QUARTERLY PROGRESS REPORT
RESEARCH AND DEVELOPMENT ACTIVITIES
FIXATION OF RADIOACTIVE RESIDUES
MAY, JUNE, JULY, 1968

OCTOBER 1968

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MAY, JUNE, JULY, 1968

By

Members of the Staff
of Chemical Technology Department
Chemistry and Metallurgy Division

Edited by
A. M. Platt

October 1968

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PREVIOUS QUARTERLY REPORTS

<u>Quarter</u>	<u>Year</u>	<u>Report Number</u>	<u>Reference*</u>
	<u>1959</u>		
First		HW-60584	59-1
Second		HW-61635	59-2
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Fourth		HW-63949	59-4
	<u>1960</u>		
First		HW-65209	60-1
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	<u>1961</u>		
First		HW-69176	61-1
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	<u>1962</u>		
First		HW-73412	62-1
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	<u>1963</u>		
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	<u>1964</u>		
First		HW-81862	64-1
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Third		HW-84467	64-3
Fourth		HW-84603	64-4
	<u>1965</u>		
First		BNWL-76	65-1
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Third		BNWL-170	65-3
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	<u>1966</u>		
First		BNWL-276	66-1
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Third		BNWL-365	66-3
Fourth		BNWL-410	66-4

* Reference notation used in this report.

PREVIOUS QUARTERLY REPORTS (contd)

<u>Quarter</u>	<u>Year</u>	<u>Report Number</u>	<u>Reference*</u>
	<u>1967</u>		
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	<u>1968</u>		
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* Reference notation used in this report.

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QUARTERLY PROGRESS REPORT
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INTRODUCTION

This progress report is the thirty-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

During the report period, approximately 2,400,000 Ci of radioactivity were processed in the sixth phosphate glass solidification run and approximately 360,000 Ci were processed in the third spray solidification run. Phosphate glass Run PG-6 was made using sulfate-free Purex Waste 1 (PW-1) feed while SS-3 was made using sulfate-containing Purex Waste 2 (PW-2) feed.

The high rare earth concentration (35 wt% as oxide) of PW-4m waste tends to raise the melting point and increase nonhomogeneity of spray solidifier melts. Satisfactory phosphate melts for the spray solidifier have only been achieved by adding sodium and iron to the point where the final melt resembled PW-1 spray solidifier product. Some borosilicate systems have been investigated, of which several have been prepared which have "stick" temperatures above 700 °C and melting points in the range 925 to 975 °C.

Three successful vacuum batch transfers of phosphate glass melts were made in engineering scale equipment to test advanced solidification techniques.

The water-nitric acid separation characteristics of an analog computer model of Waste Solidification Engineering Prototype (WSEP) were verified by comparison with actual WSEP data. Data from a pot calciner run have also been used to adjust the parameters of the model to match data on solids entrainment and ruthenium volatility.

Steady-state cooling capacities of the two WSEP product pot furnaces were calculated. The highest planned heat generation rate of 25 kW in a stainless steel pot is within the calculated furnace limits.

PHOSPHATE GLASS SOLIDIFICATION

LABORATORY STUDIES

Corrosion from Fluoride-Bearing Feed Solutions - R. F. Maness

Corrosion of the denitrator-evaporator (all titanium construction) by fluoride-bearing feed solution was estimated by exposing titanium coupons to 100 gal/tonne PW-1 solution (Meq/P = 1.0) containing 0.1M HF. The solution contained 0.5M $\text{Al}(\text{NO}_3)_3$ with an equivalent reduction in the $\text{Fe}(\text{NO}_3)_3$ content. The test was made in all-Teflon[®] equipment with semi-continuous addition of feed to solution concentrated to a boiling point of 133 °C. The exposure time was 19 hr. Titanium corrosion rates in the liquid and in the vapor phases were 5.0 and 1.3 mils/month, respectively. A titanium coupon exposed to the boiling condensate collected from the above test corroded at a rate of 16 mils/month. Based upon this value, the fluoride content of the condensate was estimated to be about 5×10^{-3} M.

[®]

E. I. du Pont de Nemours and Co., Inc., Wilmington, Delaware.

A phosphate glass run was made with the concentrated feed solution produced above. The run was made at 1100 °C with semicontinuous feed addition to an alumina crucible equipped with an overflow weir. Platinum specimens were exposed to the melt and to the vapor phase. A Nionel specimen was exposed in the off-gas line immediately above the condenser. The platinum specimen exposed to the melt corroded at a rate of 0.23 mils/month (50 hr exposure) with no evidence of embrittlement. The specimen exposed to the vapor phase evidenced no weight loss. However, the specimen fractured on bending, indicating embrittlement. The Nionel specimen exposed in the off-gas line corroded uniformly at a rate of 6.1 mils/month. Only about 15% of the condensate was collected. The remainder flashed off during feed addition.

RADIOACTIVE DEMONSTRATION

Phosphate Glass Run PG-6 - J. L. McElroy, J. N. Hartley, M. R. Schwab

During Run PG-6, waste equivalent to 0.7 tonnes of 20,000 MWd/tonne power reactor fuel was processed to produce approximately 174 kg of glass product. The 62 liters of radioactive glass produced a self-generating heat density of approximately 131 W/liter (8100 W total).

A total of 616 liters of sulfate-free FW-1 type feed (889 liters/tonne) containing 2,560,000 Ci of radioactivity including approximately 8000 Ci of radoruthenium were fed to the denitrator in 50 hr at an average feed rate of 12.3 liters/hr. Table 1 lists pertinent data for Run PG-6 as well as for all WSEP runs to date.

The sulfate-free FW-1 feed used in PG-6 was made using radio-cerium solution from B-plant as a source of radioactivity. Without Purex 1 WW in the feed no foaming problems occurred in the denitrator-evaporator as had happened in previous runs. Antifoam B* was not added. This further supports the previous evidence that small (<100 ppm) quantities

