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QUARTERLY PROGRESS REPORT
RESEARCH AND DEVELOPMENT ACTIVITIES
FIXATION OF RADIOACTIVE RESIDUES
MAY, JUNE AND JULY 1970

August 1970

AEC RESEARCH &
DEVELOPMENT REPORT

BNWL-1494

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ERRATA

Page v. The author of the section beginning on page 9 and titled "Spray Solidification Run SS-13(IPM)" is W. R. Bond.

Page 11. In the ninth entry under the heading Operational Data the units of Average Processing Rate should be tonne/day.

The first entry under Filled Pot Data should read Total Curies in Feed to Solidifer.

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RESEARCH AND DEVELOPMENT ACTIVITIES
FIXATION OF RADIOACTIVE RESIDUES
May, June, and July 1970

Edited by

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August 1970

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PREVIOUS QUARTERLY PREPORTS

<u>Quarter</u>	<u>Year</u>	<u>Report Number</u>	<u>Reference*</u>
	<u>1959</u>		
First		HW-60584	59-1
Second		HW-13635	59-2
Third		HW-63048	59-3
Fourth		HW-63949	59-4
	<u>1960</u>		
First		HW-65209	60-1
Second		HW-66571	60-2
Third		HW-67334	60-3
Fourth		HW-68193	60-4
	<u>1961</u>		
First		HW-69176	61-1
Second		HW-70517	61-2
Third		HW-71374	61-3
Fourth		HW-72324	61-4
	<u>1962</u>		
First		HW-73412	62-1
Second		HW-74375	62-2
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>		
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>		
First		BNWL-267	66-1
Second		BNWL-306	66-2
Third		BNWL-365	66-3
Fourth		BNWL-410	66-4

* *Reference notation used in this report.*

<u>Quarter</u>	<u>Year</u>	<u>Report Number</u>	<u>Reference*</u>
	<u>1967</u>		
First		BNWL-434	67-1
Second		BNWL-507	67-2
Third**		BNWL-677	67-3
Fourth		BNWL-771	67-4
	<u>1968</u>		
First		BNWL-824	68-1
Second		BNWL-889	68-2
Third		BNWL-923	68-3
Fourth		BNWL-1003	68-4
	<u>1969</u>		
First		BNWL-1074	69-1
Second		BNWL-1186	69-2
Third		BNWL-1263	69-3
Fourth		BNWL-1311	69-4
	<u>1970</u>		
First		BNWL-1401	70-1

* *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

LIST OF FIGURES	vii
LIST OF TABLES	vii
INTRODUCTION	1
SUMMARY	1
OVERALL STATUS OF WSEP RADIOACTIVE DEMONSTRATIONS -	
K. J. Schneider	2
SPRAY SOLIDIFICATION	3
Radioactive Demonstrations	3
Auxiliary Process System Performance	
During Run SS-12(IPM) - J. N. Hartley	3
Spray Solidification Run SS-13(IPM) -	
W. R. Wood	9
Auxiliary Process System Performance	
During Run SS-13(IPM) - J. N. Hartley	13
WSEP AUXILIARY STUDIES	18
Process Control of WSEP Auxiliary System -	
R. D. Dierks and J. N. Hartley	18
LABORATORY STUDIES	20
Corrosion Rates in Boiling Waste Solutions -	
R. F. Maness	20
Corrosion Rates in Rising Level Glass Melts -	
R. F. Maness	20
Studies of the Brookhaven Phosphate Glass Melter	
Failure - R. F. Maness	21
FISSION PRODUCT VOLATILITY STUDIES	21
Volatility of LMFBR Fission Products - M. R. Schwab	21
EVALUATION OF SOLIDIFIED WASTE PRODUCTS	22
Nonradioactive Laboratory Studies	22
Dissolution of WSEP Waste Products - M. R. Schwab	22
Product Measurement, Testing and Storage	23
Solids Storage Engineering Test Facility (SSETF) -	
R. J. Thompson	23

LIST OF FIGURES

1.	WSEP Auxiliary Process System	4
2.	Radioruthenium Accumulation in WSEP Auxiliaries During Run SS-12(IPM)	6
3.	Basic Instrumentation Control of WSEP Auxiliaries	14
4.	Auxiliary Evaporator and Fractionator Operating Parameters	15

LIST OF TABLES

1.	Overall Status of WSEP Radioactive Demonstrations	2
2.	Radionuclide Distribution in WSEP Auxiliaries for Runs SS-11, SS-12(IPM), and SS-13(IPM)	5
3.	Average Concentration of Radionuclides in Process Off-Gas During Runs SS-11, SS-12(IPM), and SS-13(IPM)	7
4.	Summary Data for WSEP Spray Solidification Run SS-13(IPM)	11
5.	PW-4m Feed Composition for Spray Solidification Run SS-13(IPM)	12
6.	SSETF Environmental Test Summary	24

QUARTERLY PROGRESS REPORT
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INTRODUCTION

This progress report is the forty-sixth 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, 30 radioactive demonstrations in WSEP have been completed with simulated wastes containing approximately 48 MCi of mixed radionuclides being converted to solids.

