This document consists of 4 pages and 0 figures.

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M--1079

3331

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5. W. C. Johnson
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W. F. Underwood
11. J. B. Sutton
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August 22, 1944

To: J. P. Sinclair Department: Operating

From: W. Q. Smith Department: Operating

In re: SUMMARY OF RUNS 194 TO 201 - 205 BUILDING

Run number	1 - 201	194 - 201
% Overall yield	85.3	88.2
% Original product to Room D	90.2	93.3
% Product to waste in canyon	8.5	13.7
% Product to waste in Room D	3.9	4.0
% Material balance	97.7	105.9

Trial runs of the Hanford separations procedure were continued through this series using a cerium-zirconium, cerium scavenger process in place of the cerium-zirconium, cerium-zirconium precipitation in the decontamination cycles. Losses in these wastes showed improvement over the previous series, and decontamination appeared to be as efficient as before.

The roof samples are being changed so that the sample is taken directly in the sample test tubes, thus eliminating the sampler wells and transfer pipettes, both of which are possible sources of contamination from previous sampler operations. This change should assist in obtaining more consistent analyses.

The Hanford concentration of HNO_3 to dissolve the K_2CO_3 - KOH metathesized cake from the crossover step was used on runs 200-201 and apparently was adequate to affect solution of the lanthanum hydroxide, although considerable manganese dioxide was left undissolved.

DEPARTMENT OF ENERGY DECLASSIFICATION REVIEW	
1st Review - Date: 4-10-97	Determination [Circle Number(s)]
Authority: ADC ADD	1. Classification Retained
Name: [illegible]	2. Classification Changed To:
2nd Review - Date: 4/11/97	3. Contains No DOE Classified Information
Authority: ADD	4. Coordinate With:
Name: [illegible]	5. Classification Cancelled
	6. Classified Information Bracketted
	7. Other (Specify):

W. Q. Smith

This document contains [illegible] the national defense of the [illegible] meaning of the Espionage [illegible] transmission or the [illegible] manner to an unauthorized [illegible] law.

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Individual Run Summaries

Run numbers	194	195	196	197	198	199	200	201
Date started	7-28	7-31	8-1	8-2	8-3	8-3	8-4	8-5
Age of metal in days	57	60	61	64	61	64	66	61
Beta counts per gram UNH X 10^8	1.13	1.14	0.95	1.63	1.60	0.91	1.83	1.27
Gamma counts per gram UNH X 10^5	3.11	3.72	3.25	5.77	4.82	3.50	4.23	3.93

Yield

Run numbers	186-193	194-201	194	195	196	197	198	199	200	201
% Orig.prod.in extr.ppt. (24P)	93.9	92.8	91.2	98.4	87.5	89.8	99.2	92.6	91.7	91.8
% Orig.prod.after 1st by-prod.ppt. (51R)	97.8	102.8	110.5	107.0	**97.5	100.8	106.2	98.4	98.1	104.7
% Orig.prod.in 1st prod.ppt. (54P)	89.5	96.1	96.1	96.7	96.0	98.4	95.7	93.6	99.7	91.3
% Orig.prod.after 2nd by-prod.ppt. (41R)	101.3	92.4	96.1	97.8	90.2	90.4	92.5	90.4	90.9	---
% Orig.prod.in 2nd prod.ppt. (44P)	87.6	89.6	94.7	90.4	86.4	88.6	86.7	88.1	91.5	90.7
% Orig.*(Aver.) prod.in 2nd prod.ppt. (154P)	90.5	95.8	98.8	96.5	97.6	98.4	103.3	94.9	94.9	82.2
% Orig.*(Aver.) prod.after LaF_3 by-pr.ppt. (151R)	91.7	94.6	103.3	90.3	98.4	91.0	103.5	96.8	85.6	88.0
% Orig.*(Aver.) prod.in LaF_3 pr.slurry (151R-153W)	89.4	93.3	102.0	89.2	97.2	89.8	102.6	95.1	84.5	86.1
% Orig.*(Aver.) prod.in sol.of LaF_3 pr.slurry (D1P)	85.8	96.2	107.8		87.9		96.9		92.2**	
% Orig.*(Aver.) prod.in oxid.sol.1st cy. (D-1-R)	99.0	96.5	94.0		89.5		109.4		93.1**	
% Orig.prod.in prod.cake sol.1st cy. (CP)	90.4	94.7	94.7**							
% Orig.prod.in oxid.prod.sol.final cy. (FR)	89.9	82.6	82.6**							
% Orig.prod.in final cake slurry (FP)	81.9	88.2	88.2***							

* Basis here is 1/8th of total of orig. prod. for the 8 runs since all were collected in 45 tank and 1/8th removed for each run.

