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QUARTERLY PROGRESS REPORT
RESEARCH AND DEVELOPMENT ACTIVITIES
FIXATION OF RADIOACTIVE RESIDUES
MAY, JUNE, AND JULY, 1969

August 1969

AEC RESEARCH &
DEVELOPMENT REPORT

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MAY, JUNE, and JULY, 1969

Edited by

A. G. Blasewitz

Chemical Technology Department
Chemistry and Metallurgy Division

August 1969

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PREVIOUS QUARTERLY REPORTS

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	<u>1959</u>		
First		HW-60584	59-1
Second		HW-13635	59-2
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	<u>1960</u>		
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Third		HW-75220	62-3
Fourth		HW-76262	62-4
	<u>1963</u>		
First		HW-77299	63-1
Second		HW-78188	63-2
Third		HW-70140	63-3
Fourth		HW-80526	63-4
	<u>1964</u>		
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Second		HW-83320	64-2
Third		HW-84467	64-3
Fourth		HW-84603	64-4
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* *Reference notation used in this report.*

PREVIOUS QUARTERLY REPORTS

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	<u>1967</u>		
First		BNWL-434	67-1
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Fourth		BNWL-771	67-4
	<u>1968</u>		
First		BNWL-824	68-1
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Third		BNWL-923	68-3
Fourth		BNWL-1003	68-4
	<u>1969</u>		
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* Reference notation used in this report.

** *Reports for the period July through October, 1967. Reporting periods are now February-April, May-July, August-October, and November-January.*

CONTENTS

FIGURES AND TABLES	vi
INTRODUCTION	1
SUMMARY	1
OVERALL STATUS OF WSEP DEMONSTRATIONS	2
SPRAY SOLIDIFICATION	3
Engineering Studies	3
Development Spray Solidification Runs	3
Radioactive Demonstrations	7
Spray Solidification Run SS-10	7
PHOSPHATE GLASS SOLIDIFICATION	15
Laboratory Studies	15
Corrosion Studies	15
Radioactive Demonstration	15
Phosphate Glass Solidification Run PG-7	15
INTERMEDIATE AND LOW LEVEL WASTE TREATMENT	18
Melter Condensate from Phosphate Glass Process	18
Development Studies	18
Integrated Plant Studies	20
Product Measurements, Testing, and Storage	21
Solids Storage Engineering Test Facility (SSETF).	21
Characterization of WSEP Solidified Waste	21

FIGURES

1.	Mode A and B Arrangement for SS-10	12
2.	Sulfuric Acid, Nitric Acid, and Ruthenium Concentrations in Distillate as a Function of Time (Run 2H)	19
3.	Phosphate Glass Product Effective Thermal Conductivity	23
4.	Spray Solidifier Product Effective Thermal Conductivity	24

TABLES

1.	Overall Status WSEP Radioactive Demonstrations	3
2.	Summary of Development Spray Solidification Runs	4
3.	PW-4m Feed Composition for Development Spray Solidifier Runs	6
4.	Summary Data for WSEP Spray Solidifier Run SS-10	8
5.	Activity Distribution for Run SS-10	13
6.	Ru-106 Activity Found In E-111 Off-Gas Sample Packages During SS-10	14
7.	Summary of Distillation Demonstration Unit (DDU) Hot Runs	18
8.	SSETF Environmental Test Plan	22

QUARTERLY PROGRESS REPORT
RESEARCH AND DEVELOPMENT ACTIVITIES
FIXATION OF RADIOACTIVE RESIDUES

May, June, and July, 1969

INTRODUCTION

This progress report is the forty-second 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

To date, 23 radioactive demonstrations in WSEP have been completed with simulated wastes containing approximately 32 MCi of mixed radionuclides being converted to solids.

During the report period, approximately 2.6 MCi of radioactivity were processed in the tenth spray solidification run, and approximately 2.7 MCi were processed in the seventh phosphate glass run. Spray solidification Run SS-10 processed a simulated Purex Waste 4m (PW-4m) and was the last in the first series of spray solidification runs. Run SS-10 completed that phase of the Waste Solidification Demonstration program wherein the basic engineering feasibility of all three solidification processes has been established. Although the spray solidification system is certainly not optimized, the performance data have shown that major processing factors can be adequately controlled. Phosphate glass Run PG-7 was a simulated Purex

Waste 2 (PW-2) and is the first in the final series of phosphate glass demonstrations. The PW-2 glass effective thermal conductivity of $0.9 \text{ W/(m}^2)(^{\circ}\text{C/m)}$ during air storage increased to $1.85 \text{ W/(m}^2)(^{\circ}\text{C/m)}$ when most of the glass was molten during the pot filling operation in the receiver furnace.

