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## **Multibarrier Waste Forms**

### **Part III: Process**

### **Considerations**

**R. O. Lokken**

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**October 1979**

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MULTIBARRIER WASTE FORMS  
PART III: PROCESS CONSIDERATIONS

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Richland, Washington 99352



## SUMMARY

The multibarrier concept for the solidification and storage of radioactive waste utilizes up to three barriers to isolate radionuclides from the environment: a solidified waste inner core, an impervious coating, and a metal matrix. The coating and metal matrix give the composite waste form enhanced inertness with improvements in thermal stability, mechanical strength, and leach resistance. Multibarrier Waste Forms Part I: Development describes research and development that resulted in one-liter demonstration encapsulations of four multibarrier waste forms of nonradioactive, simulated waste compositions.<sup>(1)</sup> Multibarrier Waste Forms Part II: Characterization and Evaluation evaluates the four waste forms with respect to thermal stability, mechanical strength, and leach resistance.<sup>(2)</sup>

Preliminary process flow rates and material costs were evaluated for four multibarrier waste forms with the process complexity increasing thusly:

- Glass marbles (least complex)
- Uncoated supercalcine
- Glass-coated supercalcine
- PyC/Al<sub>2</sub>O<sub>3</sub>-coated supercalcine (most complex)

This report discusses the process variables and their effect on optimization of product quality, processing simplicity, and material cost. An expanded engineering feasibility study is being conducted at Pacific Northwest Laboratory by R. L. Treat on ten alternative waste fixation processes. These studies include in-depth analysis of remote adaptability, process complexity and costs, and material requirements and costs.



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MULTIBARRIER WASTE FORMS  
PART III: PROCESS CONSIDERATIONS

INTRODUCTION

The Radioactive Waste Immobilization Program conducted by the Pacific Northwest Laboratory (PNL) for the Department of Energy (DOE) has as one of its objectives the development of processes for converting high-level liquid waste (HLLW) from alternative fuel cycles and U.S. defense programs to solid forms demonstrated to be physically, chemically, and radiolytically stable and inert. During the past 20 years, numerous waste forms and processes have been proposed for solidification of nuclear wastes. The waste forms have ranged from simple concepts such as direct storage of calcine to complex forms utilizing crystalline products, coatings, and metal matrices. Because of the increasing number of options during recent years, increased difficulty is encountered in differentiating the options and making realistic comparisons.

A major part of PNL's program has been directed towards development of low-melting (1000 to 1150°C) borosilicate glasses.<sup>(3,4)</sup> However, to provide for waste streams not readily vitrifiable and to ensure that other options for encapsulation of fission product waste were considered, an effort was initiated in 1973 to develop alternative waste forms based upon a multibarrier concept (Figure 1). The multibarrier concept aims to separate the radionuclide-containing inner core material and the environment by the use of coatings and metal matrices. The resultant composite waste form exhibits enhanced inertness due to improved thermal stability and mechanical strength, and the added barriers improve leach resistance.

The feasibility of producing lab-scale multibarrier waste forms was demonstrated by the encapsulation of one-liter volumes of simulated waste glass marbles and coated and uncoated supercalcine.<sup>(5)</sup> Following lab-scale demonstration of the multibarrier concept, an evaluation of the waste forms must consider both the level of inertness achieved (risk analysis) and the process engineering of producing the waste forms in a full-scale operation.

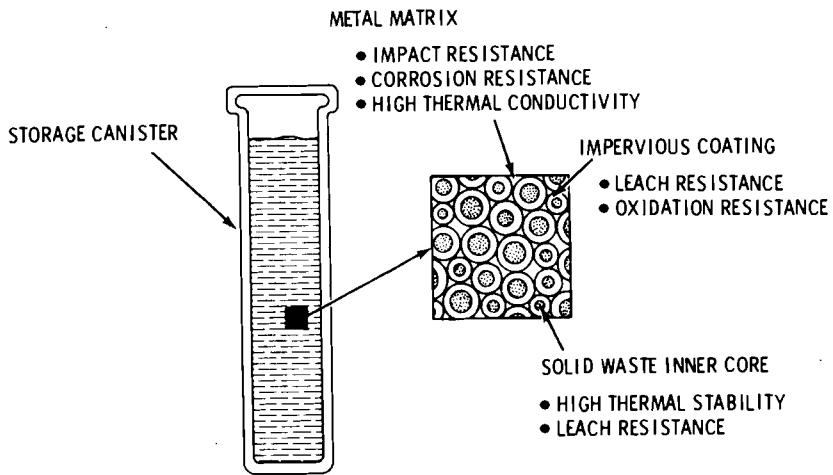


FIGURE 1. Multibarrier Concept for Isolating Nuclear Waste

As the required level of inertness or stability increases, the technological complexity of the processes would generally be expected to increase, approaching the limits of engineering feasibility. This general trend has not been characterized in sufficient detail to identify an optimum tradeoff between waste form stability and complexity.

Lutze<sup>(6)</sup> and Ross et al.<sup>(7)</sup> reviewed the properties of crystalline and glass waste form options and concluded that either glass or crystalline materials can be shown to have some advantage when a single property is considered. However, the differences are small and each material has both assets and liabilities. With proper design, either type of waste form can be used to solidify and contain radioactive waste. Lamb and Cole<sup>(8)</sup> have developed a pelletized waste form for high-level zirconia waste calcines which exhibits cesium leach rates 10 to 30 times less than those of a comparable glass. Comparing metal-matrix waste forms, Jardine and Steindler<sup>(9)</sup> concluded that metal-matrix encapsulation of commercial wastes may serve to increase the effective thermal conductivity of the waste form and to produce composites having reduced rates and improved mechanical strength. Similar conclusions have been reported by Lamb<sup>(10)</sup> and Van Geel et al.<sup>(11)</sup>

