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QUARTERLY PROGRESS REPORT
RESEARCH AND DEVELOPMENT ACTIVITIES
FIXATION OF RADIOACTIVE RESIDUES
JULY-OCTOBER, 1967

A. M. PLATT

MARCH, 1968

AEC RESEARCH &
DEVELOPMENT REPORT

Author		Title		Date	

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RICHLAND, WASHINGTON

operated by

BATTELLE MEMORIAL INSTITUTE

for the

UNITED STATES ATOMIC ENERGY COMMISSION UNDER CONTRACT AT(45-1)-1830

PRINTED BY/ FOR THE U. S. ATOMIC ENERGY COMMISSION

3 3679 00061 0974

BNWL-677
UC-70, Waste Disposal
and Processing

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By

Members of the Staff
of the Chemistry Department
and
Environmental and Radiological Sciences Department

Edited by
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Chemical Development Section

March 1968

FIRST UNRESTRICTED
DISTRIBUTION MADE

APR 2 '68

PACIFIC NORTHWEST LABORATORY
RICHLAND, WASHINGTON

Printed in the United States of America
Available from
Clearinghouse for Federal Scientific and Technical Information
National Bureau of Standards, U.S. Department of Commerce
Springfield, Virginia 22151
Price: Printed Copy \$3.00; Microfiche \$0.65

PREVIOUS QUARTERLY REPORTS

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INTRODUCTION

This progress report is the thirty-fourth 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

During pot calcination Runs PC-4 and PC-5, 650,000 and 1,150,000 Ci of radioactivity were successfully solidified in calcine. This corresponded to 2600 W of self-generating heat in a 12 in. pot for Run PC-4 and 4300 W in the 8 in. pot used for Run PC-5. Temperature differences between the pot centerline and wall were 312 and 495 °C, respectively, for the two pots.

Heat generation rates ranging from 1.1 to 4.8 kW were measured in five Waste Solidification Engineering Prototypes (WSEP) waste pots. Results were within 90% of estimates based on material balances.

The spray solidifier melter furnace, which failed during startup of WSEP Run SS-3, was repaired remotely and successfully tested by heating to 1210 °C.

Results of work performed in the developmental spray calciner indicate:

1. During continuous melting, PW-2 waste containing excessive flux can foam out of the heated receiver pot as a result of sulfate volatilization.

2. Modification of the PW-1 composition may be required to alleviate problems of excessive calciner filter pressure drop.
3. The use of finely divided NaPO_3 as a flux for melting directly in the waste container increases the corrosion of the container; the total wall corrosion still amounted to only 0.02 in. well within the design allowance of 0.125 in.

Three Glass Melter Condensate treatment methods were studied in detail in order to provide a basis for full level demonstration of treatment methods for a secondary condensate generated in the Phosphate Glass Solidification Process. These methods were 1) sulfate precipitation with barium, calcium, and lead; 2) hydrous oxide scavenging; and 3) volatilization of ruthenium from permanganate treated Glass Melter Condensate. A combination of ruthenium volatilization, hydrous oxide scavenging, and a phosphotungstate precipitation step for cesium removal followed by asphalt incorporation has potential as a highly effective treatment method.

POT SOLIDIFICATION

RADIOACTIVE DEMONSTRATION - RUNS PC-4 AND PC-5 -

J. L. McElroy

Pot calcination runs PC-4 and PC-5 were made using Purex Waste-1 (PW-1) feed. This waste is normally free of sulfate; however, because of excessive sodium in the Purex 1WW and ^{144}Ce solutions available for feed makeup, an equivalent amount of sulfate was added to prevent formation of sodium and cesium oxides which would volatilize. The resultant feed compositions (Table I) were 0.26 and 0.35M in sulfate for PC-4 and PC-5, respectively.

A major objective of the runs was to demonstrate a Mode C operation (see Figure 1) and determine the degree of retention of the fission products resulting from this type of operation. Mode C consists of feeding the pot directly from the boiling waste evaporator and continuously recycling all of the pot condensate directly to the evaporator. Waste is continuously supplied to the evaporator to replenish that fed to the pot.

TABLE I. Feed Compositions for Runs PC-4 and PC-5 in WSEP

Component	Concentration, M ^(a)	
	Run 4	Run 5
H ⁺	--	--
Fe ⁺³	0.82	0.80
Cr ⁺³	0.012	0.012
Ni ⁺²	0.005	0.005
Al ⁺³	0.11 ^(b)	0.13 ^(b)
Na ⁺	0.474	0.64
NO ₂ ⁻ -NO ₃ ⁻	6.5	6.5
PO ₄ ⁻³	0.003	0.003
U ⁺⁶	0.01	0.01
SiO ₃ ⁻²	~0.01	~0.01
Hg ⁺²	--	--
F ⁻	--	--
Fission Products		
Cs ⁺ (added K ⁺)	0.042	
Sr ⁺²	0.0155	
Ba ⁺²	0.0195	
(Y + Re) ⁺³	0.12	
ZrO ⁺²	0.065	
MoO ₄ ⁻² + Tc as MoO ₄ ⁻²	0.079	
Ru ⁺²	0.0032	
Ru ⁺² (added Mn ⁺²)	0.0288	
Rh ⁺² (added Co ⁺²)	0.0074	
Pd ⁺² (added Ni ⁺²)	0.0172	
Ag ⁺² + Cd ⁺² (added Cu ⁺²)	0.0016	
Te (added SO ₄ ⁻²)	0.0064	
Additive to Feed		
SO ₄ ⁻²	0.26	0.35
Pertinent Data at Final Volume		
M _T ⁺	4.28	4.45
Oxide weight/liter feed, kg	0.167	0.167
Final adjusted waste volume, liters/tonne	370	410
Sp. gr. at 25 °C	1.37	1.35
Vol% solids	6	13
Heat in feed, W/liter	2.4	8.3

(a) Molarities are at 378.5 liters/tonne

(b) Aluminum was used as an iron substitute due to excess aluminum in the Purex waste stock used for feed makeup.

