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REPORT

**QUARTERLY PROGRESS REPORT
RESEARCH AND DEVELOPMENT ACTIVITIES
FIXATION OF RADIOACTIVE RESIDUES
APRIL - JUNE, 1965**

Edited By

A. M. PLATT

SEPTEMBER, 1965

RELEASED FOR ANNOUNCEMENT
IN NUCLEAR SCIENCE ABSTRACTS

RICHLAND, WASHINGTON

PACIFIC NORTHWEST LABORATORY operated by BATTELLE MEMORIAL INSTITUTE

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APRIL-JUNE, 1965

By

Members of the Staff
Chemistry Department

Edited by

A. M. Platt

Chemical Development

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QUARTERLY PROGRESS REPORT
RESEARCH AND DEVELOPMENT ACTIVITIES
FIXATION OF RADIOACTIVE RESIDUES
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INTRODUCTION

This progress report is the twenty-sixth in a series presenting research and development activities in the field of radioactive wastes.

Experimental work charged to programs other than those of the Division of Reactor Development is included for general interest and completeness; such work is identified in the headings.

SUMMARY

The samples of phosphate glass produced from Purex waste in the Hot Cell Glass Experiment have not shown appreciable change in leachability over the seven-month period since they were made. However, they are showing evidence of separation of a minute second phase.

Laboratory studies of "glass" formulations have indicated that the properties of the glass are affected by the precious metal and fission product contents of the glasses.

Flowsheet development with simulated NFS 1st and 2nd cycle waste and added phosphoric acid was completed with a successful 40-hr spray calciner run. Metallic cation equivalent to phosphorous atom ratio (M^+/P) equal to 3.1 was the most successful composition.

A feed composed of a sulfate-bearing condensate adjusted to melt with caustic was not operable in the spray calciner-melter. Other tests included successful thermal shock cleaning of the calciner wall, an acceptable air vibrator to replace an air hammer, and wire filters.

Fabrication of the spray calciner system for WSEP is complete and equipment testing has started. Design of the phosphate glass system is

complete and fabrication is nearly done. Design of WSEP-related services in the FRPP is essentially done, and installation is well underway.

Design verification testing of the pot calciner prototype and its related WSEP hardware has been successfully completed. Two additional runs, a pot calciner run (CPC-8) with NFS 1st and 2nd cycle waste, and a rising level glass run (CPG-1) with Purex FTW-65 type waste were made this quarter.

Preliminary fission product release tests were made with 15 g samples of simulated waste material spiked with Cs^{137} . Release fractions of up to one percent were observed during one hour at temperatures up to 1200 °C. The reaction between the molten waste material and the stainless steel containers was briefly studied.

Temperatures at Purex waste tanks in infinite arrays with 120-foot center-to-center tank spacings are compared with those for single tanks with no neighbors.

Studies are in progress to determine methods of forming insoluble sulfate melts from acid condensates generated in a glass making process.

A recycle flowsheet to improve cesium product purity was demonstrated in the laboratory. Nominal Na/Cs ratios of 8/1 were reduced to 1/3.4 by use of recycle and increased scrub volumes.

CALCINATION AND SOLIDIFICATION STUDIES

(R. L. Moore, C. R. Cooley)

HOT CELL GLASS EXPERIMENT - U. L. Upson

The samples of phosphate glass (H-2) produced from Purex 1WW high-level wastes have not yet shown appreciable change in leachability from that originally observed (viz $\sim 2 \times 10^{-5}$ g/cm²/day, see reports 64-4 and 65-1), but on metallographic examination (at age 7 months) are showing evidence of separation into two distinct phases. The new phase, although apparently solid on exposure to air, appears to have exuded from pores in the glassy matrix (Figure 1). The composition of this exudation is not known, and the facilities in the metallurgical cell are not adequate for a mechanical micro-separation for analysis. However, since the exudations are more numerous on surfaces corresponding to the top of the melt than on those of the bottom, it seems likely that they are evidence of incomplete dehydration of the phosphoric acid, or of subsequent hydration of free P₂O₅ in the melt.

If this exudation is due to un-dehydrated phosphoric acid, then it will be very difficult to produce a fully dehydrated glass in a continuous melter, since this sample was given a 30-min "soak" at 1200 °C after cessation of the feed to the melter, followed by slow cooling of the furnace; and average residence time in the melter will have to be well in excess of 30 min to produce a stable, fully dehydrated glass.

If the exudation is due to phosphoric oxide in excess of that necessary to form a metaphosphate glass, or above that firmly fixed in such a glass, several possibilities present themselves. The first and most obvious is that the feed solution contained phosphorus in excess of the metaphosphate ratio ($M^+/P = 1$), and that this excess was not fully expelled from the molten glass. If this is the case, then either very accurate feed make-up will be required, or slightly meta-deficient feeds will be required, with correspondingly higher melter temperatures being necessary for glass formation.

On the other hand, even if no appreciable excess phosphorus was initially present, it is possible that either initially, or during cooling, or later, some pyro- ($M^+/P = 2$) or ortho-phosphates ($M^+/P = 3$) were formed, either within the glass matrix, or as micro-crystalline inclusions — thus liberating free P_2O_5 .

A combination of trace residual water and $M^+/P > 1$ also is quite possible, and water competition for the P_2O_5 might enhance formation of the higher-valence phosphates. Such transitions also may be caused by radiation damage to the meta-phosphate bonds. If so, degradation of the glassy structure may be expected to continue, and to become evident in the observed leaching rates.