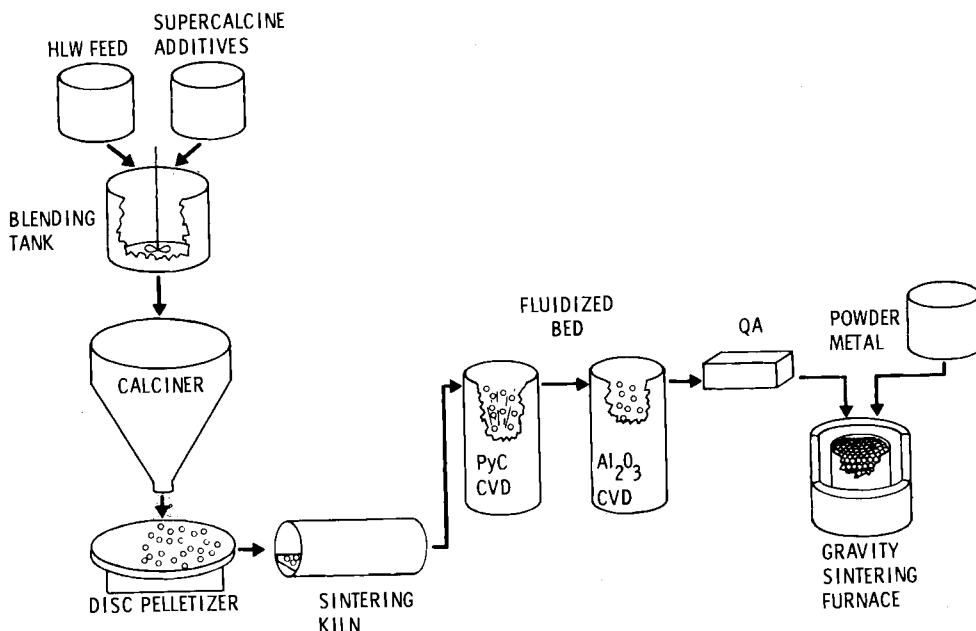
Ross<sup>(12)</sup> has reviewed 15 solidification processes and 14 current waste forms in regards to process complexity and concluded that the simplest processes are cast concrete and spent fuel encapsulation. Highly complex processes

include cermets and coated pellets in a metal matrix. In a parametric performance ranking of alternative nuclear waste encapsulants, Hench<sup>(13)</sup> has made similar rankings.

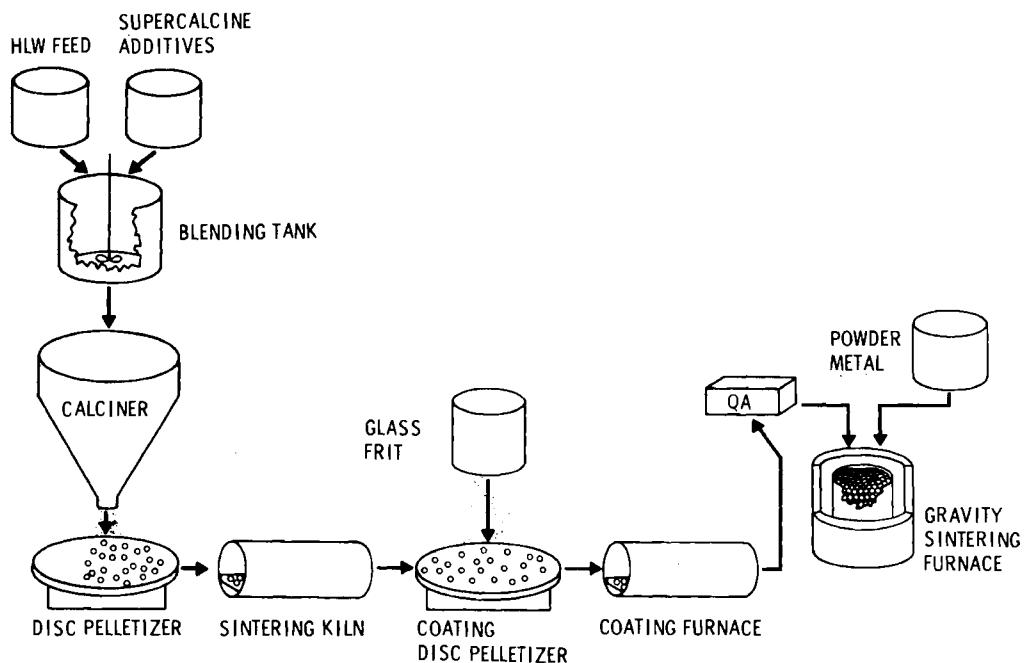
Since the goal of the multibarrier concept is to enhance the inertness of high-level waste, all process parameters and procedures should be for optimum achievement of that goal balanced against full-scale process economics and waste fixation requirements. Four waste forms were considered for the multibarrier inner core:

- Pyrolytic carbon- and alumina-coated supercalcine (CVD coating)
- glass-coated supercalcine
- uncoated supercalcine
- waste-glass marbles.