* Dow Corning Antifoam B

TABLE 1. WSEP Interim Run Summary Sheet

OPERATIONAL DATA

	1/1, PC 11/66	1/2 SS 1/67	2/3 PC 2/67	3/4 PC 2/67	2/5 SS 4/67	4/6 PC 6/67	5/7 PC 7/67	6/8 ^(h) PC 9/67	1/9 PG 11/67	2/10 PG 12/67	3/11 PG 1/68	4/12 PG 3/68	5/13 ^(r) PG 4/68	6/14 PG 5/68	3/15 SS 7/68
Solidifier/WSEP #	A	A	B	A-Modified	B-Modified	C	C	A	Part 1 A	Part 2 B	A	A	Part 1 A	Part 2 B	A
Operating Mode	PW-2	PW-1	PW-2	PW-2	PW-1	PW-1 ^(e)	PW-1 ^(g)	PW-2	PW-2 ^{(j)(k)}	PW-2 ^(m)	PW-2 ^(o)	PW-2 ^(q)	PW-1 ^(r)	PW-1	PW-2
Feed Type	400	455	400	420	455	370	410	390	478	565	586	458	540	889	590
Feed ℓ /tonne	12	12	12	8	8	12	8	8	12	8	8	8	12	8	8
Pot Dia. In.	304-L	304-L	304-L	304-L	304-L	304-L	304-L	304-L	Mild Stl.	304-L	Mild Stl.	Mild Stl.	Mild Stl.	304-L	304-L
Pot Material	43.7	37	42	31	57	42	28	43 ⁽ⁱ⁾	61.4	31	48.5	63.1	77	50	35.6
Total Run Feed Time, Hrs.	----	----	----	----	----	----	----	----	9	5	10	13	12	6	7
Startup Time (Concentration Period), Hrs.	----	----	----	----	----	----	----	----	52.4	26	32	16	65	47	43
Steady State Operation, Hrs.	----	----	----	----	----	----	----	----	11.7	15.0	14	14.4	13	2	14
Shutdown Time (Dilution Period), Hrs.	27	12.8	28	15.5	6.6	33	11	8	----	----	----	----	----	----	----
Drying + Cooling Time, Hrs.	19.5	16.3	22.4	18	13.1	25	18.4	10.7	15.4	13.6	18	11.1	9.4	11.1	12.3
Avg. Feed Rate, ℓ /hr.	----	----	----	----	----	----	----	----	21.6	19	22	16.4	20	25	24
Avg. Feed Rate During Startup, ℓ /hr.	----	----	----	----	----	----	----	----	14.3	12.6	14	11.4	7.5	10	11.5
Avg. Feed Rate During Steady State Operation, ℓ /hr.	850	603	940	553	789	1060 ^(f)	515	460	944 ^(l)	422 ^(l)	873 ⁽ⁿ⁾	697 ^(p)	725	420	616
Total Feed, liters	2.2	1.3	2.3	1.4	1.7	3	1.25	1.2	1.97	0.75	1.5	1.52	1.3	0.63	0.69
Total Feed, tonne equiv.	----	----	----	----	----	----	----	----	691	394	520	317	669	271	584
Total Feed to Melter, liters	8	16	9	9	16	9.1	9.4	7.7	10.4	13	9.2	8.5	6.1	10.5	9.4
Feed: Solid Vol Ratio	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----

PROBLEMS

None	1. Scaling on Calciner Walls 2. High Filter Δ P 3. Stalagmite in Pot 4. High Air In-Leakage	1. Solids in Feed 2. Sulfate Volatilization During Pot Drying.	None	1. High Filter Δ P 2. Feed Flow Fluctuations	1. Foaming in Pot Calciner 2. Evaporator Diptube Plugging	1. Foaming in Pot Calciner 2. Evaporator Diptube Plugging	1. Feed Pump Failures Resulted in 3-Part Run	1. $\longleftrightarrow$ Foaming in Denitrator-Evaporator 2. $\longleftrightarrow$ WSEP Evaporator Diptube Plugging 3. Feed Pump Failure Resulted in Two Part Run	1. $\longleftrightarrow$ Foaming in Denitrator-Evaporator 2. $\longleftrightarrow$ WSEP Evaporator Diptube Plugging 3. Stalagmite Formation 4. Glass Clinker in Sample Chamber	1. Melter Feed Line Plug Resulted in Two Part Run. 2. Plugging Problems in Airlift Pot Recirculation Line 3. Den. - Evap. Diptube Plugging	1. Diptube and Airlift Line Plugging Occurred Frequently	1. Solids Feeder Plugged 2. Broken Calciner Filter
------	---	---	------	--	--	--	---	---	---	---	---	---

FRACTION OF ACTIVITY DISTRIBUTED: TOTAL LESS Ru(T-Ru) AND Ru

Thru Melter Cond (T-Ru)	----	----	----	----	----	----	----	----	117 ^(s)	270 ^(s)	500 ^(s)	2	3.3	1.9	----
10 ⁻³ (Ru)	----	----	----	----	----	----	----	----	183	410	410	12	104	43	----
Thru 1st Cond (T-Ru)	4	0.53	~4.0	2.9	0.18	NA	NA	12	3.2	5.8	NA	35	16	13	110
10 ⁻³ (Ru)	140	710	~300	310	740	54	33	110	39	36	NA	22	61	17	870
Thru Evap Cond (T-Ru)	1	50	90	< 1.5	6.3	7.0	0.32	0.1	1.1	< 0.12	0.86	0.31	2.8	1.5	4.0
10 ⁻⁶ (Ru)	74	830	1420	120	4000	1700	310	350	10,340	1000	390	850	2100	1000	2300
Thru Fract Cond (T-Ru)	< 1.6	630	40	3.2 ^(c)	2.4	9.6	25	< 0.1 ^(c)	1790	11	9.6	2	35	3.3	31
10 ⁻⁹ (Ru)	< 13	400	3950	120	3000	280	17,000	170	16,600	1300	14,000	2000	670	2900	8000
Off-gas to Scrub (T-Ru)	1.6	290	1400	100	< 1	3.1	NA	10	NA	40	NA	4	0.8	NA	22
10 ⁻⁹ (Ru)	880	330	10,600	1700	250	51	NA	70	NA	9000	9300	750	1300	700	24,900
Off-gas to Stack (T-Ru)	NA	< 10	1.1	< 1.7 ^(c)	3.6	< 1.3	< 0.7	0.6 ^(c)	4.1	5.8	0.54	2	1	1	11
10 ⁻¹² (Ru)	NA	< 1000	< 2400	< 2300	< 880	< 1000	< 1300	NA	NA	NA	NA	NA	NA	NA	NA
Ratio Activity in Fract Cond to 10CFR20 (Ru)	NA	140	350	70	1.5	1800	2200	20	17,250	770	1100	400	1800	550	940
	< 16	16	6400	350	45	1000	10,000	1600	14,850	10,000	66,000	7800	1800	3200	37,000