Development spray solidification runs indicated that more than 95% of the ruthenium can be retained in the calcine from PW-4m feeds over a range of acidities if the phosphate flux is not added to the feed.

Three Distillation Demonstration Unit (DDU) runs have been made using sulfate containing condensate from phosphate glass solidification Run PG-4. At operating pressures of 30 mm Hg and lower, the sulfuric acid fraction can be distilled with a low nitric acid content and with a ruthenium content corresponding to a DF of greater than 200, a value large enough to permit incorporation of the sulfuric acid in asphalt for permanent storage. Vacuum distillation of the melter condensate has been shown on engineering scale equipment to be a viable concept for decontamination of the sulfuric acid fraction.

OVERALL STATUS OF WSEP DEMONSTRATIONS - K. J. Schneider

Table 1 shows the overall status of the radioactive demonstrations in WSEP as of July 31, 1969. A total of 23 demonstrations have been completed, 6 in the pot calcination process, 7 in the phosphate glass solidification process, and 10 with the spray solidification process. Approximately 32 MCi of mixed radionuclides representing waste from about 240,000 MWd of electrical power have been converted to solids. This amount of waste represents 91 days of all the nuclear power capacity in the United States as of March 1, 1969. The total on-line processing time for solidification of these wastes was 58 days.

TABLE 1. Overall Status WSEP Radioactive Demonstrations

	Solidification Method			Total
	Pot	Spray	Phosphate Glass	
Runs Completed	6	10	7	23
Megacuries Solidified	4.0	17.5	10.6	32
Equivalent Tonnes ^(a) Processed	11.3	14.6	10.1	36
MWd _e Represented by Waste ^(b)	75,000	98,000	67,000	40,000
Tonne/Day Rate ^(a)	0.6 to 1.0	0.5 to 0.9	0.3 to 0.7	--
Maximum kW in One Pot	5.1	12.7	9.8 ^(c)	118 ^(e)
Maximum W/R in Pot	85	205	160 ^(d)	--
Maximum Centerline Temperature in Pot, °C	940	930	780	--
Liters Solid/Tonne ^(a)	40 to 50	30 to 65	50 to 100	--

(a) Tonne is a metric tonne (1000 kg, or 2205 lb) of original fuel.

(b) Assuming 33% thermal efficiency and 20,000 MWd/tonne.

(c) True maximum has not yet been demonstrated. Calculated maximum is 11.7 kW.

(d) True maximum has not yet been demonstrated. Calculated maximum is 195 W/l.

(e) Total kilowatts encapsulated to date.

SPRAY SOLIDIFICATION

ENGINEERING STUDIES

Development Spray Solidification Runs - J. D. Moore

During nonradioactive development and WSEP spray solidification runs with PW-4m feed, between 20 and 60% of the ruthenium evolves from the feed during calcination. To determine if the ruthenium evolution from the solidifier could be reduced, four runs were made with the development spray solidifier to test methods of ruthenium retention. Run data are summarized in Table 2.

TABLE 2. *Summary of Development Spray Solidification Runs*

	<u>DSC-31</u>	<u>DSC-32</u>	<u>DSC-33</u>	<u>DSC-34</u>
Feed Type	PW-4m	PW-4m	PW-4m	PW-4m
Feed concentration, liters/tonne	506	454	454	378
Additives	Fe,Al, Na,Li, H ₃ PO ₄	ZnO, Ca(OH) ₂	ZnO, Ca(OH) ₂	HNO ₃
Feed total acidity, M H ⁺	2.4	Basic pH=10	Basic pH=10	1.7,3.4, 5.1
M ⁺ /P	2.5	--	--	--
Feed consumed, liters	125	77.2	31.9	303.9
Average feed rate, liters/hr	15.6	18.4	16.8	15.5
Calciner furnace temperature, °C	700	700	700	700
Calciner vacuum, inches of water	8	5	5	8
Atomizing gas	air	air	air	air
Atomizing gas flow, scfm	7	6.9	6.5	6.5
Filter blowback pressure, psig	40	40	40	30
Filter pressure drop, end of run, inches of water	10	5	7	4
Melter furnace temperature, °C	800	--	--	--
Product collected, kg	27.3	17.3	3.6	48
Product concentration, liters/tonne	32	--	--	--
Particulate DF (feed to condensate)	660	500	NA	NA
Ruthenium volatilized, % of total feed ruthenium	38	<2	NA	<2, <0.6, <3

 NA - Not Available

Prior to Run DSC-31, PW-4m feed (the same flowsheet used in WSEP Runs SS-9 and 10) containing phosphate flux (see Table 3 for detailed feed compositions) was acid-stripped from a total acidity of 6.05M to 2.4M. Previous experience with PW-1 feeds had shown that ruthenium release increased with feed acidity.* One hundred and sixty-eight liters of feed were cycled through a horizontal wiped-film evaporator at an average rate of 95 liters/hr during an intermittent 53 hr run in preparation for spray solidification. When the feed was processed in DSC-31, 38% of the ruthenium was volatilized to the spray solidifier off-gas system. The above reduction in feed acidity did not significantly reduce the ruthenium evolution, and apparently a much more extensive acid strip would have been required to do so.

