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QUARTERLY PROGRESS REPORT
RESEARCH AND DEVELOPMENT ACTIVITIES
FIXATION OF RADIOACTIVE RESIDUES
AUGUST, SEPTEMBER, AND OCTOBER 1970

November 1970

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DEVELOPMENT REPORT

BATTELLE
BATTELLE MEMORIAL INSTITUTE

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3000 STEVENS DRIVE, P.O. BOX 999, RICHLAND, WASHINGTON 99352

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Edited by

A. G. Blasewitz
Chemical Technology Department
Chemistry and Metallurgy Division

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<u>Quarter</u>	<u>Year</u>	<u>Report Number</u>	<u>Reference*</u>
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Second		HW-13635	59-2
Third		HW-63048	59-3
Fourth		HW-63949	59-4
	<u>1960</u>		
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	<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
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First		BNWL-1074	69-1
Second		BNWL-1186	69-2
Third		BNWL-1263	69-3
Fourth		BNWL-1311	69-4
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Second		BNWL-1494	70-2

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August-October, and November-January.

CONTENTS

LIST OF FIGURES	vii
LIST OF TABLES	vii
INTRODUCTION	1
SUMMARY	1
OVERALL SUMMARY OF WSEP RADIOACTIVE DEMONSTRATIONS -	
K. J. Schneider	2
POT SOLIDIFICATION	3
Laboratory Studies	3
• Corrosion Rates in Rising Level Glass Melts -	
R. F. Maness	3
• Rising Level Glass Developmental Studies -	
W. R. Bond and J. L. McElroy	3
Radioactive Demonstrations	6
Pot Calcination Run PC-7 -	
W. R. Bond and J. L. McElroy	6
Auxiliary Process System Performance	
During Run PC-7 - J. N. Hartley	9
Pot Calcination Run PC-8 -	
W. R. Bond and J. L. McElroy	14
Auxiliary Process System Performance	
During Run PC-8 - J. N. Hartley	15
• Rising Level Glass Run PC-9(RLG) -	
W. R. Bond and J. L. McElroy	18
WSEP AUXILIARY STUDIES	21
Process Control of WSEP Auxiliary Systems -	
R. D. Dierks and J. N. Hartley	21
LABORATORY STUDIES	24
Corrosion Rates in Boiling Waste Solutions -	
R. F. Maness	24
Ruthenium Recoveries in WSEP Solidified	
Waste Products - M. R. Schwab and F. E. Holt (WADCO)	25
EVALUATION OF SOLIDIFIED WASTE PRODUCTS	27
Nonradioactive Laboratory Studies	27
Strontium Leach Rates - F. P. Roberts	27
Characterization of Other Solidified Products -	
F. P. Roberts	27

Radioactive Laboratory Studies	28
C-Cell Testing - F. P. Roberts and C. Hampton	28
Product Measurement, Testing, and Storage	30
Core Drilling and Pot Welding - J. A. Yount	30

LIST OF FIGURES

1. WSEP Auxiliary Process System	10
2. Accumulation of Radioruthenium in WSEP Auxiliaries During Runs PC-7 and PC-8	12
3. Entrainment from Pot Calciner Durings Runs PC-7 and PC-8	17
4. Auxiliary Evaporator and Fractionator Operating Parameters	23
5. Location of Samples from PG-7 Pot	28
6. Photograph of Core 2 from PG-7 Pot	29

LIST OF TABLES

1. Overall Summary of WSEP Radioactive Demonstrations	2
2. Summary Data for WSEP Pot Solidification Runs PC-7, PC-8 and PC-9(RLG)	7
3. Feed Compositions for Pot Solidification Runs PC-7, PC-8, and PC-9(RLG)	8
4. Radionuclide Distribution in WSEP Auxiliaries for Runs PC-7 and PC-8	11
5. Average Concentration of Radionuclides in Process Off-Gas During Runs PC-7 and PC-8	13
6. Radionuclide Recoveries in Phosphate Glass Melt Samples	26
7. Radionuclide Recoveries in Spray Solidified Waste Melt Samples	26

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INTRODUCTION

This progress report is the forty-seventh 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

The full level radioactive demonstrations were successfully completed in WSEP with the ninth pot solidification demonstration. A total of 33 runs was made in which 52 million curies of radio-nuclides were converted to solids.

During the report period, the seventh, eighth, and ninth pot solidification demonstration runs in WSEP were completed. During Runs PC-7 and PC-8, simulated Purex Wastes containing 2.6 megacuries of radioactivity were reduced to a calcine product. During the ninth and final pot solidification run the rising level glass concept was demonstrated in which a simulated Purex Waste was fed to the pot where it was continuously concentrated, calcined and fused to a borosilicate glass product.

OVERALL SUMMARY OF WSEP RADIOACTIVE DEMONSTRATIONS

K. J. Schneider

The final demonstration of solidification of high level radioactive waste in WSEP was completed in October 1970. The overall summary of the radioactive demonstrations in WSEP which was started in November 1966 is shown in Table 1. A total of 33 demonstrations have been completed: 9 with the pot solidification process, 11 with the phosphate glass solidification process, and 13 with the spray solidification process. Approximately 52 megacuries of mixed radionuclides representing waste from about 335,000 MWd of electrical power have been converted to solids. This amount of waste represents 48 days of the 7000 megawatts of nuclear power capacity expected to be installed in the United States as of December 31, 1970. The total on-line processing time for solidification of these wastes was 75 days.

TABLE 1. Overall Summary of WSEP Radioactive Demonstrations

	Solidification Methods			Total
	Pot	Spray	Phosphate Glass	
Runs Completed	9	13	11	33
Megacuries Solidified	7.7	25.3	19	52
Equivalent Tonnes ^(a) of Fuel Processed	13.8	16.6	9.8	40
MWd _e Represented by Waste ^(b)	92,000	156,000	87,000	335,000
Tonnes of Fuel/day, Equivalent	0.3 to 1.0	0.5 to 1.1	0.3 to 0.7	---
Maximum kW in One Pot	5.1 ^(c)	12.7	12.0 ^(d)	192 ^(e)
Maximum W/l in Pot	93 ^(c)	205	200 ^(d)	---
Maximum Centerline Temperature in Pot, °C	1012	930	830	---
Liters Solid/Tonne ^(a)	32 to 50	30 to 100	50 to 100	---

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

b. Assuming 33% thermal efficiency.

c. For a 6-inch-diameter pot, the heat was 4.7 kW at 138 W/l.
For a 6-inch-diameter pot containing rising level glass, the heat rate density in one pot was 250 W/l.

d. For a 6-inch-diameter pot, the heat was 9.5 kW at 315 W/l.

e. Totals from all processes.