(1) NaOH added to PC-4 only

FIGURE 1. Equipment Arrangement for Pot Solidification Mode C for PC-4 and 5

Run PC-4

During PC-4, a total of 650,000 Ci, including 47,000 Ci of radio-ruthenium and a ^{90}Sr spike of 30,000 Ci were fed to the 12 in. pot in 42 hr. The ^{90}Sr spike was added for the purpose of providing a long-term radiation exposure to the calcine. The average feed rate was 26 liters/hr. The feed rate was higher than expected by approximately 25%, primarily because there was concentrating and a resulting volume reduction of the feed in the waste evaporator prior to feeding to the pot.

During the first few hours of feeding, a severe foaming condition was encountered in the pot. Approximately 20 liters of concentrated feed was expelled from the pot through the safety relief seal pot. The relief system functioned normally and no plugging occurred. After feeding water to the pot for approximately 1 hr, the run was continued successfully. A reduced feed rate was used (35 liters/hr versus the original 55 liters/hr) to maintain the liquid (or foam) level lower in the pot to allow more freeboard for foaming. No further foaming was observed, however.

After the feed was shut off, the pot was dried and then cooled by forced-air cooling of the furnace susceptor. The drying and cooling required 33 hr. The resulting overall processing rate, not including pot changeout time, was 14.4 liters/hr, which is equivalent to processing nearly one tonne (0.94) per day of power reactor waste.

Wastes equivalent to 2.96 tonnes of 4.7 year old 20,000 MWd power reactor fuel were calcined to reach a self-generating heat density of 25 W/liter in the calcine (2600 W total in pot). At steady state with the 12 in. diam pot suspended in 38 °C air, the pot wall to centerline temperature difference was 312 °C with a maximum centerline temperature of 477 °C. Additional operating parameters and collected data for runs PC-4 and PC-5 are listed in Table II.

One of the principal features of the Mode C equipment arrangement is recycle of the pot condensate as a blend with incoming feed to the pot. Thus a constituent which is volatilized from the pot will continue to

TABLE II. WSEP Pot Solidification Operating Parameters and Results

Operational Data	Run Number	
	PC-4	PC-5
Date	6/67	7/67
Feed type	PW-1 ^(a)	PW-1 ^(b)
Feed, liters/tonne	365	410
Pot diameter, in.	12	8
Pot material	304-L	304-L
Run feed time, hr	42	28
Average feed rate, liters/hr	26	18.6
Drying + cooling, hr	33	11
Total feed, liters	1080	520
Total feed, tonne equiv.	2.96	1.27
Feed: solid vol ratio	10	8.7
Operating mode	C	C
	1. Foaming in pot calciner	1. Foaming in pot calciner
	2. Plugging of evap. dip tube	2. Plugging of evap. dip tube
<u>Fraction of Activity Distributed: Total Less Ru (T-Ru), and Ru</u>		
Through first cond., 10^{-3}	(T-Ru) NA ^(c)	NA
	(Ru) <30	30-80
Through evap. cond., 10^{-6}	(T-Ru) 8.0	0.29
	(Ru) 1700	310
Through fract. cond., 10^{-9}	(T-Ru) 10.4	25
	(Ru) 286	17,000
Off-gas to scrub, 10^{-9}	(T-Ru) 3.0	NA
	(Ru) 51	NA
Off-gas to stack, 10^{-12}	(T-Ru) <1.3	<0.7
	(Ru) <10	NA
Ratio activity to MPC (fract. cond.)	(T-Ru) 8	56
	(Ru) 26	1,000

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BNWL-677

TABLE II. (contd)

<u>Filled Pot Data</u>	<u>Run Number</u>	
	<u>PC-4</u>	<u>PC-5</u>
Total curies in feed to pot	650, 000	1, 200, 000
Curies of Ru in feed to pot	47, 000	18, 000
Solid in pot, liters	105	55
Solid in pot, kg	166	86
Solid in pot, liters/tonne	35	43
Heat in pot, W	2, 600	4, 300
Equivalent aging time, yr	4. 7	1. 5
Centerline temperature in air, °C	477	725
Centerline temperature in water, °C	370	NA
Δ Temperature centerline to wall, °C	312	495
Total corrosion, mm	NA	NA
Welded lid	yes	yes
Pot self pressurization, psi	NA	NA
Pot leakage	NA	NA
Radiation reading, R/hr at 6 in.	3.8×10^4	4.9×10^4

(a) SO₄ and Na were 0.26 and 0.47M, vice desired values of 0.10 and 0.14M, respectively.

(b) SO₄ and Na were 0.35 and 0.64M, vice desired values of 0.10 and 0.14M, respectively.

(c) NA--Not available

increase in concentration in the feed stream until some steady state concentration is attained. With this feature, another objective of this run was to observe recycle effects on ruthenium in the system. Less than 5% of the ruthenium in the total feed to the pot was volatilized as indicated by data from stream samples of the pot condensate. Final samples of the feed remaining at the end of the run revealed no increase in ruthenium and confirmed the low volatility. In earlier pot runs with PW-2 waste, 30% of the ruthenium was volatilized. Although the reason for the low ruthenium volatility in PW-1 is not known, it is thought that the high iron content of PW-1 may be a key factor.

Additional removal of ruthenium from the gas stream from the fractionator was sought by adding sodium hydroxide to the top plate of the acid fractionator tower during PC-4. In a comparison of decontamination factors for the same operating mode, the ruthenium decontamination factor across the fractionator for PC-4 was 30 times better than that for PC-5 where no additives were used.