During the report period, the thirteenth and final spray solidification demonstration run in WSEP was completed. A simulated PW-4m waste (a low salt, high fission product waste resulting from the reprocessing of a power reactor fuel with an exposure of 45,000 MWd/tonne at 30 MW/tonne) was successfully reduced to a solid having a heat rate density of 200 W/liter via the in-pot melting technique. During Run SS-13(IPM) spray calcined PW-4m waste was melted directly with a borosilicate glass frit in the final storage container at a processing rate of about 1.1 tonne/day which is an 80% higher rate than was obtained for this waste type during previous spray solidification runs using the platinum melter. The fact that only 0.5% of the ruthenium in the feed processed was volatilized from the spray solidifier during this run verifies the results of Run SS-12(IPM) and developmental runs which indicated that

the ruthenium volatilization from the spray solidifier was significantly reduced when phosphoric acid was not added to the feed.

OVERALL STATUS OF WSEP RADIOACTIVE DEMONSTRATIONS

K. J. Schneider

Table 1 shows the overall status of the radioactive demonstrations in WSEP as of July 31, 1970. A total of 30 demonstrations have been completed: 6 in the pot calcination process, 11 in the phosphate glass solidification process, and 13 with the spray solidification process. Approximately 48 MCi of mixed radionuclides representing waste from about 318,000 MWd of electrical power have been converted to solids. This amount of waste represents 78 days of all the nuclear power capacity in the United States as of February 1, 1970. The total on-line processing time for solidification of these wastes was 66 days.

TABLE 1. Overall Status of WSEP Radioactive Demonstrations

	Solidification Methods			Total
	Pot	Spray	Phosphate Glass	
Runs Completed	6	13	11	30
Megacuries Solidified	4.0	25.3	19	48
Equivalent Tonnes ^(a) Processed	11.2	16.6	9.8	38
MWd _e Represented by Waste ^(b)	75,000	156,000	87,000	318,000
Tonne/Day Rate ^(a)	0.6 to 1.0	0.5 to 1.1	0.3 to 0.7	----
Maximum kW in One Pot	5.1	12.7	12.0	177 ^(e)
Maximum W/l in Pot	85	205	315 ^{(c) (d)}	----
Maximum Centerline Temperature in Pot, °C	940	930	780	----
Liters Solid/Tonne ^(a)	40 to 50	30 to 100	50 to 100	----
Runs to Complete	6	0	0	6

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

b. Assuming 33% thermal efficiency.

c. This value is for 6-in. diameter pots, whereas the comparable values for the pot and spray solidifiers are for 8-in. diameter pots.

d. For 8-in. diameter pots, the maximum is 195 W/l.

e. Totals to date from all processes.

SPRAY SOLIDIFICATIONRADIOACTIVE DEMONSTRATIONSAuxiliary Process System Performance During Run SS-12(IPM) -
J. N. Hartley

During Run SS-12(IPM) (the solidifier performance was discussed in the previous quarterly^{*}) the WSEP auxiliaries were operated in a Mode A arrangement as illustrated in Figure 1. The radionuclide distributions for Runs SS-11, SS-12(IPM), and SS-13(IPM) are summarized in Table 2.

The accumulation of radionuclides in the auxiliaries was generally typical of previous runs except for the accumulation of radoruthenium in the auxiliary evaporator. Ruthenium volatilization from the solidifier was only 1.9% of the ruthenium in the feed to the solidifier. This represents the lowest ruthenium volatilization for any spray solidification run through Run SS-12(IPM). Figure 2 shows the accumulation of radoruthenium in the WSEP auxiliaries.

During Run SS-12(IPM) the solidifier condenser off-gas contained an average of $2.9 \times 10^{-5} \mu\text{Ci}/\text{cm}^3$ of ^{106}Ru which is a factor of about 200 less than the ^{106}Ru concentration during Run SS-11. This is attributed to the decrease in ruthenium volatilization from the spray solidifier during Run SS-12(IPM). The concentration of $^{144}\text{Ce-Pr}$ remained about the same during Runs SS-11 and SS-12(IPM) and averaged $6.8 \times 10^{-5} \mu\text{Ci}/\text{cm}^3$ during Run SS-12(IPM).