POT SOLIDIFICATIONLABORATORY STUDIESCorrosion Rates in Rising Level Glass Melts - R. F. Maness

Corrosion rates of 304L and 310 stainless steel were determined in PW-4m rising level glass melts containing increasing amounts of B_2O_3 (22 to 31 wt%), Na_2O (17 to 24 wt%), and SiO_2 (10 to 14 wt%) at 900, 950, and 1000 °C. Corrosion rates of 304L in the most corrosive melt were 2.8, 5.1, and 9.7 in./yr at 900, 950, and 1000 °C, respectively. Corrosion rates of 310 were lower in all cases by a factor of about 2.

Rising Level Glass Developmental Studies - W. R. Bond and J. L. McElroy

Laboratory tests were made with the rising level glass additive stream to determine if it could be satisfactorily prepared and handled. The additive stream was to contain sodium, boron, and silica such that when it was combined with PW-4m waste and melted, a product containing 10 wt% Na_2O , 22.5 wt% B_2O_3 , 17.5 wt% SiO_2 , and 50 wt% PW-4m waste oxides would result. When the additive was prepared with 105 g/liter B_2O_3 , 71 g/liter $NaNO_3$, and 66 g/liter $SiO_2 \cdot xH_2O$ (76.5 wt% SiO_2), there were excessive solids present that were difficult to suspend and that would not dissolve on heating to 75 °C. When 43.7 g/liter of Ludox® (49 wt% colloidal silica) was used instead of the $SiO_2 \cdot xH_2O$, there were crystals present that completely dissolved when the solution was heated to 60 °C. Since H_3BO_3 crystallized out of the solution at temperatures below this, it would be necessary to extensively insulate and provide heating of the additive lines in WSEP. To avoid this situation, another alternative was investigated in which the sodium and boric acid would be added directly to the feed tank and Ludox only would

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comprise the additive stream. The Ludox[®] would not have to be heated and could readily be metered. The addition of Ludox to the feed tank was not considered since feed pump failures had been experienced in the past with silica-containing feeds.

In the laboratory, H_3BO_3 and $NaNO_3$ were added to PW-4m waste to give concentrations of 1.69M and 0.85M, respectively, based on 378 liters/tonne waste. The solution properties of the adjusted waste were investigated as a function of solution concentration and temperature. It was found that with the waste at 567 liters/tonne, H_3BO_3 started crystallizing from solution when the temperature dropped to the 45 to 50 °C range while at 756 liters/tonne crystallization occurred in the 40 to 45 °C range. In both cases the solution appeared manageable as long as their respective temperatures did not drop below that at which H_3BO_3 crystallization would start to occur.

A pump loop test was then initiated with PW-4m waste containing H_3BO_3 since difficulties in the past had been experienced with phase separation in the feed tank with similar type feeds. One hundred and forty liters of PW-4m feed was made up at 700 liters/tonne and contained additives of 1.69M H_3BO_3 and 0.85M $NaNO_3$ (concentrations based on 378 liters/tonne). The solution was maintained at 50 °C with continuous agitation in the feed tank and was pumped with an in-line pump similar to the feed pump used in WSEP.

The feed appeared to be very homogeneous; however, the failure of the pump seals after only 8 hours of service resulted in loss of all the solution. The test was repeated with a new pump since the first pump had seen prior service, and the seal failure could not be contributed directly to this particular type feed. The second test was carried out for 100 hours without

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the occurrence of any solution or pump difficulties. During this test, the feed was routed briefly through a control valve and flowmeter similar to the ones in WSEP and no particular feed flow control problems were noted.

Based on the results of these tests, it was decided that for Run PC-9(RLG) the PW-4m feed would be made up at 700 liters/tonne and would include the H_3BO_3 and $NaNO_3$ additives with Ludox[®] as a separate additive stream. The feed would be maintained in the feed tank at 50 to 60 °C under continuous agitation.

Other laboratory tests were made to determine whether it was feasible to consider running the PW-4m feed including additives film-wise down the outside of the pot centerline thermowell to gain an increased boil-off capacity (from increased heat transfer area).

In one test the additives and PW-4m feed at 378 liters/tonne were slowly poured into a mixing funnel (equal volume of additive solution per volume of feed) and were observed as they ran down through a clear piece of tubing that simulated the PC-9(RLG) feed system. The two streams appeared to mix readily. Tests with the mixed feed flowing down the outside of an unheated 3/4-inch stainless steel pipe indicated that the feed would form a film and that it did not become too concentrated to flow after being concentrated by factors of 2, 3, and 4. However, the concentration factor of 4 was probably in excess of where the waste could be expected to flow for very long under the actual conditions expected in the PC-9(RLG) pot. The mixed feed solution was easily water-flushed off the pipe at all concentrations.

These tests indicated that if film feeding were used during Run PC-9(RLG), the evaporation of the feed as it flows

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down the thermowell should add to the pot capacity, and a cake or sludge should not form on the thermowell if the feed rate is adequately controlled.

RADIOACTIVE DEMONSTRATIONS

Pot Calcination Run PC-7 - W. R. Bond and J. L. McElroy

The thirty-first radioactive demonstration in WSEP was completed with pot calcination Run PC-7. Run PC-7 was the first in the second series of pot calcination runs and also the first pot calcination run made in nearly 3 years.

During Run PC-7, a total of 500 liters of simulated PW-4m waste at 384 liters/tonne were fed to the pot in 39 hours. Another 10 hours were required to dry and cool the pot. The 60 liters of calcine product contained approximately 1,400,000 curies of fission product radioactivity (including 4400 curies of radoruthenium) to give an internal heat generation rate of approximately 5600 watts in the 8-inch diameter, stainless steel receiver pot. A summary of the data for Run PC-7 is given in Table 2 and the feed composition for the run is given in Table 3.

