QUARTERLY PROGRESS REPORT
RESEARCH AND DEVELOPMENT ACTIVITIES
FIXATION OF RADIOACTIVE RESIDUES
AUGUST, SEPTEMBER, OCTOBER, 1968

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Edited by
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<u>Quarter</u>	<u>Year</u>	<u>Report Number</u>	<u>Reference*</u>
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First		HW-60584	59-1
Second		HW-61635	59-2
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Fourth		HW-63949	59-4
	<u>1960</u>		
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Fourth		HW-72324	61-4
	<u>1962</u>		
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Second		HW-74375	62-2
Third		HW-75290	62-3
Fourth		HW-76262	62-4
	<u>1963</u>		
First		HW-77299	63-1
Second		HW-78188	63-2
Third		HW-79140	63-3
Fourth		HW-80526	63-4
	<u>1964</u>		
First		HW-81862	64-1
Second		HW-83320	64-2
Third		HW-84467	64-3
Fourth		HW-84603	64-4
	<u>1965</u>		
First		BNWL-76	65-1
Second		BNWL-139	65-2
Third		BNWL-170	65-3
Fourth		BNWL-213	65-4
	<u>1966</u>		
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Second		BNWL-306	66-2
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* Reference notation used in this report.

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TABLE OF CONTENTS

TABLES AND FIGURES	vii
INTRODUCTION	1
SUMMARY	1
SPRAY SOLIDIFICATION	3
LABORATORY STUDIES	3
Flowsheets for Processing PW-4m in the Spray Solidifier	3
Ruthenium Volatility Studies	4
Corrosion Behavior of Materials for High Temperature Melt Processing	6
ENGINEERING STUDIES	7
Development Spray Solidification Runs	7
New Freeze Valve Cooling Coil Assembly	11
RADIOACTIVE DEMONSTRATION	14
Spray Solidification Run SS-4	14
Spray Solidification Run SS-5	19
PHOSPHATE GLASS SOLIDIFICATION	24
LABORATORY STUDIES	24
Zircaloy-2 Corrosion	24
STUDIES ON AUXILIARIES TO SOLIDIFICATION SYSTEMS	24
Reaction Kinetics of Sucrose Added to the Evaporator	24
EVALUATION OF SOLIDIFIED WASTE PRODUCTS	26
NONRADIOACTIVE LAB STUDIES	26
Study of the High Temperature Storage of Solidified Waste Products Containing Sulfate	26
Product Characterization - Continuation of Labo- ratory Evaluation of PW-2 Phosphate Glass Samples	26
PRODUCT MEASUREMENTS, TESTING AND STORAGE	33
Solids Storage Engineering Test Facility	33

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INTRODUCTION

This progress report is the thirty-eighth in a series presenting research and development activities in the field of radioactive wastes.

Experimental work charged to programs other than those of the Division of Reactor Development and Technology is sometimes included for general interest and completeness; such work is identified in the headings.

SUMMARY

Spray solidification Run SS-4 was completed with 500 liters of sulfate containing PW-2 waste being reduced to 56 liters of ceramic solid in 40.5 hours. The 1,300,000 curies of fission product activity interfused in the solidified waste produced an internal heat generation rate of about 5400 watts. Operation of the spray solidification systems was quite smooth, and all the equipment performed satisfactorily. The temporary loss of the mount for the calciner vibrator resulted in an interruption of the run, but a new floating-type vibrator was placed on the calciner and the run was successfully completed.

A new method has been developed to control the discharge of melt from the spray solidifier melter. The melt in the discharge valves is solidified by an air stream. To discharge the melt, the cooling air is turned off and the heat from the melter furnace thaws the material in the freeze valve. This method of melt discharge was successfully used during spray solidification Run SS-5. During SS-5, 887 liters of sulfate-free PW-1 waste, containing 2,000,000 curies of radioactivity, was successfully reduced to 66 liters of ceramic solid in 53.2 hours. All spray solidifier systems performed satisfactorily.

Investigation of PW-4m feed formulations indicates that a phosphate rather than a borosilicate flowsheet will be favored for processing PW-4m in the spray solidifier. Among reasons for this are that rare earth precipitates form in the PW-4m borosilicate melts and extreme difficulty is encountered in preparing and handling the borosilicate feeds.

In a PW-4m run with the development spray calciner, a PW-4m flowsheet was found suitable for spray solidification when the borate flux was added separately as a solid to the melter rather than including it in the calciner feed.

SPRAY SOLIDIFICATIONLABORATORY STUDIESFlowsheets for Processing PW-4m in the Spray Solidifier -
J. E. Mendel

A large number of nonradioactive PW-4m melts of the borosilicate type were prepared. Although the laboratory investigation is continuing, present indications are that a phosphate rather than borosilicate flowsheet will be favored for processing PW-4m in the spray solidifier. Some reasons for this conclusion are:

- Rare earth precipitates were found in most of the PW-4m borosilicate melts.
- Feed preparation and handling are more difficult for the borosilicate than for the phosphate flowsheet.
- Borosilicate products were more leachable than phosphate products.

The range of additives used in preparing the PW-4m borosilicate melts included:

<u>Additive</u>	<u>Concentration Range, M^(a)</u>
Boron	3.0-9.0
Silicon	0-2.0
Alkali metal (Li, Na, or K)	0-3.0
Alkaline earth (Ca or Ba)	0-1.6
Other cations (Al, Fe, or Mo)	0-0.6

a. Moles per liter at 378 liter/tonne

It is noteworthy that all of the 69 compositions tested produced mobile melts at temperatures ranging from 850 to 1100 °C. In contrast, experience with the phosphate system has shown that comparatively greater restrictions on composition are necessary in order to obtain fluid melts in this temperature range.

A basic problem with the PW-4m borosilicate melts was non-homogeneity in the form of a precipitate containing rare earths. PW-4m contains 0.274M fission product rare earths. The precipitate was heavier than the bulk glassy phase and collected at the bottom of the melt. Segregation of this rare earth enriched phase in the waste storage containers would probably be undesirable, and efforts to define a PW-4m borosilicate composition range which totally avoids the precipitate were unsuccessful.