** 3.3% removed as samples from D1-S and D1-P included here and in material balances.

*** 1.0%, total in samples through this series, is included here and in material balances.

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Measured Losses

Run number	186-193	194-201	194	195	196	197	198	199	200	201
Extr. ppt. effluent (23MS)	2.9	2.4	1.1	3.3	4.1	1.5	2.5	2.9	1.9	2.2
1st By-product cake (71-BP-1	3.7	2.9	1.6	3.6	3.1	2.2	3.0	3.8	3.6	1.6
1st Prod.ppt. effluent (53WS)	2.3	1.0	1.6	1.1	1.1	1.1	1.0	0.7	0.7	0.8
2nd By-product cake (71-BP-2)	4.4	3.0	2.7	3.1	3.6	2.8	1.6	3.7	3.5	3.3
2nd Prod.ppt.effluent (43W)	2.1	0.8	1.6	0.5	0.5	0.9	0.3	0.6	1.4	0.4
3rd By-product cake (71-BP-3)	1.6	1.0	0.9	0.7	0.7	2.6	0.9	0.7	1.0	0.4
LaF_3 By-product cake (71-BP-4)	1.2	1.4	1.0	0.7	1.6	1.8	1.5	1.2	1.2	2.1
LaF_3 Prod. ppt. effluent (153W)	2.3	1.3	1.4	1.1	1.1	1.6	1.0	1.8	1.2	1.6
Total to waste in cells	20.4	13.7	12.0	14.2	16.4	14.1	11.7	15.4	14.6	12.5
Prod. in 1st metath. liquor (D3-CW)	0.7	0.9	1.1		0.7		0.8		0.9	
Prod. in 1st metath. wash (D3-WW)	0.6	0.5	0.4		0.4		0.5		0.7	
Prod. in 1st conc. cy. by-pr. cake (D4-BP)	0.8	0.8	1.2		0.7		1.0		0.2	
Prod. in 1st conc.cy.prod.ppt.waste (D3-W)	0.7	0.7	1.1		0.9		0.4		0.5	
Prod. in 2nd metath. liquors (CW-2 & WW-2)	0.9	1.0	1.5		1.1		0.6		0.9	
Prod. in final conc.cy.by-pr. cake (FBP)	0.0	0.0					3.7 (recycled)			
Prod. in final conc.cy.prod.ppt. waste (FW)	0.03	0.06					0.06			
Total to waste in Room D	3.7	4.0					4.0			

Material Balance

number	186-193	194-201	194	195	196	197	198	199	200	201
Through 1st by-pr.ppt. (51R)	104.6	108.0	113.3	113.8	105.4	104.6	111.6	105.2	103.8	108.5
Through 2nd by-pr.ppt. (41R)	114.7	101.8	103.1	108.9	102.8	98.0	100.5	101.6	100.6	---
Through 4th by-pr.ppt. (151R)	109.9	107.0	113.3	103.3	113.8	103.9	114.2	110.4	98.8	98.8
Through sol.of LaF_3 pr.ppt.slurry (D1-P)	107.3	111.4	122.4		104.3		111.9		107.3	
Through 1st conc.cycle (CP)	114.6	112.3				112.3				
Through final conc. cycle (FP)	105.9	105.9				105.9				

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Gamma dF

Run number	186-193	194-201	194	195	196	197	198	199	200	201
Through extr.ppt. (24P)	0.8	0.8	1.0	0.6	0.9	1.2	0.5	0.8	1.0	0.7
Through 1st by-pr.ppt. (51R)	2.4	2.3	2.3	2.5	2.4	2.5	2.1	2.4	2.0	2.3
Through 1st prod.ppt. (54P)	3.7	3.5	3.6	3.5	3.3	3.9	3.4	3.2	3.6	3.2
Through 2nd by-pr.ppt. (41R)	3.8	4.0	4.0	3.9	4.2	4.2	3.5	4.1	4.1	4.3
Through 2nd prod.ppt. (44P)	5.2	5.4	5.0	4.8	5.2	6.0	5.7	5.6	5.2	5.4
Composite of 2nd prod.ppt. (45P)	5.6	5.3								
Through LaF_3 Prod. slurry (D1-S)		6.7*	5.9		6.7		6.8		6.6	
Through sol. of LaF_3 prod.slurry (D1-P)	6.3*									
Through 1st conc.cycle (CP)	6.6	---	---							
Through final conc.cycle (FP)	8.0	7.7	7.7							

* dF's on first double runs were omitted in the average since the FBP's are recycled in these.

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M-1062

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8/7/44

To: W. C. Kay Department: Operating

From: W. Q. Smith Department: Operating

AEC RESEARCH AND DEVELOPMENT REPORT

In re: SUMMARY OF RUNS 186 TO 193 - 205 BLDG.

Run number	1 - 193	84 - 193	186 - 193
% Overall yield	85.3	88.8	81.9
% Original prod. to Room D	90.0	92.2	89.4
% Prod. to waste in Canyon	8.2	8.0	20.4
% Prod. to waste in Room D	3.9	3.6	3.7
% Material balance	97.3	100.3	105.9

This series of runs was a duplication of the Hanford separations procedure. Inactive fission products were added to the metal solution, two decontamination cycles were made using cerium-zirconium scavengers in the by-product precipitations and a lanthanum fluoride by-product precipitation was made in the crossover step. An average decontamination factor of 5.6 was obtained through the two cycles and waste losses were as normally expected with the use of scavengers. The product loss in the lanthanum fluoride by-product cake was moderate, averaging 1.6%.

Correction

In the previous report, the product content of the sample of 54P, Run 183, was not included in the results of the final cycle. Taking this into account, the overall yield becomes 89.0% and the material balance 107.7%. Likewise, the results for CP, FR and FP under yield and Cp and FP under percent material balance are increased 1.4%.

DEPARTMENT OF ENERGY DECLASSIFICATION REVIEW
1st Review - Date: 4-10-97
Authority: ☐ ADC ☒ ADD
Name: M Thacker
2nd Review - Date: 4/11/97
Authority: ADD
Name: George Dowling
1. Determination [Circle Number(s)]
2. Classification Retained
3. Classification Changed To:
4. Contains No DOE Classified Information
5. Coordinate With:
6. Classification Cancelled
7. Classified Information Bracketed
8. Other (Specify):

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CLASSIFICATION CHANGED TO CONFIDENTIAL
DATE 1-28-[illegible]
Chief, Declassification Branch

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Individual Run Summaries

Run numbers	186	187	188	189	190	191	192	193
Date started	7-17	7-17	7-18	7-19	7-19	7-21	7-21	7-22
Age of metal in days	54	54	53	50	50	52	52	52
Beta counts per gram UNH x 10^8	1.63	1.37	1.70	1.73	1.50	2.01	1.7	1.6
Gamma counts per gram UNH x 10^5	4.65	4.26	5.4	4.53	4.19	7.2	5.87	5.3