Average concentrations of radionuclides in the process off-gas for Runs SS-11, SS-12(IPM), and SS-13(IPM) are presented in Table 3. The off-gas from the solidifier condenser is filtered through a high efficiency filter before entering the evaporator.^{*} The off-gas leaving the

* BNWL-1401, 70-1

FIGURE 1. WSEP Auxiliary Process System

**TABLE 2. Radionuclide Distribution in WSEP Auxiliaries
for Runs SS-11, SS-12 (IPM), and SS-13 (IPM)**

	Fraction of Equivalent Feed Radioactivity for Total Run in Effluent Streams					
	SS-11 Mode A		SS-12 (IPM) Mode A		SS-13 (IPM) Mode B Modified(a)	
	¹⁰⁶ Ru	¹⁴⁴ Ce-Pr	¹⁰⁶ Ru	¹⁴⁴ Ce-Pr	¹⁰⁶ Ru	¹⁴⁴ Ce-Pr
Solidifier Condensate Receiver (TK-117)	----	----	----	----	4.9×10^{-3}	1.1×10^{-5}
Evaporator (TK-113)	7.3×10^{-1}	1.3×10^{-4}	1.9×10^{-2}	1.4×10^{-4}	----	----
Fractionator (TK-115)	2.1×10^{-3}	$<4 \times 10^{-7}$	1.4×10^{-3}	2.4×10^{-6}	3.4×10^{-4}	4.7×10^{-6}
Fractionator Distillate Receiver (TK-116)	5.3×10^{-6}	8×10^{-10}	2.0×10^{-6}	NDG	3×10^{-6}	NDG
Off-Gas Scrubber (TK-118)	1.9×10^{-5}	7.7×10^{-9}	3.1×10^{-5}	NDG	2.6×10^{-6}	NDG
Off-Gas from Scrubber	1.8×10^{-8}	1.4×10^{-11}	1.0×10^{-8}	3.2×10^{-11}	1.1×10^{-8}	1.5×10^{-10}
Off-Gas to Stack	$<2 \times 10^{-10}$	3×10^{-12}	$<4 \times 10^{-10}$	7.5×10^{-10}	2.2×10^{-10}	$<7.4 \times 10^{-10}$
Ratio (Fractionator Distillate Radioactivity to 10CFR20)	1.6×10^4	2.2×10^2	1.1×10^4	1.5×10^2	8×10^3	3×10^3
Ratio (Scrubber Off-Gas Radioactivity to 10CFR20)	4.9×10^2	3.8×10^1	1.9×10^2	7.0×10^1	3.9×10^2	5.8×10^1
Ratio (Stack Off-Gas Radioactivity to 10CFR20)	$<4 \times 10^{-4}$	7.8×10^{-3}	$<4 \times 10^{-4}$	$<9 \times 10^{-4}$	$<4 \times 10^{-4}$	$<2 \times 10^{-1}$

NDG - No detectable gain in radioactivity

a. Solidifier condensate was accumulated in a separate condensate receiver (TK-117)

FIGURE 2. Radioruthenium Accumulation in WSEP Auxiliaries During Run SS-12(IPM)

TABLE 3. Average Concentration of Radionuclides in Process Off-Gas During Runs SS-11, SS-12 (IPM), and SS-13 (IPM)

SS Run Number	Solidifier Condenser		Off-Gas from		Evaporator Condenser		Fractionator Condenser	
	Off-Gas, $\mu\text{Ci}/\text{cm}^3$		Condenser Filter, $\mu\text{Ci}/\text{cm}^3$		Off-Gas, $\mu\text{Ci}/\text{cm}^3$		Off-Gas, $\mu\text{Ci}/\text{cm}^3$	
	^{106}Ru	$^{144}\text{Ce-Pr}$	^{106}Ru	$^{144}\text{Ce-Pr}$	^{106}Ru	$^{144}\text{Ce-Pr}$	^{106}Ru	$^{144}\text{Ce-pr}$
11	5.7×10^{-3}	4.3×10^{-5}	2.8×10^{-6}	2.6×10^{-6}	3.4×10^{-7}	9.3×10^{-7}	5.0×10^{-6}	3.3×10^{-7}
12 (IPM)	2.9×10^{-5}	6.8×10^{-5}	2.2×10^{-6}	4.4×10^{-5}	8.7×10^{-7}	4.8×10^{-5}	5×10^{-7}	7×10^{-7}
13 (IPM)	3×10^{-4}	5×10^{-4}	3.2×10^{-6}	4.0×10^{-4}	8.3×10^{-6}	6.7×10^{-4}	2.0×10^{-7}	3.8×10^{-8}

SS Run Number	Off-Gas from High		Final Off-Gas from Scrubber			
	Efficiency Filter, $\mu\text{Ci}/\text{cm}^3$		$\mu\text{Ci}/\text{cm}^3$		Ratio to 10CFR20	
	^{106}Ru	$^{144}\text{Ce-Pr}$	^{106}Ru	$^{144}\text{Ce-Pr}$	^{106}Ru	$^{144}\text{Ce-Pr}$
11	4.4×10^{-8}	1.3×10^{-8}	9.8×10^{-8}	1.5×10^{-8}	4.9×10^2	3.8×10^1
12 (IPM)	---	2×10^{-9}	3.6×10^{-8}	2.0×10^{-8}	1.9×10^2	7.0×10^1
13 (IPM)	1.7×10^{-8}	2.1×10^{-8}	7.8×10^{-8}	2.3×10^{-8}	3.9×10^2	5.8×10^1

solidifier condenser off-gas filter contained an average of 2.2×10^{-6} $\mu\text{Ci}/\text{cm}^3$ of ^{106}Ru and 4.4×10^{-5} $\mu\text{Ci}/\text{cm}^3$ of $^{144}\text{Ce-Pr}$. These values indicated average instantaneous decontamination factors (DF's) across the filter of 8 for ^{106}Ru and 1.5 for $^{144}\text{Ce-Pr}$. These DF's were considerably less than during Run SS-11. This is possibly an indication that the filter had partially failed.