Feed solutions were prepared by dissolving the required amount of B_2O_3 in the required amount of alkali metal hydroxide and adding PW-4m to this solution. Silicon, when used, was added as SiO_2 flour and the other cations as nitrates. The feed solutions were viscous and somewhat gelatinous and proved difficult to handle in the engineering-scale equipment.

The flowsheet that will be used for the first phosphate-fluxed PW-4m spray solidifier runs will be related to the PW-1 flowsheet, which has proven quite troublefree. The additives will be 1.2M Fe, 0.75M Li, 0.75M Na and 2.70M PO_4 (where M equals moles per liter of PW-4m at 378 liter/tonne). Although the melt properties are similar to the PW-1 product, the fission product content is higher, 24.7 wt% in PW-4m product versus 17.8 wt% in PW-1 product.

Ruthenium Volatility Studies - D. W. Brown

One series of feeds was calcined at 375 °C to determine the effect of the neutralization of the feed on ruthenium volatility. A PW-2 spray solidifier flowsheet feed with 0.016M Ru was used and the acid was neutralized with sodium hydroxide. Figure 1 is a summary of the data. A continued increase in ruthenium retention occurs as the percent of nitric acid neutralized is increased. If 100% excess sodium hydroxide is added (200% neutralized) less than 1% of the ruthenium is volatilized.

FIGURE 1. Neutralization of PW-2 Acid with Sodium Hydroxide and Its Effect on Ruthenium Volatility

A second set of PW-2 spray solidifier feeds, 0.008M Ru, were calcined to determine the effect of sugar in the feed solution on ruthenium volatility. This method also successfully reduced the ruthenium volatility as shown by the following data:

<u>Sugar in Feed, M</u>	<u>% Ruthenium Volatilized</u>
0.0	23
0.26	14
0.51	2.7

No direct comparison can be made between the two sets of data because the two base feed solutions were of different ruthenium concentrations. Additional tests are planned so that a comparison can be made.

Corrosion Behavior of Materials for High Temperature

Melt Processing - R. F. Maness

The corrosion behavior, as well as the thermal and mechanical shock resistance, of Haynes Metal Ceramic LT-1 (77 Cr-23 Al₂O₃) and of chromium (both deposited by flame spraying) was determined during the formation of phosphate glass and spray solidifier type PW-1 melts. Melts were formed at 1000 °C in containers of the two materials. Corrosion after 100 exposure hours was not excessive as evidenced by visual observation and the containers, partially filled with melt, retained their integrity when cycled between 1000 °C and room temperature. However, corrosion of both materials was severe at 1150 °C. Specimens of Hastelloy D* and Duriron** corroded severely when exposed to PW-1 spray solidifier melt at 1050 °C.

A 50 Cr-50 Ni alloy specimen exposed to PW-1 phosphate glass melt at 900 °C for 80 hours corroded at a rate of about 240 mils/year. PW-1 and PW-2 spray calciner melts both corroded the alloy at a rate of about 100 mils/year. Corrosion rates of the 50 Cr-50 Ni alloy at 800 °C in PW-1 spray solidifier and phosphate glass type melts, and in PW-2 spray solidifier melt, were 7, 17, and 60 mils/year, respectively, as determined in a 145 hour test. The alloy may be useful as a material of construction for thermocouple wells in containers containing melts with molten cores. One-eighth-inch thick specimens of 310 stainless steel, Hastelloy X,[†] and Inconel 600^{††} exposed to PW-2 spray calciner melt at 900 °C were consumed after 165 exposure hours.

* Hastelloy D is a registered trademark of the Union Carbide Corp.

** Duriron is a registered trademark of the Duriron Co.

† Hastelloy X is a registered trademark of the Union Carbide Corp.

†† Inconel 600 is a registered trademark of the International Nickel Co, Inc.

ENGINEERING STUDIES

Development Spray Solidification Runs - J. D. Moore

Three runs with the cold semiworks spray solidifier were made during the quarter. Run DSC-19 was made to produce 100 pounds of simulated PW-1 calcine for Oak Ridge National Laboratory. The calcine will be used as base material for development of high thermal conductivity glass for solidification of fast reactor fuel reprocessing wastes. Runs DSC-20 and 21 tested simulated PW-4m with borate flux in an attempt to develop a feed composition from this waste type suitable for spray solidifier operation. PW-4m, containing 1.5M Li and B_2O_3 added as a solid flux to the melter in an equivalent concentration of 3.0M (based on 378 liters/tonne feed), provides a workable feed which forms a glass product containing greater than 95% of the feed ruthenium.

A. Run DSC-19

The feed rate and concentration were varied in DSC-19. For the first 9 hours of the run, an average rate of 15 liters/hour was maintained with PW-1 feed concentrated to 303 liters/tonne (see Table I). For the last 7.7 hours, 227 liters/tonne feed was spray-dried at an average rate of 18 liters/hour to hasten completion of the run. The calciner internal temperatures indicated very smooth operation. With the calciner furnace temperature at 700 °C, the internal temperatures were 15 to 20 °C cooler at 18 liters/hour than at 15 liters/hour. The equilibrium calciner filter pressure drop was 13% higher (5.75 versus 6.5 inches of water) at the higher feed rate. Particulate decontamination factors (DF) remained relatively constant from 500 to 600 throughout the run.

Calcine product was collected in an unheated stainless steel drum connected to the calciner outlet. A total of 120 pounds of fine, dry homogeneous, brown powder was produced.