The feed was started to the pot initially at approximately 30 liters/hr and averaged 25 liters/hr during the first 12 hours of the run. However, as the liquid level reached the top of the pot (50 to 60 liter level) difficulties were encountered with the pot liquid level (thermal probe) indicator. Attempts to control the feed rate automatically were unsuccessful due to the poor response of the level indicator. Although switching from a steam to an increased air purge of the pot pressure tap (condensate was thought to have been dropping on the level probe) seemed to remedy the situation, this was only temporary. The feed was interrupted for 2.5 hours when the liquid level became too high as indicated by the level reaching the pot pressure tap. Feed was terminated when at a rate of 4 to 5 liters/hr

TABLE 2. Summary Data for WSEP Pot Solidification Runs PC-7, PC-8 and PC-9(RLG)

Operational Data	PC-7	PC-8	PC-9(RLG)
Date	8/3/70-8/5/70	9/8/70-9/10/70	10/12/70-10/15/70
Operating Mode	A	A	A
Pot Diameter, in./material	8/304L SS	6/304L SS	6/310 SS
Furnace Temperature, °C	900	900	950
Feed Type	PW-1	PW-4m	PW-4m
Feed Concentration, liters/tonne	384	459	706
Total Feed to Pot, liters	500	340	393
Additive Stream Type	None	None	Diluted Ludox® (21.5 wt% Silica)
Total to Pot, liters	----	----	40
Feed Time, hours	39(a)	36	43.2(b)
Calcination and Cooling Time, hours	10	8	----
Total Run Time, hours	49	44	43.2(b)
Average Feed Rate, liters/hour	13.7(c)	9.4	9(c)
Overall Processing Rate, tonne/day	0.64	0.4	0.3(c)
Equivalent Waste to Pot, tonne	1.3	0.74	0.56
Volume Reduction, feed volume/product volume	8.3	10	22(d)
Volume Reduction if Feed were at 378 liters/tonne	8.2	8.2	11.8(d)
Filled Pot Data			
Product Type	Calcine	Calcine	Borosilicate Glass
Total Curies in Feed to Pot	1,400,000	1,200,000	1,300,000
Total Curies of Ru in Feed to Pot	4,400	2,100	1,600
Product in Pot, liters	60	34	~16(plus ~2 as calcine)(d)
Product in Pot, liters/tonne	46	46	32
Heat Generation Rate in Pot, Watts	5,600	4,700	4,600
Equivalent Aging Time, years	3.0	2.0	1.5
Q _L Temperature Under Free-Air Convective Cooling, °C	1,012	848	720(e)
Q _L to Wall Temperature Difference, °C	759	585	332(e)
Effective Thermal Conductivity of Product Under Free-Air Convective Cooling, W/(m ²)(°C/m)	0.32	0.34	1.1(e)

a. Feed was interrupted 2.5 hours during this period.

b. Actual time feed was on. Due to eight interruptions in feed during the run 58.2 hours actually elapsed between initial feed on and final feed off.

c. Based on actual feeding time.

d. Product volume estimated from pot temperatures.

e. Data with pot in air-cooled furnace as free-air data was not available.

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**TABLE 3. Feed Compositions for Pot Solidification Runs
PC-7, PC-8, and PC-9(RLG)**

Component	Run No. Feed Type	PC-7 PW-1	PC-8 PW-4m	PC-9(RLG) PW-4m
		Concentration, M ^(a)		
H ⁺		2.1	2.5	2.7
Fe ⁺³		0.8 ^(b)	0.06 ^(c)	0.064 ^(c)
Cr ⁺³		0.12	0.012	0.012
Ni ⁺²		0.005	0.008	0.008
Al ⁺³		0.13 ^(b)	0.1 ^(c)	0.1 ^(c)
Na ⁺		0.138	0.24 ^(c)	0.1
PO ₄ ⁻³		0.003	0.003	0.003
NO ₃ ⁻		6.5	6.5	6.5
MoO ₄ ⁻² + Tc (added MoO ₃)		0.079	0.161	0.161
Sr ⁺²		0.0155	0.036	0.036
Ba ⁺²		0.0195	0.041	0.041
Rb ⁺ + Cs ⁺ (added K ⁺)		0.042	0.092	0.092
(Y + RE) ⁺³		0.12	0.274	0.274
ZrO ₂ ⁺² + Nb (added ZrO ₂ ⁺²)		0.065	0.143	0.143
Ru ⁺⁴		0.0032	0.0032	0.0032
Ru ⁺⁴ (used Fe ⁺³ or Al ⁺³)		0.0288	0.079	0.079
Rh ⁺² (added Co ⁺²)		0.0074	0.013	0.013
Pd ⁺² (added Ni ⁺²)		0.0172	0.043	0.043
Ag ⁺² + Cd ⁺² (added Cu ⁺²)		0.0016	0.0041	0.0041
<u>Additive to Feed</u>				
PO ₄ ⁻³		0.06 ^(d)	0.107 ^(d)	-----
B ₂ O ₃		-----	-----	0.836
NaNO ₃		-----	-----	0.84
<u>Additive to Pot</u>				
SiO ₂ (as a 21.5 wt% colloidal suspension)		-----	-----	0.76

a. Molarities are at 378 liters/tonne.

b. The nominal Fe and Al concentrations for PW-1 waste are 0.93 and 0.001M respectively; however excess Al is the feed stock solutions from the reprocessing plant was used as a partial substitute for Fe.

c. The nominal Fe, Al and Na concentrations for PW-4m waste are 0.005, 0.001 and 0.1M respectively; however, excesses of these materials were present in the feed stock solutions from the reprocessing plant.

d. Added to suppress volatilization of alkali metal oxides.

the liquid level again became excessive. Only 78% of the originally planned feed was consumed indicating a much lower volume reduction than anticipated. (Calculated calcine density of 1.07 versus an expected 1.4 kg/liter.) No difficulties with foaming were experienced during the run indicating that the addition of antifoam to the pot was effective in preventing foaming.