Yield

Run numbers	170-177	178-185	186-193	186	187	188	189	190	191	192	193
% Orig. prod. in Extr. ppt. (24P)	93.1	97.2	93.9	92.4	92.8	95.9	87.0	92.6	101.7	90.4	93.[illegible]
% Orig. prod. after 1st by-pr. ppt. (51R)	99.9	98.2	97.8	99.2	99.6	98.7	101.3	93.7	100	96.5	92.[illegible]
% Orig. prod. in 1st pr. ppt. (54P)	88.7	94.9	89.5	97.2**	93.8	90.0	84.7	89.1	96.2	74.5	91.[illegible]
% Orig. prod. after 2nd by-pr. ppt. (41R)	92.2	93.4	101.3	161.1	93.5	93.3	76.0	107.1	100.8	96.4	86.0
% Orig. prod. in 2nd pr. ppt. (44P)			87.6	96.1	95.0	87.0	79.0	91.8	87.9	87.0	79.[illegible]
% Orig.*(Aver.) prod. in 2nd pr. ppt (154P)			90.5	87.8	84.6	88.4	89.5	86.8	89.9	87.8	109.0
% Orig.*(Aver.) prod. after LaF_3 by-pr. ppt. (151R)			91.7	87.5	84.3	87.9	91.8	84.7	85.4	112.7	98.[illegible]
% Orig.*(Aver.) prod. in LaF_3 pr. slurry (151R-153W)			89.4	85.9	83.2	86.5	81.8	83.9	87.3	110.7	90.[illegible]
% Orig.*(aver.) Prod. in sol. of LaF_3 pr. slurry (DIP)	92.7	93.0	85.8	100.8		76.1		71.9		93.8	
% Orig.*(Aver.) prod. in oxid. sol. 1st cy. (D-1-R)	94.8	99.8	99.0	108.5		85.4		77.4		124.5***	
% Orig. prod. in prod. cake sol. 1st cy. (CP)	94.0	97.9	90.4				90.4				
% Orig. prod. in oxid. prod. sol. final cy. (FR)	91.4	87.2	89.9				89.9				
% Orig. prod. in final cake slurry (FP)	93.7	89.0	81.9				81.9				

*Basis here is 1/8th of total of orig. prod. for the 8 runs since all were collected in 45 tank and 1/8th removed for each run.

**33.4% removed as sample from 54P is included here and in all subsequent figures affected by this sample.

***2.1% removed as sample from D-1-S is included here and in all subsequent figures affected by this sample.

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Measured Losses

Run number	170-177	178-185	186-193	186	187	188	189	190	191	192	193
Extr. ppt. effluent (23WS)	1.7	4.0	2.9	2.8	4.5	3.7	3.2	3.8	1.9	2.3	2.1
1st By-product cake (71BP-1)	3.7	3.6	3.7	3.3	3.9	3.6	3.4	4.9	4.1	2.7	3.5
1st Prod. ppt. effluent (53WS)	2.0	3.0	2.3	2.4	1.6	3.2	3.1	2.4	1.7	1.7	2.1
2nd By-product cake (71-BP-2)	2.0	2.4	4.4	2.6	4.2	4.8	3.5	4.3	5.8	4.3	5.6
2nd Prod. ppt. effluent (43W)	2.5	2.1	2.1	1.8	1.9	0.7	1.0	3.1	2.6	4.9	0.3
3rd By-product cake (71-BP-3)			1.6	1.3	1.2	.2	1.1	1.2	3.3	1.2	1.4
LaF_3 By-product cake (71-BP-4)			1.2	1.4	0.8	1.4	1.0	1.2	2.0	0.9	1.2
LaF_3 Prod. ppt. effluent (153W)			2.3	1.7	1.1	1.6	7.0	2.0	1.1	1.0	1.7
Total to waste in cells	11.5	15.1	20.4	16.9	16.5	20.9	22.5	18.1	17.9	21.8	18.3
Prod. in 1st metath. liquor (D3-CW)	0.5	0.7	0.7	0.7		0.7		0.6		0.9	
Prod. in 1st metath. wash (D3-WW)	0.3	0.3	0.6	0.5		0.6		0.7		0.6	
Prod. in 1st conc. cy. by-pr. cake (D4-BP)	0.5	0.7	0.8	2.3		0.5		0.2		0.4	
Prod. in 1st conc. cy. prod. ppt. waste (D3-W)	0.9	0.8	0.7	1.4		0.4		0.4		0.5	
Prod. in 2nd metath. liquors (CW-2 & WW-2)	1.1	1.0	0.9	1.3		0.7		0.6		1.0	
Prod. in final conc. cy. by-pr. cake (FBP)	0	0	0	5.7				recycled			
Prod. in final conc. cy. prod. ppt. waste (FW)	0.1	0.01	0.03	0.03							
Total to waste in Room D	3.3	3.8	3.7	3.7							

Percent Material Balance

Run number	170-177	178-185	186-193	186	187	188	189	190	191	192	193
Through 1st by-pr. ppt. (51R)	105.1	106.8	104.6	104.8	108.1	106.0	107.8	102.3	106.0	101.4	97.6
Through 2nd by-pr. ppt. (41R)	101.4	106.3	114.7	172.0	107.5	108.6	89.1	120.3	114.3	107.1	99.5
Through 4th by-pr. ppt. (151R)			109.9	102.7	99.7	107.3	107.3	100.7	112.2	132.5	115.0
Through sol. of LaF_3 pr. ppt. slurry (D1-)	104.4	109.2	107.3	118.7		99.1		96.1		115.4	
Through 1st conc. cycle (CP)	97.5	116.7	114.6	114.6							
Through final conc. cycle (FP)	108.7	107.7	105.9	105.9							