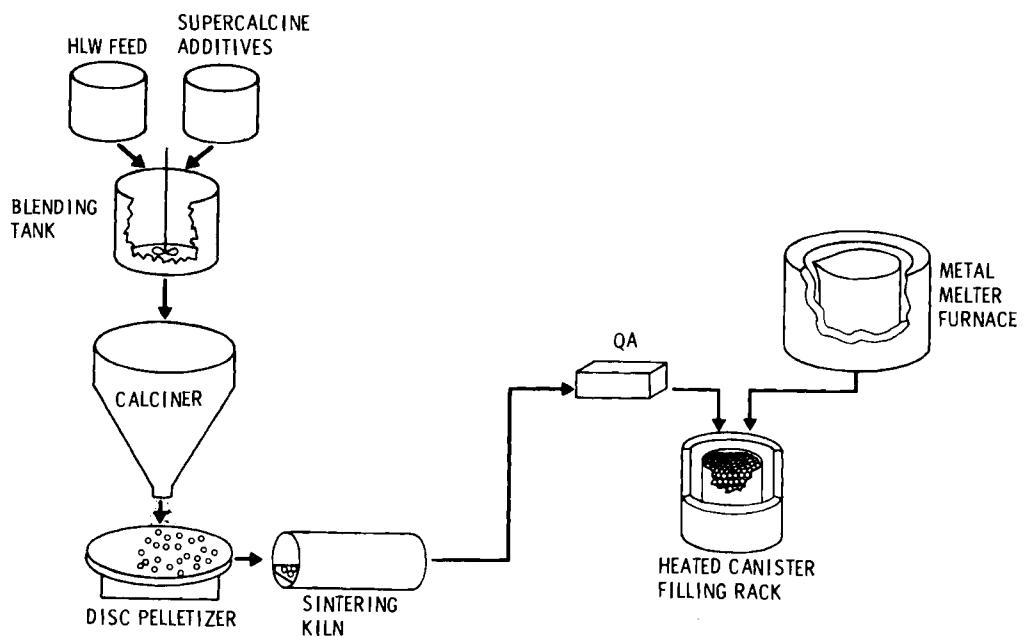
The processes for producing these waste forms in an inert encapsulated product were examined with the given consideration that requirements for increasing inertness usually increase the technological complexity of the waste fixation processes. Within each of the processes are particular parameters that require refinement to assure overall optimization of the process and results. Process flow charts for the production of the four multibarrier waste forms are shown schematically in Figures 2, 3, 4, and 5.



**FIGURE 2.** PyC/Al<sub>2</sub>O<sub>3</sub>-Coated Supercalcine Process Flowsheet



**FIGURE 3.** Glass-Coated Supercalcine Process Flowsheet



**FIGURE 4.** Uncoated Supercalcine Process Flowsheet

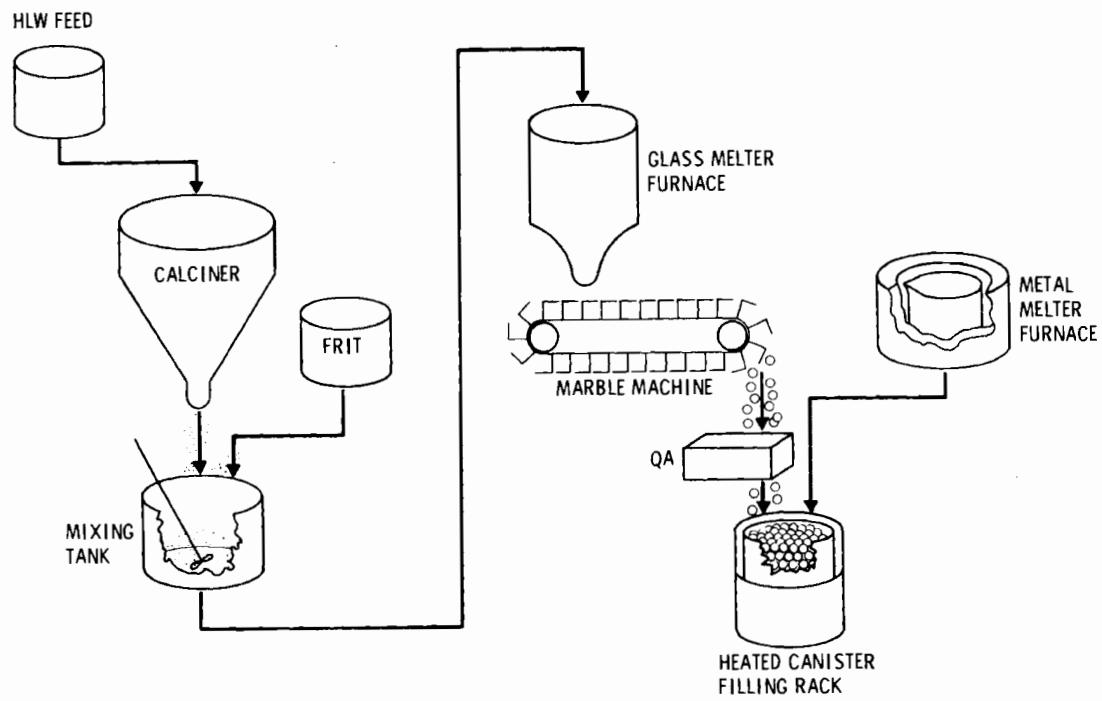
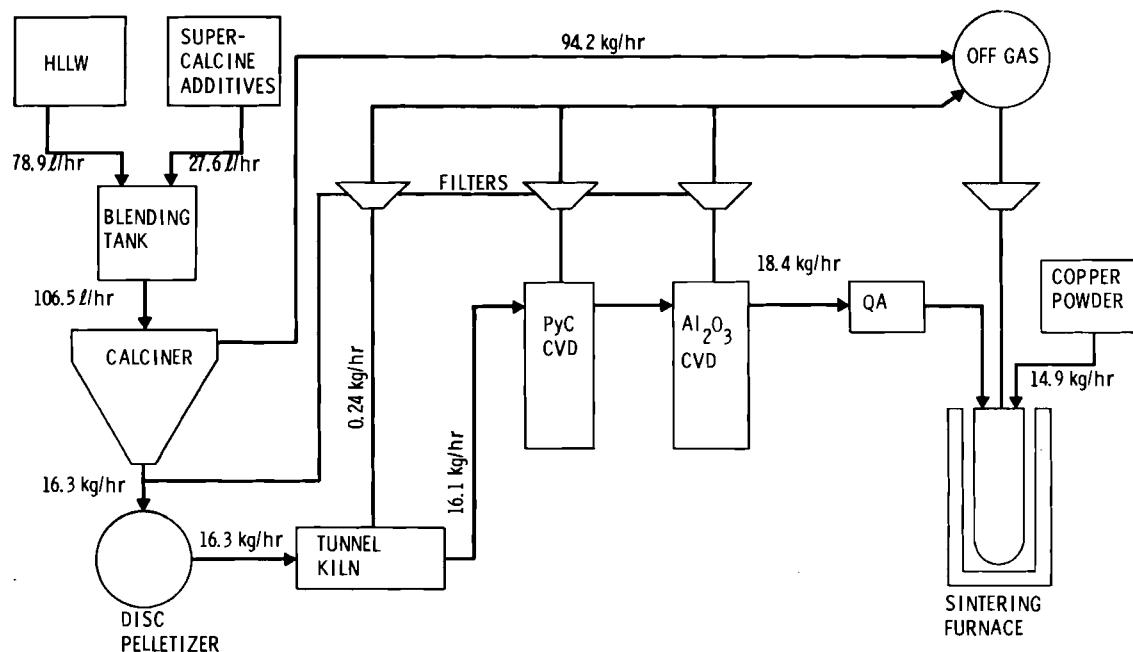