The calcine centerline temperature of 1012 °C in air and 925 °C in water exceeded thermal maximum (900 °C centerline) for both air and water cooling. This resulted since the 0.26 to 0.32 W/(m²)(°C/m) effective thermal conductivity of the calcine was lower than the anticipated value of 0.4 W/(m²)(°C/m). The lower than anticipated values for volume reduction and thermal conductivity indicated that the calcine was very porous. This decrease may be attributed to not having sulfate or a higher sodium concentration present in the feed. All previous WSEP radioactive pot calciner runs contained considerable amounts of these two constituents.

Approximately 6% of the ruthenium in the feed to the pot was volatilized to the pot condensate using a Mode A type operation. This agrees with the approximately 5% volatilization that occurred during the earlier PW-1 runs (excess sulfate and sodium was present in the early runs) with a Mode C type of operation* (feed from the evaporator to the pot with condensate returned to the evaporator for recycle). It was speculated at that time that the low volatilization may have been due to the mode of operation.

Auxiliary Process System Performance During Run PC-7 - J. N. Hartley

During Run PC-7, the WSEP auxiliaries were operated in a Mode A arrangement as illustrated in Figure 1. The radio-nuclide distributions for Runs PC-7 and PC-8 are summarized in Table 4.

*BNWL-677, 67-3

FIGURE 1. WSEP Auxiliary Process System

TABLE 4. Radionuclide Distribution in WSEP Auxiliaries for Runs PC-7 and PC-8

	Fraction of Equivalent Feed Radioactivity for Total Run in Effluent Streams			
	PC-7 Mode A		PC-8 Mode A	
	^{106}Ru	$^{144}\text{CePr}$	^{106}Ru	$^{144}\text{CePr}$
Evaporator (TK-113)	6.1×10^{-2}	1.5×10^{-3}	1.1×10^{-2}	1.2×10^{-2}
Fractionator (TK-115)	2.6×10^{-3}	4.6×10^{-7}	1.4×10^{-3}	2.2×10^{-6}
Fractionator Distillate Receiver (TK-116)	4.6×10^{-4}	NDG	2.8×10^{-4}	1.7×10^{-8}
Off-Gas Scrubber (TK-118)	2.3×10^{-5}	NDG	2.0×10^{-5}	NDG
Off-Gas from Scrubber	4.8×10^{-8}	4.6×10^{-10}	5.2×10^{-7}	3.0×10^{-9}
Off-Gas to Stack	$<6.0 \times 10^{-10}$	$<1.0 \times 10^{-12}$	$<6.0 \times 10^{-8}$	$<9.0 \times 10^{-13}$
Ratio (Fractionator Distillate Radioactivity to 10CFR20)	2.0×10^3	---	2.7×10^4	7.5×10^2
Ratio (Scrubber Off-Gas Radioactivity to 10CFR20)	22	94	3.9×10^2	4.3×10^2
Ratio (Stack Off-Gas Radioactivity to 10CFR20)	$<4.0 \times 10^{-4}$	2.0×10^{-3}	$<1.0 \times 10^{-3}$	$<2.0 \times 10^{-2}$

NDG - No detectable gain in radioactivity

The overall accumulation of radionuclides in the auxiliaries was generally typical of previous WSEP runs. Only 6.1% of the ruthenium and 0.15% of the nonvolatiles represented by $^{144}\text{CePr}$ in the feed to the pot accumulated in the WSEP auxiliary evaporator. Figure 2 shows the accumulation of ^{106}Ru in the WSEP auxiliaries for Run PC-7 and PC-8.

Decontamination factors (DF's) across the solidifier condenser were 2.6×10^2 for ^{106}Ru and 4.3×10^3 for $^{144}\text{CePr}$ based on the average concentration of radionuclides in the solidifier condenser off-gas. The average concentration of radionuclides in the process off-gas at various points in the auxiliary systems are tabulated in Table 5. During Run PC-7 the solidifier condenser off-gas filter was bypassed to determine if the filter is needed with the pot calcination process. Since there was low volatilization of ruthenium, very little ruthenium carried through the off-gas system in the gas phase and did not result in additional contamination of the auxiliary system.

FIGURE 2. Accumulation of Radioruthenium in WSEP Auxiliaries During Runs PC-7 and PC-8

TABLE 5. Average Concentration of Radionuclides in Process Off-Gas During Runs PC-7 and PC-8

PC 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	$^{144}\text{CePr}$	^{106}Ru	$^{144}\text{CePr}$	^{106}Ru	$^{144}\text{CePr}$	^{106}Ru	$^{144}\text{CePr}$
7	1.4×10^{-4}	7.5×10^{-5}	---	---	1.3×10^{-5}	2.8×10^{-5}	1.9×10^{-5}	3.6×10^{-8}
8	1.0×10^{-3}	6.1×10^{-3}	4.8×10^{-6}	8.3×10^{-6}	3.6×10^{-7}	1.6×10^{-5}	6.8×10^{-6}	4.6×10^{-8}

PC Run Number	Off-Gas from High ^(a)		Final Off-Gas from Scrubber			
	Efficiency Filter, $\mu\text{Ci}/\text{cm}^3$		$\mu\text{Ci}/\text{cm}^3$		Ratio to 10CFR20	
	^{106}Ru	$^{144}\text{CePr}$	^{106}Ru	$^{144}\text{CePr}$	^{106}Ru	$^{144}\text{CePr}$
7	3.1×10^{-6}	2.7×10^{-5}	1.1×10^{-8}	3.8×10^{-8}	55	95
8	1.5×10^{-5}	1.2×10^{-5}	7.9×10^{-8}	1.7×10^{-7}	4.0×10^2	4.3×10^2

a. The high concentration of radionuclides in the off-gas from the process filter is probably due to internal contamination of sampling line.