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	Gamma dF								
Run number	185–193	186	187	188	189	190	191	192	193
Through extr.ppt. (24P)		0.8	0.8	0.9	1.0	0.6	0.9	0.9	0.7
Through 1st by-pr.ppt. (51R)		2.3	2.3	2.3	2.3	2.3	2.4	2.3	2.4
Through 1st prod.ppt. (54P)		3.3	3.6	3.3	3.9	3.8	4.1	3.8	3.8
Through 2nd by-pr.ppt. (41R)		3.6	3.4	3.6	4.1	3.3	4.2	4.7	3.8
Through 2nd prod.ppt. (44P)		4.5	4.0	5.4	4.4	5.6	5.9	5.9	5.9
Composite of 2nd prod.ppt. (45P)	5.6								
(154P)		5.2	5.3	5.6					
Through 3rd by-pr.ppt. (141-OP)		5.0		5.9	5.7	6.3	6.0	5.6	3.4
Through 4th by-pr.ppt. (151R)		5.0	5.5	5.7	5.5		5.1	5.3	7.9
Through sol. of LaF_3 prod. slurry (D1-P)		4.3		6.3		6.3		6.4	
Through 1st conc. cycle (CP)					6.6				
Through final conc. cycle (FP)					8.0				

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DUPLICATION OF HANFORD SEPARATIONS PROCEDURE

This series of runs was made to test the separations procedure to be used at Hanford. In order to do this in Clinton equipment, it was necessary to collect the product cake solutions of all eight runs after the second decontamination cycle in a tank installed in Cell 4 especially for this purpose (tank No. 45). After the eight runs were collected at this point, Cells 4, 5 and 6 were given a HNO_3 flush, and Cell 4 a $KMnO_4$ flush in addition. Solution was taken from the collection of product cake solutions to make up each run for the crossover step, 1/8th of the total weight being used.

Hanford concentrations of inactive fission products were added to the metal solution and a two cycle cerium-zirconium double precipitation scavenger procedure was used for decontamination. For the crossover step, a regular $BiPO_4$ by-product precipitation was made in Cell 6, except that the HNO_3 concentration was made to 1¼N instead of 1N to allow for dilution caused by numerous jettings and by making a LaF_3 by-product precipitation. The effluent from the $BiPO_4$ by-product cake was reoxidized in 41 using 0.01M $KMnO_4$ at 75° for one-half hour followed by a by-product LaF_3 precipitation (preformed) at 35° in 0.2N HF with 125 mg La per liter, centrifuging, reprecipitating and centrifuging. The effluent was then reduced in 51 at 50-35° with 0.06N oxalic acid for one hour, followed by product precipitation in 0.5NHF with 50 mg La per liter, centrifuging, reprecipitating and centrifuging.

Decontamination through the first two cycles, based on the results of a sample of the collection of eight runs in 45 was 5.6. Waste losses were as normally expected with the use of scavengers. LaF_3 by-product cake losses were moderate, averaging 1.6% and the LaF_3 product effluent waste losses were normal.

Because of the low activity of the solution of LaF_3 product slurry (D-1-P), some difficulty was experienced in obtaining the decontamination factors on these by the regular procedure. Therefore, on runs 190-191 and 192-193, a larger sample of the LaF_3 product slurry (D-1-S) was run. These results compared quite favorably with the D-1-P dF's: 6.4 against 6.3 and 6.6 against 6.4 respectively.

W. Q. Smith

WQS:dp

[redacted]

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7. I. Perlman
8. C. D. Coryell
9. G. E. Boyd
10. M. F. Acken
11. J. B. Sutton
12. W. A. Dew
13. E. J. Murphy
14. E. J. Murphy
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3331

June 30, 1944

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Department: Operating

To: W. C. Kay

Department: Operating

From: W. Q. Smith

AEC RESEARCH AND DEVELOPMENT REPORT

In re: SUMMARY OF RUNS 146 TO 153 - 205 BLDG.

	1-153	84-153	146-153
Run number			
% Overall yield	82.1	86.5	93.8
% Original prod. to Room D	92.1	96.8	97.7
% Prod. to waste in canyon	7.0	5.6	5.5
% Prod. to waste in Room D	4.3	3.8	3.3
% Material balance	93.4	95.9	102.7

Oxidation difficulties are still being met in the concentration cycle, but low losses have been obtained by reoxidation with an increased amount of zirconium and dichromate. Studies are being made of reduction temperatures in Cell 4 using manganous nitrate as a catalytic agent along with the oxalic acid solution. The waste losses have increased only a small amount, if at all, due to these changes. Studies of .5N vs. 1N HF, and coformed vs. preformed lanthanum fluoride precipitations were made during the last series of runs. These indicated that .5N HF is satisfactory, and that a preformed precipitate (present procedure) is superior to a coformed precipitate.

The handling characteristics and operating procedure for the various scavenger precipitates under consideration were studied in a series of plant runs in Cells 5 and 6. These tests indicated fairly conclusively what the limitations are for the use of the scavengers, and it is believed that sufficient information is available for the proper handling of any of the scavengers now under consideration. The preferred operating procedure and a summary of the conclusions reached will be given by F. B. Vaughan in the semi-monthly report from the operating department.