Run PC-5

During PC-5, a total of 1,150,000 Ci, including 18,000 Ci of radio-ruthenium was fed to the 8 in. pot in 28 hr at an average feed rate of 19 liters/hr. Foaming of the feed in the pot was evident during the run; however, no feed was expelled from the pot as had happened during PC-4. Although the pot operating level was not maintained lower than normal for an 8 in. pot, a sufficient freeboard for foaming was maintained, especially during the high boil-off portion of the run, by limiting the initial feed rate to 25 liters/hr.

In the later stages of the run, when poor heat transfer through the calcine causes low feed rates, the foaming made the level control sufficiently difficult so that the feed was turned off after only 85% of the intended feed was in the pot. However, based on the weight and volume of calcine obtained, it was later estimated that no more than an additional 5% could have been processed, even under optimum conditions. The

temperature-type level probe in the pot was completely unreliable during the last half of the run; however, this performance may have resulted from foaming in the pot.

Heat generation during PC-5 (4300 W was approximately 85% of that required for a thermal maximum pot. (Thermal maximum is the maximum amount of heat generated by radioactivity that can be contained in a pot of solidified waste without exceeding an internal pot temperature of 900 °C or a pot wall temperature of 425 °C.) A series of furnace temperature reductions (as shown in Figure 2) were used to insure that all of the calcine within the pot had been heated to greater than 700 °C to make certain of complete denitration and also to prevent temperatures in excess of 905 °C where volatility of some constituents is possible. In addition to the 28 hr feeding period, 11 hr were required to complete the drying and cooling portion of the run.

As in Run PC-4, the process equipment was set up for Mode C operation, and the low volatility of ruthenium from the pot during PC-4 was similarly observed during PC-5 where approximately 3% of the ruthenium fed to the pot was volatilized. This low amount was insufficient for determining recycle data from the evaporator samples.

During both PC-4 and PC-5 the only significant problem encountered with the auxiliary equipment was plugging of the specific gravity dip tube in the waste evaporator. Plans to purge the dip tube with steam during PC-5 were foiled when the dip tube became completely plugged midway through the run. The evaporator sparger also became plugged with solids at some time during these two runs.

COMPUTER DATA LOGGING - R. D. Dierks

Main frame problems have largely been eliminated from the operation of the GE-412 process control computer. However, failure of the computer van air conditioning equipment compromised the performance of the machine on run PC-5 and to some extent on Run PC-6. Power consumption data were logged successfully during the last half of

FIGURE 2. Temperature Profiles PC-5

Run PC-5 and for all of Run PC-6. To test the reliability of the computer system, the computer was not shut down following the completion of the second half of Run PC-6, but continued to monitor the experimental equipment in the normal manner, with the exception that the peripheral equipment (typewriters, etc.) was not allowed to function. The computer operated for 26 days but was again shut down by an air conditioner failure. The reliability of the air conditioning system is being improved.

A third program to monitor the Continuous Phosphate glass process experiments has been assembled and stored on drum memory along with the programs for monitoring the Spray Solidification experiments and the Pot Calcination experiments. By the execution of a suitable startup code, any of the three programs can be brought into operation from the MONITOR program. The GE-412 Computer program written to perform the data processing function for the ultrasonic pot wall thickness testing was placed in drum memory and incorporated into the OPERATOR program library as Program 16.

SPRAY SOLIDIFICATION

EFFECT OF EXCESS NaPO_3 IN THE SPRAY SOLIDIFIER PW-2 FLOWSHEET - J. E. Mendel

The melt foaming observed in the pilot plant Run DSC-7 was duplicated in the laboratory by using an excess of NaPO_3 . Run DSC-7 was a spray solidifier PW-2 flowsheet run and nominally used 1.27M NaPO_3 added through a solids feeder to the melter. In the laboratory tests, no foaming occurred when 1.27M NaPO_3 was used; but foam sufficient to increase the melt volume by a factor greater than three formed when 2.3M NaPO_3 was used. NaPO_3 concentrations of at least 2.3M, as indicated by melt analysis, were reached in Run DSC-7 due to unsteady performance of the solids feeder.

Foaming did not occur in platinum vessels at temperatures up to 975 °C and with as much as 2.6M NaPO_3 . The foaming started only after the melt was poured into stainless or carbon steel containers. The steel

containers are normally held at 700 °C. The possibility of holding the steel containers at temperatures lower than 700 °C was investigated. Although melts containing 2.6M NaPO_3 could be poured into steel containers at 500 °C, the melt was too viscous to flow and fill the pots evenly. Foaming occurred when the containers were heated up to the slump point of the melt.

An interaction between the sulfate-containing melt and the steel surfaces is indicated because the foaming did not occur in platinum. Preliminary experiments indicate foaming can be almost completely avoided by coating the steel surface with a ceramic.*

DEVELOPMENTAL SPRAY CALCINER RUNS - W. R. Bond

Nonradioactive runs were continued to optimize the operation of the spray solidifier and to test flowsheets. The following developmental spray runs were made during the quarter:

- A. Run DSC-7 was carried out to evaluate a PW-2 flowsheet with solid NaPO_3 flux fed directly to the platinum melter and to test carbon steel final container.
- B. Run DSC-8 was made to test in-pot melting of PW-1 using NaPO_3 flux.
- C. Run DSC-9 was essentially a rerun of DSC-8, using larger granules of NaPO_3 to avoid having the flux stick to hot surfaces.

Conditions and results for Run DSC-7 are presented in Table III. The run was terminated after 8.5 hr when the melt receiver pot foamed over. Thirty-five percent of the pot's contents foamed either through the sample chamber into the melter furnace or out through the sample chamber window where it solidified as an extremely porous, greenish solid. Earlier in the run, there was an indication of foaming since the melt discharging from the melter was quite frothy.