The auxiliary evaporator operated on about 5M HNO_3 . Instantaneous DF's across the evaporator from bottoms to condensed overheads varied from a low of 48 to 4.4×10^3 for ^{106}Ru and a low of 3.0×10^2 to 3.0×10^4 for $^{144}\text{CePr}$. The fraction of equivalent feed radioactivity that accumulated in the fractionator was 2.6×10^{-3} for ^{106}Ru and 4.6×10^{-7} for $^{144}\text{CePr}$ which indicated cumulative DF's across the evaporator of 30 for ^{106}Ru and 3.5×10^3 for $^{144}\text{CePr}$.

The instantaneous ruthenium DF's across the fractionator varied from a low of 36 to 6.2×10^3 . No $^{144}\text{CePr}$ was detectable in the fractionator distillate. The concentration of ^{106}Ru in the accumulated fractionator distillate was above the 10CFR20 release limits by a factor of 2×10^3 .

The fraction of equivalent ruthenium that accumulated in the off-gas scrubber was 2.3×10^{-5} while 4.8×10^{-8} escaped in the scrubber off-gas. The concentration of radionuclides in the scrubber off-gas was above 10CFR20 release limits by factors of 22 for ^{106}Ru and 94 for $^{144}\text{CePr}$. After the scrubber off-gas was filtered twice more and diluted by the building ventilation air, the radionuclide concentrations in the off-gas to the stack were less than 0.2% of the 10CFR20 release limits.

Pot Calcination Run PC-8 - W. R. Bond and J. L. McElroy

The thirty-second radioactive demonstration in WSEP was successfully completed with pot calcination Run PC-8. Run PC-8 was the last standard pot calcination run as the following and final run in WSEP was a rising level glass run.

During Run PC-8, a total of 340 liters of simulated PW-4m waste at 459 liters/tonne were fed to the pot in 36 hours. Another 8 hours were required to dry and cool the pot. The 34 liters of calcine product contained approximately 1,200,000 curies of fission product radioactivity (including 2100 curies of radoruthenium) to give an internal heat generation rate of

approximately 4700 watts (138 W/liter) in the 6-inch diameter, stainless steel receiver pct. A summary of the data for Run PC-8 is given in Table 2 and the feed composition for the run is given in Table 3.

The feed was started to the pot at 18 liters/hr and was terminated at a feed rate of 3 liters/hr. The average feed rate was 9.4 liters/hr. When the pot liquid level reached the upper portion of the pot, attempts were made to control the feed rate automatically with the thermal-probe liquid-level indicator; however, as in Run PC-7, this was unsuccessful due to the poor response of the control system. For this run, the steam-purged pressure tap was extended down to the pot fill level (34 liters) and served very well during the latter portion of the run as a level indicator. No difficulties with foaming were experienced during the run indicating that the addition of antifoam was effective. This was particularly noteworthy in view of the small diameter (6 inches) of the pot. The volume reduction of the PW-4m feed was similar to that of the PW-1 feed in PC-8 (a factor of 10) with an apparent calcine density of 1.1 kg/liter being attained.

The average calcine centerline and wall temperatures of 848 and 263 °C, respectively, under the condition of free-air cooling indicated an effective thermal conductivity of $0.34 \text{ W/(m}^2)(^\circ\text{C/m)}$.

Auxiliary Process System Performance During Run PC-8

J. N. Hartley

Again as in Run PC-7 the auxiliaries were operated in a Mode A arrangement. During Run PC-8, only 1.1% of the ruthenium fed to the pot volatilized and accumulated in the auxiliary evaporator while 1.2% of the nonvolatiles represented by $^{144}\text{CePr}$ were entrained. This entrainment was a factor of 7 greater than that occurring during Run PC-7. As seen in

Figure 3, the entrainment from the pot calciner occurred mainly during the initial part of the run for both Runs PC-7 and PC-8; however, this does not explain the factor of 7 increase in entrainment during Run PC-8. Further evaluation is necessary to explain the higher entrainment during PC-8.

The auxiliary evaporator operated with an average bottoms acidity of 6M HNO_3 and ranged from 4 to 8M HNO_3 . Instantaneous DF's across the evaporator varied from less than 10 to greater than 10^4 and were typically greater than 10^3 for ^{106}Ru . Instantaneous DF's across the evaporator were typically greater than 10^4 for $^{144}\text{CePr}$. The fraction of equivalent feed radioactivity that accumulated in the acid fractionator was 1.4×10^{-3} for ^{106}Ru and 2.2×10^{-6} for $^{144}\text{CePr}$. These values were in general agreement with those of Run PC-7 except that the fraction of equivalent $^{144}\text{CePr}$ that accumulated in the fractionator during Run PC-8 was a factor of 5 greater than that during Run PC-7. These values indicated cumulative DF's across the evaporator of 82 for ^{106}Ru and 5.4×10^3 for $^{144}\text{CePr}$.

Instantaneous DF's across the fractionator ranged from a low of 16 to 3.3×10^2 for ^{106}Ru and from 4.6×10^2 to 3×10^3 for $^{144}\text{CePr}$. The concentration of radionuclides in the final aqueous effluent (fractionator distillate) was above 10CFR20 release limits by factors of 2.7×10^4 for ^{106}Ru and 7.5×10^2 for $^{144}\text{CePr}$.

The fraction of equivalent ruthenium that accumulated in the off-gas scrubber was 2.0×10^{-5} while 5.2×10^{-7} escaped in the scrubber off-gas. There was no accumulation of $^{144}\text{CePr}$ in the off-gas scrubber. The concentration of radionuclides in the scrubber off-gas was above 10CFR20 release limits by factors of 3.9×10^2 for ^{106}Ru and 4.3×10^2 for $^{144}\text{CePr}$. The radionuclide concentration in the final off-gas to the stack were less than 2% of the 10CFR20 release limits.

FIGURE 3. Entrainment from Pot Calciner During Runs PC-7 and PC-8

Rising Level Glass Run PC-9(RLG) - W. R. Bond and J. L. McElroy

The thirty-third and final radioactive demonstration in WSEP was completed with rising level glass Run PC-9(RLG). During Run PC-9(RLG), 393 liters of simulated PW-4m waste at 706 liters/tonne were fed to the pot where it was continuously concentrated, calcined and fused to a borosilicate glass product (50 wt% waste oxides). Sodium and boric acid additives necessary for fusion were added directly to the feed tank while the required silica additive was blended in-line with the feed as an aqueous suspension just prior to entry into the pot. To increase the processing rate of the 6-inch diameter pot from the 3 liter/hr rate which would be otherwise attained, the feed was added filmwise down the pot centerline thermowell. The intermediate thermowell in the pot was to be used as an indicator of the level within the pot. A summary of the data for Run PC-9(RLG) is given in Table 2 and the feed composition for the run is given in Table 3.