FILLED POT DATA

Total Curies In Feed to Pot	85,000	83,000	430,000	375,000	419,000	650,000	1,150,000	1,300,000	316,000	399,000	1,540,000	1,107,000	2,354,000	2,560,000	333,000
Curies of Ru In Feed to Pot	9200	5700	35,000	32,000	5300	47,000	18,000	36,000	13,000	19,600	10,200	30,912	42,200	8000	25,700
Solid in Pot, liters	105	43	105	48	110	60	55	60	105	41.3	62	113.3	62	113.3	40
Solid in Pot, kg	151	133	141	86.4	152	166	86	79.5	290	118	213	306	174	102	102
Solid in Pot, ℓ /he	50	32	45	47	32	41	43	50	48	46.4	50.7	61	108	94	65
Heat in Pot, watts	600 (420) ^{(a)(b)}	330	1700 (1500)	1770 (1800)	(2100)	(4300)	(4300)	5100 (4850)	1300	(1700)	3300	(4200)	8800	9800 (8100)	1600
Equiv. Aging Time, yrs.	50	40	6	3.7	3.8	4.7	1.5	1.0	5.8	2.3	1.6	0.5	0.2	0.2	3.8
ℓ Temp. in Air, $^{\circ}$ C	140	170	485 ^(d)	410 ^(d)	315 ^(d)	477	725	940	320 ^(d)	415 ^(d)	655 ^(d)	510 ^(d)	730 ^(d)	780 ^(d)	318 ^(d)
ℓ Temp. in Water, $^{\circ}$ C	NA [*]	NA	NA	NA	NA	370	NA	850	140	NA	430	NA	NA	NA	NA
Δ Temp. ℓ to Wall, $^{\circ}$ C	70	30	235	220	65	312	495	700	90	140	255	175	350	380	81
Total Corrosion, mm	None	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Welded Lid	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Pot Self Pressurization, psi	< 1	NA	< 1	< 1	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Pot Leakage, atm cc/sec	15 $\rightarrow$ 7 psi/90 Days	NA	NA	NA	NA	NA	NA	NA	1.1x10 ⁻⁸	2.4x10 ⁻⁸	4.6x10 ⁻⁸	NA	NA	NA	NA
Radiation Reading, R/hr at 6 in.	2.5x10 ⁴	2.5x10 ³	2.4x10 ⁴	3.9x10 ⁴	1.1x10 ⁴	3.8x10 ⁴	4.9x10 ⁴	5.7x10 ⁴	9.5x10 ³	4.8x10 ⁴	4.1x10 ⁴	3.7x10 ⁴	4.7x10 ⁴	NA	NA

*NA-Not Available

- (a) Values in Brackets are from Pot Calorimetry Data
(b) Calorimetry Value is After 3 Months Decay
(c) Corrected Because of Foreign Contamination to give DF Approximately Equal to that for Ru
(d) Values are with Pot Setting in Cool Furnace
(e) SO₄ and Na were 0.25 and 0.47M. Vice Desired Values of 0.10 and 0.14M. Respectively
(f) 60 Liters Expelled from Pot due to Foaming
(g) SO₄ and Na were 0.35 and 0.64M. Vice Desired Values of 0.10 and 0.14M. Respectively
(h) PC-6 was a 3 part run. All DF Data is for 1st Part Only
(i) Feeding Extended to Obtain a Maximum Fill
(j) Al Substituted for 37% of the Nominal 0.445M Fe
(k) Fe Substituted for 90% of the Ru
(l) 12% of Total Feed to Denitrator lost due to Foaming
(m) Al Substituted for 65% of the Nominal 0.445M Fe
(n) 27% of Total Feed to Denitrator lost due to Foaming
(o) Al Substituted for 40% of Nominal 0.445M Fe
100% of Nominal Radioactive Strontium Added
20% of Nominal Ruthenium Level Added
(p) 50% of Total Feed to Denitrator lost due to Foaming
(q) Al Substituted for 59% of Nominal 0.445M Fe
Rare Earths were 6 Times Nominal 0.12M R. E.
(r) U⁺⁶ was 0.05M. Versus Desired 0.01M, 0.24M H₃PO₄ Added to Compensate for Excess U⁺⁶
(s) Activity Includes Feed Losses from Denitrator from Overflow to Melter Condensate Receiver

of organics (probably dibutyl phosphate, DBP, from degraded tributyl phosphate, TBP) in the Purex 1WW probably was the cause of foaming in previous runs. The sodium and phosphate concentrations in the PW-1 feed were 2.6 and 6.23M, respectively (at 378 liters/tonne).

Several plugging problems occurred throughout the run, but did not cause any delays. Dip tube and airlift line plugging occurred frequently, and required constant observations and frequent correction to avoid any interruption in the process. The main plugs that occurred during the run were:

- 1) The denitrator-evaporator airlift air line plugged twice during startup and once during shutdown.
- 2) The airlift pot weight factor dip tube plugged shortly after startup and was not unplugged until after the run.
- 3) The denitrator weight factor and specific gravity dip tubes were kept clear by hourly steam blowdowns but would start to plug if more than an hour elapsed between blowdowns.
- 4) The airlift recirculation line required frequent backing out of the plug valve as well as acid flushes to remove solids from this area.

During Run PG-6, 1.7% of the radioruthenium and 1.3% of other radioactive species that were fed to the denitrator were volatilized or entrained from the denitrator. Likewise, approximately 4.3% of the radioruthenium and 0.2% of all other radioactive species that were fed to the melter were volatilized or entrained.

The fraction of feed activity in the final condensate stream from the acid fractionator was 2.9×10^{-6} for radioruthenium and 3.3×10^{-9} for all other radioactive species. This represents activity which is above CFR20 limits⁽¹⁾ by factors of 3200 and 550, respectively.

(1) AEC's Rules and Regulations, 10CFR20, Table II, Column II.

With the pot sitting in the cool furnace, the 9800 W of internal heat produced a centerline temperature of 780 °C and a centerline-to-wall temperature difference of 380 °C. These values gave an effective thermal conductivity for the product of 1.0 W/m °C [0.61 Btu/(hr)(ft)(°F)].

SPRAY SOLIDIFICATION

LABORATORY STUDIES

Flowsheets for Processing PW-4m in the Spray Solidifier - J. E. Mendel

The third high level radioactive waste composition to be included in the WSEP Program will be PW-4m. It has been defined as the waste resulting from the "clean" reprocessing of fuel with an exposure of 45,000 MWd/tonne @ 30 MW/tonne. The principal differences between PW-4m and the two other WSEP waste compositions are:

	<u>PW-4m</u>		<u>PW-1</u>		<u>PW-2</u>	
	<u>wt%</u>	<u>mole %</u>	<u>wt%</u>	<u>mole %</u>	<u>wt%</u>	<u>mole %</u>
FP oxides	85.7	76.7	37.2	36.2	26.4	16.4
Na ₂ O	7.6	18.3	4.8	8.0	15.1	24.4
Fe ₂ O ₃	3.1	2.8	54.8	53.8	18.6	11.7
SO ₃	---		---		36.6	45.7

Table 2 shows the composition of the simulated PW-4m being used in laboratory testing.

Indications are that PW-4m can be processed by the phosphate glass process without great difficulty. Melts with M⁺/P ratios of 0.9 and 1.0 were smooth and easy to pour at 775 °C (M⁺ included 1.5M sodium which was added to lower the viscosity and melting point).

