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REDUCTION OF LIQUID RADIOACTIVE WASTES TO SOLIDS: SCOUTING TESTS

C. W. Hancher

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CHEMICAL TECHNOLOGY DIVISION
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REDUCTION OF LIQUID RADIOACTIVE WASTES TO SOLIDS: SCOUTING TESTS

C. W. Hancher

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ABSTRACT

Scouting experiments indicated that calcination of highly active Darex and Sulfex decladding and Purex extraction wastes will not release hazardous amounts of fission products to the atmosphere. Simulated wastes containing up to 90 curies/liter of activity were evaporated and calcined to 750°C and the off-gases passed through a condenser and a series of caustic scrubbers. Less than 0.1% of the fission products was released, of which ruthenium was 10-90%. Fission product release was lower from neutralized than from acidic wastes. Fission products were leached appreciably, 0.05% of the beta and 0.02% of the gamma, from the calcination solid with water in 96 hr.

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1.0 INTRODUCTION

This report describes scouting experiments on evaporation and calcination of radioactive liquid wastes. Storage of radioactive wastes as solids should be less hazardous than liquid storage and may prove to be less expensive. The degree of volatility of fission products during calcination is an important factor in determining the feasibility and cost of calcination. This work and the suggested flowsheet represent an initial scouting effort in the ORNL program for "pot" calcination of high radioactive level wastes.[1,2]

Seven experiments were made with waste simulating that from Darex (dilute aqua regia and Sulfex sulfuric acid) decladding of stainless steel—clad fuel and Purex extraction (tributyl phosphate from nitric acid solution) of uranium fuel. The wastes were spiked to intermediate levels of activity, up to 4×10^{13} each beta and gamma counts/min/ml (90 curies/liter), with raw waste from Purex processing of irradiated fuel. One run was made with raw Purex waste of 8×10^{13} each beta and gamma counts/min/ml. Because of the high level of activity, experiments could not be as extensive as was desired with the equipment available. In one experiment kaolinite was added in an attempt to decrease the water leaching of fission products from the calcination product.

The author acknowledges the assistance of the group of ORNL Analytical Chemistry Division, who determined the fission product values in the various samples.

2.0 FLOWSHEET

A suggested flowsheet for converting liquid radioactive wastes to solid prior to storage consists in evaporating and calcining the waste (Fig. 1). Neutralization of the waste prior to evaporation may decrease

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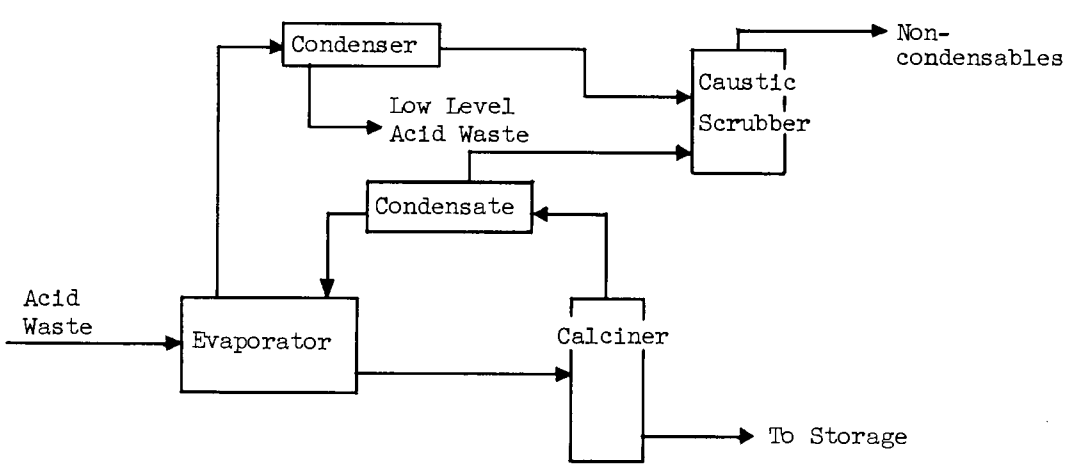

Fig. 1. Suggested flowsheet for evaporation and calcination of liquid radioactive wastes.

the volatility of the fission products, and addition of clay may decrease the leachability of fission products from the calcination cake.

