpected problems arise with one or both of the primary selections which prevent their use, or in case the emphasis on water solubility becomes an overriding factor. Approximately 20% of the developmental effort should be devoted to study of the backup compounds. This would shift to a full-scale effort if the primary compounds are dropped from consideration at any time during the program.

ACKNOWLEDGEMENTS

The evaluation of the various compounds and selection of the primary and secondary candidates for encapsulation was essentially a group effort of a number of people from the Chemistry and Chemical Engineering Department of Battelle Northwest. Those persons making contributions to the effort include R. E. Burns, G. Jansen, Jr., and L. K. Mudge.

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