* Sauereisen sealing cement, No. 33, powder, product of Sauereisen Cements Company, Pittsburgh, Pa.

TABLE III. Run Conditions and Results for Run DSC-7

Feed Type	PW-2
Atomizing Gas	Air
Calciner Furnace Temp, °C	680
Melter Furnace Temp, °C	950-1000
Melt Temp, °C	880-910 ^(a)
Receiver Pot Temp, °C	650-750
Feed Consumed, liters	131
Feed Concentration, liters/tonne	454
Feed On-Time, hr	8.5
Average Feed Rate, liters/hr	15.4
Solid Flux (NaPO ₃) Added, kg	17.4
Solid Flux (NaPO ₃) Desired, kg	14.0
Product Collected, kg	46.9
Pot Material	Mild Steel
Pot Diameter, in.	8

(a) These temperatures were noted near the surfaces of the melt and thus the temperature lower in the melt may have ranged higher than this but was not detected due to the thermocouple being shorted out at this point.

The cause of the foaming has been attributed to sulfate volatilization. The presence of only 2.5% of the total sulfur in the condensate and caustic scrub solution indicates that the overall volatilization was apparently not excessive and probably occurred only periodically during the run. Table IV presents a comparison between the analyzed and desired concentration of the major components in the feed. Although there are some deviations, they would not promote sulfate volatilization. In Table V, the analysis of three melt samples taken of the melter discharge during the run reveals a loss of sulfate during periods of relatively high NaPO₃ flux addition.

It appears that the foaming resulted from the particular combination of a relatively high flux concentration, a relatively high melt temperature, and the large length-to-diameter ratio of the receiver pot. The discharge of a melt having a high gas content into the receiver pot, sulfate volatilization in the receiver pot, and the relatively long escape

TABLE IV. Comparison of Analyzed and Desired Feed Composition for Run DSC-7

<u>Element</u>	<u>Concentration, moles/liter at 100 gal/tonne</u>	
	<u>Analyzed</u>	<u>Desired</u>
Fe	0.425	0.445
Na	0.92	0.930
Li	1.49	1.17
Ca	0.74	0.6
Al	0.23	0.25
P	1.38	1.62
S	0.79	0.87

path, and small escape area for gas leaving the pot caused the viscous melt to raise out of the pot like bread dough.

Other observations made during the run were:

1. The atomization of the PW-2 feed was adequate as no gross deposition of calcine occurred in either the calciner barrel or cone.
2. During post-run inspection of the calciner, it was noted that the spray nozzle air tip was partially plugged with calcined feed which reduced the orifice flow area substantially. Calcined feed was also noted inside the air tip. Testing of the nozzle revealed that the calcine had reduced the air flow at a given nozzle pressure by 40% and also deflected the spray 10 degrees off vertical. As no decrease in air flow was noted during the run and since no localized deposition was noted on the calciner walls, it was concluded that the blockage occurred after the run and was the result of inadequate flushing of the nozzle as indicated by the presence of feed solution in the feed line. This emphasizes the importance of adequately flushing the nozzle with water immediately after discontinuing feed.
3. Control of the solids addition rate was ragged because the desired solid addition rate was not established immediately

TABLE V. Melt Sample Analyses (Concentrations referenced back to feed at 100 gal/tonne)

Sample Number	Total Sodium, moles/liter	Sodium Added as NaPO_3 , (a) moles/liter	Sulfur, moles/liter	Deviation of Sulfur Content from Feed Composition, %	Appearance of the Solidified Samples
M1	3.5	2.6	0.66	-17	Very porous Dark green
M2	2.6	1.7	0.65	-16	Very porous Dark green
M3	1.6	0.7	0.85	+8	Dense Light green

(a) 1.27 moles/liter desired

and a period of overfeeding was experienced during the first half of run. This points out the necessity of establishing the addition rate shortly after the start of the run.

4. No bridging or plugging problems were experienced at the solids addition port in the melter cover as the solid NaPO_3 particles (3-8 mesh) had sufficient velocity to prevent sticking on the hot surfaces of the port. Plugging experienced in the past probably resulted from melt splashing up into the port.
5. An ultrasonic scan of the mild steel receiver pot after the run indicated that several areas on the pot wall had been reduced in thickness by as much as 15 mils. Considerable oxidative attack was noted on the outside of the pot. The pot had been at temperature for approximately 12 hr.

Run conditions and results for Runs DSC-8 and 9 are presented in Table VI. Excessive pressure drops developed across the spray calciner off-gas filters when simulated PW-1 waste was calcined. The pressure drop during Run DSC-8 ranged between 17 and 19 in. of water while Run DSC-9 was terminated when the pressure drop reached 23 in. of water. An overall decontamination factor of only 90 for particulates compared to instantaneous values of over 600 indicated that a burst of calcine passed through the filters at the start of Run DSC-8, even though the filters were coated with calcine from a previous run. At the start of Run DSC-9

TABLE VI. Run Conditions and Results for the Spray
Calciner Pot Melting Runs

Run No.	DSC-8	DSC-9
	PW-1 ^(a)	PW-1 ^(a)
Feed type	Air	Air
Atomizing gas	Air	Air
Calciner furnace temp, °C	700	700
Pot furnace temp, °C	850	850
Total feed, liters	109.8	63.8
Feed concentration, liters/tonne	378.5	378.5
Feed on-time, hr	8.0	4.7
Average feed rate, liters/hr	13.7	13.6
Solid flux (NaPO ₃) added, kg	41.8	26.3
Average NaPO ₃ concentration, moles/liter ³ of feed	3.7	4.0
Product collected, kg	55.6	33.0 ^(b)
Feed-to-product volume ratio	5.4	--
Product concentration, liters/tonne	70.3	--
Pot material	304-L SS	304-L SS
Pot diameter, in.	8	8

(a) Contained no melt-forming additives

(b) Includes 4.4 kg of calcine removed from the filters

gross quantities of calcine passed through the filters which were clean prior to the run and were deposited throughout the off-gas system. As was apparent by the existence of a very dense, 1/4 in. thick coating of calcine on the filters after the run, a very impermeable filter coat then formed which resulted in the high pressure drop terminating the run. The calcine produced during both these runs was much finer than that produced during previous runs in which melt-forming additives were added to the feed.