FIGURE 5. Waste-Glass Marble Process Flowsheet

## PROCESS FLOW RATES

Flow rate calculations have been performed for four sample multibarrier waste form processes. It was assumed that radioactive waste would be processed at a rate equivalent to 5 metric tons (t) of uranium per 24-hr operating day. Solidification processes follow the final concentration of HLLW to a volume of 378.5 l/t of uranium. The solid waste content of HLLW is 58.7 kg/t of uranium. Figures 6, 7, 8, and 9 show process flow sheets and calculated flow rates for PyC/Al<sub>2</sub>O<sub>3</sub>-coated supercalcine, glass-coated supercalcine, uncoated supercalcine, and waste-glass marble processes, respectively.

Annual canister and matrix requirements for the processes are presented in Table 1. The annual requirements were based upon the maximum allowable centerline temperature (based on either the softening temperature of the cores or the melting temperature of the matrix) and a maximum allowable canister diameter of 0.61 m for repository storage. Canister length was held constant at 3 m. Annual material costs for components of the multibarrier waste products are listed in Table 2. These costs do not include process costs.



**FIGURE 6.** PyC/Al<sub>2</sub>O<sub>3</sub>-Coated Supercalcine Process Flow Rates

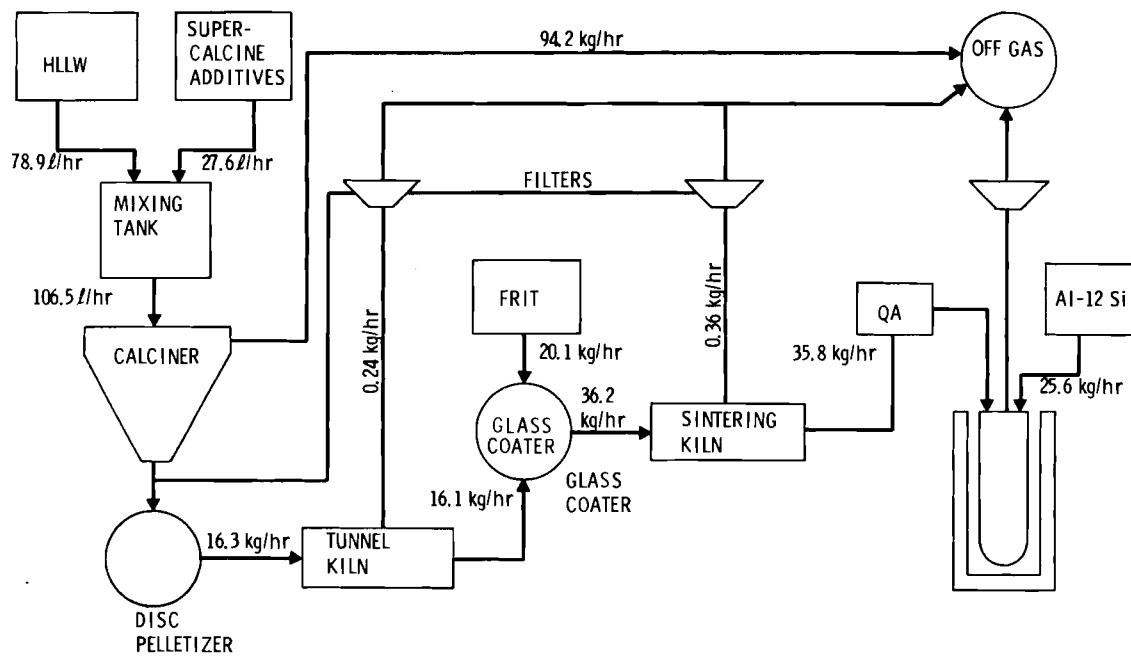


FIGURE 7. Glass-Coated Supercalcine Process Flow Rates

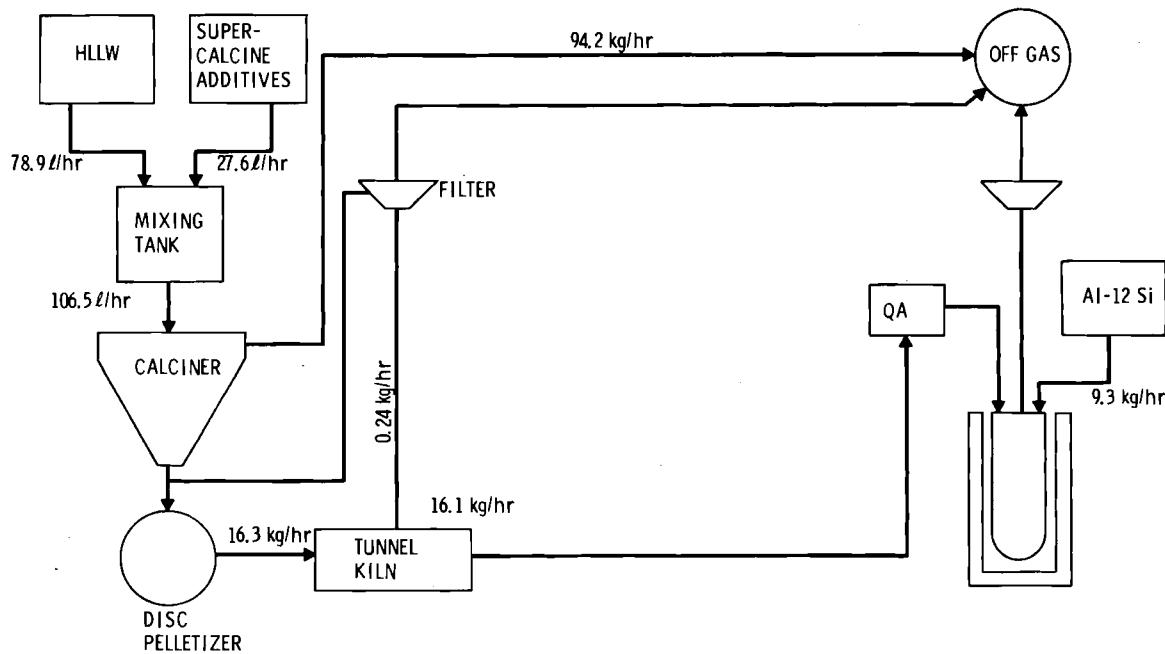


FIGURE 8. Uncoated Supercalcine Process Flow Rates

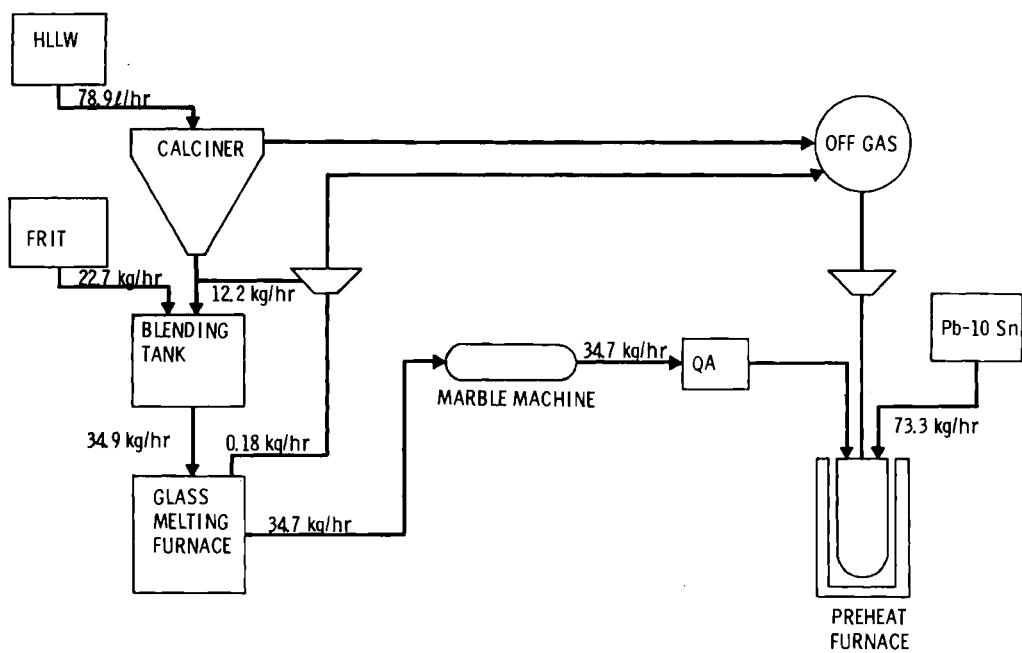


FIGURE 9. Waste-Glass Marble Process Flow Rates

TABLE 1. Annual Canister and Matrix Materials Requirements for Various Multibarrier Waste Products