The auxiliary evaporator operated on about 5M HNO_3 . Instantaneous ruthenium DF's across the evaporator ranged from 1.8 to 2.2×10^3 . These values were considerably less than during Run SS-11. There was again, as in most previous spray solidification runs, no apparent correlation between evaporator overhead acidity and instantaneous ruthenium DF's. This is attributed to an increased overhead acidity from oxides of nitrogen not being scrubbed out completely by the solidifier condenser and by internal contamination of the system. The fraction of equivalent feed ruthenium accumulated in the fractionator was 1.4×10^{-3} which indicated a cumulative ruthenium DF of 14 across the evaporator, while the fraction of $^{144}\text{Ce-Pr}$ accumulated was 2.4×10^{-6} which indicated a cumulative DF for nonvolatiles of 74.

The fraction of equivalent feed radioactivity that accumulated in the fractionator distillate receiver was 2×10^{-6} for ^{106}Ru representing a cumulative DF across the fractionator of 7.5×10^2 . There was no detectable gain of $^{144}\text{Ce-Pr}$ in the fractionator distillate receiver. The concentration of radionuclides in the fractionator distillate (final aqueous effluent) was above 10CFR20 release limits by factors of 1.1×10^4 for ^{106}Ru and 1.5×10^2 for $^{144}\text{Ce-Pr}$ representing nonvolatiles.

The fraction of equivalent feed radioactivity that accumulated in the off-gas scrubber was 1.0×10^{-8} for ^{106}Ru , agreeing with data from Run SS-11. The final process off-gas

leaving the scrubber contained an average of $3.6 \times 10^{-8} \mu\text{Ci}/\text{cm}^3$ of ^{106}Ru and $2.0 \times 10^{-8} \mu\text{Ci}/\text{cm}^3$ of $^{144}\text{Ce-Pr}$ representing an equivalent fraction of feed radioactivity of 1.0×10^{-8} for ^{106}Ru and 3.2×10^{-11} for $^{144}\text{Ce-Pr}$. These values are above 10CFR20 release limits by factors of 1.9×10^2 for ^{106}Ru and 7.0×10^1 for $^{144}\text{Ce-Pr}$. However, after the scrubber off-gas was filtered twice and combined with building ventilation air, the radioactivity released with the off-gas to the stack was well below 10CFR20 release limits.

Spray Solidification Run SS-13(IPM) - W. R. Bond

The final spray solidification run, SS-13(IPM), was successfully completed during the quarter using the in-pot melting technique wherein spray calcined PW-4m waste was melted with a borosilicate glass frit directly in the final storage container. As in Run SS-12(IPM)* the WSEP spray calciner was coupled directly to the receiver pot via a special spool-piece and the solid frit was added directly to the pot.

During the thirteen spray solidification runs, a total of 25 MCi of radioactivity was interfused into 710 liters of solid product. The spray solidification equipment will be disassembled and inspected after the WSEP runs are complete.

During Run SS-13(IPM), a simulated waste equivalent to that resulting from the reprocessing of 0.88 tonne of power reactor fuel (45,000 MWd/tonne at 30 MW/tonne) with an equivalent aging time of 1.0 years was processed to produce 151 kg of product. The 50 liters of radioactive solid produced a heat rate density of 200 W/liter (10,400 W total based on analytical data and 9660 W total based on pot calorimetry) in an 8-in. diameter 304L stainless steel receiver pot. The steady-state pot centerline temperature in the air-cooled

* BNWL-1401, 70-1.

furnace was 824 °C, and the centerline-to-wall temperature difference was 403 °C. These values indicate an effective thermal conductivity of 1.28 W/(m²)(°C/m) [0.74 Btu/(hr)(ft²)(°F/ft)] for the product. As during Run SS-12(IPM) the effective thermal conductivity of the product while molten during processing was approximately 3 to 4 times higher than this and consequently pot centerline temperatures did not exceed 1040 °C despite pot wall temperatures of 900 to 950 °C.

The calciner was fed 308 liters of PW-4m feed at 352 liters/tonne in 20 hr. The resulting calcine was combined with 106 kg of borosilicate glass frit in the receiver pot. The feed containing 2,830,000 Ci of radioactivity including 10,700 Ci of radioactive ruthenium was reduced in volume by a factor of 6.2, or 6.6 on a 378 liters/tonne basis. A summary of run data for SS-13(IPM) is presented in Table 4 and the feed composition for the run is given in Table 5.