The liquid is evaporated and the residue calcined, with the temperature gradually increasing to approximately 750°C. The off-gas is passed through a condenser, and the noncondensable portion is scrubbed with caustic prior to release. The condensed off-gas, containing fission products, is recycled to the evaporator. The calcined solid is stored.

3.0 EXPERIMENTAL WORK

3.1 Results

The maximum fission product recovery from the off-gas was 0.5% of the amount in the original waste, ruthenium being the most volatile (Table 1). Fission products in the noncondensable off-gas from Purex and Sulfex type wastes were a factor of 10 to 100 lower than in that from Darex type waste. Neutralization of the waste decreased the noncondensable off-gas activity by a factor of 10 to 100 with all three wastes.

The calcination cake activity reading at 1 ft was 500 to 100 r. The addition of kaolinite clay to the waste did not prevent water leaching of the fission product from the calcined cake. When the solid was leached 96 hr in cold water, appreciable radioactivity was found in the leach liquor (Fig. 2, Table 2), 0.05% of the beta and 0.02% of the gamma.

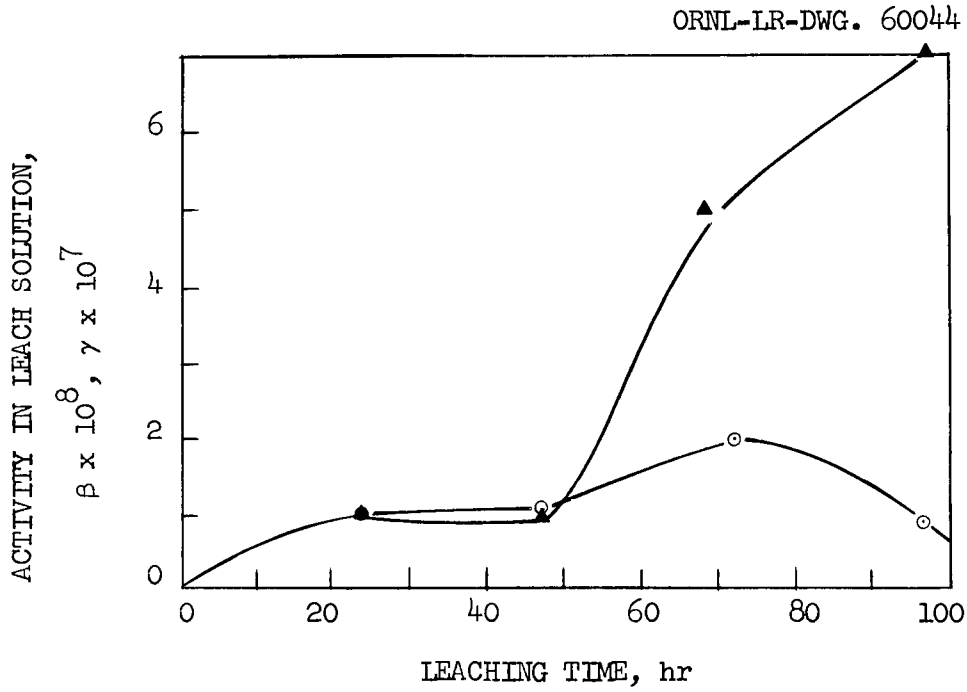

Fig. 2. Leaching of solid calcination cake with 200 ml of water at room temperature.