250X

FIGURE 1

Surface of Purex 1WW Phosphate Glass at Age 7 Months

In a separate study, a portion of a platinum melter liner used in one of the HCGE runs has been subjected to successive dissolutions in concentrated HCl and in aqua regia. Gamma analysis of these solutions has indicated that, the fission-product contaminants had diffused quite evenly into the platinum (at least for the first 0.002 in. removed to date), and that probably complete dissolution and electrochemical separation would be required for satisfactory platinum recovery. Most surprisingly, ruthenium is not a major contaminant. Ce^{144} and Cs^{137} appear to constitute the bulk of the contamination--making the prospect of electrochemical recovery more promising.

LABORATORY STUDIES

Effect of Varying Composition on Properties - G. B. Barton

As reported last quarter (65-1) a series of samples from a sulfate-free system was prepared. The drip temperature measurements have been extended to include the materials heated to 1200 °C and then slow cooled, or heated to 1200 °C and chill cast. These data are summarized on Figure 2.

Comparing the data that have been obtained that cover samples of the same thermal history (i.e., chill cast after melting at 1200 °C there) is not a significant difference between those with and without sulfate. There are some detailed differences but the major patterns are still similar. However, the samples that are expected to show the major difference (melted at 1000 °C, then chill cast) are still in process of measurement, so the definitive answer is not in hand yet as to whether or not elimination of sulfate will produce melts with superior properties.

It would be desirable to know somewhat more concerning the effects of thermal history. For example, what is the relation of cooling rate to properties? This will be examined in more detail when equipment for programmed heating and cooling is obtained.

Effect of Fission Product Mixture - G. B. Barton

There are two questions that were examined:

- (1) Is it necessary to include the precious metal ions in the fission product simulation mix?

FIGURE 2

Drip Temperature [10% Fission Product Oxides, 90% Process Oxides (Sulfate free) and Phosphoric Acid for the Indicated Ratio]

(2) Is it necessary to examine more than one fission product spectrum?

The experimental program set up to give answers to these questions was based on three fission product mixtures representing (a) low exposure natural uranium in a thermal flux, (b) 22,000 MWD/MT uranium in a fast reactor, and (c) Pu recycled to equilibrium at 10,000 MWD/MT. The variation in fission products is indicated by the ratio of some of the groups of elements to zirconium, the most abundant single element (Table I).

This work was well under way when the decision was made to use a Yankee fuel at 20,000 MWD/MT as the basis for WSEP runs. This fission product composition [calculated by H. H. Van Tuyl with his ISOGEN code (HW-83785)] is Rb + Cs 0.690, Sr + Ba 0.527, Y + Rare Earths 1.912, Ru + Rh + Pd 0.928.

TABLE I
RATIO OF INDICATED GROUPS OF ATOMS TO THOSE OF ZIRCONIUM

Element Group	Fuel Type		
	a	b	c
Rb + Cs	0.562	0.890	0.674
Sr + Ba	0.507	0.675	0.522
Y + Rare Earths	1.706	2.748	2.140
Ru + Rh + Pd	0.590	0.649	1.355

Samples were prepared with these fission products mixed with the Purex process components at 5, 10, and 20% levels both with and without the precious metals and at M^+/P ratios of 2.3 and 1.0. After preparation of the powder the materials were subjected to the following thermal history: M^+/P 2.3 powder was loaded into crucibles (groups of four consisting of two of each of the pair of compositions with and without precious metals), heated 1 hr at 200 °C, 1 hr at 325 °C, 1 hr at 700 °C, and 2 hr at 1000 °C. The furnace was then shut off and allowed to cool. One pair of samples was removed from the crucibles, crushed, sieved, and measured. The other samples were reheated to 1200 °C for one hour then poured onto a steel plate. This produced a glass, which was then crushed, sieved, and measured. The M^+/P 1.0 material is still in process of measurement, and will be described with the results next time.

The results on the M^+/P 2.3 mixtures are given in Table II. It can be seen that the drip temperature is markedly affected both by composition and thermal history, but the compositional effect in the slow-cooled material is largely that of total fission products rather than precious metals. However, for the chill cast samples there is a marked decrease in drip temperature upon inclusion of the precious metals. The solubility-thermal history - precious metal relation is reversed from the drip temperature property. The effect of fission product concentration on solubility was markedly increased by the addition of the precious metals.

TABLE II
 PROPERTIES OF MIXTURES OF VARIOUS FISSION PRODUCT TYPES
 - WITH AND WITHOUT THE PRECIOUS METALS -
 WITH A FIXED PUREX PROCESS COMPOSITION (64-2)

FP Type		Thermal History							
		Melted 1000 °C Slow Cooled				Melted 1200 °C Then Chill Cast			
		Without Precious Metals		With Precious Metals		Without Precious Metals		With Precious Metals	
	Concen- tration	Drip Tem- perature	Percent Solubility	Drip Tem- perature	Percent Solubility	Drip Tem- perature	Percent Solubility	Drip Tem- perature	Percent Solubility
(a)	5	799	3.29	822	3.49	927	0.35	918	0.40
	10	782	2.69	777	2.44	959	0.44	934	0.52
	20	767	3.48	771	4.63	964	0.24	950	0.24
(b)	5	860	4.52	858	0.70	914	0.34	875	0.41
	10	775	3.16	850	0.79	938	0.29	877	0.29
	20	742	3.77	741	14.8	956	0.37	861	0.46
(c)	5	839	3.09	845	0.85	909	0.45	851	0.096
	10	755	2.57	761	27.5	939	0.28	860	0.30
	20	763	3.67	729	25.2	961	0.49	856	0.27

So, the tentative answers to the above two questions are (1) that the precious metals should be included and (2) that at least some measurements must be made to relate fission product distribution as determined by the fuel processed to the properties that are used to decide suitability of the mixture for the purpose.