The spray calciner performed satisfactorily during Run SS-13(IPM) at an average feed rate of 15.4 liters/hr. The in-pot melting process easily kept up with the calciner at a melt-forming rate of approximately 2.5 liters/hr. For this run the flowsheet was modified such that excess aluminum in the feed stock solutions was substituted for the zinc and calcium additives used during Run SS-12(IPM) and this increased the waste concentration in the product from 17 to 28 wt%. The increased product waste concentration and the 2.5 liters/hr melt formation rate resulted in an average processing rate of 1.05 tonne/day during Run SS-13(IPM) which is 80% higher than the highest processing rate attained for the PW-4m waste during previous spray solidification runs using the platinum melter.

Ruthenium volatilization from the solidifier during Run SS-13(IPM) was only 0.5% of the ruthenium in the feed to the solidifier. This represents the lowest ruthenium volatility

TABLE 4. Summary Data for WSEP Spray Solidification
Run SS-13(IPM)

Operational Data

Date	5/11/70-5/12/70
Operating Mode ^(a)	Modified Mode B
Feed Type	PW-4m
Feed, liters/tonne	352
Total Feed to Solidifier, liters	308
Total Feed Time, hr	20
Average Feed Rate, liters/hr	15.4
Average Melt Forming Rate, liters/hr	2.5
Average Processing Rate, liters/hr	1.05
Equivalent Waste to Solidifier, tonne	0.88
Volume Reduction, feed volume/product volume	6.2
Volume Reduction if Feed Were at 378 liters/tonne	6.6

Filled Pot Data

Total Curies in Feed to Pot	2,830,000
Total Curies of Ru in Feed to Solidifier	10,700
Product in Pot, liters	50
Product in Pot, liters/tonne	57
Heat Generation Rate in Pot, W	10,000 ^(b)
Equivalent Aging Time, years	1.0
Cl Temperature in "Air-Cooled" Furnace, °C	824
Cl to Wall Temperature Difference, °C	403
Effective Thermal Conductivity of Product in an Air-Cooled Furnace, W/(m ²)(°C/m)	1.28

a. See section on auxiliary operation during SS-13(IPM).

b. Based on the average of feed analysis and pot calorimetry data.

TABLE 5. PW-4m Feed Composition for Spray Solidification
Run SS-13(IPM)

<u>Component</u>	<u>Concentration, M^(a)</u>
H ⁺	0.43
Fe ⁺³	0.06 ^(b)
Cr ⁺³	0.012
Ni ⁺²	0.008
Al ⁺³	0.31 ^(b)
Na ⁺	0.19 ^(b)
PO ₄ ⁻³	0.003
NO ₃ ⁻	3.59
SO ₄ ⁻²	----
MoO ₄ ⁻² + Tc as MoO ₄ ⁻²	0.161
Sr ⁺²	0.036
Ba ⁺²	0.041
Rb ⁺ + Cs ⁺ (added K ⁺)	0.092
(Y + RE) ⁺³	0.43 ^(b)
ZrO ⁺² + Nb as ZrO ⁺²	0.143
Ru ⁺⁴	0.0024
Ru ⁺⁴ (used Fe ⁺³)	0.08
Rh ⁺² (added Co ⁺²)	0.013
Pd ⁺² (added Ni ⁺²)	0.043
Ag ⁺² (added Cu ⁺²)	0.0016
Cd ⁺² (added Cu ⁺²)	0.0025
<u>Additive to Feed</u>	
None	
<u>Additive to Pot</u>	
Borosilicate glass frit	320 g/liter

a. Molarities are at 378 liters/tonne.

b. The nominal Fe, Al, Na, and RE concentrations for PW-4m waste are 0.05, 0.001, 0.10, and 0.274M respectively; however, excesses of these materials were present in the feed stock solutions from the processing plant.

for any of the spray solidification runs and is due to the absence of phosphoric acid in the feed to the solidifier, since all the glass-forming flux was added directly to the melt pot.

The performance of the spray calciner off-gas filters was very satisfactory during the run as the filter pressure drop was maintained at 3.6 in. of water and only 0.001% of the particulates were entrained from the calciner.

Auxiliary Process System Performance During Run SS-13(IPM) -
J. N. Hartley

Modified Mode B operation of the WSEP auxiliaries was used during Run SS-13(IPM). This included routing the solidifier condensate to a separate receiving tank as illustrated in Figure 1.