TABLE I. Summary of Development Spray Solidification Runs

	DSC-19		DSC-20	DSC-21
	<u>Part 1</u>	<u>Part 2</u>		
Feed Type	PW-1	PW-1	PW-4m	PW-4m
Feed Concentration, liters/tonne	303	227	378	447
Additives	none	none	Li ⁺ , SiO ₂ to liquid B ₂ O ₃ to melter	Li ⁺ , B ₂ O ₃ to liquid
Feed Consumed, liters	136	137	48	10 (estimate)
Average Feed Rate, liters/hr	15	18	11.4	11 (estimate)
Calciner Furnace Temperature, °C	700	700	700	700
Calciner Vacuum, inches of water	5	5	7	10
Atomizing Gas	Air	Air	Air	Air
Atomizing Gas Flow, scfm	6	5.6	6	6.5
Filter Blowback Pressure, psig	40	40	30	30
Filter Pressure Drop, End of Run, inches of water	5.75	6.5	4.3	2.5
Melter Furnace Temperature, °C	--	--	1000-1100	--
Product Receiver Furnace Temperature, °C	--	--	800	800
Product Collected, kg	23.4	31.1	16.8	2 (estimate)
Product Density, grams/cm ³	--	--	2.6	3.2
Particulate DF (Feed to Condensate)	600	500	360	3300
Ruthenium Retention in Product, %	<5	<5	>95	not determined

The calciner internal walls were relatively clean with no deposits exceeding 1/8-inch thickness.

A ruthenium material balance gave a poor recovery as less than 5% of the feed ruthenium was found in the product and only 13% was found in the condensate and scrub solution.

B. Runs DSC-20 and 21

Runs DSC-20 and 21 were made to compare the spray solidifier performance with PW-4m feed with B_2O_3 flux added as a solid to the melter during DSC-20 and added to the liquid feed to the calciner during DSC-21. Feed for DSC-20 (see Table II) was simulated 378 liters/tonne PW-4m with 1.5M Li and 0.8M silica added to the liquid feed, and with 3.0M B_2O_3 added in crystalline form to the melter. For DSC-21, the silica was omitted since it caused severe wear to the feed pump bearings in the previous run. Its presence in the feed was to improve product solubility, but it was found to have had little effect. The B_2O_3 was added to the liquid feed in DSC-21, but it did not completely dissolve and a heavy crystalline precipitate formed when the feed was cooled below 50 °C.

The solid B_2O_3 addition mode proved to be more successful than the liquid addition. In DSC-21, with the flux in the liquid feed, the feed formed a thick paste in the storage tank after about 55 minutes of spray solidifier operation. The paste was formed when the feed was gradually cooled to 40 °C while circulating through the pump loop back to the tank. The handling problems with this feed makes it infeasible for further testing at this time.

Silica was the only insoluble precipitate in the feed for DSC-20 and without it the feed would probably have run smoothly. As the silica began to wear the pump bearings, the feed line pressure oscillated between 25 and 45 psig (normal setting was 35 psig). These pressure fluctuations produced large droplets

TABLE II. Feed Compositions for Development Spray Solidifier Runs
(Bases: 378 liters/tonne Concentration)

	DSC-19	DSC-20	DSC-21
Waste Type	PW-1	PW-4m	PW-4m
H ⁺	3.52M	4.81M	4.81M
Fe ⁺³	0.959	0.124	0.124
Cr ⁺³	0.012	0.012	0.012
Ni ⁺²	0.005	0.008	0.008
Al ⁺³	0.001	0.001	0.001
ZrO ⁺²	0.065	0.143	0.143
Na ⁺	0.138	0.342	0.342
SiO ₃ ⁻²	0.010	0.010	0.010
NO ₃ ⁻	7.5	6.66	6.66
PO ₄ ⁻³	0.003	0.003	0.003
MoO ₄ ⁻²	0.065	0.13	0.13
Sr ⁺²	0.0155	0.036	0.036
Ba ⁺²	0.0195	0.041	0.041
Rb + Cs (used K ⁺)	0.0416	0.092	0.092
(Y + RE) ⁺³	0.12	0.274	0.274
Ru ⁺⁴	0.0032	0.0082	0.0082
Rh (used Co ⁺²)	0.0074	0.013	0.013
Ag ⁺	0.0008 (used Cu ⁺²)	0.0016	0.0016
Cd ⁺²	0.0008 (used Cu ⁺²)	0.0025	0.0025
Te (used SO ₄ ⁻²)	0.0064	0.014	0.014
Tc	0.014 (used Mn ⁺²)	0.031 (used MoO ₄ ⁻²)	0.031 (used MoO ₄ ⁻²)
Pd (used Ni ⁺²)	0.0172	0.043	0.043
<u>Additives</u>			
LiOH		1.258	1.258
SiO ₂		0.8	--
B ₂ O ₃		3.0 (solid to melter)	3.0

in the feed atomization pattern which formed calcine deposits up to 3/4-inch thick in the calciner cone below the spray nozzle. Sixty-mesh B₂O₃ crystals were added to the melter through a nozzle in the calciner cone. Moisture condensation in the crystals at the nozzle outlet plugged the addition port after 2 hours. For the remaining 2 hours of the run, solids were added manually through a port in the melter top. The solids plug could have been prevented by continuously purging the solids addition line with heated air.

The calciner filter pressure drop was relatively low in both runs, although longer duration runs would have yielded more useful data.

Melt from DSC-20 was a homogeneous dark olive-green glass with a density of 2.6 g/cm^3 and a melting point of 850°C . Product from DSC-21 contained small light-colored inclusions. It melted at 950°C and had a density of 3.2 g/cm^3 . The higher density and melting point, and crystalline structure was due to a borate deficiency in the melt. This occurred because of borate feed handling problems described above.

Greater than 95% of the feed ruthenium was retained in the melt from DSC-20. Since DSC-21 was so short, no ruthenium analytical data was obtained.

New Freeze Valve Cooling Coil Assembly - J. D. Kaser

A new method has been developed to control the discharge of melt from the spray solidifier melter. The melt in the valves is solidified by air cooling coils. To discharge the melt, the cooling air is turned off to either the weir overflow valve or the drain valve and the heat from the melter furnace thaws the material in the valves. A complete cooling coil assembly has been fabricated, tested, and installed in B-Cell.