Waste Form	Matrix	Maximum Centerline Temperature, °C(c)	Canister Dia, (d) m	Number of Canisters Required/yr	Total Volume of Waste Product, m <sup>3</sup> /yr	Waste Loading vol%	Volume of Matrix, m <sup>3</sup> /yr
Reference Glass	--	500	0.15	1310	74	35	--
Reference Glass	--	800	0.23	623	74	35	--
Glass Marble <sup>(a)</sup>	Pb-10Sn	300	0.55	169	123	21	49
Uncoated <sup>(a)</sup>	Al-12Si	600	0.61	71	63	45	25
Supercalcine							
Glass-Coated <sup>(b)</sup>	Al-12Si	600	0.61	77	69	41	28
Supercalcine							
PyC/Al <sub>2</sub> O <sub>3</sub> -Coated <sup>(a)</sup>	Cu (65%)	800	0.61	94	84	34	34
Supercalcine							
PyC/Al <sub>2</sub> O <sub>3</sub> -Coated <sup>(a)</sup>	Cu (65%)	800	0.61	141	126	21	76
Supercalcine							

(a) 60% packing

(b) 40% packing

(c) Maximum temperature is based on either the softening temperature of glass or the service temperature of the matrix

(d) Maximum selected diameter of 0.61 m for repository compatibility

TABLE 2. Annual Component Materials Costs for Various Multibarrier Waste Products

Inner Core Waste Form	Encapsulation Matrix	Total Volume of Waste Product, m <sup>3</sup> /yr	Annual Materials Costs, (a) \$1000/yr	Additives	Coating	Matrix	Canister	Total
Reference Glass(b)	--	74	359	--	--	--	255	614
Glass Marble(c)	Pb-10Sn	123	359	--	--	826	140	1325
Uncoated(c)	Al-12Si	63	295	--	--	80	65	440
Supercalcine								
Glass-Coated(c)	Al-12Si	69	295	17	--	87	71	470
Supercalcine								
PyC/Al <sub>2</sub> O <sub>3</sub> -Coated(c)Cu (65%)		84	295	19	--	473	87	874
Supercalcine								
PyC/Al <sub>2</sub> O <sub>3</sub> -Coated(d)Cu (65%)		126	295	19	--	1064	130	1508
Supercalcine								

(a) Material costs only - does not include process costs; based on 1979 cost with 1979 dollars.