Table 1. Fission Products Volatilized from Simulated Process Wastes

Volume of feed to evaporator: 200 ml

Waste	Activity in Feed, c/min			Activity, % of that in feed: In Condensate			Activity, % of that in feed: In Noncondensable Off-gas		
	β	γ	Ru	β	γ	Ru	β	γ	Ru
Darex (Run 60)	$2x10^{12}$	$2x10^{12}$	$2x10^{11}$	0.02	0.1	0.02	$6x10^{13}$	$4x10^{-3}$	$7x10^{-2}$
Darex (Run 63)	$4x10^{13}$	$4x10^{13}$	-	Lost	Lost	-	$2x10^{-8}$	$1x10^{-7}$	-
Darex + NaOH (Run 62)	$2x10^{12}$	$2x10^{12}$	$2x10^{11}$	0.003	0.007	0.01	$6x10^{-5}$	$4x10^{-4}$	$2x10^{-4}$
Sulfex (Run 61)	$1x10^{12}$	$1x10^{12}$	$1x10^{11}$	0.02	0.3	0.1	$3x10^{-5}$	$1x10^{-4}$	$3x10^{-4}$
Purex (Run 66)	$2x10^{12}$	$2x10^{12}$	$2x10^{11}$	0.04	0.06	0.04	$1x10^{-4}$	$2x10^{-4}$	$4x10^{-4}$
Purex (Run 67)	$8x10^{13}$	$8x10^{13}$	$4x10^{12}$	0.001	0.01	0.03	$1x10^{-4}$	$2x10^{-4}$	$2x10^{-3}$
Purex + NaOH (Run 64)	$2x10^{12}$	$2x10^{12}$	$2x10^{11}$	0.003	0.002	0.02	$3x10^{-5}$	$7x10^{-5}$	-
Purex + NaOH + clay (Run 65)	$4x10^{13}$	$4x10^{13}$	$2x10^{12}$	0.005	0.01	0.1	$7x10^{-6}$	$2x10^{-5}$	$4x10^{-5}$

Table 2. Leaching of Fission Products from Calcination Solid

Activity of feed to evaporator: $4x10^{11}$ β and $4x10^{11}$ γ c/min/ml
Volume of liquid feed to evaporator: 200 ml

Time, hr	Activity in Leach Liquor, c/min/ml: Gross β	Gross γ	Sr β	Ce β	Cs γ	Ru γ	Zr + Nb γ
0	-	-	-	-	-	-	-
24	$1x10^{8}$	$1x10^{7}$	$9x10^{7}$	$2x10^{6}$	$6x10^{6}$	$1x10^{6}$	$4x10^{6}$
48	$1x10^{8}$	$9x10^{6}$	-	-	-	-	-
72	$2x10^{8}$	$5x10^{7}$	-	-	-	-	-
96	$0.7x10^{8}$	$7x10^{7}$	-	-	-	-	-

3.2 Equipment and Procedure

The waste was calcined, in 200-ml batches, in a muffle furnace with a maximum operating temperature of 1000°C. The 4- by 4- by 4-in. (inner dimensions) furnace was lined with a 4-in.-high, 3-in.-dia, schedule 40 pipe, in which the calcination vessel, a 600-ml stainless steel beaker, was placed. Off-gas escaped through a 0.25-in.-dia hole in the liner cover and passed to a glass water-cooled condenser. Noncondensables went to the bottom of a packed-bed scrubber, a 1-in.-dia glass pipe 24 in. high, packed with 20 in. of 0.25-in.-dia Raschig rings, and was removed from the top. Ten per cent sodium hydroxide was pumped into the top of the column by a Sigma motor (finger) pump and drawn from the bottom. Gas leaving the scrubber was bubbled through two glass bubblers, each containing 200 ml of 10% sodium hydroxide, and then went to the plant radioactive off-gas system. The experimental equipment scheme is shown in Fig. 3 and the compositions of the solutions used in Table 3.

The heating cycle for the calcination was 10 to 12 hr. The waste mixture was heated to approximately its boiling point and held at this temperature until 70 to 80% of the original mixture appeared as condensate.