An objective of Run SS-13(IPM) was to demonstrate automatic control of the WSEP auxiliaries. This included using cascade control loops with the evaporator weight factor controller cascaded to the evaporator tube bundle steam controller and the fractionator specific gravity controller cascaded to the fractionator tube bundle steam controller. Figure 3 shows the basic control instrumentation used for the WSEP auxiliaries. During the run 2M HNO_3 was fed to the evaporator at about 14 liters/hr to simulate solidifier condensate since the actual solidifier condensate was collected separately. During the automatic control periods steady operation was not obtained as illustrated by the evaporator and fractionator operating parameters shown in Figure 4. One main problem was that the fractionator specific gravity signal was adversely affected by the fractionator boilup which resulted in poor control. Also twice during the run the evaporator dip tubes were inadvertently vented causing an upset in automatic control.

FIGURE 3. Basic Instrumentation Control of WSEP Auxiliaries

FIGURE 4. Auxiliary Evaporator and Fractionator Operating Parameters

Auxiliary tests are planned for determining more completely the automatic control requirements for the WSEP auxiliaries. Automatic control of the WSEP auxiliaries is planned for the remaining WSEP runs.

The accumulation of radionuclides in the auxiliaries during Run SS-13(IPM) was generally the same as Runs SS-11 and SS-12(IPM) as shown in Table 2.

During Run SS-13(IPM) the solidifier condenser off-gas contained an average of 3×10^{-4} $\mu\text{Ci}/\text{cm}^3$ of ^{106}Ru and 5×10^{-4} $\mu\text{Ci}/\text{cm}^3$ of $^{144}\text{Ce-Pr}$ while the off-gas leaving the condenser filter contained 3.2×10^{-6} $\mu\text{Ci}/\text{cm}^3$ of ^{106}Ru and 4×10^{-4} $\mu\text{Ci}/\text{cm}^3$ of $^{144}\text{Ce-Pr}$. These values indicated a DF of 94 for ruthenium and a DF of 1.3 for $^{144}\text{Ce-Pr}$ across the high efficiency filter. The low DF for $^{144}\text{Ce-Pr}$ indicates that either the filter had failed or there was internal contamination of gas sample lines. The concentration of $^{144}\text{Ce-Pr}$ in the off-gas from the filter increased by a factor of about 10 over that during Run SS-12(IPM). Visual inspection of the condenser off-gas filter after Run SS-13(IPM) revealed that the filter media had partially slipped out of the filter canister as the result of the failure of the adhesive which bonded the media to the canister walls. It was therefore possible for a portion of the off-gas to by-pass the filter media which would result in the low DF observed above. The inlet side of the filter was darkened by the presence of ruthenium and other radionuclides while the outlet remained the same color as new filtering media. There was no evidence of corrosion of the aluminum filtering media supports as has been the case with the high efficiency process filter prior to the off-gas scrubber.

Prior to this modified Mode B run, 450 liters of Purex 1WW containing 370,000 Ci of total radioactivity including 23,000 Ci of rutherenium were transferred to the auxiliary evaporator. During the run the evaporator bottoms were slowly acid stripped. Instantaneous ruthenium DF's across the evaporator were typically greater than 10^4 while instantaneous $^{144}\text{Ce-Pr}$ DF's were typically greater than 10^5 . There was no decrease in DF's when the feed was started to the solidifier. The equivalent fraction of feed ruthenium accumulated in the fractionator was 3.4×10^{-4} indicating a cumulative evaporator ruthenium DF of 3.6×10^3 while the fraction of feed $^{144}\text{Ce-Pr}$ accumulated was 4.7×10^{-6} indicating a cumulative DF for nonvolatiles of about 10^4 .

The fraction of equivalent feed radioactivity that accumulated in the fractionator distillate receiver was 3×10^{-6} for ^{106}Ru representing a cumulative DF across the fractionator of 3.7×10^2 . There was no detectable gain of $^{144}\text{Ce-Pr}$ in the fractionator distillate receiver. Instantaneous ruthenium DF's across the fractionator were typically greater than 10^2 while instantaneous $^{144}\text{Ce-Pr}$ DF's were greater than 10^5 .

The fractionator distillate (final aqueous effluent) contained about 8×10^{-5} Ci/liter of ^{106}Ru and 6×10^{-5} Ci/liter of $^{144}\text{Ce-Pr}$. These values were above 10CFR20 release limits by factors of 8×10^3 for ^{106}Ru and 3×10^3 for $^{144}\text{Ce-Pr}$ representing nonvolatiles.

The equivalent fraction of feed ruthenium accumulated in the off-gas scrubber was 2.6×10^{-6} while there was no detectable gain of $^{144}\text{Ce-Pr}$ in the scrubber. The final process off-gas leaving the scrubber contained an average of 7.8×10^{-8} $\mu\text{Ci/cm}^3$ of ^{106}Ru and 2.3×10^{-8} $\mu\text{Ci/cm}^3$ of $^{144}\text{Ce-Pr}$. These values are above 10CFR20 release limits by factors of 3.9×10^2 for ^{106}Ru and 5.8×10^1 for $^{144}\text{Ce-Pr}$. As in all

previous WSEP runs the scrubber off-gas was filtered and combined with building ventilation air before being released to the atmosphere and as a result the radioactivity released with the off-gas was well below 10CFR20 release limits.