Since acid-stripping is very time consuming with the available evaporators, both in the nonradioactive laboratory and in the WSEP, chemical neutralization of the feed was tried. Simulated PW-4m was made basic (pH of 10) by adding Ca(OH)_2 (0.65M) and ZnO (0.5M). When diluted to 454 liters/tonne, the feed contained about 70 vol% solids (the precipitate was a fine, easily suspended solid), but a relatively steady feed flow was nevertheless achieved in Runs DSC-32 and 33. No flux was added to the feed, so the calcine was collected in a barrel connected to the bottom of the calciner. Less than 2% of the ruthenium was volatilize from the feed during these runs.

In DSC-34 the effect of feed acidity on ruthenium retention in the absence of phosphate ion was studied. Three PW-4m feed batches were prepared with total acidities of 1.7M, 3.4M, and 5.1M, respectively. No additives other than

*BNWL-1003, 68-4.

TABLE 3. *PW-4m Feed Composition for Development Spray Solidifier Runs. Basis: 378 liters/tonne concentration*

<u>Chemicals</u>	
Fe ⁺³	0.124M ^(a)
Cr ⁺³	0.012
Ni ⁺²	0.051
Al ⁺³	0.001
Na ⁺	0.342 ^(b)
SiO ₃ ⁻²	0.01
PO ₄ ⁻³	0.003
NO ₃ ⁻	1.932
H ⁺	0.037

<u>Fission Products</u> ^(e)	
MoO ₄ ⁻²	0.161
Sr ⁺²	0.036
Ba ⁺²	0.041
Rb + Cs (used K ⁺)	0.092
(Y + RE) ⁺³	0.274
ZrO ⁺²	0.143
Ru ⁺⁴	0.0082 ^(c)
Rh (used Co ⁺²)	0.013
Ag ⁺	0.0016
Cd ⁺²	0.0025
Te (used SO ₄ ⁻²)	0.014
Pd (used Ni ⁺² , included above)	0.043
Te (used MoO ₄ ⁻² , included above)	0.031

<u>Additives</u>	<u>DSC-34</u>				
	<u>DSC-31</u>	<u>DSC-32-33</u>	<u>Part 1</u>	<u>Part 2</u>	<u>Part 3</u>
Fe ⁺³	0.9	--	--	--	--
Al ⁺³	0.2	--	--	--	--
Li ⁺	0.75	--	--	--	--
Na ⁺	0.75	--	--	--	--
P O ~ - ~	2.8	--	--	--	--
Ca ⁺²	--	0.65	--	--	--
ZnO	--	0.5	--	--	--
OH ⁻	1.5	1.3	--	--	--
H ⁺	5.6 ^(d)	--	0.83	2.33	3.83
NO ₃ ⁻	3.3 ^(d)	--	0.83	2.33	3.83

(a) Nominal Fe concentration is 0.05M. Excess is Ru substitute.

(b) Nominal Na concentration is 0.1M. Excess is from using Na₂MoO₄ · 2H₂O for Mo addition.

(c) Nominal Ru concentration is 0.082M.

(d) Concentrations before acid-stripping.

(e) Simulated with nonradioactive chemicals.

the nitric acid necessary to adjust the feed acidities were used. About 100 liters from each batch were fed to the spray calciner. Powder was collected in a product pot connected to the calciner. Less than 3% of the ruthenium was evolved from all three feed batches with no significant difference in the amount released from any of the three.

From the results of these tests, it appears that ruthenium can best be retained in the product if the phosphate flux is added as a separate stream to the melter. Since the ruthenium evolution from phosphate-free liquid feeds is minimal with acidities as high as 5.1M, acid-stripping of the radioactive waste during feed preparation would not be necessary. Further flowsheet development is necessary, however, to find the type and amount of flux necessary to form a good solid product.