Table 3. Compositions of Simulated Feed Used in Waste Calcination Study*

All solutions made up to 1 liter

Chemical	Mol. Wt.	Amount	Conc., M
		Purex	
$Fe(NO_3)_3 \cdot 9\ H_2O$	404	202 g/liter	0.5
Na_2SO_4	142	21.3 g/liter	0.15
H_2SO_4	98	35.5 ml of 18 M	0.60
HNO_3	63	353 ml of 15.6 M	5.50
		Darex	
$Ni(NO_3)_2 \cdot 6\ H_2O$	291	19.8 g/liter	0.075
$Fe(NO_3)_3 \cdot 9\ H_2O$	404	275 g/liter	0.68
$Ca(NO_3)_3 \cdot 9\ H_2O$	400	64 g/liter	0.16
$Al(NO_3)_3 \cdot 9\ H_2O$	375	46.1 g/liter	0.123
HCl	36.5	1 ml of 1 M	0.001
HNO_3	63	189 ml of 15.6 M	2.95
		Sulfex	
$Fe_2(SO_4)_3$	400	340	0.86
$Cr_2(SO_4)_3$	392	130	0.33
$Ni(SO_4)$	155	6.0	0.04
H_2SO_4	98	75 ml of 18 M	1.30

*Prepared according to formulas in CF-58-4-45, "Manual for the Preparation of Simulated Fuel Reprocessing Waste Solution," by W. J. Lacy, June 20, 1958.