The optimum combination of additive fluxing and modifying agents was sought for processing PW-4m in the spray solidifier process. The goal is to find compositions which form calcines that are not "sticky" at 700 °C, and thus will not build deposits on the walls of the spray calciner, yet which melt at approximately 900 °C. Somewhat higher

TABLE 2. Composition of PW-4m at 100 gal/tonne*

<u>Inert Chemicals</u>	<u>Molarity</u>
Fe	0.05
Cr	0.012
Ni	0.008
Al	0.001
Na	0.322 **
U	0.010
NO ₃	6.50
P	0.003
<u>Fission Products</u>	
Rb (K)	0.0141
Cs (K)	0.0783
Sr	0.0356
Ba	0.0410
RE	0.274
Zr	0.143
Mo	0.130
Tc (Mo)	0.031
Ru (Mn)	0.0816
Rh (Co)	0.0133
Pd (Ni)	0.0425
Ag (Cu)	0.0016
Cd (Cu)	0.0025
Te (SO ₄)	0.0144

* Substitutions, as shown in parentheses, are being made for some of the fission product elements.

** Nominal Na is 0.10M. Excess is due to addition of Mo as Na₂MoO₄.

melting points can be accommodated in the spray solidifier process, but experience indicates that a melting point of 900 °C or below permits optimum operation. Nearly 100 PW-4m melts have been prepared. Phosphorus,

boron, and silicon, singly and in combination, were used as fluxing agents. Lithium, sodium, iron, aluminum, zinc, and molybdenum in various combinations were tested to modify the melt characteristics.

The relatively high rare earth concentration (35 wt% as oxide) tends to raise the melting point and increase nonhomogeneity in PW-4m melts. In order to make phosphate-based PW-4m melts which could be processed in the spray solidifier it was necessary to add sodium and iron until the final melt resembled PW-1 spray solidifier product.

Investigation of some borosilicate melts was also started. Although further optimization is required, several PW-4m borosilicate melts have been prepared which have "stick" temperatures above 700 °C and melt in the range of 925 to 975 °C.

Corrosion from Fluoride-Bearing Feed Solutions - R. F. Maness

A fluoride-bearing PW-2 solution (0.03M fluoride) was concentrated to a boiling point of 128 °C and fed semicontinuously to a 304L pot held at 700 °C. The calcine produced was then fed semicontinuously to a 304L melter pot held at 850 °C. The off-gas, during both operations, was pulled through a 304L sintered SS filter held at 300 °C. The total exposure time was 33 hr. The filter evidenced a weight loss of 0.5%. No attack was apparent from microscopic examination. Platinum specimens exposed to the melt and to the vapor phase during the melting operation were unattacked. The fluoride content of the condensate collected during the calcination operation was estimated to be 5×10^{-3} M.

Platinum Melter Corrosion - R. F. Maness

The small electric furnace used to operate the spray calciner melter freeze valve has been subject to frequent failure. Burners operating on a propane-air mixture could be used to operate this valve; therefore, experiments were made to determine if platinum embrittlement occurred under these operating conditions. Aliquots of PW-2 melt were placed periodically on a section of 10 mil thick platinum sheet

which was heated to about 1000 °C with a torch burning a propane-air mixture. The specimen was allowed to cool to room temperature at 30 min intervals and was then flexed to determine ductility. After 36 cycles the specimen was cleaned and examined microscopically. One area of the specimen appeared to contain surface cracks. However, examination of this area after sectioning, polishing, and etching revealed no cracking or other preferential attack. The experiment was continued with the remaining portion of the specimen using a Meeker burner rather than a torch. The specimen fractured after two additional cycles. An experiment similar to that described above was made using, in addition to a propane-air burner, a reducing flame from a small torch burning propane with insufficient oxygen. The platinum specimen fractured after 10 cycles.

Ruthenium Volatility Studies - D. W. Brown

The effect of hypophosphorous acid, H_3PO_2 , on ruthenium volatility in the PW-1 spray solidification process was studied on a laboratory scale. Data indicate that 1M hypophosphorous acid lowers the ruthenium volatility to below the detectable limits of the analysis, in this case less than 3% ruthenium.

ENGINEERING STUDIES

Development Spray Calciner Run DSC-18 - J. D. Moore

A spray solidification run (DSC-18) was made with the cold semi-works spray solidifier to fill a special test pot with simulated PW-2 melt. The pot will be used for studies of gas evolution from storage pots with molten cores. The 12 in. diam unheated pot was filled uniformly with about 40 liters of melt by batch-dumping from the melter freeze valve and weir during a 29.5 hr run.

The spray calciner, operating at 700 °C, was fed with simulated 137 gal/tonne high sulfate (PW-2) waste at an average rate of 11 liters/hr (0.5 tonne/day). This rate reached the capacity of the melter operating at a furnace temperature of 925 °C. Air atomization of feed through an internal-mix nozzle produced a fine homogeneous powder feed to the melter. Operation of the system was satisfactory.

After the run, the calciner internal walls were coated with an apparently steady-state layer of powder between 1/8 and 3/16 in. thick and the calciner filters were covered with a 1/16 in. coating. The filter pressure drop increased gradually from 1-1/4 to 5 in. of water with a filter blow-back pressure of 35 psig. For the last quarter of the run, the blow-back pressure was increased to 40 psig, and the filter pressure drop gradually decreased from 5 to 4-1/4 in. of water at the end of the run, thereby providing reasonably good control of filter pressure drop. The particulate decontamination factor (DF) across the filters varied from a low of 150, 2 hr after the feed was on, to 2600 after 12 hr when a steady-state coating of powder had formed on the clean filters. The DF remained at 2600 to the end of the run.

A new solids feeder was successfully tested for addition of sodium metaphosphate flux to the melter. The feeder consists of two 1-1/2 in. ball valves spaced about 18 in. apart in a pipe connected to a diagonal nozzle in the calciner cone. The valve handles are coupled together to an air-driven piston which opens and closes the valves. When one valve is open, the other is closed thus maintaining a seal of the calciner at all times. Sodium metaphosphate chips are fed into the pipe from a vibrating auger feeder. Once every 5 min a timer actuates the piston which simultaneously opens the bottom valve and closes the top valve dumping the accumulated chips into the melter. Total flux addition with this equipment was within 2% of the desired amount. No solids plugging was encountered.

Melt was drained in 4 to 5 liter batches at a batch rate of about 10 liters/hr without the formation of stalagmites in the product pot. When batch-dumping the melt through the melter freeze valve, foamy melt discharging from the freeze valve bridged across to the freeze valve furnace liner, plugged off the outlet, and burned out the furnace. The foamy melt was caused by evolution of oxides of sulfur when the melt temperature was raised excessively to 910 °C to drain through the freeze valve. The tip of the freeze valve was too cold to completely melt the plug so the tip was probed with a rod to start the melt flow.

Subsequent batch dumping of melt through the weir also indicated that the weir tip was not adequately heated. Heating the weir tip with an oxyacetylene torch or manually rodding the weir tube initiated draining of the melter. Modifications to the freeze valve and weir tube assemblies will be made accordingly.