Since the cost of precious metals for pilot plant scale operation becomes large, it appears desirable to determine this relation on a laboratory scale, and then make the adjustments and interpolations indicated.

Some effort is being devoted by our Statistics Group to see if these relations can be reduced to mathematical expressions in which the various constituents of the waste can be assigned coefficients. If this could be realized it would simplify processing of variable wastes by indicating the proper adjustment of composition to yield the desired properties.

COLD SEMIWORKS STUDIES

Spray Calciner-Melter Development - J. D. Moore

NFS 1st and 2nd Cycle Waste - H_3PO_4 Flowsheet Development

Testing of the spray calciner-melter for operable flowsheets using NFS 1st and 2nd cycle waste (Table III) was completed. Eight runs investigating feeds with metallic cation equivalent to phosphorous atom ratios

TABLE III

FEED COMPOSITIONS TESTED

	NFS 1st and 2nd Cycle Waste 247 gal/tonne U, M	NFS 1st and 2nd Cycle Waste (High Na ⁺) 247 gal/tonne U, M	Purex Waste-2 100 gal/tonne U, M	Purex Waste-2 Adjusted to Melt 200 gal/tonne U, M	Recycle Sulfate Condensate - Spent Caustic, M
Fe ⁺³	0.571*	0.571*	0.502*	0.251*	0.0012
Cr ⁺³	0.001	0.001	0.024	0.012	-----
Ni ⁺²	0.001	0.001	0.10	0.005	-----
Al ⁺³	-----	-----	0.001	1.375	-----
Na ⁺	0.35	2.62	0.930	1.815	0.266
SO ₄ ⁻²	0.35	0.35	0.870	0.435	0.133
PO ₄ ⁻³	-----	-----	0.006	1.928	-----
SiO ₃ ⁻²	-----	-----	0.010	0.005	-----
ZrO ⁺²	0.031	0.031	0.065	0.0325	-----
MoO ₄ ⁻²	0.024	0.049	0.065	0.0325	-----
(Y + Rare Earths) ⁺³	0.055	0.055	0.1185	0.0592	-----
Rb + Cs (used K ⁺)	0.015	0.015	0.042	0.021	-----
Sr + Ba (used Ca ⁺²)	0.015	0.015	0.035	0.0175	-----
Ru + Rh + Pd (used additional Fe ⁺³)	0.011	0.011	0.057	0.028	-----
H ⁺	6.57	6.57	0.382	4.126	1.800
NO ₃ ⁻	8.147	10.367	1.56	4.4136	1.8036
CO ₃ ⁻²	0.0075	0.0075	0.021	0.0105	-----
OH ⁻	-----	-----	-----	1.2	-----
Li ⁺	-----	-----	-----	1.2	-----
Sugar	-----	-----	0.265	-----	-----

*Includes additional Fe⁺³ used as substitute for Ru, Rh, and Pd.

(M^+/P) ranging from 1.9 to 3.1 indicated that feed with M^+/P equal to 2.7 and 3.1 are completely operable under the conditions tested (see Table IV). Solids in feed with M^+/P equal to 3.1 remained in suspension longer than solids in the other feeds. For this reason, only this feed could be metered accurately into the calciner. Powder deposition on the calciner wall was minimal ($<1/8$ -in. thick) with this feed. Filter performance was adequate with all feeds tested. Feed with M^+/P equal to 2.1 produced a black glassy solid. All other solids were gray, crystalline, ceramic materials.

Feed with M^+/P equal to 3.1 was successful in a continuous 40-hr run. The filters operated well with a two-minute blow-back pulse cycle. Melt was continuously produced at a rate of $0.6 \text{ ft}^3/\text{day}$ at 1000 to 1050 °C.

Simulated NFS 1st and 2nd cycle waste (247 gal/tonne U) containing high sodium ion (Table III) with phosphate added to give M^+/P equal to 2.7 was tested in a seven-hour run (SC-121). Partially melted deposits formed on the calciner walls and the filters plugged. Deposits were formed by the presence of a high concentration of NaNO_3 which melts at 308 °C. (This deposition could be minimized by the addition of sugar to the feed which would reduce the nitrate.)

Purex Waste-2 Flowsheet Development

Studies with Purex Waste-2 (Table III) flowsheets were started. Purex Waste-2 (PW-2) is a combined first and second cycle waste and is one of the potential feeds for demonstration in the Waste Solidification Engineering Prototypes.