Initial operating experience including its successful use in WSEP run SS-5 has shown that this method bears high promise as a simple and reliable replacement for the previously used electric heaters. The electric heater system had experienced an excessive failure rate and consequently this change should provide a significant improvement in the reliability and operability of the solidifier system.

Figure 2 shows a top view of the cooling coil assembly. The coils were wound from 1/4 inch outside diameter Hastelloy X* tubing. The coils are as long as possible without touching any part of the melter, with the valve tips extending about 1 inch below the coils. The cooling coil for the drain valve is 4 inches long and the coil for the overflow weir valve is

* Haynes Stellite Corporation

Neg 0683606-3

FIGURE 2. Top View of Freeze Valve Cooling Coil Assembly

2 inches long. The coils have an inside diameter of 1 inch and are wound without gaps between turns. The cooling air flows from the bottom to the top of the coils and is then exhausted from the assembly.

The coils are not intended to freeze the melt in the valves while the melter is draining rapidly. Rather, the coils seal the valves after flow has stopped. At the beginning of a run, the slow trickle of melt into the cooled valves forms the initial seal.

The outside of one coil is insulated with 1/2 inch thick Cerafelt* covered with platinum foil. This insulation reduces the interaction between cooling coils. Without insulation, cooling air flowing in one coil may freeze both valves. The coil assembly is surrounded by 1/2 to 1 inch thick Cerafelt to minimize heat loss from the valves when the cooling air is off. Insulation below the coils (not shown) is supported by the stainless steel plate which is shown. The melt falls through the large holes in the plate. To further reduce heat loss the entire assembly was lined with platinum foil (not shown).

Experience has revealed that molten waste will rapidly corrode Hastelloy X at the operating temperatures of the melter (900 to 1200 °C) if metal and melt come in contact. Furthermore, the platinum valves may be attacked if the valves, the melt, and the alloy coil are all in contact with each other. To prevent such contact, a platinum wire cage is suspended inside each coil.

After the assembly was fabricated, the temperatures at the valve tips were measured both inside and outside the valve at various cooling air rates. The thermocouples measuring the temperatures outside the valves were permanently installed for control purposes. Tests were run at two temperatures: 920 °C, corresponding to PW-2 operation, and 1150 °C, corresponding to PW-1 and PW-4 operation. The test results are summarized in

* *Johns-Manville, Inc.*

Table III. These tests show the decreasing valve temperatures with increasing cooling air flow, and the moderate interaction between coils. The valve tips must be above 750 °C to thaw PW-2 waste and above 925 °C to thaw PW-1 waste. In actual operation (WSEP Run SS-5), the valve tip temperature ran about 50 °C higher than the test data at the same operating conditions. This difference may be attributed to the platinum foil lining and to the fact that during the tests the coils radiated heat through the drain holes at the bottom of the assembly to a cold laboratory floor, while in actual operation the coils see the inside of the receiver pot which is about 700 °C.

*TABLE III. Final Tests of Freeze Valve Cooling Coil Assembly
(Assembly not Lined with Platinum Foil)*

Furnace Temp, °C	Drain Valve Tip, °C		Weir Tip, °C		Air Flow, scfm	
	Inside	Outside	Inside	Outside	Drain Valve	Weir
920	750	750	750	760	0	0
	730	730	675	690	0	0.7
	725	725	645	665	0	1.3
	670	670	730	<600	0.7	0
	<600	600	705	<600	1.3	0
1150	980	990	975	985	0	0
	960	960	875	900	0	0.7
	950	950	845	885	0	1.3
	900	920	950	960	0.7	0
	800	850	920	930	1.3	0

RADIOACTIVE DEMONSTRATION

Spray Solidification Run SS-4 - W. R. Bond, M. R. Schwab,
and J. L. McElroy

The fourth radioactive demonstration of the spray solidification process was successfully completed with Run SS-4, WSEP Run-16. During SS-4, 500 liters of PW-2 waste at 560 liters/tonne were reduced to 56 liters of calcine in 40.5 hours. The 1,300,000 curies of fission product

activity (including 3,500 curies of ruthenium) in the solidified waste produced an internal heat generation rate of about 5400 watts. The centerline temperature of the 8-inch diameter mild steel receiver in the air-cooled furnace was 510 °C with a pot wall temperature of 290 °C. The 220 °C temperature drop gave an effective thermal conductivity of $1.1 \text{ W/(m}^2)(\text{°C/m})$ [$0.66 \text{ Btu/(hr)(ft}^2)(\text{°F/ft})$] for the solidified waste product. Table IV presents a summary of the data for Run SS-4.

The operation of the spray solidification systems was quite smooth. The principal process factors (atomization, filter operation, flow control) performed very satisfactorily. A new two-filter blowback assembly (two filters blown back simultaneously) produced much lower pressure surges than observed in previous runs with a three-filter blowback arrangement. Air blowback was used during Run SS-4 instead of steam due to a continuous air blowback on one filter bank caused by a solenoid valve becoming locked open. A new vent line between the process condenser and the auxiliary evaporator provided ample vacuum in the solidifier even with the continual air blowback.

The only difficulty which occurred during SS-4 was a failure of the mounting of the vibrator on the spray calciner (the welds holding the base of the mounting to the spray calciner broke loose). This necessitated an interruption of the run while a floating-type vibrator was fabricated and placed on the calciner.

The batch dumping of the molten product into the unheated receiving pot was completed without stalagmite formation. The melter was dumped about every three hours by heating the freeze valve with a propane torch. An interesting feature of the PW-2 product was the sensitivity of the flux-to-feed addition ratio on the sulfate volatility. Each time the melter was dumped, sulfate fumes were observed coming from the flowing product.