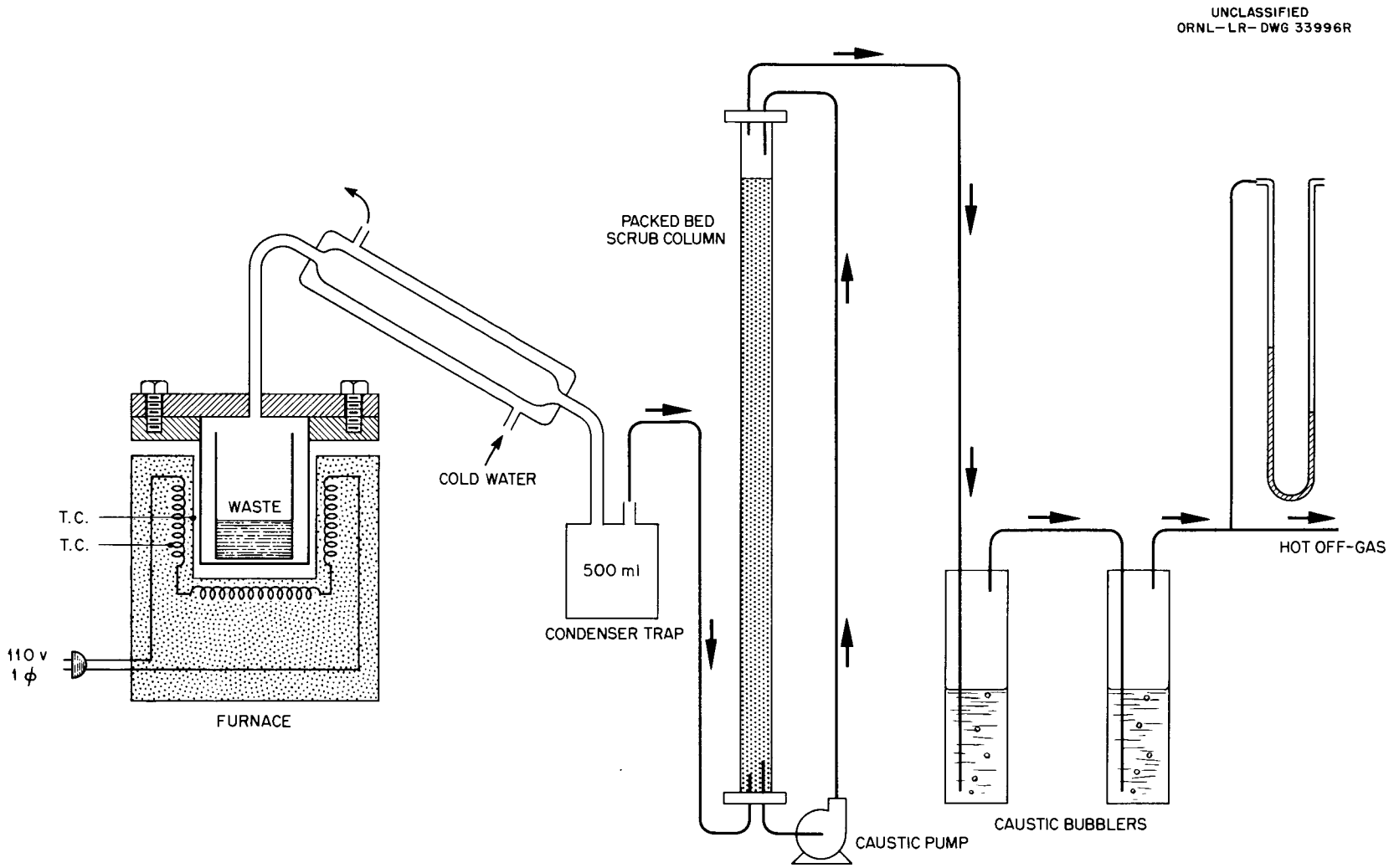

Fig. 3. Experimental setup for radioactive waste reduction to solids.

At this point the rate of condensate production decreased or stopped, and the temperature was increased to 700 to 800°C and was held there 2 to 3 hr. During the initial heating, while the liquid was boiling, the condensate receiver was emptied and sampled four or five times.

The packed-bed scrubber, containing approximately 400 ml of 10% sodium hydroxide, was sampled after each condensate sample was taken. The caustic bubblers were sampled only at the end of each run.

In run 65, in which clay was added, 194 g of Georgia kaolinite, 15 g of boric acid, and 28 g of sodium hydroxide* were added to 100 ml of raw Purex waste, plus 200 ml of water to permit mixing. After the calcination, the cake was leached by adding 100 ml of water, all of which was absorbed by the sintered solid the first day. The next day another 100 ml was added, and the liquid was sampled every 24 hr.

When neutralized waste was run, sodium hydroxide was added and the mixture was stirred prior to calcination.

3.3 Analytical Procedures

The gross gamma activity was determined with a deep-well crystal gamma spectrometer system and the gross beta by taking a small amount of the sample, evaporating to dryness, and counting with a Geiger-Mueller tube system. The individual fission products were separated by chemical procedures and then counted. The counting efficiencies for the various isotopes varied from 4% for the strontium to between 17 and 22% for the other fission products reported.

4.0 REFERENCES

1. Chemical Technology Division Annual Progress Report for Period Ending August 31, 1960, ORNL-2993 (Sept. 26, 1960).

2. Chemical Technology Division Annual Progress Report for Period Ending May 31, 1961, ORNL-3153 (in press).

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*Formula proposed by T. Tamura, Health Physics Division, ORNL.

5.0 APPENDIX

Details of the various runs are given below.

	Volume, ml	Activity, c/min/ml						
		Gross β	Gross γ	Sr β	Ce β	Cs γ	Ru γ	Zr + Nb γ
Simulated Darex decladding waste, run 60								
Feed	200	$9x10^9$	$1x10^{10}$	$1x10^9$	$4x10^9$	$4x10^8$	$1x10^9$	$1x10^{10}$
Condensate	110	$3x10^6$	$2x10^7$	$3x10^5$	$9x10^5$	$8x10^4$	$4x10^5$	$2x10^7$
	40	$3x10^5$	$3x10^6$	$3x10^4$	$7x10^4$	$2x10^4$	$2x10^4$	$5x10^6$
	20	$4x10^5$	$4x10^6$	$1x10^3$	$3x10^2$	$1x10^4$	$5x10^5$	$3x10^6$
NaOH in wash column	400 ml total	325	1600	3	8	34	40	-
		470	1660	42	161	63	1270	-
		525	1790	7	-	34	1070	-
		275	845	9	5	20	478	-
		200	955	7	8	28	470	-
NaOH in bubbler	200*	$2x10^5$	$2x10^6$	$9x10^2$	$3x10^3$	$6x10^2$	$3x10^5$	$1x10^6$
	200*	$3x10^5$	$2x10^6$	$5x10^2$	$4x10^2$	$1x10^3$	$4x10^5$	$1x10^6$
	200	Lost	-	-	-	-	-	-

*Samples contaminated.