(b) 500°C centerline

(c) 60% packing

(d) 40% packing

## OPTIMIZATION OF PROCESS STEPS

The purpose of investigating various waste forms is to provide the basis of optimizing the process economics and the inertness of the waste. Achieving over-all optimization entails optimizing all process step procedures. This would include supercalcine pelletization, application of coatings, waste loading, and matrix material selection, each of which will influence the others.

In the coated supercalcine processes the pellet core diameter and porosity must be controlled through operational parameters of the pelletizing process. The pellet diameter selection must be based on six main considerations:

- pelletizing efficiency,
- volume shrinkage during sintering,
- sintering time,
- coating ease,
- temperature gradients within the pellets, and
- waste loading of coated pellets.

Similarly, the desired pellet porosity must be determined with consideration of:

- pellet strength,
- thermal conductivity,
- coating efficiency,
- waste loading, and
- process limitations.

Larger pellets, while increasing waste loading by requiring less fractional coating, increase sintering time and contain larger temperature gradients within the pellets. Larger pellets require the use of a CVD drum coater for applying  $\text{Al}_2\text{O}_3$  coatings instead of a fluidized/vibrating bed coater as was used to apply both PyC and  $\text{Al}_2\text{O}_3$  coatings to particles 3 mm in diameter. Pellets with near zero porosity would have maximum strength, thermal conductivity, and the volumetric waste loading; however, CVD coating is simplified if there is a certain amount of porosity to allow for thermal

expansion differences of coatings and cores. A factor that must also be considered is the minimum porosity obtainable through the pelletizing and sintering process.

Glass or glaze coating is primarily a hand operation and would be difficult to automate. It would be ideal if a glass frit coating could be applied in the disc pelletizer as suggested in Figure 3. Development of this technique at this date has not been successful. A dipping operation using a glaze slip was used to produce samples for encapsulation demonstration. This technique would not be readily adaptable to remote facilities. No alternative processes have been identified for glass coating.

Optimization of coating thickness requires iterative evaluation of inertness and economics. Increasing coating thickness improves inertness but at the same time increases cost and coating time. Thermal conductivity and waste loading are decreased when coating thickness is increased.

Waste loading in coated pellets, defined as the fraction of waste in the entire pellet, varies with pellet diameter and coating thickness. The waste loading in uncoated supercalcine pellets is ~75 wt% with a pellet density of  $3.1 \text{ g/cm}^3$  (73% TD-theoretical density). For comparative purposes, Figure 10 shows the relationship between pellet diameter, coating thickness, and the weight of waste in a cylindrical canister 10 feet (304.8 cm) in length by 1 foot (30.5 cm) diameter for three waste forms. Waste loading of glass marbles, which contain no coating, is independent of marble diameter and has a value of 35 wt%. In all cases it is assumed the waste-form pellets are spherical and arranged in hexagonal close-packed configuration within the canister.

Matrix material selection is governed by numerous conditions and restrictions: 1) thermal conductivity, 2) waste loading, 3) maximum allowable temperature, 4) strength, 5) economics, 6) weight, 7) fabrication methods and 8) matrix/waste form interactions. The maximum temperature allowed within the canister is determined by the lowest temperature at which either the metal matrix begins to melt or the pellets and/or coatings begin to soften. Metal selection is closely dependent on canister sizing and waste loading. A higher waste loading per pellet gives rise to higher waste loadings per canister and

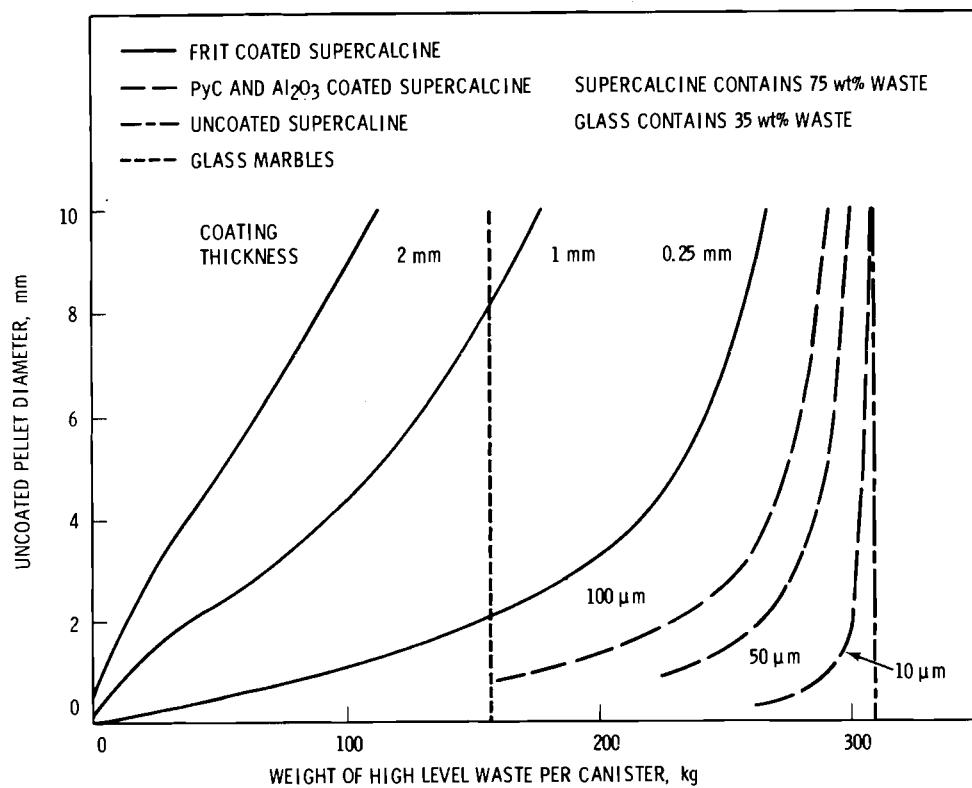


FIGURE 10. Waste Contained in a Canister 10 ft x 1 ft Diameter

subsequently higher volumetric heat generation. Larger heat generation dictates the need for smaller-diameter canisters, which lower waste loading and decrease temperature gradients, and/or high-melting temperature metals, and/or metals with higher thermal conductivities. The addition of strength, weight, and cost as selection considerations greatly complicates the optimization process.