TABLE IV. Summary Data for WSEP Spray Solidifier Runs

Operational Data	SS-4	SS-5
Solidifier Run/WSEP Run	4/16	5/17
Solidifier	Spray	Spray
Date	9/68	10/68
Operating Mode	A	A
Feed Type	PW-2 (a)	PW-1
Feed, liters/tonne	560	446
Pot Diameter, in.	8	8
Pot Material	Mild Stl.	Mild Stl.
Total Run Feed Time, hr	40.5	53.2
Average Feed Rate, liters/hr	12.3	16.7
Total Feed, liters	500	887
Total Feed, tonne equivalent	0.89	2.0
Feed: Solid Volume Ratio	8.9	13.3
Feed: Solid Volume Ratio at 378 liters/tonne Basis	6.0	11.4
Filter ΔP , inches of Water	7.0 (b)	6.0

Problems

SS-4 Weld on vibrator mounting plate failed; new unit installed and run continued.

SS-5 Power lead to melter furnace lost insulation and began arcing; insulation replaced with 3 1/2 hr delay in run.

Filled Pot Data

Total Curies in Feed to Pot	1,300,000 (c)	2,000,000 (d)
Total Curies of Ru in Feed to Pot	3500 (c)	17,000 (d)
Solid in Pot, liters	56	66
Solid in Pot, kg	160 (e)	214 (f)
Solid in Pot, liters/tonne	63	33
Heat in Pot, W(g)	5400	8200
Equivalent Aging Time, years	0.8	1.1
$\bar{C}$ Temp in Air-Cooled Furnace, $^{\circ}\text{C}$	510	633
$\bar{C}$ to Wall Δ Temp., $^{\circ}\text{C}$	218	278
Effective Thermal Conductivity, $\text{W}/(\text{m}^2)(^{\circ}\text{C}/\text{m})[\text{Btu}/(\text{hr})(\text{ft}^2)(^{\circ}\text{F}/\text{ft})]$	1.1 [0.66]	1.1 [0.64]
Welded Lid	Yes	Yes
Radiation Reading, R/hr at 6 in.	3.8×10^4	—

- a. See Table V for feed composition.
- b. Approximately 3 inches of ΔP was due to locked open blowback solenoid.
- c. By melt analysis
- d. By feed analysis
- e. Calculated from lab density of 2.84 kg/liter and roddeed volume in pot.
- f. Calculated from lab density of 3.24 kg/liter and roddeed volume in pot.
- g. By feed volume ratio; actual value pending pot calorimetry data.

The flux (sodium hexametaphosphate) was added batchwise as a solid to the melter every half hour to maintain a flux-to-feed ratio of 87 grams NaPO_3 per liter of feed at 555 liters/tonne. The sulfate fumes became very dense each time the flux-to-feed ratio was allowed to exceed the nominal 87 g/liter, namely up to 90 g/liter. Conversely, the least sulfate fuming occurred for a flux-to-feed ratio of 85 g/liter. While the flux is required to lower the melting point of the calcine, the melting point did not appear to be as sensitive to the flux-to-feed ratio as was the sulfate volatility.

The surface of the solid product was smooth and level on inspection of the receiving pot following the run.

The feed composition for Run SS-4 is given in Table V. The feed differed from a standard PW-2 waste formulation in the amount of aluminum present. The excess of aluminum in the Purex 1WW waste solution used to prepare the feed was used for 13% of the iron requirements in the PW-2 feed. The aluminum replaced 0.059M Fe of the total iron requirement of 0.475M (including 0.0288M Fe substituted for ruthenium) at 378 liters/tonne.

The distribution of the feed radioactivity following Run SS-4 is given in Table VI. About 11% of the radio-ruthenium fed to the solidifier was found in the auxiliaries following the run. While the 11% ruthenium volatility value agreed with stream sample data from the process condenser, only 23% additional ruthenium was found (by melt analysis) in the product. The remaining 66% of the radioruthenium fed to the solidifier was not found although additional laboratory analyses are pending.

Instantaneous decontamination factors (DF) were determined for ruthenium across the WSEP auxiliary equipment. Instantaneous ruthenium DF's for the evaporator to the evaporator overheads ranged from 500 to 2500. Instantaneous ruthenium DF's for the fractionator to the fractionator overheads ranged from 10 to 30.

TABLE V. PW-2 Feed Composition for Run SS-4

Component	Molarity, M ^(a)	Lab Analysis, M ^(a)
H ⁺	----	5.5
Fe ⁺³	0.445	0.415 ^(d)
Cr ⁺³	0.024	
Ni ⁺²	0.01	
Al ⁺³	0.001	
Na ⁺¹	0.93	1.02
U ⁺⁶	0.01	
NO ₂ ⁻¹ -NO ₃ ⁻¹	5.37	7.14 ^(b)
SO ₄ ⁻²	0.87	0.66 ^(c)
PO ₄ ⁻³	0.006	
SiO ₃ ⁻²	0.01	
Cs ⁺ (added K ⁺)	0.042	
Sr ⁺²	0.0155	
Ba ⁺²	0.0195	
(Y + RE) ⁺³	0.120	0.23 ^(g)
ZrO ⁺²	0.065	
MoO ₄ ⁻² + Tc as MoO ₄ ⁻²	0.079	
Ru ⁺⁴	0.0032	0.0020
Ru ⁺⁴ (added Fe ⁺³)	0.0288	(d)
Rh ⁺² (added Co ⁺²)	0.0074	
Pd ⁺² (added Ni ⁺²)	0.0172	
Ag ⁺¹ + Cd ⁺² (added Cu ⁺²)	0.0024	
TeO ₄ ⁻² (added SO ₄ ⁻²)	0.0064	
Additives to Feed		
Al ⁺³	0.25	0.31 ^(d)
Ca ⁺²	0.60	
Li ⁺¹ (as hydroxide)	1.17	
Na ⁺¹ (added as NaPO ₃ solid directly to melter)	1.27	
PO ₄ ⁻³ (total including 1.61 as H ₃ PO ₄)	2.88	1.64 (in feed)
Final Feed		
Volume, liters		569
Waste Equivalents		1.01
[M _T ⁺ / for Feed (for melt; includes NaPO ₃ additive)	6.11 (7.38)	
[M _T ⁺ /P for Feed (for melt; includes NaPO ₃ additive)	3.80 (2.56)	
Sp Gr at 25 °C		1.36
% Solids		30
Total Radioactivity, Ci/liter ^(e)		3200
Radioruthenium, Ci/liter ^(e)		31
Total Heat, W/liter ^(f)		11

a. Molarities are at 378 liters/tonne basis.

b. Includes nitrate from additives.

c. Presence of insoluble sulfates (probably calcium sulfate) gives lower value than known to be actually present.

d. Excess aluminum in IWW waste replaces 0.059M Fe in the total iron requirement of 0.475M (including 0.0288M Fe substituted for ruthenium).

e. By feed analysis. Radioactivity in product was determined by analysis of the product.

f. As determined by calorimetry measurement in feed tank.

g. Rare earth analysis has history of being off as much as a factor of 2.