Two runs were made without the melter to produce PW-2 calcine for laboratory corrosion and glass composition studies. Feed concentration was 100 gal/tonne U with sugar added (0.265 molar) for nitrate reduction. A large accumulation of powder backed up above the powder collection pot during the first run. This was caused by condensate formation on the powder sampler which was not insulated. During the second run, the calciner

TABLE IV
SUMMARY OF SPRAY CALCINER-MELTER RUNS

Run No.	Feed Type	M ² /2	Feed Rate gal/hr	Calciner Furnace Temperature, °C	Melter Furnace Temperature, °C	Run Duration, hr	Feed Nozzle	Filter Type	Filter Blow-back Cycle, min	Blow-back Steam Pressure, psig	Initial Filter Pressure Drop in H ₂ O	Final Filter Pressure Drop in H ₂ O	Wall Deposit Thickness, in.
SC-121	NFS 1st & 2nd Cycle, Hi Na	2.7	3	700	1070	5	Spraco Systems Set-up No. 5	(15) Porous SS 8 - 20 u	1	85	12	>25	Heavy
SC-122	NFS 1st & 2nd Cycle, Lo Na*	2.7	5	700	1100	5.2	" "	" "	1	85	17	19	1/16
SC-123	" "	3.0	5	700	1100	5.2	" "	" "	1	85	13	17	Thick at bottom of calciner.
SC-124	" "	2.4	5	700	1100	5.5	" "	" "	1	85	14	18	Plugged bottom of calciner.
SC-125	" "	2.4	5	700	1050	6.7	Spraco Nozzle and Set-up No. 5	" "	2	85	11	19	1/4
SC-126	" "	1.9	5	700	1050	6.7	" "	" "	2	85	15	16	Plugged calciner outlet.
SC-127	" "	1.9	4	700	1025	5.7	Spraco Nozzle	" "	2	85	12	15	Large build up at calciner bottom.
SC-128	" "	2.1	5	700	1090	3.6	Spraco Systems Set-up No. 5	" "	2	85	21.5*	>25*	Plugged bottom of calciner.
SC-129	" "	3.1	5	700	1115	40	" "	" "	2	85	17	16	<1/8
SC-130	Sulfate Conden- sate Recycle	---	5	700	985	2.9	" "	" "	2	85	3.75	>25	Fine
SC-131	Purex Waste-2	---	5	700	---	4.8	" "	(4) 100 u wire filters & (11) porous SS	2	85 (1st 1-1/2 hr) 50 (last 1.8 hr)	2.5	17.5	Plugged calciner outlet.
SC-132	" "	---	5	800	---	4.6	" "	" "	5 (1st 1-1/2 hr) 2 (last 1.6 hr)	50	1	>25	1/16
SC-133	Purex Waste-2, Adjusted	4.2	3	550	1050	4.1	" "	(4) 50 u wire filters & (11) porous SS	2 (1st 1/2 hr) 10 (last 3.6 hr)	75	<1	3.75	1/16 - 1/8
SC-134	NFS 1st Cycle	2.7	3	700	1050	2.9	" "	" "	3	85	8.7	20	1/16

*Due to high air in-leakage into calciner.

wall temperature was raised 100 °C and the sampler was insulated. No powder build-up formed. Product was a red-brown, free-flowing powder without any agglomerates.

Another variation of PW-2 was tested for retention of sulfate as suggested by laboratory results. A feed concentration of 200 gal/tonne U was adjusted with Al^{+3} , Li^{+} , Na^{+} , and PO_4^{-3} (Table III) to melt at 600 °C. Product is currently being analyzed for sulfate content. Feed rate of three gallons per hour approached the melter capacity with the melter furnace operating at 1050 °C. Melt discharged steadily from the melter weir. Forty and one-half pounds of a dark brown, semi-vitreous product were collected during the run. A uniform coating of powder less than 1/8-in. thick covered the inside of the calciner walls. A low pressure drop across the filters was maintained with a ten minute filter blow-back cycle using 75 psig steam.

NFS 1st Cycle Waste - H_3PO_4

A scheduled 48-hr run (SC-134) was started using simulated 122 gal/tonne U NFS 1st cycle waste (65-1). Phosphate was added in a concentration to give M^{+}/P equal to 2.7. Feed flow was erratic but filter pressure drop reached an equilibrium value of 20 in. of water. Wall deposition was a uniform 1/16-in. coating of powder. The run was terminated after three hours when the copper lead wires of the resistance furnace on the melter oxidized and parted. Also, the Bakelite terminal board on the furnace became overheated and began to char and crack.

Sulfate Condensate Recycle Test

One approach to handling volatile sulfate from solidification processes is to produce a solid from the condensate. Condensate containing sulfate may be converted to a sulfate salt melt by combining with spent caustic scrubber solution. Operability was tested in the spray calciner-melter. Sufficient sodium was added to the condensate to form stoichiometric sodium sulfate. From the start, the pressure drop across the filters increased until the vacuum in the calciner began to drop. The feed was stopped and the filters were cleaned with nitric acid. The feed was started again

but the filters soon plugged again with fused sodium nitrate and the run was terminated. No visible deposits were formed on the calciner walls.

New Equipment Testing

A 20 kilowatt resistance furnace for 1300 °C operation was installed on the platinum melter. Heating elements were Kanthal A-1. During the first 100 hr of operation at temperatures not exceeding 1100 °C, several separate failures occurred: The external furnace temperature became too high and terminal boards, wire seals, and copper lead wire failed. Heating elements fractured near the junction with a lead out post.

Four wound-wire filters were substituted for 4 of the 15 porous stainless filters of the spray calciner during two runs. Wire spacing was 100 μ and filter area was the same as that of the filters. Off-gas filtration was inadequate since the condensate samples contained up to 1 vol% of solids. A coating of powder was allowed to form on the filters in order to increase off-gas filtration efficiency, but solids content of subsequent condensate samples was not reduced appreciably. The 100 μ spaced filters were replaced with four similar wound-wire filters with 50 μ wire spacing and equal filter area. A low pressure drop across the filters was maintained in two runs with the 50 μ filters. No solids were visible in the condensate samples. Condensate is being analyzed.