RADIOACTIVE DEMONSTRATION - W. R. Bond, J. N. Hartley, and
W. K. Winegardner

Spray Solidification Run SS-10

During Run SS-10, a simulated waste equivalent to that resulting from the reprocessing of 1.23 tonne of power reactor fuel (45,000 MWd/tonne at 30 MW/tonne) with an equivalent aging time of 1.8 years was reduced to 58 liters of ceramic product. The radioactive ceramic produced a heat rate density of 127 W/liter (7370 Btu/hr total based on pot calorimetry) in an 8-in. diameter mild steel melt receiver. The steady-state pot centerline temperature in the air-cooled furnace was 611 °C, and the centerline-to-wall temperature difference was 262 °C. These values indicate an effective thermal conductivity of $1.25 \text{ W/(m}^2)(\text{°C/m}) [0.72 \text{ Btu/(hr) (ft}^2)(\text{°F/ft})]$ for the ceramic product.

The solidifier was fed 750 liters of PW-4m feed [additives of 0.94M $\text{Fe}(\text{NO}_3)_3$, 0.16M $\text{Al}(\text{NO}_3)_3$, 0.75M LiOH , 0.97M NaOH , and 2.80M H_3PO_4 based on 378 liters/tonne] at 611 liters/tonne in 56 hr at an average feed rate of 13.5 liters/hr. The feed contained 2,580,000 Ci of radioactivity including 12,700 Ci of ruthenium. A summary of run data for SS-10 is presented in Table 4.

TABLE 4. Summary Data for WSEP Spray Solidifier Run SS-10

<u>Operational Data</u>	
WSEP Run	22
Date	4/69
Operating Mode	67% A - 33% B
Feed Type	PW-4m
Feed, liters/tonne	611
Pot Diameter, in.	8
Pot Material	Mild Steel
Total Run Feed Time, hr	55.5
Average Feed Rate, liters/hr	13.5
Total Feed, liters	750
Total Feed, tonne equivalent	1.23
Feed: Solid Volume Ratio ^(a)	11.4
Feed: Solid Volume Ratio at ^(a) 378 liters/tonne Basis	7.1
Filter AP, in. of water	5-9

Problems

Deposition of calcine in the bottom conical section of calciner and filter chamber.

<u>Filled Pot Data</u>	
Total Curies in Feed to Pot ^(b)	2,270,000
Total Curies of Ru in Feed to Pot ^{(b)(c)}	6,150
Solid in Pot, liters	58

TABLE 4. (Contd)

Solid in Pot, liters/tonne ^(a)	55
Heat in Pot, w ^(d)	7,370
Equivalent Aging Time, years	1.8
£ Temp in Air-cooled Furnace, °C	611
£ to Wall A Temp., °C	262
Effective Thermal Conductivity, W/(m ²) (°C/m) [Btu/(hr) (ft ²) (°F/ft)]	1.25 [0.72]
Welded Lid	Yes
Radiation Reading, R/hr at 6 in.	25,000

(a) Twelve percent of product assumed retained in solidifier.

(b) By feed analysis and assuming 12% of activity retained in solidifier.

(c) Forty-five percent of total ruthenium volatilized from solidifier.

(d) By pot calorimetry data.

During the run, the flow of calcine from the spray calciner into the melter was difficult to maintain. The flow was more erratic than in previous runs, and occasional chunks of calcine were noted entering the melter, particularly during filter blowback. Early in the run the feed was interrupted for 3-1/2 hr to rod out what was thought to be a partial calcine bridge in the conical bottom outlet of the calciner. However, the rodding dislodged very little calcine, and increased vibration of the calciner was required for the remainder of the run to maintain calcine flow to the melter. The filter pressure drop also increased from 5 to 9 in. of water during the run. Raising the filter blowback pressure from 40 to 60 psig did not stop the increasing trend in filter pressure drop, but it may have slowed it. This is the first time during the last seven spray solidification runs that a significant increase in filter pressure drop has occurred during a run. An inspection of the inside of the solidifier was made to determine, if possible, the

cause of the difficulties experienced with the calcine flow and filter pressure drop. Two calcine deposits were found in the conical bottom section of the calciner; the main deposit was located in the area directly below the calciner barrel and a smaller one was located in the area below the filters. Considerable deposition was noted in the filter chamber as caked calcine was lodged around the bottom section of the filters. This explains the erratic calcine flow during filter blowback and the increase in filter pressure drop during the run. The calciner walls were relatively clean. In all cases, the deposits appeared to be the result of wet calcine depositing and drying to form a cake. This suggests inadequate feed atomization, the reason for which is currently under investigation. Based on calorimetry tests of the pot (7370 W by calorimetry versus 8350 W by feed analysis) and volume reduction data from Run SS-9*, it is estimated that approximately 12% of the feed or about 20 kg of calcine accumulated in the calciner during the run. A thorough flush of the system will be required prior to the next spray solidifier run.

The PW-4m melt was batch discharged from the melter via the freeze-drain valve into the unheated mild steel receiver. The melt was quite viscous as in Run SS-9*, but an adequate fill of the receiver was achieved.