All of the ruthenium in the feed to the calciner was volatilized to the off-gas condensate.

RADIOACTIVE DEMONSTRATION

Spray Solidification Run SS-3 - J. L. McElroy, W. R. Bond,
M. R. Schwab, and J. N. Hartley

The fifteenth WSEP pilot plant demonstration of the solidification of high-level radioactive wastes was completed with the third spray solidification Run SS-3. During Run SS-3, 362 liters of high-sulfate waste (@ 590 liters/tonne) containing 333,000 Ci of radioactivity including 26,000 Ci of radoruthenium were fed to the spray solidifier in 35.6 hr at an average rate of 10.1 liters/hr. (See Table 1, for SS-3 data.) A total of 40 liters of ceramic solid was produced with an internal heat generation rate of 40 W/liter corresponding to 38% of thermal maximum for the product. The total of 1600 W of fission product heat and the centerline-to-wall temperature difference of 81 °C gave an effective thermal conductivity of 1.28 W/m °C [0.74 Btu/(hr)(ft)(°F)] for the ceramic product.

An internal-mix spray nozzle was used to spray the PW-2 type feed [additives of 1.61M H_3PO_4 , 1.17 LiOH, 0.6M $\text{Ca}(\text{NO}_3)_2$, 0.25 $\text{Al}(\text{NO}_3)_3$ at 378 liters/tonne] into the solidifier. Feed atomization with unheated air prevented nozzle plugging as in the previous run, SS-2. In addition to the feed additives, solid sodium hexametaphosphate was added to the melter batchwise to maintain an additive composition of 1.27M NaPO_3 at 378 liters/tonne feed concentration, to reduce the melting point to ~700 °C. A gravity flow, mechanically vibrated solids feeder was used to add the solid flux (sodium metaphosphate). A heated air purge line was added to the solids feeder during the run to minimize condensation of solidifier vapors and subsequent plugging in the solids feed line where it enters the cone of the calciner.

Run SS-3 was plagued by equipment failures, and after several attempts to maintain a continuous operation, the run was terminated due to equipment failures. Early in the run before the feed was started to the solidifier, the freeze valve and weir heaters failed, the feed tank agitator failed, and the feed pump began leaking, in that order. Without adequate feed agitation, the heavy solids in the feed would have settled and the run was shut down until the agitator and feed pump were repaired. The run was started without the weir and freeze valve heaters by using a propane torch on the freeze valve to batch drain the product from the melter to the pot receiver. After about 8 hr of feeding, the melter solids addition line plugged at the calciner and the run was shut down.

Because of a potential for embrittlement in the platinum freeze valve in the presence of a propane flame, the original 12 in. diam pot receiver was replaced with an 8 in. pot, thus shortening the run and requiring only half the original product planned.

During the second part of Run SS-3 (second pot receiver), the feed was shut off intermittently (after 1 to 8 hr of operation) to periodically flush out plugs of entrained calcine in the vent and drain lines from the solidifier condenser (E-111) with nitric acid. Significant quantities of solids in the off-gases (11% of the total calcine produced) indicated a failed solidifier off-gas filter. Failure of one of the 316L sintered SS filters has since been visually verified. Investigations to determine the cause of failure are presently underway. Another shut-down was necessary to repair the solidifier vibrator (used to maintain the heated walls of the calcine free from calcine deposition). The run was terminated after about 32 hr of total feed-on time (including about 8 hr with the first pot receiver) when the solidifier off-gas line plugged at the inlet to the solidifier condenser and could not be cleared while "on-line."

Approximately 87% of the ruthenium in the feed to the solidifier was volatilized. As yet, no quantitative data are available on sulfate

losses from the melt. However, on two occasions when the melt temperature exceeded 890 °C, sulfate fumes were observed evolving from the melt as it drained from the melter into the receiver pot.

DEVELOPMENT STUDIES ON SOLIDIFICATION PROCESSES

ADVANCED SOLIDIFICATION TECHNIQUES

Feasibility of Vacuum Batch Transfer of Melts from Simulated Purex-Type Waste - J. N. Hartley (Not charged to Division of Reactor Development)

This investigation is being made to provide information for assessing the feasibility of a promising new concept of a batch melting solidifier for treating high level radioactive waste. The batch solidifier consists of an evaporator chamber above a batch melter as an integral unit. The waste is concentrated and melted batch-wise, then transferred to the final storage container. Vacuum batch transfer of melt through a heated tube to a final receiving vessel was tested.

Three successful transfers were made using phosphate glass melts of simulated wastes. A fourth transfer was started using a sulfate containing ceramic melt from the spray solidifier but due to an equipment failure, was discontinued.

Figure 1 shows the major features of the vacuum transfer equipment. Glass or ceramic is melted by an induction furnace. The molten glass or ceramic is then transferred through a heated tube to a final mild steel receiving pot.

Run VT-1

The first transfer was made using a sulfate-free (PW-1) phosphate glass melt. A foamup of melt in the receiving pot after transferring 30 kg of melt caused the transfer to be stopped. The cause of the foamup was an excessive vacuum (29 in. mercury) in the receiving pot. The 13.3 liters of glass product was transferred to the mild steel receiving vessel in 0.65 min at an average rate of 20.4 liters/min.

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FIGURE 1. Vacuum Transfer Equipment Arrangement

BNWL-889

The transfer tube was heated to 850 °C before the transfer was started and the melt was held at 900 °C. After the transfer, the unheated receiving pot wall temperature had increased to 350 °C.

Run VT-2

The second vacuum transfer was successfully completed by transferring 44 kg of PW-1 phosphate glass melt. Thirty liters of glass were transferred in 2.4 min at an average rate of 12.5 liters/min. This run used 15 in. of mercury vacuum instead of the 29 in. used in the previous run. No transfer problems occurred with the transfer tube heated to 1000 °C, although this was 150 °C above operating conditions and resulted in a weakening of the 304L transfer tube. The melt was at 935 °C before the transfer started and after the transfer was complete, the receiving pot wall temperature had increased to 445 °C.

Run VT-3

The third transfer was successfully completed using a phosphate glass melt from a sulfate-containing (PW-2) feed. During the transfer, 30 kg of melt were transferred to a mild steel receiving pot. The 13 liters of glass were transferred in 1.2 min at an average rate of 10.8 liters/min. The transfer rate was about half the rate in previous transfers, due to a heater failure on the tip of the transfer tube where the average temperature was only 200 °C as compared to ~850 °C for the rest of the tube. This caused some buildup of glass in the pipe. No other problems occurred. The melt was at 925 °C when the transfer started and the vacuum was at 15 in. of mercury.

Run VT-4

The fourth transfer was started using a sulfate-containing ceramic melt from the spray solidifier, but was discontinued because of equipment failure. More experimental work is planned with this type melt.