Testing of a new external-mix atomizing feed nozzle was carried out in three runs (SC-125, 126, 127). Feed atomization performance with this nozzle is at least as good as the standard nozzle for the spray calciner. Since the new nozzle outlet could not be extended below the top flange of the calciner without modification, feed back-mixed around the nozzle and calcined to plug the nozzle.

The pneumatic hammer was replaced by a floating piston type air vibrator. The spray calciner internal walls were maintained free from excessive powder build-up by the air vibrator during four runs (SC-131 to 134). The vibrator performance was at least equal to that of the pneumatic hammer. Its maintenance frequency appears to be much less than that of the hammer.

A thermal shock technique for cleaning the calciner walls between runs has been successfully tested several times. Wall cleaning could be required if gross malfunction of the calciner occurred. With the calciner walls heated to 700 °C, a tank cleaning nozzle mounted on the end of a six-foot length of stainless steel tubing is inserted through a port in the top of the calciner. Water is sprayed through the nozzle onto the hot walls. Wall deposits spall off and drop into the cold melter. The melter is then heated to boil off the excess water and melt the residual deposition.

WASTE SOLIDIFICATION ENGINEERING PROTOTYPES

(W. H. Swift, C. R. Cooley, C. E. King)

Design and Fabrication Status - K. J. Schneider, G. Rey, M. O. Rankin,
and V. P. Kelly

The previously reported (62-3) design, development, procurement, and fabrication of the Waste Solidification Engineering Prototypes (WSEP) is continuing. The WSEP will provide engineering scale demonstrations of promising solidification processes with actual high-level wastes. The prototypes will be installed in the Fuels Recycle Pilot Plant (FRPP), now under construction and scheduled for completion in 1965. Three solidification prototypes are currently scheduled for demonstration beginning in 1966: a pot, a spray, and a phosphate glass solidification.

Fabrication and procurement of the prototype spray calciner is complete. The three-zone resistance heated furnace for the spray calciner was received in a damaged condition, and will be returned to the manufacturer for repair after the design verification tests. The platinum melter has been received, but the resistance-heated melter furnace has been delayed by the manufacturer. Initial melter tests may be conducted with an existing resistance-heated furnace. Installation of the spray calciner in the 321 Building for design verification tests has been completed in June. Initial tests are underway.

Design of the phosphate glass solidification system is complete. Onsite fabrication is about 90% done. Manufacturer delays have resulted in expected delivery of the melter furnace and the platinum melter early next quarter.

Onsite fabrication of the furnace rack to contain the six-zone resistance-heated glass receiver and pot calciner furnace is about 90% done. Manufacturer's design of the furnace includes using zone cooling by air with provisions for water injection if required. Delivery is expected early next quarter.

The scope of the welding station has been modified to combine the wall thickness measurements. Procurement and fabrication of the welding station and other auxiliary equipment such as material handling and viewing equipment, jumpers, and control system wiring is nearly complete.

Design Verification Tests - J. L. McElroy, F. L. Mourich and G. Rey

Design verification testing of the pot calciner prototype and its related WSEP hardware has been successfully completed.

Two additional runs, a pot calciner run (CPC-8) with NFS 1st and 2nd cycle waste, and a rising level glass run (CPG-1) with Purex FTW-65 type waste were made this quarter. Feed compositions used are listed in Table V.

In addition to determining operating characteristics of NFS 1st and 2nd cycle waste, operability of a heat sensitive (thermocouple) liquid level probe in a pot containing a shortened draft tube was an objective of Run CPC-8. The short draft tube permitted operation at a lower liquid level and allowed higher wall temperatures near the top of the pot. This provided a good indication of the operating level and permitted a high average feed rate to be maintained. By controlling individual zone temperatures of the six-zone induction heated furnace it was possible to control the rate of rise of the pot internal temperatures so that all calcine temperature went from 600 to 800 °C as a group. This procedure minimizes the time the calcine is subjected to high temperatures in the lower zones and should help provide uniform drying of the calcine throughout all zones. The control of temperature rise in the present test was to gain experience in controlling temperature rise rates which may be difficult under conditions of internal heat generation from fission products.

The rising level glass run (CPG-1) was made using an 8-in. pot, modified to allow feed flow as a film down the centerline thermocouple well (3/4-in. Sch 80 pipe). This had a four-fold purpose: (1) to decrease spattering and therefore dust entrainment; (2) to increase the evaporation rate by increasing the radiant heat transfer from the pot walls to the

TABLE V
FEED COMPOSITIONS

Run Number	CPC-8	CPG-1
Feed Type	NFS 1st & 2nd Cycle, <u>M</u>	Purex FTW-65, <u>M</u>
H ⁺	6.57	3.00
Fe ⁺⁺⁺	0.56	0.11
Cr ⁺⁺	0.001	0.02
Ni ⁺⁺	0.001	0.01
Al ⁺⁺⁺		0.33
Na ⁺	0.66	1.33
Li ⁺		0.89
NO ₃ ⁻	8.48	3.10
SO ₄ ⁻⁻	0.35	0.16
PO ₄ ⁻⁻	0.01	1.33
SiO ₃ ⁻⁻		0.02
Sr ⁺⁺ (1)	0.009	0.01
Ba ⁺⁺ (1)	0.006	0.01
ZrO ⁺⁺	0.031	0.02
MoO ₄ ⁻⁻	0.024	0.03
Cs ⁺	0.015 ⁽²⁾	0.02 ⁽³⁾
Y + Rare Earths	0.055	0.05
Ru ⁺⁺	0.001	0.03 ⁽⁴⁾
M ₂ O moles/liter ⁽⁵⁾	1.4	2.0
Specific gravity at 25 °C	1.34	1.28
Volume percent solids	5	30 (gelatinous)
Volume, gal/tonne	247	80

(1) Sr⁺⁺ and Ba⁺⁺ added as Ca⁺⁺.