The feed was started to the pot at 15 liters/hr without any difficulties, however, it was apparent shortly after the start of the run that in addition to feed flowing down the centerline thermowell, the feed was also wetting the intermediate thermowell as indicated by a decrease in the top intermediate pot temperatures. After 2.5 hours of feeding, the pot feed was switched from waste to water due to the progressive decrease in the top intermediate pot temperatures. While feeding water for 40 minutes the intermediate temperatures increased to where they were initially, but started decreasing once waste feed was again started to the pot. The intermediate temperatures eventually leveled out and feeding progressed smoothly; however, it was noted that with exception of the bottom zone of the pot, the intermediate temperatures were not responding to the rising pot level as in early nonradioactive runs. This made it difficult to determine the pot liquid inventory. The

intermediate temperatures only responded when it was apparent that melt had formed. After approximately 25 hours of feeding, oscillations in the pot pressure indicated that liquid was contacting the pot pressure tap (located 18 inches down from the top of the pot) and a reduction in feed rate (13 to 10 liters/hr) was necessary to correct the situation. However, 5 hours later it was necessary to terminate feeding for approximately 30 minutes as the result of high liquid level indications. During the next 26 hours, feeding to the pot was interrupted five times due to high liquid level in the pot and as a result only 55 liters of feed were fed to the pot in 12 hours of actual feeding during this period. During one of these interruptions the pot was allowed to bake for approximately 7 hours so that any calcine deposits or bridges in the upper portion of the pot might be melted down, but no improvement in pot capacity was noted. The run was terminated following the last interruption in feed.

Inspection of the pot after the run revealed a partial calcine plug in the pot neck (approximately 75% reduction in cross-sectional area) and considerable calcine (2 to 3 liters) above the pot baffle (the baffle is 4 inches in diameter and centered 13 inches down from the top of the pot). The calcine in the pot neck and a portion of that on the baffle was most likely due to entrainment. Indications of high entrainment were apparent early in the run as the absolute filter downstream of the solidifier condenser started to plug immediately after feed was started and had to be bypassed. Also approximately 20 hours into the run the vent system downstream of the condenser started plugging and had to be flushed. However, analysis of the accumulated condensate indicated that only 0.4% of the radioactivity fed to the pot during the run was entrained from it which was not any higher than in previous pot caliner runs. The cause of the apparent plugs in the vent system has not been resolved at this time.

The 393 liters of feed added to the pot during the run was only 65% of the total planned. The feed contained 1,300,000 curies of fission product radioactivity (including 1600 curies of radioruthenium) and had a heat generation rate of approximately 4600 watts. The feed rate averaged 11.3 liters/hr for the first 86% of the run with an overall average feed rate of 9.1 liters/hr for the entire run. The glass level in the pot was estimated at 34 inches or 16 liters based on pot temperatures. This was only 86% of the product volume anticipated from the volume of feed added, indicating that the remaining 14% of the material was lodged in the upper regions of the pot.

The average glass centerline and wall temperatures of 720 and 388 °C, respectively, in the air-cooled furnace indicated an effective thermal conductivity of $1.1 \text{ W/(m}^2)(^\circ\text{C/m)}$ based on a heat rate density of 250 W/liter. This thermoconductivity is in the same range as that of the BNL phosphate glass and the in-pot melting borosilicate glass products. During processing, the rising level glass product behaved as the other products in that the effective thermoconductivity increased significantly when it became molten. At an average product temperature of 900 °C the rising level glass product had an effective thermoconductivity of approximately $2.0 \text{ W/(m}^2)(^\circ\text{C/m)}$. This is approximately 40% lower than that observed for the in-pot melting borosilicate glass product at the same temperature and may be due to the higher viscosity (poorer mixing) of the rising level glass product because of its higher waste oxide content.

Although the planned volume of feed was not processed, the performance during the run was basically satisfactory particularly in view of the fact that the rising level glass process had not received the high degree of development on a nonradioactive basis which had been accorded all other WSEP solidification techniques. High points of the run were

(1) a borosilicate glass product having a relatively high thermal conductivity was produced using the rising level glass concept, (2) film-feeding down the pot centerline thermowell gave an approximately three-fold increase in capacity; however, it must be assured that the feed does not wet other internal pot surfaces other than the centerline well, and (3) the boric acid was added to the feed tank without causing any solution handling problems by maintaining the solution temperature above 60 °C.

WSEP AUXILIARY STUDIES

PROCESS CONTROL OF WSEP AUXILIARY SYSTEMS -

R. D. Dierks and J. N. Hartley

During Runs PC-7, 8, and 9, the operation of the evaporator and the acid fractionator were, for the most part, automatically controlled by three interacting, cascade control loops. One loop maintained the volume of the evaporator bottoms constant by regulating the steam flow to the evaporator tube bundle (evaporator boil-up) as a function of the weight factor of the evaporator contents. A second loop maintained the acid concentration constant in the evaporator by controlling the rate at which recycle stripwater was added to the evaporator in proportion to the conductivity of the condensed overhead vapors from the evaporator. As the overhead vapors were in equilibrium with the evaporator bottoms, the conductivity or acidity of the condensed vapors was an indirect, but accurate, measure of the bottoms acidity. Thus as the overhead acidity increased, the recycle stripwater flow rate increased, increasing the volume of the evaporator bottoms, the boilup rate and the acid removal rate from the bottoms, thereby reducing the acidity in the bottoms. Boilup in the fractionator was controlled as a function of the weight factor of the fractionator contents and as the recycle increased to the evaporator, the increased evaporator boilup supplied increased feed

to the fractionator, which in turn increased its boilup, supplying an increased volume of condensate to replenish the recycle surge tank (fractionator condenser).