	Volume, ml	Activity, c/min/ml Gross β	Gross γ	Sr β	Ce β	Cs γ	Ru γ	Zr + Nb γ
Run 63								
Feed	200	$2x10^{11}$	$2x10^{11}$	$2x10^{10}$	$9x10^{11}$	$1x10^{10}$	$2x10^{10}$	$1x10^{11}$
Condensate	0							
NaOH wash column	400	580	125					
NaOH bubbler	200	250	1600					
	200	290	662					
Simulated Darex decladding waste + NaOH, run 62								
Feed	200	$9x10^9$	$9x10^9$	$1x10^9$	$4x10^9$	$4x10^9$	$1x10^9$	
Condensate	40	$1x10^6$	$2x10^6$	$5x10^4$	$6x10^5$	$2x10^5$	$5x10^5$	-
	40	$7x10^4$	$1x10^5$	$4x10^3$	$1x10^4$	$1x10^4$	$4x10^4$	-
	40	375	$4x10^4$	$1x10^3$	$2x10^4$	$6x10^3$	$1x10^4$	-
	40	0	$7x10^5$	$1x10^5$	$3x10^4$	$9x10^4$	$6x10^5$	-
NaOH wash column	400 ml total	130	649		1800		110	-
		75	693		1100		115	-
		360	1900		1100			-
		40	0		1500			-
NaOH bubbler	200	1500	$1x10^4$	440	1300	2000	574	-
	200	1500	$2x10^4$	485	940	9000	832	-
	200	1300	$1x10^4$	219	1800	1500	1400	-

	Volume, ml	Activity, c/min/ml						
		Gross β	Gross γ	Sr β	Ce β	Cs γ	Ru γ	Zr + Nb γ
Simulated Sulfex decladding waste, run 61								
Feed	200	$6x10^9$	$6x10^9$	$7x10^8$	$1x10^9$	$4x10^9$	$5x10^8$	$4x10^9$
Condensate	35	$4x10^6$	$3x10^7$	$4x10^5$	$5x10^5$	$6x10^6$	$5x10^5$	$2x10^7$
	35	$8x10^5$	$6x10^6$	$2x10^5$	$5x10^4$	$8x10^5$	$7x10^4$	$4x10^6$
	35	$4x10^5$	$2x10^6$	$7x10^4$	$5x10^4$	$2x10^5$	$3x10^4$	$2x10^6$
	35	$1x10^6$	$6x10^7$	$8x10^4$	$3x10^5$	$2x10^5$	$2x10^6$	$4x10^7$
NaOH wash column	400 ml total	70	190				60	
		160	173				45	73
		0	155				36	43
		80	74				29	12
NaOH bubbler	200	550	2200	44	55	175	589	1200
	200	350	1600	60	177	52	423	1000
	200	180	969	21	33	47	180	642

	Volume, ml	Activity, c/min/ml						
		Gross β	Gross γ	Sr β	Ce β	Cs γ	Ru γ	Zr-Nb γ
Simulated Purex extraction waste, run 66								
Feed	200	$9x10^9$	$9x10^9$	$1x10^9$	$1x10^9$	$4x10^9$	$1x10^9$	$5x10^9$
Condensate	70	$4x10^6$	$6x10^6$	$3x10^4$	$8x10^4$	$5x10^5$	$6x10^6$	-
	70	$5x10^6$	$9x10^6$	$3x10^4$	$1x10^5$	$6x10^4$	$8x10_6$	-
	20	Lost	-	-	-	-	-	-
NaOH wash column	400 ml total	$2x10^3$	$9x10^3$	$3x10^4$	$6x10^2$	$1x10^3$	$2x10^3$	$6x10^3$
		$6x10^3$	$9x10^3$	$3x10^4$	$3x10^3$	$2x10^3$	$3x10^3$	$3x10^3$
		$2x10^3$	$5x10^3$	-	$5x10^2$	$2x10^3$	$2x10^3$	$2x10^3$
		$2x10^3$	$9x10^3$	-	-	$2x10^3$	$1x10^3$	$4x10^3$
NaOH bubbler	200	Lost						
	200	$2x10^3$	$2x10^3$	-	-	-	-	-
	200	Lost						
Raw Purex extraction waste, run 67								
Feed	200	$4x10^{11}$	$4x10^{11}$	$1x10^{10}$	$4x10^{10}$	$4x10^9$	$1x10^{10}$	$1x10^{11}$
Condensate	50	Lost						
	80	$5x10^6$	$7x10^6$	$3x10^4$	$2x10^5$	-	$7x10^6$	-
	20	Lost						
NaOH wash column	400 ml total	$6x10^3$	$1x10^4$	-	-	-	$1x10^4$	-
		$5x10^4$	$8x10^4$	$3x10^2$	$3x10^2$	-	$8x10^4$	-
NaOH bubbler	200	$4x10^5$	$7x10^5$	$8x10^2$	$1x10^4$	$3x10^3$	$1x10^5$	-
	200	$1x10^3$	$3x10^3$	$2x10^2$	$2x10^2$	$1x10^3$	$1x10^3$	94
	200	Lost						