(2) Cs⁺ added as Na⁺.

(3) Cs⁺ added as K⁺.

(4) Ru⁺⁺ added as Mn⁺⁺.

(5) Feed cations expressed as calcined oxide.

film; (3) to maintain process stability by positive control of the feed entry into the melt; (4) to confine liquid interface corrosion in the pot to the thermowell. With a pot wall temperature of 850 °C, a calculated additional increase in boilup rate of 15 liters/hr is possible due to the film, decreasing linearly to 3 liters/hr as the pot is filled to level. Heating only the bottom three of six furnace zones during the run gave an initial boilup increase of 9 liters/hr and about 5 liters/hr of the overall average rate was attributed to evaporation from the film. The pot operating level was determined by interpretation of spaced internal and external pot thermocouples, located as shown in Figure 3. Temperature profiles which resulted during the run are shown in Figure 4.

Fifty liters of a uniform, granite-like material (Figure 5) were produced with no apparent corrosion of the 304-L stainless steel pot. Mild etching of the centerline probe did occur.

Partial refluxing of vapors from the off-gas line to another vessel successfully kept the off-gas line clean. However, the pot neck was nearly plugged with calcine and although no excessive pot internal pressures occurred during the run, further tests on refluxing to the pot are necessary to determine if the washing action will prevent calcine accumulation and the formation of a potential plug. Table VI summarizes the pertinent data for Runs CPC-8 and CPG-1.

Table VII summarizes the performance of prototype equipment. The GE-412 computer has been successfully used in several prototype pot calcination runs to perform on-stream material balances, routine calculations, and logging of data. A 5 to 30 liters/hr airlift was added to the prototype and successfully used for feeding during the rising level glass run.

Titanium corrosion of the evaporator and fractionator was detected following completion of the pot calciner tests. Fluoride concentrations of 0.017M and 0.006M were found in the fractionator bottoms from run CPC-6 and in a sample of titanium dioxide deposited on the fractionator tube bundle.

NOTES:

1	Startup and Checkout	6	Bottom Zone Temperature Reduced Since Melt Is About 2 Zones Deep
2	All Centerline Temperatures Indicate Wetting by Feed	7	Hold Lines Show Typical Pattern as Melt Level Rises in the Pot
3	Available ΔT for Heat Transfer from Pot Wall to Either the Centerline Thermocouple Probe or to Pool of Liquid	8	Furnace Cooling Started at 50 SCFM Air per Zone
4	Boiling Point Elevation due to Waste Concentration	9	Rate of Cooling Changes due to Fusion of Glass
5	First Indication of Melt, 6 IW & 5 C Show Melt Before 6 C Because of Pot Bottom Heat Losses		

FIGURE 4

Run CPG-1 Rising Level Glass with Purex FTW-65

FIGURE 3
8-in. Rising Level Glass Pot Details

FIGURE 5

Calciner Pot Filled with Purex FTW-65 Waste by Rising Level Glass Process

TABLE VI
POT CALCINER AND RISING LEVEL GLASS
DESIGN VERIFICATION TESTS

Run Number	CPC-8	CPG-1
Feed Type	NFS 1st & 2nd Cycle	Purex FTW-65
Operating Method	Cold Wall	Rising Level
Method of Level Detection	Thermal Probe	Thermocouples
Pot Diameter, inches	8	8
Draft Tube Diameter, inches	3	--
Net Power, kilowatts	36	30
Furnace Operating Temperature, °C	900	850
Initial Feed Rate, liters/hour	40	18
Final Feed Rate, liters/hour	5	7
Feed on Time, hours	41	62
Volume of Feed, liters	797	614
Average Feed Rate, liters/hour	19.4 ⁽¹⁾	9.9
Feed Added Prior to Scaling, liters ⁽²⁾	418	--
Furnace on Time, hours	50	68
Average Over-all Rate, liters/hour	15.9	9.0
Average Boilup Rate, liters/hour	18.3 ⁽³⁾	9.8 ⁽⁴⁾
Calcine Net, kilograms	89	--
Glass Net, kilograms	--	123
Pot Fill Volume, percent Full	100	80
ρ_B of Calcine or Glass, kilograms/liter	1.49	2.46
Volume Reduction of Glass	--	12.3

(1) Does not include 115 liters of 1M NaNO_3 added as separate stream.

(2) Feed addition rate drops off rapidly due to heat flux limitations following scaling.

(3) Includes NaNO_3 additive.

(4) 50 liters of water added prior to feed.