HEAT TRANSFER STUDIES

Pot Furnace Cooling Capacities - J. D. Kaser

To assist in planning future WSEP runs at higher levels of radio-activity, the steady-state cooling capacities of the induction heated (5A) pot furnace, and the resistance heated (6A) pot furnace were calculated as functions of pot wall temperature. One of these two furnaces contains the 8 ft long product receiving pots during waste solidification in WSEP.

Heat balances between the pot wall and the various surfaces within the furnaces were made assuming a uniform temperature at each surface and neglecting the heat losses at the top and bottom of the furnaces. Accounting for the effect of two cooling air streams in the furnace (indirect air blasting at the outside of the susceptor wall and direct forced air within the susceptor) resulted in a system of independent nonlinear equations which were solved by an iterative technique and programmed for the computer.

The results of these calculations are plotted in Figures 2 and 3. Curves showing calculated wall temperatures for pots hanging in air are included for convenient comparison. Table 3 compares relevant WSEP run data with calculated temperatures read from Figure 2. The table shows fair agreement between calculated and observed values, considering the uncertainties in temperature and heat generation rate measurements, and in the values of the heat transfer coefficients used in the calculations. The centerline temperatures were estimated from the calculated pot wall temperatures by using a thermal conductivity value of 0.43 Btu/(ft)(hr)(°F) obtained from preliminary phosphate glass process run data.

Corrosion experience to date indicates that a stainless steel pot wall probably should not exceed approximately 850 °C during processing and that a carbon steel pot should not exceed 700 °C. In these cases, the calculations indicate that heat generation limits for a stainless steel pot is 33 kW in 5A furnace and about 28 kW in 6A furnace. The corresponding limits for a mild steel pot would be 25 and 20 kW. The highest

FIGURE 2. Steady-State Pot Temperatures During Cooling in the WSEP 5A Furnace

FIGURE 3. Steady-State Pot Temperatures During Cooling in the WSEP 6A Furnace

potential heat generation rate now planned for WSEP is 25 kW using a stainless steel pot in 5A furnace which is within these calculated furnace limits. Since the melting points of the fusible solid wastes range between 700 and 850 °C, the pot cores will remain molten during both furnace cooling and air storage at heat generation rates exceeding 5 to 19 kW.

TABLE 3. Comparison of Measured and Calculated Pot Temperatures

<u>WSEP Run No.</u>	<u>Heat Gen. Rate, kW</u>	<u>Wall Temperature, °C</u>		<u>Cooling Condition</u>
		<u>Observed</u>	<u>Calculated</u>	
PC-4	2.6	165	160	Hanging in air outside furnace
PC-5	4.3	230	240	↓
PC-6	5.1	240	250	
PG-5	8.8	345	400	In furnace. Outer cooling air only
PG-6	8.1	420	420	↓

COMPUTER SIMULATION - C. R. Cole, P. C. Owzarski, and J. L. McElroy

The initial simulation of the WSEP off-gas train and pot calciner has been completed. Figure 4 shows the various elements included in the simulation. The present simulation of the WSEP Plant is capable of any of the three modes of operation (A, B, or C). The present WSEP simulation can be preprogrammed to operate in any desired manner or it can be operated manually as the actual plant might be. The model is now ready to be used to predict the results of any method of operation of the pot calciner.

Heat and material balances are performed in each of the units simulated. Nitric acid separation where it occurs is computed from water-nitric acid equilibrium data and enthalpy data on liquid and vapor mixtures of water-nitric acid. Solids entrainment in the units in which it occurs is assumed to be proportional to a power function of vaporization rate out of that unit and to the solids concentration in that unit.

FIGURE 4. Units of the WSEP System Which Are Included in the Present Computer Simulation Model

Ruthenium volatility is computed as a power function of the nitric acid concentration in the liquid and is proportional to the ruthenium concentration of the liquid.

The water-nitric acid separation characteristics of the model were verified by comparisons with data from a nonradioactive WSEP run where acid feed of known concentration was fed to the evaporator and the separation characteristics of the evaporator and fractionator were recorded. Figure 5 shows the comparison between the model and the actual WSEP data for this run.

Data from WSEP Run PC-2 were used to adjust the parameters of the model in order to match the observed data on solids entrainment and ruthenium volatility. Figure 6 shows a comparison between the actual WSEP data on solids entrainment and ruthenium volatility for Run PC-2 and the simulation's match of this data.

WASTE FROM CHEMICAL PROCESSING OF FAST REACTOR FUELS - E. A. Coppinger

Waste from Fast Reactor Fuels

The radioactive waste resulting from chemical processing of fast reactor fuels will be different than wastes from processing of thermal reactor fuels. The higher fuel exposure and specific power in fast reactors will result in much higher fission product contents and heat generation rates in the waste. The fission product spectrum and amounts of neptunium and transplutonium isotopes will also be different in the waste due to the different irradiation conditions, higher Pu/U ratio in the fuel, and higher neutron energy in a fast reactor. The reprocessing scheme for fast reactor fuels may also result in differences in the chemical composition of the waste. A very short study was undertaken to provide a limited assessment of the possible aqueous waste resulting from processing of fast reactor fuels.

FIGURE 5. Comparison Between the Computer Acid Run Simulation and Actual WSEP Data

FIGURE 6. Comparison Between Actual WSEP Data for Run PC-2 for Solids and Ruthenium Radioactivity Levels and the Levels Computed by the Model

RADIOACTIVE SOLIDS

FIGURE 6. (contd)

Approximate heat generation rates, fission product concentrations, and changes in fission product spectrum in fast reactor fuel have been determined and are compiled in Tables 4 through 7. The amounts of neptunium and transplutonium isotopes in a conceptual fast reactor design have been obtained from available calculations and are also listed.

TABLE 4. Approximate Fission Product Content in Irradiated Fuel^(a)

Element	Fission Product Content, g/MT U + Pu	
	Fast Reactor Core Fuel ^(b)	PWR Fuel ^(c)
Se	166	46
Rb	609	318
Sr	1310	837
Y	677	432
Zr	7584	3418
Nb	60	8.8
Mo	8800	3228
Tc	2384	789
Ru	8535	2068
Rh	2490	400
Pd	6660	970
Ag	756	44
Cd	440	52
In	42	1.6
Sn	436	48
Sb	160	15
Te	1666	446
Cs	10830	2567
Ba	3540	1350
La	3058	1160
Ce	7110	2408
Pr	3125	1072
Nd	10168	3638
Pm	1015	124
Sm	2470	686
Eu	407	157
Gd	323	64
Tb	30	1.4
Dy	20	0.46

(a) Does not include gases or elements present in quantities less than 10 g/MT.

(b) 100,000 MWd/MT at 200 MW/MT, 180 day cooling.