Prior to the run, the simulated PW-4m waste, including additives, was acid stripped for approximately 50 hr in the waste evaporator (TK-113) to determine the effect of reduced acidity on ruthenium volatility from the spray solidifier. As a result of the stripping operation, the total acidity of the feed was reduced from 7.5M to 3.2M (based

*BNWL-1074, 69-1.

on 378 liters/tonne). A greater reduction of the total acidity had been planned, but due to frequent plugging of the evaporator dip tubes the stripping operation was curtailed prematurely.

During the last third of Run SS-10, operation was changed from Mode A to Mode B by feeding Purex high level waste (lWW) from the evaporator feed tank (TK-112) to the waste evaporator (TK-113). Figure 1 shows the Mode A and B arrangement for SS-10. The activity distribution during each mode of operation and for the total run is presented in Table 5. Forty-five percent of the ruthenium in the feed processed during Mode A was volatilized from the solidifier and indicates that the reduction of the total acidity of the feed prior to the run did not significantly reduce ruthenium volatility from the solidifier. Only 0.08% of the CePr-144 representing the nonvolatiles in the feed processed during Mode A was entrained from the solidifier. Although Table 5 indicates some differences in distribution of activity downstream of the evaporator (TK-113) during Mode B, these differences are not considered significant based on data from previous Mode A runs.*

During SS-10 work continued on gas sampling techniques that will permit better definition of the type, form, and extent of activity present in the process off-gas stream. The gaseous effluent from the scrubber (TK-118) was sampled for both particulate and volatile forms of activity by routing a side stream of the effluent through first glass fiber filters and then a 2M KOH solution. Essentially all of the activity was deposited on the filters. Calculations

*BNWL-1074, 69-1 and BNWL-1003, 68-4.

FIGURE 1 Mode A and B Arrangement for SS-10

TABLE 5. Activity Distribution for Run SS-10

Fraction of Feed Radioactivity to Solidifier During each Mode of Operation (d) and Total Run in the Effluent Streams						
	Mode A		Mode B		Total Run	
	(Ru)	(T-Ru)	(Ru)	(T-Ru)	(Ru)	(T-Ru)
Evaporator Bottoms (TK-113) (a)	4.5×10^{-1}	7.9×10^{-4}	2.1	2.4×10^{-1}	1.0	7.9×10^{-2}
Fractionator Bottoms (TK-115)	4.0×10^{-3}	2.3×10^{-7}	7.8×10^{-3}	7.1×10^{-7}	5.3×10^{-3}	3.9×10^{-7}
Fractionator Con- densate (TK-116) (b)	0.6×10^{-6}	ND	13.0×10^{-6}	5.6×10^{-10}	4.8×10^{-6}	1.9×10^{-10}
Off-Gas to Scrubber	3.4×10^{-5}	2.1×10^{-10}	2.7×10^{-5}	$<17.0 \times 10^{-10}$	3.2×10^{-5}	$<7.0 \times 10^{-10}$
Off-Gas to Stack	---	---	---	---	$<2.0 \times 10^{-10}$	1.5×10^{-12}
Ratio Fractionator Condensate Activity to 10CFR20	---	---	---	---	4.9×10^3	1.3×10^2
Ratio Stack Activity to 10CFR20	---	---	---	---	$<4 \times 10^{-4}$	$3.4 \times 10^{-3(c)}$

(T-Ru) Fraction of total feed activity less ruthenium

ND Not Detected

(a) Fraction of feed activity for Mode B and Total Run includes the activity fed to TK-113 from from TK-112 during Mode B.

(b) Essentially all the condensate was accumulated during the Mode B portion of the run and accounts to a large extent for the higher fraction accumulated during Mode B.

(c) For gross beta conservatively calculated as Sr-90 and Y-90.

(d) See Figure 1 for Mode A and B arrangement.

suggest that an order of magnitude for the Ru-106 concentration in the process off-gas was 10^{-8} $\mu\text{Ci}/\text{cm}^3$, the same as that observed during SS-9. The CePr-144 concentration was calculated to be about 10^{-7} $\mu\text{Ci}/\text{cm}^3$, about 100 times higher than that observed during SS-9.

During SS-10 the process off-gas stream was also sampled downstream of the process condenser (E-111) using a technique similar to the one discussed above, i.e., a portion of the off-gas from the condenser was bypassed first through a glass fiber filter and then bubbled, via a fitted plastic cylinder, through a 2M KOH solution. The Ru-106 activity associated with five different filter-caustic scrubber sample package assemblies is listed in Table 6. Calculations, based on the total Ru-106 activity deposited in those five sample packages, account for over 100% of the ruthenium activity that accumulated in the fractionator (about 70 Ci). Sampling of the gaseous effluent streams will continue during the phosphate glass demonstration runs.