WSEP AUXILIARY STUDIES

PROCESS CONTROL OF WSEP AUXILIARY SYSTEM - R. D. Dierks and J. N. Hartley

Nitric acid and water recovery from the solidifier off-gas in WSEP is dependant on both efficient decontamination in the evaporator and acid concentration in the fractionator. Decontamination data from the evaporator and fractionator have been compromised by the inability to maintain constant acid conditions in the evaporator bottoms. While the evaporator overhead conductivity monitor has provided an excellent indication of the overhead and bottoms acid concentration, attempts to manually adjust boilup and stripwater recycle controller set points to maintain the overhead conductivity at some fixed value and observe the constraints of liquid volume and acid concentration of the two vessels as feed conditions vary have not been successful. See Figure 4 for evaporator and fractionator operation during Run SS-13(IPM).

Six standard, three-mode controllers were used in three cascade loops to control the auxiliary equipment as shown in Figure 3. One controller sensed the evaporator weight factor and cascaded its output to the setpoint of the controller on the evaporator tube bundle steam flow. Another similar system controlled the steam flow to the fractionator tube bundle as an inverse function of fractionator specific gravity. A third system controlled the stripwater recycle flow rate as a direct function of the overhead conductivity.

During the initial automatic control test of the auxiliaries it was found that the specific gravity measurement in the fractionator was a function of the boilup rate and could not be used for controlling the steam flow. While a control system based on a weight factor measurement was adequate to maintain stable operating conditions, it did not implement the primary function of the system, namely to maintain the fractionator bottoms at 10M HNO_3 .

During subsequent auxiliary tests and in analog simulations, the very long time constants of the evaporator and fractionator were realized, explaining the instabilities that had been observed during these early tests. To avoid overshoot and subsequent loss of water from the auxiliary system to the fractionator distillate receiver, the weight factor controllers were operated with zero reset. However, it was found that the 3-mode controllers available could not be operated as proportional-only instruments and some long term cycling resulted. To eliminate this problem, the General Electric 412 process control computer that has been used as a data logging device was programmed to set the set points on the controllers for the steam flow to the evaporator and fractionator tube bundles. The computer was programmed to calculate the set points in direct proportion to the deviation of the measured weight factors from the desired operating weight factor. In addition, the computer was programmed to limit the steam flow to minimum values to maintain boiling in the evaporator and fractionator at all times, and to limit the steam flow to maximum values to avoid pressurization of the auxiliary evaporator and fractionator by excessive boilup. In addition, the computer program provided the capability of periodically venting the weight factor and specific gravity dip tubes in the evaporator to avoid plugging of the dip tubes during operation.

The last test of the auxiliary system indicated that automatic operation of the system could be expected during the forthcoming pot calcination runs.

LABORATORY STUDIES

CORROSION RATES IN BOILING WASTE SOLUTIONS - R. F. Maness

Corrosion rates of potential waste evaporator and storage tank materials of construction (304L SS, 310 SS and titanium) are being determined in boiling PW-1, PW-2, PW-4m, and LMFBR type waste solutions. The tests are being made with solutions 6M in nitric acid and with solutions in which the acidity has been reduced from 6 to about 2M by reaction with formaldehyde. The corrosion resistance of the test alloys in these solutions were titanium greater than 310 SS greater than 304L SS. Results to date for 304L SS range from a high of about 0.24 in./yr, in PW-1 (6M HNO_3) to a low of about 0.024 in./yr, in PW-4m (2M HNO_3). This study will be expanded in time to include feed solutions containing glass-forming additives.

CORROSION RATES IN RISING LEVEL GLASS MELTS - R. F. Maness

Corrosion rates of pot materials (304L SS and 310 SS) are being determined in PW-4m rising level glass melts containing increasing amounts of B_2O_3 (22 to 31%), Na_2O (17 to 24%), and SiO_2 (10 to 14%). Results to date at 900 °C indicate the melt which contained the least amount of additives was the most corrosive. Corrosion rates of 304L SS and of 310 SS in this melt were 2.82 and 1.26 in./yr, respectively, at the interface position, whereas corresponding rates in the melt containing the largest amount of additives were 1.36 and 0.36 in./yr. Additional tests are in progress.

STUDIES OF THE BROOKHAVEN PHOSPHATE GLASS MELTER FAILURE -

R. F. Maness

A metallographic examination was made of a platinum specimen removed from the phosphate glass melter which partially collapsed after 40 months service at Brookhaven National Laboratory. Creep tests reported by Brookhaven indicated the long process exposure reduced the creep resistance of the platinum. Optical and electron probe analyses at Pacific Northwest Laboratories showed the grain boundaries to be quite clean. More sensitive analytical techniques (e.g., ion-probe) might be more productive in explaining the reduced creep resistance.