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CLASSIFICATION CHANGED TO CONFIDENTIAL
DATE: 6-24-56
H. F. Carroll
Chief, Declassification Branch

DEPARTMENT OF ENERGY DECLASSIFICATION REVIEW

1st Review - Date: 4-10-97
Authority: ☐ADC ☒ADD
Name: M Theles
2nd Review - Date: 4/11/97
Authority: ADD
Name: Jerry Flaherty

Determination [Circle Number(s)]
1. Classification Retained
2. Classification Changed To:
3. Contains No DOE Classified Info
4. Coordinate With:
5. Classification Cancelled
6. Classified Information Bracketted
7. Other (Specify):

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Individual Run Summaries

Run number	146	147	148	149	150	151	152	153
Date started	6-14-44	6-15-44	6-16-44	6-17-44	6-18-44	6-19-44	6-20-44	6-21-44
Age of metal in days	45	46	47	36	37	38	38	39
Beta counts per gram UNH X 10^8	2.21	2.28	2.07	4.07	[illegible]	2.03	3.1	3.09
Gamma counts per gram UNH X 10^6	2.1	1.096	1.05	1.02	1.53	9.9X10^{-2}	1.6	1.313

Yield

Run number	84-153	146-153	146	147	148	149	150	151	152	153
% Orig. prod. in Cell 2 prod. cake sol. (24P)	86.7	93.1	85.2	82.4	98.0	102.3	103.6	93.4	90.6	89.5
% Orig. prod. in Cell 2 oxid. prod. sol. (31R)	96.8	103.9	100.7	109.5	108.1	108.5	109.9	101.3	95.4	100.1
% Orig. prod. in Cell 3 prod. cake sol. (34P)	86.3	91.5	89.7	78.3	88.1	90.2	108.1	92.1	95.1	88.1
% Orig. prod. in Cell 3 oxid. prod. sol. (41R)	97.8	99.0	87.4	90.7	104.5	109.1	109.7	96.2	103.0	90.0
% Orig. prod. in Cell 4 slurry (41R-43W)	96.8	97.7	85.5	89.2	103.7	107.6	108.6	95.5	102.4	88.8
% Orig. prod. in sol. of Cell 4 slurry (D-1-P)	85.1	91.5	94.4		119.2		78.2		72.8	
% Orig. prod. in oxid. sol. 1st cycle (D-1-R)	87.9	91.2	93.7		104.3		92.4		73.0	
% Orig. prod. in prod. cake sol. 1st cycle (CP)	89.9	86.0				86.0				
% Orig. prod. in oxid. prod. sol. final cy. (FR)	87.0	96.8				96.8				
% Orig. prod. in final cake slurry (FP)	86.5	93.8				93.8*				

*Includes 0.1% product taken in samples.

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MEASURED LOSSES
(Percentage of starting material)

Run number	84-153	146-153	146	147	148	149	150	151	152	153
Cell 2 waste effluent (23WS)	2.2	1.5	1.5	1.0	2.1	0.6	1.4	2.0	1.8	1.4
Cell 2 by-product cake (71BP-1)	0.7	0.9	0.9	0.7	0.9	1.7	1.1	0.9	0.5	0.4
Cell 3 waste effluent (33WS)	1.2	1.4	11	1.8	1.2	1.7	1.5	1.2	1.2	1.2
Cell 3 by-product cake (71BP-2)	0.5	0.6	0.4	0.6	1.7	0.4	0.5	0.4	0.5	0.5
Cell 4 waste effluent (43W)	1.0	1.2	1.9	1.5	0.8	1.5	1.0	0.6	1.0	1.2
Total to waste in cells	5.6	5.5	5.8	5.6	6.8	5.9	5.5	5.2	5.0	4.7
Prod. in 1st metath. liquor (D3=CW)	0.5	0.6	0.5		0.7		0.5		0.6	
Prod. in 1st metath. wash (D3=WW)	0.5	0.3	0.4		0.2		0.4		0.3	
Prod. in 1st conc. cycle by-prod. cake (D4=BP)	0.9	0.7	0.7		1.1		0.9		0.4	
Prod. in 1st conc. cycle prod. ppt. waste (D3=W)	0.5	0.4	0.4		0.6		0.4		0.3	
Prod. in 2nd metath. liquors (CW-2 & WW-2)	1.1	1.3	1.4		1.1		1.3		1.2	
Prod. in final conc. cycle by-prod. cake (FBP)	0		4.2 (Recycled)							
Prod. in final conc. cycle prod. ppt. waste (FW)	0.3	0.05	0.05							
Total to waste in Room D	3.8	3.3	3.3							

Percent Material Balance

Run number	84-153	146-153	146	147	148	149	150	151	152	153
Through Cell 2 (31R)	99.6	106.3	103.1	111.2	111.2	110.8	112.5	104.3	97.8	102.0
Through Cell 3 (41R)	102.4	103.3	91.2	94.8	110.4	113.5	114.2	100.7	107.5	93.5
Through solution of Cell 4 slurry (D-1-P)	92.0	79.9	100.9		126.3		98.1		78.5	
Through first concentration cycle (CP)	92.6	89.3	89.3							
Through final concentration cycle (FP)	95.9	102.7	102.7							

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Gamma dF

Run Number	146	147	148	149	150	151	152	153
Through Cell 2 prod.ppt. (24P)	1.0	0.9	0.8	0.7	1.0	0.8	0.9	1.0
Through Cell 2 by-prod.ppt. (31R)	2.1	2.1	2.3	1.9	2.2	2.1	1.03	2.04
Through Cell 3 prod.ppt. (34P)	3.5	3.3	3.3	3.3	3.4	3.1	3.4	3.3
Through Cell 4 by-prod.ppt. (41R)	2.5	2.8	3.5	3.1	3.1	3.8	3.6	4.0
Through solution of Cell 4 slurry(D-1-P)	4.6		4.7		4.6		4.3	
Through first conc.cycle (CP)	6.2							
Through final conc.cycle (FP)	7.1							

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DISCUSSION

OXIDATION - FIRST CONCENTRATION CYCLE

As in the previous two series, difficulties were met in the oxidation of charges 146-147, 148-149, and 152-153 of this group. In order to obtain satisfactory oxidation, the zirconium and dichromate solutions were increased according to the following schedule:

Run Number	Zirconium used in normal oxid.	Dichromate used in normal oxid.	Additional Zirconium used	Additional Dichromate used	Unoxidized	By-product loss
146-147	25 lbs.	37 lbs.	25 lbs.	--	2.1%	0.7%
148-149	25 lbs.	37 lbs.	40 lbs.	4 lbs.	6.7%	1.1%
152-153	25 lbs.	37 lbs.	--	5 lbs.	0.6%	0.4%

Usually the losses in the by-product cake were less than the oxidation check indicated was unoxidized.