Condensate samples taken during Run DSC-8 indicated that less than 4% of the ruthenium in the feed was being evolved compared to the 50 to 60% experienced with feeds containing melt-forming additives.

The corrosion of the 304-L SS container during Run DSC-8 caused a 0.02 in. reduction in wall thickness as measured by an ultrasonic pulse echo method. This was well within the design corrosion allowance of 0.125 in. for an initial wall thickness of 0.33 in. Corrosion was immeasurably small during runs with coarsely granulated flux including Run DSC-9.

The increasing corrosion in Run DSC-8 is believed due to relatively pure NaPO_3 adhering to the walls. Laboratory tests show the pure flux is highly corrosive. The NaPO_3 solid flux used in this run was finely divided and tended to deposit on the hot surfaces. Coarse NaPO_3 normally used does not deposit on the hot surfaces.

SPRAY NOZZLE ATOMIZATION STUDIES - J. D. Kaser

Runs made in the prototype spray calciner have frequently resulted in heavy deposition on the inside surfaces. This suggests incomplete drying of the spray droplets. One way to improve drying is to reduce and control droplet size. A more complete understanding of the fundamental parameters affecting atomization is also highly important for developing accurate scaleup factors for the calcination equipment. Consequently, an investigation has been in progress to evaluate the parameters affecting atomization.

The spray calciner may be operated with either steam or air as the atomizing gas. It would be desirable to measure directly the effect of steam versus air on spray droplet size; however, this is not feasible due to condensation of the steam and/or heating and evaporation of the liquid drops by the steam. Consequently, an indirect approach was taken and the effects of the noncondensable gases--helium and argon--were measured and compared to air. The most variable gas property here is density, while the viscosities of these three gases are nearly the same (ca. 2×10^{-4} cP).

Water was atomized by these gases in an internal mix pneumatic atomizing nozzle. As in earlier experiments, the water droplets were captured in kerosene filled optical cells, photomicrographed, sized, and

counted. The experiments were performed at constant volumetric gas and liquid flow rates, while varying only the kind of atomizing gas used. The results are tabulated in Table VII.

TABLE VII. Effect of Atomizing Gas on Drop Size

Nozzle: Spraying Systems Company Set Up #42

Gas Rate: 6.75 scfm

Water Rate: 20 liters/hr

Temperature of gas and liquid: approximately 25 °C.

<u>Gas</u>	<u>Number Median Size, microns</u>	<u>Mass Median Size, microns</u>
Air	28	84
	21	71
	26	74
Argon	27	80
	40	90
Helium	35	148
	36	139

The data tabulated above represent repeated experiments which were performed to estimate error. The statistical "t" test applied to this data shows that the difference in mass median drop size for helium and air is real and not due to chance (probability >99.5%). Whereas, the difference between air and argon drop diameters may be due to chance, since the probability that this difference is real is only about 85%.

If it is assumed that drop size is proportional to gas density or molecular weight, then these results indicate that atomizing with steam would yield a mass median diameter of about 120 μ compared to about 80 μ for air at the same volume flow rate. Thus, to maintain a constant capacity, the steam rate should be higher than the air rate.

As a logical next step, these three gases will be compared at a constant mass flow rate rather than at a constant volumetric rate.

RADIOACTIVE DEMONSTRATION

During Run SS-3 start-up, the melter furnace stopped heating while operating at 1150 °C. Electrical continuity checks showed that the furnace elements were sound, but that four of the six copper leads from the furnace junction box to individual phase terminals were electrically open. The leads, which were routed inside the furnace cooling jacket, were found to be badly burned by excessive heat from the furnace. The furnace had been energized intermittently for 503 hr, including 107 hr at 1150 °C or above.

All six of the copper leads were replaced and re-routed outside the furnace jacket in order to keep them cool. The furnace was then heated to 1210 °C over a 6 hr period. Phase current and power readings taken at intermediate temperature settings proved that the furnace was fully repaired.

WASTE SOLIDIFICATION CONDENSATE TREATMENT

A. J. Shuckrow and B. W. Mercer

Upward revisions in estimates of ruthenium levels in Glass Melter Condensate have prompted a re-examination of previously studied treatment methods as well as new lines of investigation. Basically, three types of treatment operations are under study: (1) sulfate precipitation with barium, calcium, and lead, (2) hydrous oxide scavenging, and (3) volatilization of ruthenium from permanganate treated Glass Melter Condensate.

Sulfate precipitates have been found to contain significant quantities (~10%) of ruthenium even after several washings. A rather large volume of precipitate is formed in each of the three reactions. Incorporation of calcium sulfate in emulsified asphalt has been found to be possible up to 60% solids. Sulfate precipitates have also been incorporated in cement and leach tests are now in progress. Radiation damage due to high predicted ruthenium levels may prohibit asphalt incorporation. Cement mortars, being relatively insensitive to radiation damage, can accommodate higher activity levels. However, leach rates from cement are expected to be much higher than from asphalt.

Simulated Glass Melter Condensate made 0.024M in KMnO_4 is susceptible to volatilization treatment of ruthenium. Boiling the solution causes the oxidation of ruthenium to RuO_4 and the volatilization of this compound from solution. Laboratory observations indicate that a ruthenium DF of 25 can be realized with a 10 min boiling time. A manganese oxide precipitate forms during the heating process. This precipitate contains very little Ru, Cs, or Sr but about 80% of the Zr and Ce are carried by the precipitate.