TABLE VII
WSEP EQUIPMENT PERFORMANCE

Equipment Piece	Construction Material	Conditions			Total Operating Hours	Remarks
		Temperature, °C	Pressure in H ₂ O	Chemical		
Feed Tanks	SS 304 L	60	-5	Darex, Purex, TBP-25 and NFS type wastes	800	Agitators used for 183 hr.
Condensate Tanks	SS 304 L	25	-18	H ₂ O, Dilute HNO ₃	800	---
Caustic Scrubber	SS 304 L	25-40	-20	Caustic	800	Circulating pump used for 93 hr.
Evaporator	A-55 Titanium	110	-18	Boiling Feed	500	Mild titanium corrosion caused by presence of fluoride ion. Most predominant during CPC-6
Fractionator	A-55 Titanium	115	-18	8M HNO ₃	500	Mild titanium corrosion caused by presence of fluoride ion. Most predominant during CPC-6.
Pot Calciner Condenser	A-55 Titanium	110	-18	HNO ₃	800	Localized vapor entry corrosion up to 1/16-in. during runs CPC-3 and CPC-4.
Feed Pump	SS 304 L	60	--	Feeds	468	Graphite bearing excessively worn after 468 hr.
Induction Furnace	Hastelloy X	900	--	--	687	Delaminated insulation partially replaced with alumina silicate fiber insulation at 450 hr. Motor generator bearing replaced at 577 hr.
GE-412 On-Line Computer	--	--	--	--	450	---
Calciner Pots	SS 304 L, non-corrosion tested	100-900	-18	Concentrated Feed	488	No warping, bending or detectable corrosion.

The source of this unexpected fluoride is being investigated. Six-hour laboratory tests have shown that in 6M boiling HNO_3 , fluoride concentrations of 0.005M and 0.025M will produce titanium corrosion rates of 8.5 and 347 mils/month, respectively.

Waste Container Storage - W. V. DeMier and M. E. Spaeth

In the WSEP demonstration, containers of solidified radioactive waste are to be transferred to an intermediate storage station. At that station (Rack 4A) calorimetric measurement of the heat generation rate will be made.

Since containers will be loaded to safe thermal maximums, a guide has been determined for maximum steady-state temperatures reached by cylindrical radioactive waste containers under various conditions of storage. Criteria was that container centerline temperature must not exceed 900°C (1652°F) and/or the wall temperature must not exceed 427°C (800°F). The centerline and wall temperature limits used are assumed to be critical temperatures which should not be exceeded in the handling or storage of heat generating waste containers in the WSEP.

Table VIII shows the maximum tolerable amount of heat generating materials allowed in an eight inch diameter waste containing cylinder for various storage media. Figure 6 shows the effect of heat generation on permissible storage conditions.

TABLE VIII
PERMISSIBLE HEAT GENERATION RATE
FOR 8-in. diam. WASTE CONTAINERS

<u>Storage Medium</u>	<u>Material in Waste Cylinder</u>	<u>Maximum Tolerable Heat Generating Materials, (Btu/hr-ft³)</u>
Sand	Glass	1,700
Air	Glass	22,200
Water	Glass	92,500
Sand	Calcine	1,700
Air	Calcine	12,250
Water	Calcine	15,500

- Region to left of curve designates conditions, below the assumed critical conditions, for that storage medium.
- Temperature assumed critical when centerline reaches 1652 °F (900 °C) or wall temperature reaches 800 °F (427 °C).
- Assumed thermal conductivity of calcine is 0.3 and glass is 1.0 Btu/hr-ft-°F.
- Assumed thermal conductivity of sand is 0.2 Btu/hr-ft-°F.
- Waste heat density at 20,000 Mwd/t (assuming liquid right out of reactor) and 15 Mw/t at one day old is 2.82×10^5 Btu/hr-ft³ and at one week 1.35×10^5 Btu/hr-ft³.

FIGURE 6

Thermal Criticality for Various Diameter Pots Versus Heat Generation Rate for Typical Glass and Calcine Under Various Conditions of Storage

HAZARDS OF SHIPPING SOLIDIFIED HIGH LEVEL WASTE

(A. E. Albrethsen)

The airborne release of fission products from high level radioactive wastes during a shipping accident and the possible sustained exposure to a high temperature fire is an important consideration in the overall evaluation of the accident. The experimental studies are directed to the evaluation of airborne fission product release from high level ceramic waste material at high temperatures. The program is concerned with two mechanisms of release—volatilization and mechanical entrainment of particulates in an airstream.

Current experimental procedures involve the spiking of a 15 gram sample of a phosphate ceramic material—representative of a solidified waste from a power reactor fuel with a 20,000 MWD/T irradiation—initially fusing this material to fix the spike, and then reheating at the experimental temperature. Cs^{137} is being used as a tracer for cesium volatilization and Ce^{144} , Ru^{106} , and Sr^{85} will be used to trace the respective elements. The environmental atmosphere is controlled by a carrier gas which passes over the molten ceramic, through a quartz chimney, and through a fiber glass filter. The volatiles from the heated ceramic are collected on the quartz chimney and on the fiber glass filter. Samples of the condensed volatiles are taken after the initial fixing heat and after the second heat. Metal crucibles, 304 L stainless steel at the present, are being used in order to maintain experimental conditions similar to those in an accident with actual waste material which would be contained in a metal capsule.

During the initial heats where the apparatus was being checked out, it was found that the ceramic melts at about 975 °C. Also an appreciable amount of corrosion of the stainless steel crucibles was observed in both air and argon atmospheres. This corrosion occurred in zones of the crucible: (1) at the air-melt-crucible contact where the removal of the protective metal oxide by the melt permitted a continual oxidation of the crucible by the air, and (2) at the interface between the melt and the crucible where the metal itself reacted with the melt. cursory checks with some thin walled nickel crucibles indicated that nickel is a more inert material than the stainless steel.