A fine precipitate was observed in the oxidized solution of the Cell 4 slurry during the last series of runs. A sample of this precipitate was analyzed by the Plant Assistance Group and found to be zirconium phosphate. As yet it has not been determined whether or not the precipitate is causing interference in the oxidation.

As a result of these studies, the Plant Assistance Group has recommended that the phosphoric acid concentration during the product precipitation in Cell 3 be cut to .02M, and that the product cake in Cell 4 be washed with 250 lbs. of 6% HNO_3 in order to decrease the quantity of phosphoric acid entering Room D.

REDUCTION WITH MANGANOUS NITRATE - CELL 4

Beginning with run 146, the normal reduction using 600 lbs. of 10% oxalic acid solution was carried out in the presence of 10 lbs. of 50% manganous nitrate solution used as a catalyzing agent. The reduction temperature on runs 146-148 was 35°C in contrast to the normal 75°C. On these runs there was a slight increase in product loss in the waste (average 1.5% as compared to a normal of about 0.8%). On runs 149-153 the reduction temperature was increased to 50° and the waste losses dropped slightly to an average of 1.1%.

PRECIPITATION STUDIES - CELL 4

The results of the variables introduced in the lanthanum fluoride product precipitations of the preceding series were not made in the last report. These results are tabulated on the following page.

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Run Nos.	HF Normality	Type of Strike	Product Loss in Waste
138-139	1N	Preformed (HF added first)	0.2% - 0.4%*
140-141	0.5N	Preformed (HF added first)	0.8% - 0.6%
142-143	1N	Coformed (La added first)	2.4% - 2.2%
144-145	0.5N	Coformed (La added first)	1.0% - 1.9%

*These results are lower than the average 0.8% for 43W.

It is indicated from the above results that the present plant practice of preformed lanthanum fluoride precipitations results in lower waste losses than a coformed precipitation. The use of 0.5N HF does not appear to have increased the waste losses.

W. Q. Smith

W. Q. Smith

WQS:dp

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3331

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3.	F. B. Vaughan	13.	E. J. Murphy	23.	M. D. Whitaker
4.	O. H. Greager	14.	E. J. Murphy	24.	M. D. Whitaker
5.	W. C. Johnson	15.	W. Q. Smith	25.	M. D. Whitaker
6.	R. L. Dean	16.	W. Q. Smith	26.	C. M. Cooper
7.	I. Perlman	17.	W. E. Kirst	27.	T. Hogness
8.	C. D. Coryell	18.	W. E. Kirst	28.	T. Hogness
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June 23, 1944

To: Department: Operating

From: W. Q. Smith Department: Operating

899

In re: SUMMARY OF RUNS 138 TO 145 - 205 BLDG.

Run number	1-145	84-145	138-145
% Overall yield	81.0	85.4	94.5
% Original prod. to Room D	91.6	96.6	96.4
% Prod. to waste in canyon	7.1	5.6	7.8
% Prod. to waste in Room D	4.4	3.9	3.6
% Material Balance	92.6	94.9	106.0

The final by-product cake and final waste from this series will be recycled in the next series. Adjustment for the increase of the recycle of this series over the last series will increase the yield and material balance by 0.12%.

Studies of the lowered H_3PO_4 concentrations and slow strike-precipitations, during the extraction step, were continued in this series. Runs 138-141, precipitated with 0.4 mol H_3PO_4, showed a slight increase in the waste losses. The waste losses on runs 142-145 were higher than on the previous four runs when 0.3 M H_3PO_4 concentration was used.

Through an operating error, 13.4% of the product was lost with the metal waste of run 142. The normal metal waste loss of this run was 2.5%. Total waste loss was 15.9%.

On two of the double charges of this series, further difficulties were encountered in the oxidation step of the first concentration cycle. It was noted that during oxidation, the oxidized solution contained a fine precipitate. The tank was cleaned out thoroughly, but on runs 138-139 and 142-143 it was necessary to use additional amounts of zirconium for complete oxidation.

A study continues of multiple samples taken from the dissolver and Cell 2 precipitator using air instead of steam. Considerably better agreement is obtained between samples taken from the same solution in the same vessel and between 11 M and 21 MR samples. A more detailed report will be made in the near future.

AEC RESEARCH AND DEVELOPMENT REPORT

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Individual Run Summaries

Run number	138	139	140	141	142	143	144	145
Date started	6-5	6-6	6-7	6-8	6-9	6-11	6-12	6-12
Age metal in days	36	40	38	39	40	42	43	43
Beta counts per gram UNH X 10^8	2.71	2.37	2.15	2.25	2.00	2.1	1.94	2.24
Gamma counts per gram UNH X 10^6	1.56	1.27	1.41	1.23	1.08	.91	.84	1.46