TABLE VI. Activity Distribution for Run SS-4 (Mode A Operation)*

	Fraction of Radioactivity in Condensate				
	Ru	CePr	Cs	ZrNb	Total less Ru
Through Process Condenser (E-111)	1.1×10^{-1}	1.2×10^{-3}	1.4×10^{-2}	2.5×10^{-3}	1.2×10^{-3}
Through Evaporator Condenser (E-113)	7.4×10^{-4}	8.5×10^{-7}	5.1×10^{-5}	1.2×10^{-6}	9.4×10^{-7}
Through Fractionator Condenser (E-115)	2.7×10^{-5}	3.1×10^{-9}	3.9×10^{-8}	----	3.1×10^{-9}
Off-Gas to Scrubber	3.0×10^{-4}	7.4×10^{-10}	1.1×10^{-7}	----	1.5×10^{-9}
Off-Gas to Stack	----	----	----	----	1.0×10^{-12}
Ratio (Fract. Cond.) Activity to 10CFR20	77,000	570	14	----	----

* BNWL-507, 67-2

Spray Solidification Run SS-5 - W. R. Bond and J. N. Hartley

The seventeenth WSEP pilot plant demonstration of the solidification of high level radioactive waste was successfully completed with the fifth spray solidification Run SS-5. During Run SS-5, waste equivalent to 2.0 tonnes of power reactor fuel (20,000 MWd/tonne at 15 MW/tonne) with an equivalent aging time of 1.1 years was processed to produce 214 kilograms of ceramic product. The 66 liters of radioactive ceramic produced a heat rate density of 125 W/liter (8200 watts total based on feed analytical data) in an 8-inch diameter 304-L stainless steel melt receiver. The steady-state pot centerline temperature in the air-cooled furnace was 633 °C, and the centerline-to-wall temperature difference was 278 °C. These values gave an effective thermal conductivity of $1.1 \text{ W/(m}^2)(\text{°C/m})$ [$0.64 \text{ Btu/(hr)(ft}^2)(\text{°F/ft})$] for the ceramic product. Preliminary data for Run SS-5 is presented in Table IV.

The calciner was fed 887 liters of adjusted sulfate-free PW-1 feed (additives of 0.5M LiOH, 0.5M NaOH, and 1.71M H_3PO_4 based on 378 liters/tonne) at 446 liters/tonne in 53.2 hours at an average feed rate of 16.7 liters/hr. The feed contained 2,000,000 curies of radioactivity, including 17,000 curies of radioactive ruthenium, and was reduced in volume by a factor of 13.3 (11.4 on a 378 liters/tonne basis).

A new freeze valve cooling assembly (see Engineering Studies - New Freeze Valve Cooling Coil Assembly) was used throughout the run. The melter was batch dumped through the drain valve eleven times (about every five hours). The drain valve began to flow about two minutes after the air was turned off to the cooling coils around the valve. The use of air-cooled coils was an effective means of sealing the drain valve following each batch dump of the melt to the receiver.

Additional information on Run SS-5 will be reported in the next quarterly report.

Off-Gas Filter Failure - W. R. Bond

The spray solidifier off-gas filters were removed from the unit following Run SS-3 for inspection as an appreciable quantity of calcine (11%) passed the filters during the run. It was found that 2 of the 15 sintered powder metal (316-L SST) filters had failed. Both failures occurred just below the top circumferential weld (manufacturer's weld) which joins the porous metal to a solid collar. The collar attaches the filters to venturis through which the off-gases exit and back through which a pulse of superheated steam is directed periodically for filter cleaning. The filters failed after 400 hours of service (feed-on time). They were visually inspected after the first 300 hours and appeared in good condition at that time. One filter had a 2 inch long by 1/32 inch wide crack in the porous metal along the circumference in the weld-affected zone. A 1 to 1-1/2 inch section of porous metal was missing from the top of the second filter. The porous metal 1 to 2 inches directly below this failure appeared pitted as though numerous metal particles were missing. A sample of the porous metal (Figure 3) taken in this area was very crumbly and possessed no ductility. Another sample (Figure 4) taken 4 to 5 inches below this point was not pitted and possessed some ductility. Both samples were sent to Radiometallurgy for metallographic examination. Although the examination revealed that

Sample as Received

Neg E6480

2.5X

*Cross Section of Sample
as Etched*

Neg E6478

25X

*Cross Section of Sample
as Etched*

Neg E6470

125X

FIGURE 3. *Sample of Porous Metal Filter Taken at the Failed Area*

Samples as Received

Neg E6471

2.5X

*Cross Section of Sample
as Etched*

Neg E6435

25X

*Cross Section of Sample
as Etched*

Neg E6486

125X

FIGURE 4. *Sample of Porous Metal Filter Taken 4 to 5 Inches Below the Failed Area*

an appreciable amount of metal particles were missing from the sample taken near the failure, signs of corrosion of the remaining particles were no more pronounced than on the other sample which was considerably more intact. To date, a combination of mechanical fatigue and corrosion is suspected as the cause of the failure. Fatigue of the top of the filter could result from a pivoting action about the porous metal to solid metal weld caused by vibration. A filter manufacturer was contacted and it was recommended that a perforated support plate be installed on the inside of the filters to minimize the mechanical stressing of the porous metal. Materials of construction that may be more resistant to corrosion than 316-L SST are also being considered. The replacement set of filters now in the calciner was an existing spare set and as such is similar in most respects to the original filters.