The results of five preliminary tests with simulated waste material spiked with Cs^{137} are shown in Tables IX and X. The releases during the fixing melt (Table IX) may not be significant because the release may have occurred before the spike was dissolved in the melt. The releases during the second melt (Table X) which is intended to be indicative of an accident involving a fire infer that the accidental release could be significant and will probably have to be considered in an accident evaluation. Releases of as high as one percent were realized during a one hour heat at 1200 °C.

TABLE IX
RELEASE OF Cs^{137} FROM A 15 GRAM SAMPLE OF
SIMULATED WASTE DURING THE FIXING AND MELTING HEAT*

<u>Run</u>	<u>Preheat Time, Minutes</u>	<u>Temperature</u>	<u>Release Fraction</u>
1	90	975 °C	2×10^{-3}
2	80	975 °C	6×10^{-3}
3	50	1000 °C	3×10^{-3}
4	70	970 °C	3×10^{-3}
5	70	975 °C	2.5×10^{-3}

* Argon atmosphere was maintained. Preheat time indicates time taken to heat sample to temperature which was then maintained for one hour.

TABLE X
RELEASE OF Cs^{137} FROM A 15 GRAM SIMULATED
WASTE SAMPLE DURING A ONE HOUR HEAT

<u>Run</u>	<u>Temperature</u>	<u>Atmosphere</u>	<u>Release Fraction</u>
1	1075 °C	Argon	6×10^{-3}
2	1200 °C	Argon	4×10^{-3}
3	1075 °C	Air	6×10^{-3}
4	1200 °C	Air	1×10^{-2}
5	1150 °C	Air	1×10^{-2}

WASTE STORAGE AND TREATMENT
(B. M. Johnson, G. J. Alkire)

Heat Transfer at Buried Radioactive Waste Storage Tanks - W. E. Willingham,
G. Jansen

An important consideration in the in-tank solidification of radioactive wastes is the temperature reached in the solid cake because of heat generating fission products. All previous calculations for Purex waste tanks have been made with a 60-foot insulated radius surrounding the tank with 120-foot center-to-center spacings. New calculations with a 300-foot insulated radius have given results which approximate a solitary tank with no neighbors. This situation is quite useful for analyzing temperatures in tanks in which unusual conditions are present.

In Figure 7 the temperature profiles are presented for a horizontal plane passing through the center of a two-foot thick cake generating 32,800 Btu/hr. A temperature of 408 °F is reached in the cake for the tank with a 60-foot insulated radius, while the cake in the solitary tank reaches only 332 °F. The close approach to ambient temperature of 70 °F at radii greater than 200 feet shows a good approximation to a single tank in an infinite radius medium.

In Figure 8, the vertical temperature profiles along the axis of the tank in the two systems are compared. The temperature gradients toward the surface and the water table are quite similar, although much more heat flows radially in the larger system. For the 60-foot insulated radius 85.6% of the heat generated goes to the atmosphere and the remainder to the water table. For the solitary tank, 68.5% of the heat is distributed to the atmosphere and the water table within the 60-foot radius and the remainder outside this radius. The split between the atmosphere and the water table is the same as for the tank with 60-foot insulated radius.

Waste Solidification Condensate Treatment - B. W. Mercer

Laboratory studies were initiated to determine methods for converting sulfate in the waste solidification glass melter condensate to a relatively insoluble melt. Lead sulfate is currently being investigated as a possible

FIGURE 7
 Temperature Profile in the Horizontal Plane of Maximum Temperature

FIGURE 8

Vertical Temperature Profile at the Axis of the System

candidate for sulfate melts. Lead sulfate forms melts below 1000 °C with borax and lead oxide. The lead sulfate-borax mixture forms a two-phase melt, whereas the lead sulfate-lead oxide mixture forms a one-phase melt. Both melts are expected to have a low solubility which will be determined by a standard method at a later date. The addition of lead oxide (litharge) to the condensate forms a slurry of lead sulfate which appears to have good flow characteristics without addition of water. By comparison, calcium sulfate forms a very thick slurry under the same conditions.

Fission Product Separation and Packaging - B. W. Mercer

(Not charged to Division of Reactor Development)

Laboratory experiments were conducted to determine improvement in cesium purity as a result of recycling cesium eluates with supernatant feed to an AW-500 ion exchange column. A reduction in the amount of sodium impurity in the separated cesium is desired to permit higher cesium loadings on the zeolite in the storage canisters or reduce the additional purification needed for isotope sources.

A simulated feed solution, 4.4 M sodium and 4×10^{-3} M cesium, was used in the experimental study. The feed solution composition was based on the following assumptions.

1. Four loading and elution cycles were made with standard supernatant feed but the water washes and dilute ammonium carbonate scrubs were eliminated.
2. The eluates were evaporated to remove the ammonium carbonate and combined with a batch of standard feed for the fifth loading cycle.

The fifth loading cycle with feed containing recycled cesium was taken to 38 column volumes where 5% breakthrough occurred. Four column volumes of water and 13 column volumes of ammonium carbonate scrub were then pumped through the bed. The amount of scrub was increased over the standard flowsheet volume (8 column volumes) to effect further purification of the cesium. About 1% and 1.5% of the cesium was lost during the loading and scrub cycles respectively. The Na:Cs mole ratio by recycle was 1:3.4 as compared to 8:1 by the standard method.

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