Yield

Run number	84-145	138-145	138	139	140	141	142	143	144	145
% Orig.prod.in Cell 2 prod.cake sol.(24P)	85.8	91.8	96.7	84.8	88.3	86.5	95.6	97.9	95.6	92.5
% Orig.prod.in Cell 2 oxid.prod.sol.(31R)	95.7	102.2	105.0	84.3	105.4	112.0	92.8	104.4	107.0	107.9
% Orig.prod.in Cell 3 prod.cake sol.(34P)	85.5	89.5	92.4	75.6	92.8	94.5	78.0	94.3	93.5	97.8
% Orig.prod.in Cell 3 oxid.prod.sol.(41R)	97.6	97.6	103.2	74.2	106.3	108.6	89.0	100.1	105.3	95.9
% Orig.prod.in Cell 4 slurry (41R-43W)	96.6	96.4	102.9	73.9	105.4	108.0	86.4	98.7	104.3	94.0
% Orig.prod.in sol. of Cell 4 slurry (D1-P)	84.5	81.3	82.7		71.4		76.5		94.8	
% Orig.prod.in oxid.sol.1st cycle(D1-R)	87.5	87.9	79.2		109.9		67.7		95.4	
% Orig.prod.in prod.cake sol.1st cycle(CP)	90.5	98.4	98.4							
% Orig.prod.in oxid.prod.sol.final cy.(FR)	85.5	92.9	92.9							
% Orig.prod.in final cake slurry (FP)	85.4	94.5	94.5*							

*Includes .4% product taken in samples.

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MEASURED LOSSES
(Percentage of starting material)

Run number	84-145	138-145	138	139	140	141	142	143	144	145
Cell 2 waste effluent (23MS)	2.3	3.5	1.8	1.2	1.2	1.9	15.9	2.5	2.0	2.5
Cell 2 by-product cake (73BP-1)	0.7	0.6	0.5	0.4	0.7	0.6	0.5	0.6	0.9	0.8
Cell 3 waste effluent (33WS)	1.2	1.6	1.1	2.4	2.1	1.2	2.1	1.7	0.5	1.5
Cell 3 by-product cake (33BP-2)	0.5	1.0	0.6	3.9	0.5	0.2	0.3	0.3	0.5	0.6
Cell 4 waste effluent (43W)	1.0	1.1	0.2	0.4	0.8	0.6	2.4	2.2	1.0	1.9
Total to waste in cells	5.6	7.8	3.8	8.2	5.4	4.5	21.2	7.5	5.4	7.3
Prod. in 1st metath. liquor (D3-CW)	0.5	0.8	0.5		1.8		0.3		0.5	
Prod. in 1st metath. wash (D3-WW)	0.5	0.6	0.4		1.4		0.2		0.3	
Prod. in 1st conc. cycle by-prod. cake (D4-BP)	0.9	0.7	0.9		0.3		0.9		0.6	
Prod. in 1st conc. cycle prod. ppt. waste (D3-W)	0.5	0.6	0.9		0.4		0.5		0.3	
Prod. in 2nd metath. liquors (CW-2 & WW-2)	1.1	1.0	1.0		1.2		0.9		0.9	
Prod. in final conc. cycle by-prod. cake (FBP)	0	0	3.9 (Recycled)							
Prod. in final conc. cycle prod. ppt. waste (FW)	0.5	0.6	0.6 (Recycled)							
Total to waste in Room D	3.9	3.6	3.6							

Percent Material Balance

Run number	84-145	138-145	138	139	140	141	142	143	144	145
Through Cell 2 (31R)	98.7	106.3	107.8	85.8	107.4	114.8	109.2	107.6	111.2	111.5
Through Cell 3 (41R)	102.2	104.3	106.6	82.2	110.8	112.5	107.6	106.3	109.7	101.4
Through solution of cell 4 slurry (D-1-P)	91.1	99.5	89.6		82.2		91.0		101.8	
Through first concentration cycle (CP)	93.1	110.1	110.1							
Through final concentration cycle (FP)	94.9	106.0	106.0							

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Gamma dF

Run Number	138	139	140	141	142	143	144	145
Through Cell 2 prod. ppt. (24P)	0.9	0.9	0.9	0.9	0.8	0.7	0.7	1.0
Through Cell 2 by-prod. ppt. (31R)	2.2	1.9	2.2	2.2	2.2	2.2	2.2	2.6
Through Cell 3 prod. ppt. (34P)	3.6	2.8	3.3	3.0	3.0	3.2	3.4	3.6
Through Cell 4 by-prod. ppt. (41R)	3.8	4.0	3.8	3.9	3.9	3.4	3.5	--
Through solution of Cell 4 slurry (D-1-P)	4.8		4.4		4.6		4.8	
Through first conc. cycle (CP)				6.5				
Through final conc. cycle (FP)				7.5				

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ITGO VIII 52

DISCUSSION

Extraction Precipitation Studies, Cell 2

The studies of lowered concentrations of H_3PO_4 and slow product precipitation, in Cell 2, were continued from the previous series of runs. In the first four runs of this series, 138-141, the precipitations were made over a period of 120 minutes, as in the last series, but with a concentration of 0.4 mol. The results were slightly higher than normal (1.5% product in metal wastes).

On runs 142-145, the length of time of precipitation remained the same, but the H_3PO_4 molarity was reduced to 0.3. The waste losses at this concentration increased to an average product content of 2.5%.

As a result of these studies, a concentration of 0.6 mol H_3PO_4 and a precipitation time of two hours has been tentatively adopted for subsequent runs, in agreement with the proposed Hanford flowsheet. The decrease from 0.8 mol to 0.6 mol H_3PO_4 will result in lowered waste volumes. The period of digestion will be shortened from the normal two hours to the one hour period used during the trial runs.

High Product Loss in Metal Wastes, Run 142

Through an operating error, the product loss in the metal waste of this run was excessive. 13.4% of the product effluent from the by-product centrifugation was jetted on top of the waste which contained 2.5% product. This resulted in a waste loss of 15.9% product.

Oxidation, First Concentration Cycle

As in the previous series, difficulty was met in the oxidation of runs 138-139 and 142-143 in the first concentration cycle. It was necessary to use 50 lbs of 1.5 zirconium solution on both charges before oxidation was complete. During recent tuns, a fine precipitate has been observed in the oxidized solutions. An effort is being made to determine the composition of this precipitate with the assistance of the Research Group.