TABLE 6. Ru-106 Activity Found in E-111 Off-Gas Sample Packages During SS-10

Sample Package No.	Sampling Time, hr	Ru-106 Deposition Rate, $\mu\text{Ci}/\text{hr} \times 10^{-4}$	
		On Filter	In Caustic
2	2.0	2.05	0.06
3	0.8	2.40	ND
4	2.0	0.07	0.18
5	1.0	0.07	0.90
6	1.0	0.50	0.30

ND Not Detected.

PHOSPHATE GLASS SOLIDIFICATIONLABORATORY STUDIESCorrosion Studies - R. F. Maness

A laboratory-scale phosphate glass run was made using a PW-2 feed solution containing 0.5M NH_4NO_3 . The run was made at 1000 °C with semicontinuous feed addition to an alumina crucible equipped with an overflow weir. Total time at temperature was 33 hr, whereas feed was added semicontinuously for 22 hr. Platinum specimens were exposed to the melt phase, to the melt-vapor interface, and to the vapor phase. The platinum specimens exposed to the melt phase and to the interface were unattacked. The end of the specimen exposed to the vapor phase (about 6 in above the melt) was covered with melt as a result of splattering. Removal of the melt revealed a crack, presumably as a consequence of embrittlement under reducing conditions. No attack or embrittlement of other areas of the coupon was noted.

Platinum specimens were exposed to molten (decomposing) $(\text{NH}_4)_2\text{HPO}_4$ at 400, 600, and 800 °C and to $\text{NH}_4\text{H}_2\text{PO}_4$ at 450 and 700 °C for 16 hr periods. Corrosion was slight and no embrittlement was noted.

RADIOACTIVE DEMONSTRATIONPhosphate Glass Run PG-7 - J. L. McElroy, R. F. Drager*

During Run PG-7 simulated waste equivalent to 1.1 tonnes of power reactor fuel (20,000 MWd/tonne at 15 MW/tonne) with an equivalent aging time of 0.5 years was solidified

*On assignment from Brookhaven National Laboratory.

to produce 182 kg of glass product. The 68 liters of radioactive glass produced a heat rate density of 144 W/liter (9800 W total based on a pot calorimetry test). The heat rate density was only 80% of the planned thermal maximum that would have produced a molten core of one-half the diameter in the 8-in. diameter receiver pot.

Because of the low remelt temperature of the phosphate glass (600 to 700 °C), and because of an increased potential for corrosion, this molten core limit was established in addition to the 900 °C centerline limit and a 427 °C wall temperature limit. The steady-state pot centerline temperature of 749 °C with the pot hanging in 40 °C air indicated that the pot centerline was slightly molten. In air, a pot centerline-to-wall temperature difference of 418 °C gave an effective thermal conductivity of $0.90 \text{ W/(m}^2)(\text{°C/m})$ [$0.52 \text{ Btu/(hr)(ft}^2)(\text{°F/ft})$]. During processing, with the pot in the heated receiver furnace, the pot centerline temperature was 853 °C, and the centerline-to-wall temperature difference was 202 °C. For this condition of the nearly completely molten glass, the effective thermal conductivity increased to $1.85 \text{ W/(m}^2)(\text{°C/m})$ [$1.07 \text{ Btu/(hr)(ft}^2)(\text{°F/ft})$].

The denitrator was fed 793 liters of adjusted sulfate containing PW-2 type feed (644 liters/tonne) in 44 hr at an average feed rate of 18 liters/hr. Approximately 100 liters of the feed remained in the denitrator at the end of the run. The feed contained approximately 2,700,000 Ci of radioactivity. A commercial silicone antifoam agent was added continuously to the denitrator as in previous phosphate glass runs*, but was not completely effective. Foaming that occurred in the denitrator was initially due to a sour batch of antifoam, but foaming was still present when a fresh batch of antifoam was used. No serious operating problems resulted from the foaming.

*BNWL-834, 68-1.

Some plugging difficulties in the airlift pot recirculation line to the denitrator caused periods of excessive feed rates to the melter and resulted in foaming of the melt within the melter. On one occasion the foaming led to a slight pressurization of the melter when foam levels reached the melter viewing window and entered the off-gas line and the overflow seal pot line. The melter control temperature that was used to manually control the airlift feed rate to the melter did not always reflect the true internal melter operating conditions. The thermocouple is located near the melt surface in the melter. It would correctly indicate a period of overfeeding by a decrease in temperature and it would normally indicate a period of recovery by an increase in temperature. However, at times, increases in temperature were solely due to rising levels of foam which resulted from overfeeding. False assumptions of melter recovery during these periods then led to a continuation of resumption of feed to the melter and aggravated the foaming condition.