Computer Data Logging

A GE 412 computer has been used successfully during the report period by Engineering Demonstration personnel to assist in the execution of experimental run plans and in the post-run evaluation of these experiments. The computer has been valuable in calculating and logging routine data associated with both pre-startup activities and normal operating periods when operating personnel time was more beneficially used for other activities.

The 412 has been programmed to continuously monitor many of the process sensors of the WSEP facility, calculating and logging specific values of interest, and alarming out-of-limit conditions. Data was logged either at 15 minute intervals or on demand with typewriters or paper tape perforators. Alarming was accomplished with typewritten messages. Typewriter logged data were expressed in convenient engineering units that were directly usable by operators for controlling the progress of the experiment or by engineers

evaluating the experiment. Additional data were logged with paper tape perforators for later evaluation by engineering personnel using electronic data processing facilities. When this technique was used, certain data were machine plotted, eliminating the time consuming process of manually calculating and hand plotting of these data.

Typical logged data included:

- 1) Feed solution specific gravity, volume and dropout rate.
- 2) Auxiliary train volumes, specific gravities, temperatures, acid concentrations and flow rates for acid material balance calculations.
- 3) Furnace power consumption.
- 4) Material balance calculations.
- 5) Clock time and elapsed run time.

PHOSPHATE GLASS SOLIDIFICATION

LABORATORY STUDIES

Zircaloy-2 Corrosion - R. F. Maness

Zircaloy-2 was evaluated as a material of construction for containing PW-1 and PW-2 phosphate glass feed solutions boiling at 120, 130 and 140 °C. Corrosion rates ranged from 0.01 to 2.5 mils/month. Corrosion rates of Zircaloy-2 in boiling melter condensate ($4.2\text{M H}_2\text{SO}_4 - 2.1\text{M HNO}_3 - 0.2\text{M H}_3\text{PO}_4$) and in melter condensate plus PW-2 feed solution were 0.01 and 0.4 mils/month, respectively. These rates were appreciably lower than those found for any common material of construction evaluated to date.

STUDIES ON AUXILIARIES TO SOLIDIFICATION SYSTEMS

Reaction Kinetics of Sucrose Added to the Evaporator -

P. C. Owzarski

Sucrose will be added to the WSEP evaporator to reduce ruthenium volatility by producing reducing oxides of nitrogen.

It is necessary to understand the sucrose- HNO_3 reaction to predict transient behavior of the evaporator. Existing batch reaction data* were examined using the following best fitting kinetic models:

$$\frac{d[\text{carbon}]}{dt} = -k [\text{carbon}][\text{HNO}_3]$$

$$\frac{d[\text{HNO}_3]}{dt} = -\alpha k [\text{carbon}][\text{HNO}_3]$$

The constants k and α are dependent on temperature and concentration of various ions (Fe^{+3} most notably). The following table was computed from 24-hour batch denitration data at 100 °C. The rate constants are based on carbon concentration (moles sucrose/liter) and α is the number of moles of HNO_3 destroyed per mole of sucrose reacted.

<u>$[\text{Fe}^{+3}]$ moles/liter</u>	<u>α</u>	<u>k, liters/mole min</u>	<u>αk</u>
0	12	0.00070	0.0083
0.1	15	0.00057	0.0086
0.5	18	0.00087	0.016
0.8	20	0.0026	0.052
1.0	20	0.0028	0.056

EVALUATION OF SOLIDIFIED WASTE PRODUCTS

NONRADIOACTIVE LAB STUDIES

Study of the High Temperature Storage of Solidified Waste Products Containing Sulfate - D. W. Brown

This experiment is designed to determine whether the sulfate in solidified waste products containing sulfate will decompose under conditions of high temperature storage and result in excessive vessel pressurization.

A vessel 12 inches in diameter and 26 inches tall was filled 22.5 inches deep with Spray Solidification PW-2 (sulfate-containing) melt. This was heated at the center of the ceramic product.

An equilibrium temperature of about 500 °C at the ceramic-heater interface was held for nine days and no pressurization due to sulfate decomposition was observed. When a temperature of 600 °C was attempted, the heater burned out. A second heater was installed in the same manner and it too burned out on attempting to reach 600 °C. In both cases, there was a 15 mil gap between the heater and the platinum sheath. A third heater, with the platinum sheath drawn onto the heater, has been installed in the vessel and this should allow a temperature of 700 °C at the ceramic-heater interface.

Product Characterization - Continuation of Laboratory Evaluation of PW-2 Phosphate Glass Samples - P. L. Farnsworth and W. L. Mularz

Thermal Diffusivity

Optimum sample geometry for thermal diffusivity measurements is 0.5 inch diameter and 0.027 inch thick. One side of each sample is vapor coated with a layer of chromium and gold for

electrical and thermal contact of the thermocouple. The vapor-deposited films adhere to the substrate during heating without any detrimental effect on the measurement.

Preliminary results show that the thermal diffusivities of the samples do not vary greatly with sample history and temperature and are of a low value (see Table VII).

Thermal Conductivity

The thermal conductivity of samples 6, 9, and 10 (see Table VII) is being measured by a comparative technique using a Thermo-Physics Corporation model TC-2000 thermal conductivity apparatus. The apparatus has been calibrated and measurements are in progress. Other samples are being machined.

Dispersibility

A prototype sample (1.0 inch diameter and 0.50 inch long) was shattered by a 2.9 pound weight with a blunted steel tip (0.12 inch diameter) dropped from a height of 5 feet. The particles were sieved and classified as to size and weight. Results are shown in Table VIII and Figure 5.

The quantity of sample passing through a 230 mesh screen is less than 0.1%. Characterization will include only particles greater than 325 mesh.

Additional samples have been prepared and will be evaluated. Their geometry is 0.75 inch diameter by 0.50 inch long.