Hydrous manganese oxide formed by the oxidation-reduction reaction between Mn^{+2} and MnO_4^- at high pH (10 to 12) has been found to be effective in removing ruthenium from simulated Glass Melter Condensate. This treatment gives ruthenium DF's in the range 20 to 30 for the condensate and 10 to 20 for the evaporator bottoms from the ruthenium volatilization step. Good removals of Sr, Ce, and Zr are also obtained in this scavenging treatment. A separate step is required for cesium removal.

A possible treatment scheme for Glass Melter Condensate would be ruthenium volatilization from permanganate treated solution followed by phospho-tungstate precipitation for cesium removal and finally hydrous oxide scavenging and asphalt incorporation. Any one of these steps could be eliminated as the treatment needs of the condensate became better defined.

PRODUCT EVALUATION

HEAT RATES IN WSEP POTS - W. V. DeMier

The heat generation rate was measured in five radioactive pots and found to be within 90% of the estimates based on material balance data from each run. The results of the determinations were as follows (heat expressed as total kilowatts in pot, adjusted to account for elapsed time between pot fill date and date of in-cell calorimetry measurement):

Heat Content at Pot Fill Date, kW

<u>Run</u>	<u>Calorimetry</u>	<u>Material Balance</u>
SS-2	1.5	1.5
PC-2	1.1	1.2
PC-4	2.6	2.5
PC-5	4.3	4.3
PC-6	4.8	5.1

THERMAL CONDUCTIVITY

Samples from development run DSC-1 are being used to determine thermal conductivity of PW-1 spray solidified waste (nonradioactive) as a function of temperature. Initial determinations were made by radial heat flow techniques, using a large section of the filled pot. The conductivity as determined by this method was found to decrease from about 1.2 W/m-°C at 65 °C to 1 W/m-°C at 120 °C and remain nearly constant to a temperature of about 300 °C. (See Figure 3)

Values with more accuracy and values at higher temperatures are being obtained from a split bar comparative apparatus. Conductivities (also shown in Figure 3) determined so far with this apparatus are about 10% higher than those from the less accurate radial flow equipment.

THERMAL EXPANSION

The thermal expansion of a sample of nonradioactive solidified PW-1 waste from phosphate glass run CPG-7 was measured by G. B. Barton. The expansion coefficients closely match those for stainless steel; so no differential expansion problems are expected in pots filled with this waste. Results are summarized below. Included also are brief summaries of thermal expansion data previously reported (BNWL-306, BNWL-365) for other processes and feed types.

SIMULATED PRODUCTS - J. E. Mendel

Several leach tests of nonradioactive samples were completed during the report period. Leach rates were calculated from sodium

Process Type and Run No.	Heating of Solidified Waste		Cooling of Solidified Waste	
	Temp. Range, °C	Expansion Coefficient (α), in. / in. -°C	Temp. Range, °C	Expansion Coefficient (α), in. / in. -°C
Spray Solidifier PW-2 CSCM-7 "ceramic"	25 to 365	1.58×10^{-5} softened at 390 °C	paralleled expansion for heating cycle	
Spray Solidifier PW-1 CSCM-8 "ceramic"	25 to 200 200 to 700 700 to 820	3.13×10^{-6} 4.88×10^{-6} 3.12×10^{-5}	840 to 600 600 to 400 400 to 200	5.51×10^{-6} 4.49×10^{-6} 1.15×10^{-5}
Phosphate Glass PW-1 CPG-7 as received	25 to 380 380 to 415	1.41×10^{-5} 9.87×10^{-5}	380 to 25	1.52×10^{-5}
annealed	25 to 380 380 to 415	1.38×10^{-5} 6.02×10^{-5}	380 to 25	1.46×10^{-5}
Phosphate Glass PW-2 WSEP Run A	RT to 380 380 to 440	1.11×10^{-5} 4.14×10^{-5}	paralleled expansion for heating cycle	

For comparison: α for 304-L stainless steel = $1.98 \times 10^{-5} \frac{\text{in.}}{\text{in.} \cdot ^\circ\text{C}}$ from 20° to 890°

α for 310 stainless steel = 1.96×10^{-5} from 20° to 890°

α for carbon steel = $\sim 1.44 \times 10^{-5}$ from 0° to 650°

FIGURE 3. The Relationship of Expansion Coefficient to Temperature for Different Processes

analyses of the leach water, and lithium analyses where applicable.

Some conclusions drawn from the data are:

1. The leach procedure is flexible. Leachant volume and the time interval between leach water changes can be varied as desired until the concentration of the leached elements in the leach water begins to affect the rate. In the case of sodium and lithium, this does not occur until concentrations of at least 0.05 to 0.1 g/liter are reached.
2. Leaching periods of 8 to 12 weeks at 25 °C are sufficient to define an essentially constant leach rate.
3. Leach rates of grab samples and product from the pot were compared for spray solidifier:PW-1 product prepared in Run DSC-1A in EDL-102. Grab samples were taken during the early, middle, and later stages of the run. Their leach rates were compared with the leach rates of cubes of product cut from corresponding points in the pot. The grab samples were not given any supplementary heat treatment and may be considered to have been air-quenched. The leach rates of the three sectioned grab samples agreed closely with one another as did the leach rates of the three pot samples; however, the initial leach rates of the grab samples were a factor of about 4 higher than the pot samples. It is thought that this was because of incomplete bonding between the product and the container wall in the grab sample; more surface was actually exposed for leaching than that calculated from the samples external dimensions. The grab sample containers used in Run DSC-1A were pressed from thin sheet and the walls were wrinkled. In later runs smooth-walled grab sample containers will be used.
4. It was possible to distinguish visually a difference in the glassiness of different portions of the WSEP Run A product (BNL phosphate glass process:PW-2) when the pot was sectioned.