It had been shown in previous tests that to avoid overshoot in the steam control loops it would be necessary to operate the weight factor controller with zero reset. The General Electric 412 process control computer, normally used as a data logging tool, was programmed to accomplish this, as the reset could not be removed completely from the available three mode controllers. The process computer established the set points for the two steam flow controllers as a function of the weight factor of the contents of the two tanks.

Pertinent data obtained from Run PC-7 is shown in Figure 4. Two blown fuses in the computer output section prevented the control system from functioning prior to the start of the feed to the solidifier, accounting, in part, for the lack of control during the start up period. With the computer functioning, and the volume of the evaporator bottoms reduced to the proper value, the control of the evaporator bottoms acidity was quite effective. While the steam flow to the fractionator tube bundle was quite stable under weight factor control, it was necessary to periodically increase the weight factor set point to keep the acid concentration down as acid accumulated during the process of the run.

Control of the evaporator bottoms acid concentration was compromised in the initial part of Run PC-8 by a faulty start-up procedure, and by an unexplained failure of the overhead conductivity sensor during the remainder of the run, resulting in the recycle flowcontrol set point being manually adjusted rather than being cascaded from the conductivity measurement. The two steam flow control loops were quite stable and held their respective volumes reasonably constant. Inspection of

FIGURE 4. Auxiliary Evaporator and Fractionator Operating Parameters

the data from the fractionator system suggested that the fractionator boilup might be successfully controlled as a function of the computer calculated specific gravity, even though this system did not prove feasible during earlier tests using conventional analog control instrumentation.

Run PC-9(RLG) was again compromised by a failure of the overhead conductivity sensor during most of the run; however, when it was functioning all three of the cascade systems performed quite well. Weight factor control of the evaporator boilup was again very satisfactory. Specific gravity control of the fractionator worked quite well, producing only a mildly erratic boilup pattern. The failure of the conductivity sensor was attributed to the extremely low flow of condensate through the sensor well, resulting in the sensor being immersed in a nonrepresentative sample. Conductivity measurements were roughly correlatable with the specific gravity of the bottoms as long as the evaporator boilup remained relatively high. As the recycle rate and the evaporator boilup decreased, conductivity values stabilized while specific gravity values continued changing. In general Run PC-9(RLG) demonstrated the feasibility of completely automatic interrelated control of the acid recovery auxiliary system.

LABORATORY STUDIES

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

The corrosiveness of a simulated LMFBR type waste solution was determined in boiling tests with 304L stainless steel, 310 stainless steel, and titanium corrosion specimens. Corrosion resistance was in the order titanium > 310 > 304L. Corrosion rates varied from 0.18 to 0.0001 in./yr.

Corrosion rates of 304L stainless steel, titanium, and Zircaloy-2 were determined in simulated boiling PW-1 and PW-2 solutions which contained the glass forming additives required to form a phosphate glass melt and also in PW-1 and PW-2 solutions containing spray solidifier melt additives. Corrosion rates of 304L, titanium, and Zircaloy-2 in the PW-1 phosphate glass feed solutions after one month's exposure were about 0.18 to 0.004, and 0.006 in./yr, respectively. Corrosion rates in the PW-2 solution and in the solutions containing spray solidifier type additives were somewhat lower.

RUTHENIUM RECOVERIES IN WSEP SOLIDIFIED WASTE PRODUCTS -

M. R. Schwab and F. E. Holt (WADCO)

Tables 6 and 7 present the ruthenium recoveries in the WSEP waste products as determined with the Direct Count method described in a previous report.* The best ruthenium balances were obtained for the most recent WSEP runs since both feed samples and melt samples were available for direct counting. No feed samples remained from the earlier WSEP runs and the gamma emission analysis (GEA) data was used for these samples. The direct count data confirmed the suspected loss of ruthenium during spray solidification Run SS-4, however, the whereabouts of the missing material was not determined. The direct count method was developed as a nondestructive analysis for radioruthenium in the WSEP solidified waste product samples. Gamma emission analysis of aqueous solutions of the waste products proved inadequate due to incomplete sample dissolution and volatilization of ruthenium during the chemical dissolution procedure. Although a closed-bomb dissolution procedure has since been developed, the direct count method has proven to be rapid, nondestructive, and sufficiently accurate to

*BNWL-1401, 70-1

TABLE 6. Radionuclide Recoveries in Phosphate Glass Melt Samples

PG Run No	Melt Sample Recoveries, % ^(a)		Total Recovery, % ^(b)
	¹³⁷ Cs	¹⁰⁶ Ru	¹⁰⁶ Ru
1	52	51	62
2	58 ± 15 (c)	56 ± 10	65
3	90	82	92
4	103 ± 31	133 ± 41	135
5	122 ± 29	92 ± 13	103
6	NA (d)	24 ± 4	29
7	108 ± 17	70 ± 13	84
8	105 ± 17	60 ± 12	63
9 (f)	97 ± 15	91 ± 12	97
10 (f)	NA	NA	NA (e)
11 (f)	94 ± 17	84 ± 12	91

TABLE 7. Radionuclide Recoveries in Spray Solidified Waste Melt Samples

SS Run No	Melt Sample Recoveries, % ^(a)		Total Recovery, % ^(b)
	¹³⁷ Cs	¹⁰⁶ Ru	¹⁰⁶ Ru
1	NA (d)	NA	75
2	NA	NA	74
3	NA	NA	71
4	84 ± 20 (c)	17 ± 2	28 (g)
5	78 ± 14	17 ± 3	87
6	99 ± 12	34 ± 6	84
7	92 ± 16	14 ± 4	75
8	117 ± 29	50 ± 10	90
9	97 ± 6	19 ± 4	76
10	102 ± 13	31 ± 4	78
11 (f)	95 ± 3	18 ± 7	91
12 (IPM) (f)	135 ± 2	103 ± 14	105
13 (IPM) (f)	95 ± 4	113 ± 9	115

- a. Based on direct count data and 100% recovery of ¹⁴⁴CePr in feed to solidifier
- b. Includes ¹⁰⁶Ru found in solidifier condensates
- c. Standard deviation given as ± value where available from counting statistics
- d. NA = Not Available
- e. Feed prepared from cerium solution; essentially no ruthenium present in feed
- f. Feed samples and melt samples direct counted; all other data based on past GEA results
- g. Low value attributed to deposition of ruthenium somewhere in the system.

determine the distribution of radionuclides in the solid waste product samples. To date, all available WSEP melt samples have been direct counted and the method is presently being used to analyze the core-drill samples from the Product Evaluation Program.