FISSION PRODUCT VOLATILITY STUDIESVOLATILITY OF LMFBR FISSION PRODUCTS - M. R. Schwab

The analyses for ^{129m}Te in the solidifier effluents from WSEP Runs PG-11 and SS-11 (both LMFBR flowsheets) were completed and confirmed the nonradioactive pilot plant data previously reported.* During Run PG-11, 2.6% of the ^{129m}Te in the feed to the phosphate glass melter accumulated in the melter condensate. The nonradioactive pilot plant data indicated that from 1 to 10% of the tellurium would volatilize from the phosphate glass melter.

During Run SS-11, 0.1% of the ^{129m}Te in the feed to the spray solidifier accumulated in the WSEP auxiliary evaporator which collects the solidifier condensate. These results agreed with the nonradioactive data wherein from 0.1 to 0.5% of the tellurium volatilized from the spray solidifier.

* BNWL-1311, 69-4.

EVALUATION OF SOLIDIFIED WASTE PRODUCTSNONRADIOACTIVE LABORATORY STUDIESDissolution of WSEP Waste Products - M. R. Schwab

A complete dissolution of the solidified waste samples (core-drill samples of WSEP containers) is required for a complete analysis of the waste product constituents including the nonradioactive constituents and the low gamma-emitting or beta-emitting radionuclides.

The nondestructive direct count procedure^{*} is suitable for measuring the relative concentrations of the principle radionuclides ($^{144}\text{Ce-Pr}$, ^{137}Cs , ^{106}Ru , $^{95}\text{Zr-Nb}$) in the core-drill samples from each product storage pot. However, once the actual concentration of any one of the principle gamma-emitting radionuclides is measured (by gamma emission analysis of a totally dissolved sample), the direct count method is suitable for measuring actual concentrations in all core-drill samples from the same product receiver. Consequently, a complete dissolution of the core-drill solidified waste samples from the Product Evaluation Program is needed to fully utilize the nondestructive direct count procedure.

A study is in progress to develop a suitable total dissolution procedure for each of the WSEP waste products. Preliminary laboratory results with nonradioactive phosphate glass (PW-2) and spray solidified (PW-1) waste products indicate that the total dissolution procedure for each waste type will probably require three steps: (1) a digestion period in a sealed Parr[®] Acid Digestion Bomb, (2) dissolution in an acid solvent, and (3) treatment with HF.

* BNWL-1311, 69-4.

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This three-step procedure illustrates the extremes required to obtain an aqueous solution of the WSEP waste products. During the digestion period, the pulverized waste product sample and the solvent (KOH for phosphate glass and $\text{H}_3\text{BO}_3\text{-HCl}$ for spray solidified waste) are held in a sealed, Teflon-lined bomb for 1.5 hours in an oven at 146 °C. The digested waste-solvent slurry is dissolved in HCl at 60 to 80 °C for 1 to 3 hours after which from 10 to 20 wt% of the waste sample is still undissolved. Perchloric acid (HClO_4) and HF are added and the sample solution is heated until all fuming stops. After the HF treatment step, less than 0.1 wt% of the phosphate glass sample and less than 0.5 wt% of the spray solidified waste sample remained undissolved. Analyses for the undissolved waste product residues are not available at this time although the residues are thought to be mostly silicates.

Several other solvent combinations have been found which are suitable for each of the waste products. However, no universal solvent suitable for all waste products has been found. The phosphate glass waste products are best digested in a basic solvent while the spray solidified waste products (except for the in-pot melting flowsheet) are best digested in an acidic solvent containing borate ion. The borosilicate waste product from the in-pot melting flowsheet digests best in a basic solvent such as KOH-KF.

PRODUCT MEASUREMENT, TESTING AND STORAGE

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

One-half of the WSEP waste containers that are scheduled for environmental testing in SSETF have been transferred to the facility. Table 6 summarizes the waste containers and the maximum container centerline and wall temperature and the

maximum environmental test pod temperature. Some faulty thermocouples in PG-7 have forced limiting the temperatures of the test until the thermocouples can be replaced.

TABLE 6. SSETF Environmental Test Summary

Run Number Pot Number <u>Cubicle Number</u>	<u>Date Testing Started</u>	<u>Pod Environment</u>	<u>Maximum Temperature, °C</u>		
			<u>Pod</u>	<u>Pot Wall</u>	<u>℄</u>
SS-4 40 A-21-3	9/11/69	Water	85	85	121
PG-6 39 A-21-1	10/1/69	Water	72	----	128
PC-6 30 A-21-4	11/17/69	Air	499	----	556
SS-8 43 A-21-2	1/5/70	Air	500	----	566
SS-10 54 A-31-3	2/9/70	Water	35	39	162
PG-7 37 A-31-4	5/15/70	Air	208	----	565
SS-9 36 A-31-1	6/1/70	Air	471	490	569
PG-8 46 A-31-2	6/23/70	Water	93	97	285

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