In general, the most glassy appearing product had been heated to the highest temperature, 1000 to 1100 °C, and cooled most rapidly. The leach rate of this product was compared to that of the less glassy product which had been heated to only 800 to 1000 °C. The leach rate for the former was about 1.5×10^{-6} g/cm²-day versus approximately 9×10^{-5} g/cm²-day for the latter.

WSEP PRODUCTS

A single grab sample was obtained for leach testing from each of the first two spray solidifier runs made with radioactive waste in the WSEP. Runs SS-1 and SS-2 were similar runs in which PW-1 was processed (see BNWL-507 for run details). The grab samples had a circular cross section, were about 1 1/2 in. long, and contained approximately 10 cm³ of product. Wafers, 1/4 in. thick, cut with a diamond blade, were used for the leach tests. The results are shown in Table VIII.

As was observed in the cold tests, lithium leached at a somewhat faster rate than sodium. Other workers have found cesium to be less leachable than sodium; however, this was not the case with the SS-1 and SS-2 products, from which cesium leached at about the same rate as sodium. Antimony had a leach rate of the same magnitude as the alkali metals. Ruthenium could not be detected in the leachant and, therefore, had a leach rate at least 10 times lower than the alkali metals. Cerium leached at a rate which was less than 1/100th that of the alkali metals.

The leach rates calculated for Run SS-2 were uniformly lower than for Run SS-1 although the runs were very similar. The difference is thought to lie in the type of grab sample container used. A pressed container which had uneven walls that apparently did not bond well with the product was used in Run SS-1; a smooth-walled container was used in Run SS-2. The rates calculated for Run SS-2 are thought to be most representative.

TABLE VIII. WSEP Product Leach Rates^(a)

	Run SS-1					
	Leach Rates, ^(b) g/cm ² -day					
	¹³⁷ Cs	¹³⁴ Cs	¹²⁵ Sb	¹⁴⁴ Ce	Na	Li
First Week	2.6 x 10 ⁻³	1.4 x 10 ⁻³	2.6 x 10 ⁻³	<5.0 x 10 ⁻⁶	3.0 x 10 ⁻⁴	1.1 x 10 ⁻³
Second Week	7.5 x 10 ⁻⁴	4.0 x 10 ⁻⁴	3.8 x 10 ⁻⁴	1.3 x 10 ⁻⁶	1.3 x 10 ⁻⁴	3.7 x 10 ⁻⁴
Third Week	5.0 x 10 ⁻⁴	2.2 x 10 ⁻⁴	5.7 x 10 ⁻⁴	4.7 x 10 ⁻⁷	3.0 x 10 ⁻⁴	2.5 x 10 ⁻⁴
Fourth Week	4.9 x 10 ⁻⁴	2.4 x 10 ⁻⁴	1.9 x 10 ⁻⁴	6.8 x 10 ⁻⁷	4.4 x 10 ⁻⁵	2.6 x 10 ⁻⁴
Fifth Week	3.9 x 10 ⁻⁴	2.1 x 10 ⁻⁴	1.9 x 10 ⁻⁴	<5.0 x 10 ⁻⁷	5.2 x 10 ⁻⁵	2.1 x 10 ⁻⁴
Sixth Week	3.8 x 10 ⁻⁴	2.0 x 10 ⁻⁴	1.2 x 10 ⁻⁴	<5.0 x 10 ⁻⁷	5.8 x 10 ⁻⁵	2.0 x 10 ⁻⁴
Seventh Week	2.8 x 10 ⁻⁴	1.4 x 10 ⁻⁴	9.5 x 10 ⁻⁵	<5.0 x 10 ⁻⁷	4.7 x 10 ⁻⁵	1.7 x 10 ⁻⁴
Eighth Week	2.9 x 10 ⁻⁴	1.5 x 10 ⁻⁴	2.5 x 10 ⁻⁴	<4.0 x 10 ⁻⁷	4.4 x 10 ⁻⁵	1.6 x 10 ⁻⁴
Ninth Week	2.9 x 10 ⁻⁴	1.5 x 10 ⁻⁴	9.5 x 10 ⁻⁵	<4.0 x 10 ⁻⁷	4.7 x 10 ⁻⁵	1.7 x 10 ⁻⁴
Tenth Week	3.2 x 10 ⁻⁴	1.6 x 10 ⁻⁴	9.5 x 10 ⁻⁵	<4.0 x 10 ⁻⁷	5.0 x 10 ⁻⁵	1.7 x 10 ⁻⁴
Eleventh	2.7 x 10 ⁻⁴	1.3 x 10 ⁻⁴	9.5 x 10 ⁻⁵	<3.0 x 10 ⁻⁷	3.3 x 10 ⁻⁵	2.0 x 10 ⁻⁴
Twelfth Week	2.8 x 10 ⁻⁴	1.4 x 10 ⁻⁴	1.9 x 10 ⁻⁴	<3.0 x 10 ⁻⁷	4.7 x 10 ⁻⁵	1.9 x 10 ⁻⁴