	Volume, ml	Activity, c/min/ml						
		Gross β	Gross γ	Sr β	Ce β	Cs γ	Ru γ	Zr-Nb γ
Simulated Purex extraction waste + NaOH, run 64								
Feed	200	$8x10^9$	$8x10^9$	$8x10^8$	$4x10^9$	$4x10^7$	$8x10^8$	$5x10^9$
Condensate	35	$9x10^4$	$2x10^5$	$2x10^4$	$1x10^4$	$2x10^4$	$3x10^4$	$4x10^4$
	35	$2x10^5$	$8x10^4$	$4x10^4$	$5x10^4$	$2x10^4$	$2x10^4$	$4x10^4$
	35	$1x10^6$	$4x10^5$	$2x10^5$	$2x10^5$	$1x10^5$	$5x10^5$	$7x10^4$
	40	$1x10^5$	$8x10^4$	$4x10^4$	$5x10^4$	$2x10^4$	$2x10^4$	$4x10^4$
NaOH wash column	400 ml total	1500	2000					
		400	1200					
		400	2500					
		600	2300					
NaOH bubbler	200	240	460					
	200	180	317					
	200	490	1000					

	Volume,	Activity, c/min/ml						
		Gross β	Gross γ	Sr β	Ce β	Cs γ	Ru γ	Zr-Nb γ
Simulated Purex waste + NaOH + clay, run 65								
Feed	100	$4x10^{11}$	$4x10^{11}$	$1x10^{10}$	$4x10^{10}$	$4x10^{9}$	$2x10^{10}$	$1x10^{11}$
Condensate	30	$3x10^{7}$	$6x10^{7}$	$1x10^{5}$	$9x10^{4}$	$3x10^{6}$	$5x10^{7}$	$5x10^{6}$
	70	$4x10^{6}$	$6x10^{6}$	$4x10^{4}$	$1x10^{4}$	$5x10^{5}$	$5x10^{6}$	$1x10^{5}$
	70	$1x10^{6}$	$4x10^{6}$	$3x10^{4}$	$8x10^{3}$	$2x10^{5}$	$3x10^{6}$	$3x10^{5}$
	70	$9x10^{6}$	$2x10^{7}$	$1x10^{5}$	$4x10^{5}$	$4x10^{5}$	$1x10^{7}$	$9x10^{5}$
NaOH wash column	400 ml total	$1x10^{4}$	$1x10^{4}$	$1x10^{3}$	$1x10^{3}$	$3x10^{3}$	$3x10^{3}$	$8x10^{3}$
		$7x10^{3}$	$1x10^{4}$	$1x10^{3}$	$1x10^{3}$	$4x10^{3}$	$2x10^{3}$	$7x10^{3}$
		$7x10^{3}$	$3x10^{4}$	600	$1x10^{3}$	$3x10^{3}$	900	$8x10^{3}$
		$4x10^{3}$	$1x10^{4}$	800	39	$3x10^{3}$	$2x10^{3}$	$5x10^{3}$
NaOH bubbler	200	580	1500	-	67	314	714	210
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