(c) 30,000 MWd/MT at 32.5 MW/MT, 180 day cooling.

TABLE 5. Approximate Amounts of Various Fission Products at 90 Days Cooling

Nuclide	Fast Reactor Core Fuel ^(a)		PWR Fuel ^(b)	
	kCi/MT	W/MT	kCi/MT	W/MT
⁸⁹ Sr	816	2806	195.8	673
⁹⁰ Sr ^(c)	111.7	754	72	486
⁹¹ Y	1410	5180	312.3	1147
⁹⁵ Zr	3160	15790	476	2378
⁹⁵ Nb	5220	25100	783	3765
¹⁰³ Ru	1920	6816	225.7	800
¹⁰⁶ Ru ^(c)	3800	36110	345.4	3282
^{115m} Cd	31.4	115	0.1	0.4
^{123m} Sn	99	330	0.4	1.3
¹²⁶ Sn	0.004	--	--	--
¹²⁵ Sb	73.2	238	7.2	23.4
^{125m} Te	15	13	1.5	1.3
^{127m} Te ^(c)	57	108	2.7	5.1
^{129m} Te ^(c)	72	333	13.8	63.8
¹³⁴ Cs ^(d)	220	2230	144.4	1464
¹³⁷ Cs ^(c)	337	1630	98.5	476
¹⁴¹ Ce	1230	1820	188.6	279
¹⁴⁴ Ce ^(c)	3790	30926	787	6422
¹⁴⁷ Pm	1000	360	122.8	44
¹⁵¹ Sm	14.6	1.7	0.35	0.04
¹⁵⁴ Eu ^(d)	3	26.8	4.3	38.5
¹⁵⁵ Eu	144	130	4.5	4.1

(a) 100,000 MWd/MT at 200 MW/MT.

(b) 30,000 MWd/MT at 32.5 MW/MT.

(c) Watts include short-lived daughter.

(d) Not fission products. Produced from neutron capture by ¹³³Cs and ¹⁵³Eu.

TABLE 6. Approximate Amounts of Various Fission Products at 180 Days Cooling

Nuclide	Fast Reactor Core Fuel ^(a)		PWR Fuel ^(b)	
	kCi/MT	W/MT	kCi/MT	W/MT
⁸⁹ Sr	237	815	56.8	195
⁹⁰ Sr ^(c)	111	749	71.6	483
⁹¹ Y	490	1800	108.4	398
⁹⁵ Zr	1210	6046	182.3	911
⁹⁵ Nb	2346	11280	353	1697
¹⁰³ Ru	404	1434	47.5	168
¹⁰⁶ Ru ^(c)	3200	30400	291.2	2767
^{115m} Cd	7.3	27	0.02	0.07
^{123m} Sn	61	203	0.26	0.86
¹²⁶ Sn	0.004	--	--	--
¹²⁵ Sb	68.7	223	6.8	22
^{125m} Te	14.9	13	1.5	1.3
^{127m} Te ^(c)	31.6	60	1.5	2.9
^{129m} Te ^(c)	11.4	53	2.1	9.5
¹³⁴ Cs ^(d)	200	2027	133.2	1350
¹³⁷ Cs ^(c)	335	1620	97.9	473
¹⁴¹ Ce	180	266	27.6	41
¹⁴⁴ Ce ^(c)	3050	24888	632.4	5160
¹⁴⁷ Pm	950	342	115	41
¹⁵¹ Sm	14.6	1.7	0.35	0.04
¹⁵⁴ Eu ^(d)	3	26.8	4.3	38.5
¹⁵⁵ Eu	131	118	4.1	3.7

(a) 100,000 MWd/MT at 200 MW/MT.

(b) 30,000 MWd/MT at 32.5 MW/MT.

(c) Watts include short-lived daughter.

(d) Not fission products. Produced from neutron capture by ¹³³Cs
¹⁵³Eu.

TABLE 7. Neptunium and Transplutonium Isotope
Content in Fast Reactor Core Fuel^(a)

Nuclide	Amount, g/MT U + Pu		
	At Discharge	90 Day Cool	180 Day Cool
²³⁷ Np	332.6	332.6	332.6
²⁴¹ Am	1165.1	1297.7 ^(b)	1428.4 ^(b)
²⁴² Am	56.5	56.5	56.5
²⁴³ Am	337.2	337.2	337.2
Total Am	1558.8	1691.4	1882.1
²⁴² Cm ^(c)	193.9	132.3	90.1
²⁴³ Cm	0.4	0.4	0.4
²⁴⁴ Cm	5.3	5.2	5.2
Total Cm	199.6	137.9	95.7

(a) Based on equilibrium core exposure of 110,000 MWd/MT at a specific power of 56.5 MW/MT.

(b) The increase in ²⁴¹Am after discharge results from beta decay of ²⁴¹Pu. When plutonium is removed by processing before cooling for 90 or 180 days, the increase in ²⁴¹Am content in process solutions stops.

(c) Any ²⁴²Cm left in the waste stream after processing decays to ²³⁸Pu.

In conceptual fast reactor designs, the reactor is usually divided into three major zones, i. e., the core, axial blanket, and radial blanket. The axial blanket fuel and core fuel are in the same fuel pin in several design concepts. In such cases, the irradiated fuel from the core and axial blanket will be processed together and the waste will contain a blend of radioisotopes and stable fission products from these two fuels. Since the burnup of the axial blanket is typically less than 10,000 MWd/MT, the waste from the blended fuel will be fairly similar to the waste from thermal reactor fuel in terms of total fission product content and heat generation rates.

The relative amounts of ^{106}Ru and the potential by-products, palladium and rhodium, will be significantly increased in wastes from processing of fast reactor fuel. The relative amount of ^{90}Sr will be decreased. The relative amounts of cadmium, tin, antimony, and tellurium will be increased although the total amount of each will still be small. Some of these latter fission products, notably antimony and tellurium, tend to be volatile like ruthenium at high temperatures and could complicate waste solidification processes.

The chemical composition of the waste is difficult to specify quantitatively because of the uncertainties that exist in the fuel composition and fuel processing scheme. On the basis of a Purex-type process, the aqueous waste could range from relatively "clean" solutions of fission products to solutions containing moderate to high concentrations of sodium, iron, and sulfate. The waste generated during processing of carbide fuels is a possible exception. If carbide fuels are dissolved directly in acid, the waste could contain soluble organic compounds that could complicate waste solidification processes.

INTERMEDIATE AND LOW-LEVEL WASTE TREATMENT

LABORATORY STUDIES

Low-Level Waste Treatment

Distillation Demonstration Unit (DDU) - S. J. Dougherty

Assembly of the DDU has been essentially completed, and tests of the apparatus have been conducted to determine any necessary equipment modifications or additions. These tests have been conducted with boiling water at 20 and 50 mm mercury pressure and with a boiling acid mixture at 50 mm mercury. The approximate analysis of the acid mixture was:

The results of these runs have clearly indicated the capabilities of the equipment. The results show that:

- 1) The differential pressure (D/P) cells are an entirely unsatisfactory method for liquid-level measurement at the operating pressures of 10 to 100 mm mercury.
- 2) Better contact is required between the heaters and evaporator tank.
- 3) When D/P cell and heater modifications are made, the equipment will perform very well.