EVALUATION OF SOLIDIFIED WASTE PRODUCTS

NONRADIOACTIVE LABORATORY STUDIES

Strontium Leach Rates - F. P. Roberts

Strontium-90 is the isotope of most biological concern of all the fission product activities in solidified high level waste. For this reason the leachability of strontium is being measured on a series of simulated phosphate glass and spray solidifier products.

An analytical method has been developed which will measure strontium concentration in the range expected to be found in the leach test solutions, i.e., 0.01 to 50 ppm. The basis for the method is a carrier precipitation using lanthanum carbonate. The precipitation serves to concentrate the strontium and to remove the anionic impurities which interfere (mainly phosphate and sulfate). The actual measurement is by flame absorption or emission photometry. This method has proven successful and the strontium leach rate measurements are in progress.

Characterization of Other Solidified Products - F. P. Roberts

Evaluation of the pertinent physical and chemical characteristics of products from the phosphate glass and spray solidification processes are essentially complete. Work is now in progress to perform some of the same measurements on the calcine product from the pot solidification process the borosilicate products from the rising level glass and the in-pot-melting processes. In addition, some measurements are being

made on a number of simulated products from British, German, French, and Canadian processes to provide data which will permit comparison of their characteristics with U.S. processes.

RADIOACTIVE LABORATORY STUDIES

C-Cell Testing - F. P. Roberts and C. Hampton

The installation and testing of equipment in C-Cell for performing product characterization tests on radioactive specimens from WSEP pots has been completed, and testing of two core drilled samples from PG-7 is now underway.

The two core samples were drilled at 24 inches and 37 inches from the top of PG-7 pot and 90° radially from each other as shown in Figure 5. Core 1 had broken into several pieces during drilling but one satisfactory test wafer was obtained from the pot-wall side. Core 2 was intact, and was about 2.75 inches long. Three test wafers were obtained from it. A photograph of Core 2 is shown in Figure 6.

FIGURE 5. Location of Samples from PG-7 Pot

FIGURE 6. Photograph of Core 2 from PG-7 Pot

BNWL-1533

Observations of the two specimens indicate they consist mainly of devitrified glass of a reddish brown color with traces of glassy black material.

Tests performed or in progress include calorimetry, dispersibility, leachability and direct gamma scanning.

PRODUCT MEASUREMENT, TESTING, AND STORAGE

Core Drilling and Pot Welding - J. A. Yount.

Final testing and installation was completed on the core drill for the Solids Storage Engineering Test Facility (SSETF). This effort culminated a 12-month design, fabrication, and non-radioactive testing program to produce a machine that could:

- Drill both a 5/16-inch steel wall and 3 inches of a glass or ceramic product,
- Grind flat the area around the hole in the pot in preparation for welding
- Have all its components disassembled and replaced and accurately realigned with a minimum of effort
- Fit comfortably into the limited space available in SSETF.

Radioactive core drilling was started with stainless steel pot PG-7. A high speed hole saw was first used to penetrate the pot wall and expose the smooth, almost ceramic appearing product. This part of the operation went more smoothly than the nonradioactive tests, with the saw producing a clean, round, smooth cut, and tool run out at a minimum.

The drilling of the product itself was approached cautiously since in the nonradioactive tests "gumming" of the product at higher temperatures had caused plugging of the air cooling passages in the bit, subsequent overheating, and finally the destruction of the sample. Also, mislabelling by the manufacturer has resulted in a drill motor with only two-thirds the speed ideal

for phosphate glass cutting. Therefore, the actual core sampling was performed with the drill motor speed and cooling air on full and the feed at the lowest possible setting, requiring approximately 6 hours for a successful sampling 3 inches deep, compared with the usual 2 hours required.

During drilling, variations in the drill motor speed indicated cracks or voids in the product after coring approximately 2 inches into the product. Later examination of the fractured sample verified cracks in this region. A second sample was taken and proved to be continuous and of more than adequate length for the tests performed in C-Cell.

After the faces of the holes were ground flat for welding, a first attempt at sealing the holes began. Due to the high temperatures of the product during the welding process, the tightest possible fit between plug and hole is desired. A shrink-fit attempt with the first plug failed due to lack of clearance; a smaller plug was tried--it failed. Nonradioactive tests were performed to further identify the problem, and it was determined several things were interfering with the welding process:

- The high temperature of the radioactive pot caused what appeared to be off-gas problems; the intensity of these problems varied greatly depending on the nature and temperature of the product prior to welding.
- Welding plug fit-up tolerance was inconsistent.
- To avoid cracking in the weld, the 310 stainless steel pot required a considerably different technique for welding than did the 304L stainless steel pots previously tested.

Since an unsuccessful welding attempt in cell could damage the hole beyond further repair, a very carefully defined series of nonradioactive tests were undertaken to solve the problem.

To prevent the product's interference with the welding process, a tool was designed to carve the product away from the pot wall on the back side. The complete machining process was rewritten to insure the absolute minimum amount of contamination on the metal surface near the weld. The welding plug itself was redesigned with a heavier lip for more plug metal flow during the welding process, and greater relief on the inside to allow room for the product to flow as it melts while the plug is being welded in place.

Before the metal plug was inserted, the hole was refaced with a new grinding wheel and then reamed to produce a very smooth round hole of known dimensions. This allows a shrink fit (0.002 inches interference) between the plug and the hole.

One key to the problem of cracking in the welding of stainless steel is the ferrite composition of the weld. The pot presently being drilled is 310 stainless steel which is entirely devoid of ferrite. To increase the ferrite composition, and reduce the tendency to crack, a 304L stainless steel plug with certified chemical composition high in ferrite (10%) was used, and the joint was designed so the finished weld was predominantly 304L.

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