## MATRIX OPTIMIZATION

Multibarrier waste forms development considers the basic waste forms previously discussed as a basis of optimization of process economics and enhanced inertness. PyC/Al<sub>2</sub>O<sub>3</sub>-coated supercalcine may be embedded in gravity-sintered metal matrices as a consequence of the higher waste loading and subsequent heat generation of supercalcine (75 wt% waste as compared to 35 wt% for glass). Employing gravity-sintered matrices allows the use of metals with melting temperatures greater than the temperatures expected within the canisters.

Factors other than the metal's melting temperature must be considered in matrix metal optimization and selection, such as thermal conductivity, strength, chemical stability, and cost. Although stainless steel has a melting temperature of  $\sim 1500^{\circ}\text{C}$ , its thermal conductivity is only 0.25 W/cm- $^{\circ}\text{C}$ , which decreases to 0.036 W/cm- $^{\circ}\text{C}$  in a sintered condition containing 50% porosity. Using this lower value for the matrix thermal conductivity, 0.0196 W/cm- $^{\circ}\text{C}$  is the calculated composite thermal conductivity of duplex-coated supercalcine in a gravity-sintered stainless steel matrix, assuming 60 vol% packing of supercalcine pellets. The centerline temperature in a 12-in. (30.5-cm) diameter canister of this waste form is  $834^{\circ}\text{C}$ . It was assumed that the canister walls were cooled by natural convection to air at  $30^{\circ}\text{C}$ . Using a gravity-sintered copper matrix with a thermal conductivity of 0.52 W/cm- $^{\circ}\text{C}$  would increase the composite thermal conductivity to 0.155 W/cm- $^{\circ}\text{C}$  and decrease the centerline temperature to  $161^{\circ}\text{C}$ , well below the melting temperature of copper ( $1083^{\circ}\text{C}$ ). Similarly, when incorporating the coated supercalcine cores in a cast aluminum matrix (composite thermal conductivity of 0.606 W/cm- $^{\circ}\text{C}$ ), the centerline temperature is decreased to  $71^{\circ}\text{C}$ , illustrating the fact that metals with high thermal conductivities permit the use of larger-diameter canisters, assuming the canister acts as an infinite cylinder with heat transfer along the radial direction.

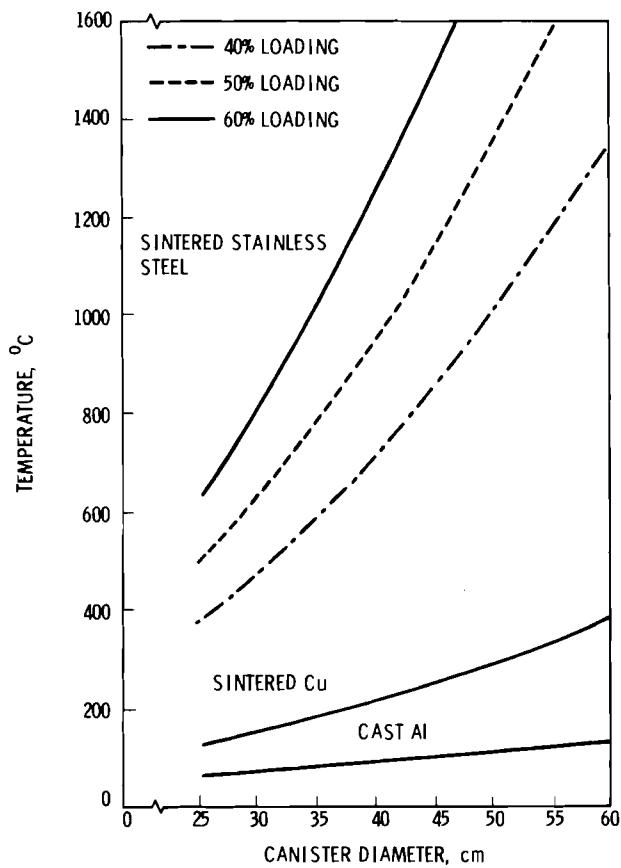
Another factor that has a major effect on canister size limitations is the volumetric waste loading. Reducing the volumetric waste loading of duplex-coated supercalcine from 60 vol% to 50 vol% in a gravity-sintered copper matrix

increases composite thermal conductivity to  $0.209 \text{ W/cm}^{-2}\text{C}$ , reduces volumetric heat generation, and decreases the centerline temperature of a 12-inch-diameter canister to  $116^{\circ}\text{C}$ . Further increases in thermal conductivity could be gained through the use of cast, 100% dense metals such as aluminum and copper alloys. Figure 11 shows the relationship of canister centerline temperatures to canister diameter for duplex-coated supercalcine in various matrices. The supercalcine cores have 4-mm diameters with 20- $\mu\text{m}$  PyC coatings and 60- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  coatings; sintered matrices are assumed to be 50% dense. Thus, it becomes apparent that achieving the greatest possible thermal conductivity through optimum volumetric waste form packing and a cast matrix allows greater waste loadings per canister based on relative heat generation and heat dissipation. For example, at a limiting temperature of  $250^{\circ}\text{C}$  for duplex-coated supercalcine in a gravity-sintered copper matrix, 50% more waste can be incorporated in a canister with 50 vol% loading than with 60 vol% loading because the former can use larger canisters, and because of the high conductivity and lower volumetric heat generation.