Although D/P cells are generally considered to be a satisfactory system for liquid-level measurement at atmospheric pressure, they are unsuitable for use in partially evacuated systems, primarily because of the admission of excessive air to the evacuated system. Because of the unsuitability of the D/P cells in measuring liquid levels, alternate methods of liquid level measurement will be used.

The poor heat transfer between the heaters and evaporator tank will be solved by placing a heat-conducting substance between the heaters and tank.

EVALUATION OF SOLIDIFIED WASTE PRODUCTS

NONRADIOACTIVE LABORATORY STUDIES

Study of Thermal History of Melts - D. W. Brown

Sixteen phosphate glass melts were prepared from a solution of PW-2 waste such that each melt had a different thermal history. Characterization tests have been run on these melts. Table 8 summarizes the experiment and the results. The greatest factor affecting the properties of the melt was the storage temperature. Samples stored at 600 °C were much more leachable than those stored at 100 °C. Porosity correlates very well to solubility within a storage temperature bracket.

TABLE 8. Summary of Thermal History on Melts and Data

Beaker No.	M. T., °C	T. A. M. T., hr	C. R., deg/hr	S. T., °C	Surface Appearance	Drip Pt., °C	Leachability % loss, 72 hr	Density, g/cm ³	Porosity, % of sample
2	1200	1	100	600	dull, very few holes	855	41.4	2.843	1.3
3	1200	25	100	600	dull, very few holes	865	35.3	2.874	2.06
10	1200	1	30	600	glossy, no holes	872	1.9	2.882	0
15	1200	25	30	600	glossy cracked	895	7.4	2.918	0
7	1000	1	30	600	very dull, many holes	800	41.4	2.988	2.25
8	1000	1	100	600	very dull, many holes	800	33.8	2.981	3.24
9	1000	25	30	600	very dull, no holes	795	39.3	2.977	2.4
16	1000	25	100	600	dull, pin holes	845	35.9	2.926	2.31
12	1200	1	30	100	glossy smooth	875	1.2	2.906	0
13	1200	25	30	100	glossy smooth	875	1.6	2.897	0
14	1200	1	100	100	glossy smooth	870	1.4	2.916	0
17	1200	25	100	100	glossy cracked	870	2.2	2.909	0
4	1000	1	30	100	dull, no holes	845	5.5	2.944	2.93
5	1000	1	100	100	glossy cracked	855	4.5	2.890	~1.0
6	1000	25	30	100	glossy smooth	825	5.1	2.814	2.81
11	1000	25	100	100	glossy smooth	915	1.5	2.897	0

M. T. melt temperature
 T. A. M. T. time at melt temperature
 C. R. cooling rate
 S. T. storage temperature

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

A vessel 12 in. in diameter by 26 in. high has been built and filled 22-1/2 in. deep with spray solidification sulfate-containing (PW-2) melt. This will be heated at the center of the melt. The pressure buildup due to decomposing sulfate and the temperature across the melt will be measured.

Product Characterization Laboratory Studies - W. Mularz

Laboratory prepared phosphate glass samples prepared from sulfate-containing (PW-2) feed are being evaluated to characterize the physical properties of the glasses.

Thermal diffusivity will be determined by a rapid thermal pulse method. Linear coefficients of thermal expansion are being measured. Preliminary data indicates a high linear coefficient of thermal expansion. Some samples are oozing a clear liquid (presumed to be a phosphate) at elevated temperatures (~400 to 500 °C).

A prototype drop weight apparatus has been developed for dispersibility measurements. It consists of a 2 in. diam plexiglass tube, a weight with a 1/8 in. diam blunt end steel tip, and a steel holder for the sample. The tube allows the weight to fall in a straight line. Sufficient clearance is allowed to prevent drag of the falling weight. When the glass samples break under impact, the particles are collected and classified by weight and size as: greater than sieve size, and subsieve size. Analysis of the subsieve sizes will be attempted with a Coulter Counter.

The thermal shock apparatus has not been developed as yet. It will consist of a tube furnace capable of 600 °C and a water bath into which the sample can fall. Thermal conductivity will be determined on samples that are 1 in. long with a 1 in. diam.

Some of the samples are porous and cracked. Hence, in the evaluation of most parameters the values obtained must be interpreted carefully.

PRODUCT MEASUREMENTS, TESTING, AND STORAGE

Solids Storage Engineering Testing - V. P. Kelly

A-Cell modifications for SSET that are being completed include:

- 1) Design and fabrication of a push-pull through-the-wall transfer mechanism.
- 2) Completion of gallery work, which includes correcting problems with manipulator monorail installation.
- 3) Installation of special mounting brackets in cell for waste pot and test pod storage.
- 4) Fabrication of an ion chamber shielding cask for in-cell radiation monitoring.

Completion of these project items is expected by fall.

SSET Equipment and Systems Fabrication and Installation

Fabrication and installation of SSET equipment is proceeding in the effort to activate SSET late this year. Status of the major items of equipment is:

- 1) Construction of 12 standard test pods is complete
- 2) Construction of four salt storage pods is complete except for assembly. With the exception of assembly of these pods, the full complement of pods for SSETF is now complete.
- 3) Design and fabrication of in-cell transfer hardware and work trays is started.
- 4) Design and installation of items is well along, including
 - a) emergency in-cell cooling system; b) emergency test pod cooling system; and c) auxiliary viewing for both the airlock and A-Cell.
- 5) Fabrication of the core driller and a second generation welding turntable is started.

Remote Handling Tests for SSETF

Remote handling design verification testing is in process to verify operability of in-cell components.

Product Characterization - R. J. Thompson

The following product evaluation data on radioactive WSEP pots were collected during the period:

- 1) PG-4 Pot Calorimetry - 4.2 kW + 10% (referenced to the shutdown date of Run PG-4).
- 2) Pot pressures for filled pots in Rack 4A

<u>Run No. for Pot</u>	<u>Pressure</u>
PC-1	No reading
SS-1	1 psig
PC-2	-10 in. Hg
PC-3	-28 in. HG
SS-2	1 psig
PC-4	12 psig
PC-5	-2 psig
PC-6	-11 in. HG
PG-1	No reading
PG-2	-30 in. Hg
PG-3	-24 in. Hg
PG-4	No pressure (leaky jumper)

The pots from PG-5, PG-6, and SS-3 were welded.

The pot wall thickness scanner is still giving erratic results. Repairs are underway.

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