With the melter operating at a furnace temperature of 1250 °C, the average melt pool temperature was nearly 1150 °C, and the melt rate to the receiver pot was 1.45 liters/hr. The melt was overflowed through the unheated melter weir until the end of the run when it was batch-dumped through the freeze valve into the receiver pot. This was done to completely empty the melter. Receiver temperatures were controlled at approximately 600 °C to prevent possible stalagmite formation.

Initial startup of Run PG-7 was delayed by failure of the melter furnace during heatup. Although the exact cause of failure has not been found it was determined that two of the three Kanthal A-1 heating elements were open. Visual inspection of the elements revealed an extreme radial

bowing out from their support brackets as though the elements had grown in length, but were restrained from growing in the lineal end-to-end direction. A new furnace, similar to the failed furnace, but with rods perpendicular to the elements was installed for PG-7 to limit the bowing.

Additional data for the run is being assembled and will be reported in the next quarterly.

INTERMEDIATE AND LOW LEVEL WASTE TREATMENT

MELTER CONDENSATE FROM PHOSPHATE GLASS PROCESS

Development Studies - W. T. McKean

A series of three hot runs have been made distilling actual sulfate containing melter condensate from phosphate glass solidification Run PG-4 in the Distillation Demonstration Unit (DDU). Distillation pressure, rate of heat input, feed volume, and feed and product transfer procedures were similar to earlier cold runs described in previous quarterly reports.* Table 7 lists the operating conditions for the three runs.

TABLE 7. *Summary of Distillation Demonstration Unit (DDU)
Hot Runs*

Run No.	Boilup, gal/hr	Pressure, mm Hg	DF ^(a)	
			H ₂ SO ₄ fraction	HNO ₃ Fraction
1H	1.8	50	75	27
2H	1.6	50	60	12
3H	1.9	30	220	34

(a) $DF (H_2SO_4 \text{ Fraction}) = \text{Ruthenium in Feed} / \text{Ruthenium in } H_2SO_4 \text{ Fraction}$.
 $DF (HNO_3 \text{ Fraction}) = \text{Ruthenium in Feed} / \text{Ruthenium in } HNO_3 \text{ Fraction}$

*BNWL-1003, 68-4, and BNWL-1074, 69-1.

Acid separation efficiencies under these operating conditions were quite good as shown in Figure 2 where the distillate concentrations of nitric acid, sulfuric acid, and ruthenium are shown as a function of distillation time. At an elapsed time of 7.3 hr, corresponding to 78% volume distilled, the distillate was switched from the nitric acid receiving tank to the sulfuric acid tank. Approximately 8% of the sulfuric acid charged to the evaporator was collected in the nitric acid tank, 75% in the sulfuric acid tank, and the 17% remained in the evaporator. All nitric acid charged to the evaporator was distilled.

FIGURE 2. Sulfuric Acid, Nitric Acid and Ruthenium Concentrations in Distillate as a Function of Time. (Run 2H)

Ruthenium evolution is directly related to the period of maximum nitric acid evolution as shown in Figure 2. As residual nitric acid is distilled from the evaporator, the ruthenium evolution subsides. Clearly, the decontamination factor (DF) of the sulfuric acid fraction is dependent on

the point of tank switching; in Figure 2 a delay in switch point to 8 hours would substantially decrease ruthenium content of the sulfuric acid fraction. Of course, along with this increase in DF of the sulfuric acid stream would go an increase in sulfuric acid content of the nitric acid fraction. Table 7 lists the DF values attained in the series of runs. In order to dispose of the sulfuric acid fraction by an asphalting procedure, it was determined that a DF (H_2SO_4 fraction) of at least 200 would be required based on an activity level of 10 Ci/gal of asphalt. On this basis the DDU concept can be used to decontaminate the sulfuric acid fraction of melter condensate. To attain the necessary DF levels, operating pressures in the range of 30 mm of Hg are required.

INTEGRATED PLANT STUDIES - G. L. Richardson

A study flowsheet that incorporates waste solidification and secondary waste recycle in a conventional Purex reprocessing plant has been developed. The flowsheet segregates condensates from product and waste concentration and acid fractionator products according to the activity level. Relatively clean solutions are recycled to the tail-end of the Purex process for feed makeups and scrub and strip solutions, while the more contaminated acid and condensate streams are used in feed dissolution and other head-end steps. During normal plant operation, the flowsheet provides more "clean" HNO_3 and condensate than is required in the process, thus providing a convenient method for bleeding excess water and acid from the system.