Improvement of the mechanical strength of the composite waste form is the second means of achieving the objective of the multibarrier concept. Composite waste forms, like all waste forms, must exhibit good impact strength in the event of an accident (during shipment to an ultimate disposal site). The overall strength of the composite, largely dependent on the strength of the metal matrix and canister, is difficult to optimize because of the complex nature of the final product and the effects of radioactive waste particles on the matrix. The overall strength of the composite, therefore, must be evaluated with respect to:

- waste-matrix geometry (volumetric waste loading),
- self heating of waste pellets,
- irradiation effects, and
- waste form/matrix/canister reactions.

The self-heating characteristic of the waste pellets may be advantageous or detrimental to the overall strength of the waste/metal composite. In extreme cases, enough heat may be generated to cause melting of the metal matrix or softening of waste glass marbles. Thermal expansion differences



**FIGURE 11.** Canister Centerline Temperatures for CVD-Coated Supercalcine in Various Matrices

between the waste pellets and the matrix may cause internal stresses in the pellets. Loss of strength may also occur as the temperature of the matrix reaches a point where annealing begins. Annealing softens metals by relieving stresses, removing defects, and creating larger grains--that is, allowing the metal to approach an equilibrium condition. Waste canisters could exhibit this behavior as a result of the self-contained, time-dependent heat generation.

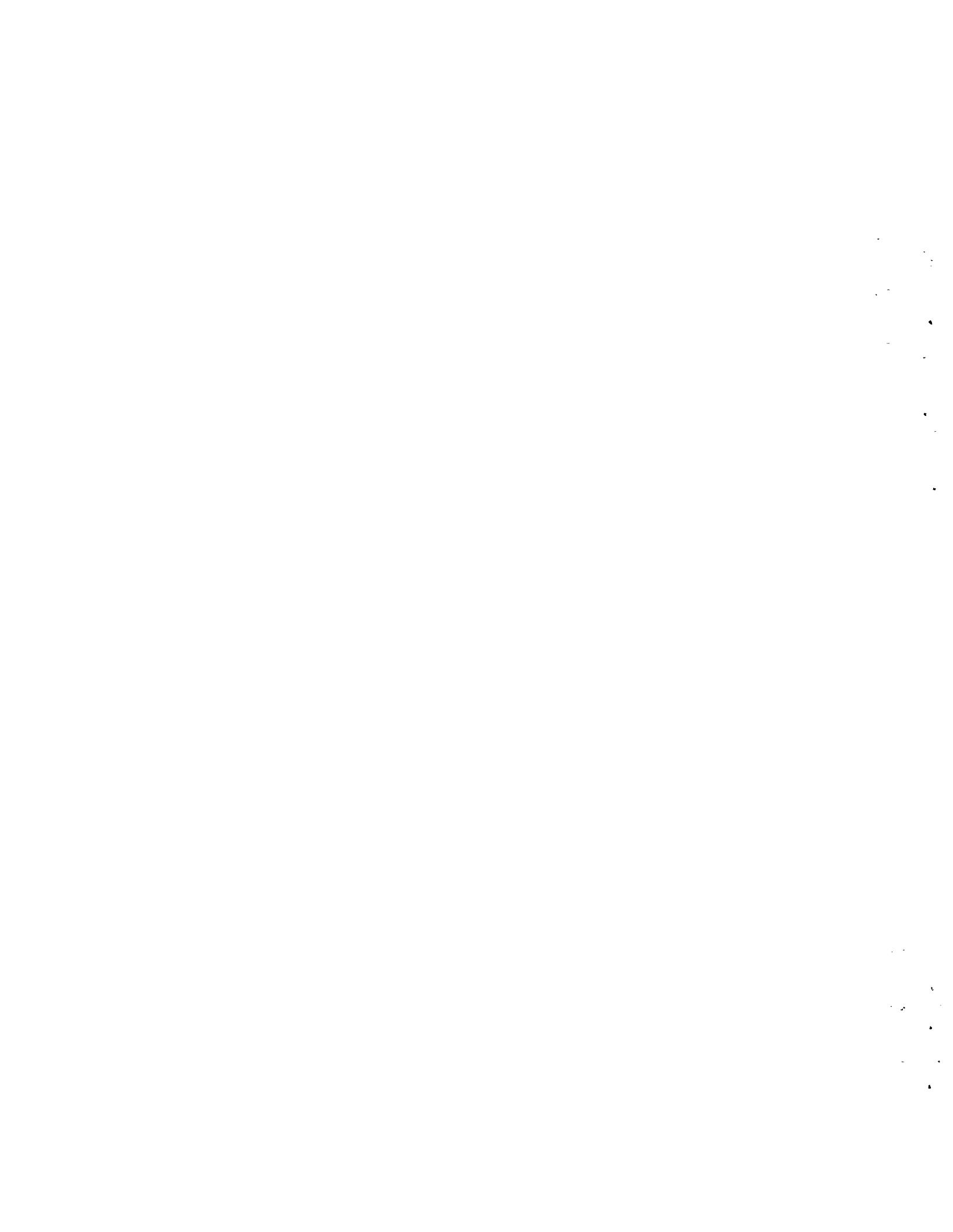
## CONCLUSIONS

In this preliminary study the process complexity of four multibarrier waste form concepts was found to increase thusly:

- Glass marbles (least complex)
- Uncoated supercalcine
- Glass-coated supercalcine
- PyC/Al<sub>2</sub>O<sub>3</sub>-coated supercalcine (most complex)

From earlier studies<sup>(2)</sup> it was found that inertness increases in a similar manner. It, therefore, becomes necessary to decide whether economics, complexity, or inertness is of greatest importance. Achieving enhanced inertness usually dictates increasing material costs, process costs, or both, in addition to increasing complexity. Selection of a multibarrier waste form will necessarily be based on a balance that considers the magnitude and relative importance of each factor with respect to the type of waste to be solidified.

Since these process considerations were limited to four multibarrier waste forms with commercial waste, additional studies must be conducted to determine their applicability to other types of waste and the remote adaptability and process costs. Alternative waste forms such as the above multibarrier concepts have received less development effort than glass; consequently further research and development is needed to define processing variables conducive to producing waste forms with improved properties.



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