TABLE VII. Sample History and Characterization of PW-2 Phosphate Glass Samples

Sample No.	Processing Temperature, °C	Time of Process, hr	Cooling Rate, °C/hr	Temperature for 8 Weeks, °C	% Solubility in H ₂ O	Density, g/ml	Porosity, %	Appearance after CLTE ^(a)	CLTE ^(a) × 10 ⁻⁶ in./in./°C			
									200 °C	300 °C	400 °C	500 °C
4	1000	1	30	100	5.57	2.94	0	some glassiness	9.8	10.6	12.8	
5	1000	1	100	100	4.46	2.89	1.0					
6	1000	25	30	100	5.07	2.81	0	slightly glassy one end	9.1	10.8	11.6	
11	1000	25	100	100	1.48	2.89	0					
7	1000	1	30	600	41.35	2.99	2.2	heavily glassed	5.8	12.6	9.4	2.3
8	1000	1	100	600	33.84	2.98	3.2	heavily glassed	6.9	10.7	9.7	8.0
9	1000	25	30	600	39.27	2.98	2.4	some glassiness porous	7.1	8.5	4.6 ^(b) 7.6	
16	1000	25	100	600	35.86	2.93	2.3	no change in appearance	10.1	13.8	14.2	11.6
12	1200	1	30	100	1.18	2.91	0	no change in appearance	10.3	9.1	7.5	
14	1200	1	100	100	1.39	2.92	0	glassy	9.4	9.8	7.1	
13	1200	25	30	100	1.63	2.90	0					
17	1200	25	100	100	2.18	2.91	0					
10	1200	1	30	600	1.93	2.88	0	no glassy appearance yellowish	9.8	11.0	12.1	11.6
2	1200	1	100	600	41.43	2.92	0	very glassy yellowish	10.8	11.8	14.4	11.7
15	1200	25	30	600	7.45	2.92	0					
3	1200	25	100	600	35.33	2.87	2.1	quite glassy one end	10.1	11.7	13.1	11.2

a. Coefficient of Linear Thermal Expansion

b. One test gave two different results

FIGURE 5. Dispersibility of PW-2 Phosphate Glass Samples,
Weight Percent Versus Size of Particles Collected

TABLE VIII. *Dispersibility of PW-2 Phosphate Glass Samples*

<u>Sieve Opening^(a)</u>		
<u>inches</u>	<u>Weight, g</u>	<u>wt%</u>
0.312	4.588	21.3
0.223	6.9618	32.3
0.157	3.1942	14.8
0.111	2.5391	11.7
0.0787	1.5720	7.3
0.0555	0.8149	3.8
0.0394	0.4085	1.9
0.0278	0.3467	1.6
0.0197	0.2851	1.3
0.0139	0.1921	0.89
0.0098	0.1893	0.88
0.0070	0.1472	0.68
0.0049	0.1357	0.63
0.0035	0.1619	0.74
0.0025	0.0258	0.11
<0.0025	0.0030	0.01
Total Weight 21.565		99.94%

a. *The particles collected passed through a larger screen size opening and rest on the indicated sieve opening.*

Coefficient of Linear Thermal Expansion (CLTE)

The linear thermal expansion of eleven samples has been measured from 200 to 500 °C. The results are tabulated in Table VII and plotted in Figures 6 and 7. The CLTE up to 300 °C is high and is comparable to iron, 12.5×10^{-6} in./in./°C. Although the composition is the same for all samples, their thermal histories vary and hence the CLTE varies. Normally, the CLTE is measured on annealed samples.

Heating of some of the samples caused gas evolution and a glassy appearance on the surface. Most notably affected were samples which were melted at 1000 °C and stored at 600 °C as indicated in Table VII.

Thermal Shock

A set of sieves will be used to classify particles which are produced by fracturing when the hot glass drops into a liquid medium at 0 °C. Because some samples are soluble in water, water will not be used as the quenching medium. Samples have been

FIGURE 6. Coefficient of Linear Thermal Expansion

FIGURE 7. Coefficient of Linear Thermal Expansion of PW-2 Phosphate Glass Samples

prepared and will be evaluated during the next quarter. Their geometries are 0.75 inch square by 0.12 inch, 0.25 inch, and 0.50 inch thick.

Crystallinity

All sixteen samples were mounted and polished. Their unetched microstructures are shown in Figure 8.

Melting and storage temperatures have the most significant effects upon the microstructure. Rate of cooling and time in the melt have less significant effect. The effect of storage time was not determined.

In samples melted at 1000 °C and 1200 °C and stored at 600 °C, the crystals are larger and more abundant than those stored at 100 °C. Porosity is greater in samples melted at 1000 °C and stored at 600 °C than in samples melted at 1200 °C and stored at 600 °C. The samples heat treated at 600 °C were softer to abrasion than the samples which were heat treated at 100 °C.

Microstructure analysis and visual observation of samples 2, 4, and 6 also revealed porosity in samples which were considered nonporous by mercury porosimeter measurements. Thin sections of the samples have been prepared and further microstructure analysis is continuing. Samples 2, 5, 8, and 14 will be examined for phase composition with electron beam microprobe spectroscopy and X-ray diffractometry.

PRODUCT MEASUREMENTS, TESTING AND STORAGE

Solids Storage Engineering Test Facility (SSETF) - D. H. Siemens and V. P. Kelly

Modification of A-Cell for SSETF has been virtually completed following installation of special in-cell mounting brackets, fabrication of the transfer mechanism and shielded ion chamber, and completion of most gallery items. Installation of the first-floor crane control outlet is not complete. However, A-Cell startup will not be delayed by this item.

Neg 06899891

FIGURE 8. Microstructure of Phosphate Glasses
all Samples Unetched 20X

Design has been completed for gallery piping and electrical systems, and handling and transfer hardware for in-cell test pods and waste pots. Fabrication and installation of equipment and systems is expected to be completed in sufficient time to allow at least two weeks for remote design verification tests prior to the projected startup this year.

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