The bulk of the excess clean acid and water is derived from evaporation and calcination of the UNH product. By definition, the overhead streams from these operations

should have only trace amounts of fission products (mostly ruthenium), together with minor amounts of entrained UNH. Final cleanup for disposal, if desired, should be easily accomplished.

Other flowsheet features that favor clean effluent streams include the use of sugar to minimize ruthenium volatilization, use of the aqueous waste stream to scrub nitric oxides and ruthenium from the waste calciner off-gas, and the use of a "fumeless" (O_2) dissolution technique to improve recovery of NO_x from the dissolver and calciner off-gases.

PRODUCT MEASUREMENTS, TESTING AND STORAGE

Solids Storage Engineering Test Facility (SSETF) - R. J. Thompson

The cubicle radiation shielding problems that were reported last quarter* have been resolved by installation of the original cubicle shielding plugs from inside the cell. Full use of SSETF is expected during the first quarter of fiscal year 1970.

The schedule of WSEP waste containers to be studied in SSETF has been revised to include a PW-2 (PC-6) and a fast reactor (PG-11) solidified waste; the schedule is given in Table 8.

Characterization of WSEP Solidified Waste - R. J. Thompson

Figures 3 and 4 are graphical summaries of effective thermal conductivity data for phosphate glass Runs 1 to 6 and spray solidifier Runs 4 to 10, respectively. A least

*BNWL-1074, 69-1.

TABLE 8 SSETF Environmental Test Plan

Test No.	Process (a) Run No.	PW Feed Type	Initial Heat, kW	Pot Material	Storage Media
1	PG-4	2	5.8	MS	Air
2	SS-4	2	5.2	MS	Air
3	PC-6	2	5.1	304L	Air
4	PG-6	1	9.6	304L	Water
5	SS-8	1	6.8	304L	Air
6	SS-9	4m	7.2	310	Water
7	SS-10	4m	7.2	MS	Air
8	PG-7	2	10.8	310	Air
9	PG-8	4m	8.5	MS	Water
10	PG-10	4m	8.5	304L	Air
11	PG-11	Fast (b) Reactor	8.5	304L	Water
12	SS-11	Fast Reactor	9.0	304L	Water
13	SS-12	4m	10.0	304L	Water
14	PC-8	4m	6.0	304L	Air
15	PC-9	4m	6.0	304L	Water
16	PC-12	1	5.1	304L	Water

(a) PC = pot calcine, PG = phosphate glass, SS = spray solidifier
 (b) Waste resulting from the processing of a fast reactor core
 fuel irradiated to 100,000 MWd/tonne at 200 MW/tonne.

FIGURE 3. Phosphate Glass Product Effective Thermal Conductivity

FIGURE 4. Spray Solidifier Product Effective Thermal Conductivity

squares regression analysis was made for the effective thermal conductivity of the solidified waste from each process. The resulting equation is presented with the data for both products. The SS-6 data were not included in the regression analysis for spray solidifier product because of the significantly different thermal history of SS-6. Storage of SS-6 at a high temperature (centerline temperature of 900 to 950 °C) for approximately 24 hr in B-Cell resulted in a significantly higher effective thermal conductivity at high temperatures for the product. The effect of the high temperature storage will be the subject of further study in the SSETF where temperature conditions can be maintained more carefully than in B-Cell.

The thermal conductivity of the products from both processes exhibits moderate temperature dependence. In general, the spray solidifier product has a higher effective thermal conductivity than phosphate glass product. However, at higher average temperatures the effective thermal conductivity of the two products is more nearly the same.

The slopes of the effective thermal conductivity curves for the individual runs do not vary greatly from the slope of the least squares regression line for the two products except for SS-6. As noted previously, the high temperature storage appears to have materially affected the spray solidifier product thermal conductivity for this run.

The following product characterization measurements were made in B-Cell during the report period.

A. Pot Calorimetry

1. SS-10: 7.13 kW on 5/2/69
2. SS-3: 0.622 kW on 5/7/69
3. SS-5: 5.11 kW on 6/2/69

4. PG-7: 9.55 kW on 7/16/69
- B. Afterfill dimensions
 1. PG-5: No significant dimension changes noted.
 2. SS-10: No significant dimension changes noted.
- C. Pot Leak Testing
 1. SS-9: 2.1×10^{-7} atm cm³/sec
 2. SS-10: 2.5×10^{-7} atm cm³/sec
- D. Kiethly Scans
 1. PG-6 1.2×10^4 R/hr max (5/12/69)
 2. SS-10 2.5×10^4 R/hr max (4/25/69)
- E. Temperature Profiles
 1. PG-6: air
 2. SS-3: H₂O
 3. SS-10: air and H₂O
 4. SS-5: H₂O
 5. PG-7: air and H₂O

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