Run SS-2						
First Week	8.7 x 10 ⁻⁵	3.6 x 10 ⁻⁵	7.6 x 10 ⁻⁵	2.5 x 10 ⁻⁷	6.9 x 10 ⁻⁵	1.7 x 10 ⁻⁴
Second Week	4.6 x 10 ⁻⁵	1.8 x 10 ⁻⁵	4.2 x 10 ⁻⁵	1.0 x 10 ⁻⁷	3.6 x 10 ⁻⁵	7.4 x 10 ⁻⁵
Third Week	4.2 x 10 ⁻⁵	1.7 x 10 ⁻⁵	1.9 x 10 ⁻⁵	5.5 x 10 ⁻⁸	2.8 x 10 ⁻⁵	5.0 x 10 ⁻⁵
Fourth Week	2.7 x 10 ⁻⁵	1.0 x 10 ⁻⁵	1.3 x 10 ⁻⁵	8.4 x 10 ⁻⁸	1.9 x 10 ⁻⁵	2.5 x 10 ⁻⁵
Fifth Week	2.3 x 10 ⁻⁵	7.9 x 10 ⁻⁶	1.3 x 10 ⁻⁵	9.5 x 10 ⁻⁸	7.2 x 10 ⁻⁵	2.5 x 10 ⁻⁵
Sixth Week	1.5 x 10 ⁻⁵	6.1 x 10 ⁻⁶	1.7 x 10 ⁻⁵	4.8 x 10 ⁻⁸	1.9 x 10 ⁻⁵	2.5 x 10 ⁻⁵
Seventh Week	1.7 x 10 ⁻⁵	6.6 x 10 ⁻⁶	1.1 x 10 ⁻⁵	2.7 x 10 ⁻⁸	1.1 x 10 ⁻⁵	1.2 x 10 ⁻⁵
Eighth Week	1.7 x 10 ⁻⁵	7.0 x 10 ⁻⁶	1.1 x 10 ⁻⁵	1.8 x 10 ⁻⁸	1.7 x 10 ⁻⁵	1.7 x 10 ⁻⁵

(a) Procedure described in BNWL-365

$$\frac{(\text{total counts leached})/\text{day}}{(\text{total initial counts})}$$
(b) Leach rate =
$$\frac{(\text{sample area, cm}^2)}{(\text{sample weight, g})}$$

POT LEAK TESTING - V. P. Kelly and T. J. Owen

Leak testing of waste pot PC-4 was attempted using a mass spectrometer (helium) leak tester connected in parallel with a 450 liter/sec roughing pump. The tester and pump are connected to the pot for pump-down to the high vacuum (0.2μ) required for test. The test was terminated after 70 hr due to other in-cell activities, without having achieved the required test vacuum. However, the results indicate that this condition is due to severe pot outgassing rather than leakage. The outgas was apparently nonradioactive since no buildup in the activity of the roughing pump suction was detected during the test.

Although not conclusive, the results of the PC-4 test point to confirmation of the feasibility of vacuum leak testing of waste pots.

POT SAMPLING BY CORE DRILLING

A limited number of samples of solidified radioactive wastes are required to complete the evaluation of the WSEP "product." Sampling by rotary core drilling using diamond-set bits is being studied for this purpose. Currently, design verification testing and development is under way, using both hydraulic and mechanical-feed drillers with single and double core barrels fitted with flat or step-face bits. Coring of both spray solidifier and phosphate glass melts has been done.

The results of these tests indicate that the mechanical feed driller with a single-tube core barrel and flat face bit will be preferred for radial coring. The flat-face bit will core both metal and glass so that bit changing will not be required during drilling. The flat-face bit also appears to require less cooling air so that the dusting and core barrel seal problem is minimized. However, dust control remains a major problem, particularly when dismantling the driller to recover the core.

Another area of concern is the selection of feeds and speeds as a function of core material to produce integral cores rather than fragmented

cores. Core fragments of irregular shape and difficult-to-measure surface area are undesirable for the solubility tests to be carried with the core sample. Future tests will be aimed at defining driller operating parameters required to produce solid cores.

A second phase of the core sampling studies is the patching of radially-cored pots by welding. Nominal 2-in. diameter plugs have been successfully welded in pot wall core holes using a remotely-operated tungsten-inert gas torch. It is expected that cored pots will be "recanned" for final storage so that pot patch welds will not require the integrity of a primary containment vessel.

SOLIDS STORAGE ENGINEERING TESTING

A-CELL MODIFICATIONS (PROJECT BAP-016)

Modifications to the cell for SSET started with installation of the stainless steel wall liner and in-the-wall process off-gas piping. Both phases are 50 to 60% complete. Shielding window frames are on site so that completion of in-cell work during December, 1967 can be expected if shielding window shipping schedules are met.

ENVIRONMENTAL TEST PODS

Assembly of six environmental test pods for SSET is essentially complete. Figure 4 shows the first two pods mounted in a shipping cradle ready for transfer to the cell. Components for an additional six pods are complete; assembly will proceed as funds become available.

SALT STORAGE EXPERIMENT

A salt storage experiment, involving storage of waste pots in environmental test pods filled with salt, have been proposed for SSET. The experiment has been proposed to supplement the data and technology which have resulted from the ORNL-conducted Project Salt Vault Program aimed at demonstrating waste disposal in salt mines.

Design criteria for, and conceptual design of, salt storage pods have been developed in conjunction with ORNL. The conceptual salt

Neg PNL 0673128-7cn

FIGURE 4. Environmental Test Pods for Solids Storage Engineering Testing

pod design, shown in Figure 4, is based on the conventional salt pod design, Figure 5. An inner carbon-steel shell will be filled with 10-in. ID by 15-in. OD salt doughnuts, and fitted with the required thermocouples to measure temperature profiles. The waste pot is lowered into place and off-gas and thermocouple lead extensions connected. The void spaces are then backfilled with crushed salt and the entire inner shell and pot assembly lowered into outer pod vessel. The outer pod vessel is equipped with cooling coils, slurry circulating connections, and an outer insulating jacket. The assembly hangs vertically downward supported by a frame which is integral with the shielding plug. The plug is inserted into sleeves in the cell wall and locked into place to provide support for the vessels as well as a pipeway for piping and thermocouple leads.

Methods of collecting salt pod off-gas and of removing the collecting salt pod off-gas and of removing the caked salt at the end of the storage period are being studied to complete the preliminary scope of salt storage equipment requirements.

FIGURE 5. Salt Storage Environmental Test Pod

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