W. Q. SMITH

By P. B. Orr

PBO/mr

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July 10, 1944

To: W. C. Kay Department: Operating

From: W. Q. Smith Department: Operating

In re: SUMMARY OF RUNS 154-161 - 205 BUILDING

SEC RESEARCH AND DEVELOPMENT REPORT

Run number	1-161	84-161	154-161
% Overall yield	82.6	86.7	88.2
% Orig. prod. to Room D	90.4	93.8	71.6
% Prod. to waste in canyon	6.9	5.6	5.3
% Prod. to waste in Room D	4.2	3.7	2.8
% Material balance	93.6	95.9	96.3

A correction should be made in the report of the last series. On page six, in the table, the HF normality for runs 142-143 should read 0.5N and runs 144-145 should be 1N.

Beginning with run 159, Cells 5 and 6 were put into use along with the other cells in a straight line operation. One $BiPO_4$ precipitation is done in each cell, as follows: A product precipitation in Cell 2, a by-product in Cell 3, a product in Cell 5, a by-product in Cell 6 and a lanthanum fluoride product in Cell 4.

Starting with run 154, the HF concentration in the lanthanum fluoride product precipitation was reduced from 1N to 0.5N. The waste losses remained normal.

Oxidation difficulties were again encountered in this series of runs. The phosphoric acid concentration of the Cell 3 by-product precipitation was reduced to 0.2M from .1M in an attempt to reduce phosphoric acid being carried into Room D. This resulted in an increase in concentration of bismuth in the effluent, such that bismuth oxalate precipitated in subsequent reductions in Cell 4. Samples of the reduced solution showed low product content and it was necessary to raise the phosphoric acid back to the original .1M in Cell 5. Overall product yield was affected little or none. Washing of the Cell 4 product cake will be substituted for the reduction of Cell 5 by-product phosphoric acid.

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Individual Run Summaries

Run Number	154	155	156	157	158	159	160	161
Date started	6-22	6-23	6-24	6-25	6-26	6-27	6-28	6-30
Age of Metal in days	40	37	38	39	40	41	42	60
Beta counts per gram UNH x 10^8	2.49	2.61	1.72	1.62	2.82	1.68	1.72	2.52
Gamma counts per gram UNH x 10^5	1.63 x 10^6	9.84	6.74	6.48	7.04	7.65	7.52	7.56

Yield

Run Number	84-161	154-161	154	155	156	157	158	159	160	161
%Orig.prod.in cell 2 prod.cake sol.(24P)	87.4	92.6	86.8	89.8	90.7	93.8	101.0	82.8	87.2	102.0
%Orig.prod.in cell 2 oxid.prod.sol.(31R)	96.7	96.2	89.8	97.0	101.2	99.7	99.7	90.3	105.0	93.5
%Orig.prod.in Cell 3 prod.cake sol.(34P)	86.5	88.2	89.1	77.8	94.3	88.5	-	79.6	90.5	96.4
%Orig.prod.in Cell 3 oxid.prod.sol.(41R)	94.8	72.8	91.0	102.0	97.2	104.0	99.2	12.7	46.7	39.4
%Orig.prod.in Cell 4 slurry (41R-43W)	93.8	71.6	89.8	100.3	96.9	102.8	98.0	11.2	46.0	37.4
%Orig.prod.in sol.of Cell 4 slurry (D-1-P)	86.4	94.0		76.0						
%Orig.prod.in oxid.sol.1st cycle (D-1-R)	88.0	88.0		88.7	129.0		52.7		118.8	
%Orig.prod.in prod.cake sol.1st cycle (CP)	89.1	83.4			105.1		51.2		106.5	
%Orig.prod.in oxid.prod.sol.final cy.(FR)	86.7	85.4				83.4				
%Orig.prod.in final cake slurry (FP)	86.7	88.2				85.4				
						88.2*				

*Includes 0.1% product removed in samples

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MEASURED LOSSES
(Percent of Starting Material)

Run Number	84-161	154-161	154	155	156	157	158	159	160	161
Cell 2 waste effluent (23WS)	2.1	1.3	2.2	0.7	1.8	1.9	0.7	1.5	1.7	0.6
Cell 3 by-product cake (71-BP-1)	0.7	0.7	0.6	0.5	0.6	1.2	1.1	0.6	0.6	0.5
Cell 3 waste effluent (33WS)	1.3	1.6	2.3	1.7	1.7	1.1	2.5	1.4	1.0	1.0
Cell 3 by-product cake (71-BP-2)	0.5	0.6	0.6	0.5	0.6	0.4	0.5	0.4	1.7	0.2
Cell 4 waste effluent (43W)	1.0	1.2	1.1	1.8	0.3	1.3	1.1	1.5	1.4	1.1
Total waste in cells	5.6	5.3	6.8	5.2	5.1	5.8	5.9	5.2	6.3	3.3
Prod.inlet metath.liquor (D3-CW)	0.5	0.3	0.4	0.4		0.3		0.2		
Prod.in 1st metath.wash (D3-WW)	0.4	0.2	0.3	0.3		0.2		0.2		
Prod.in 1st conc.cycle by-prod.cake(D4-BP)	0.9	0.6	0.4	0.8		0.6		0.6		
Prod.in 1st conc.cy.prod.ppt.waste(D3-W)	0.5	0.4	0.3	0.6		0.2		0.5		
Prod.in 2nd metath.liquors (CW-2 & WW-2)	1.1	1.2	1.3	1.7		0.6		1.4		
Prod.in final conc.cycle by-Prod.cake(FBP)	0	0		4.1 (Recycled)						
Prod.in final conc.cycle prod.ppt.waste(FW	0	0.1		0.1						
Total to waste in Room D	9.3	8.1		8.1						

Percent Material Balance

Run Number	84-161	154-161	154	155	156	157	158	159	160	161
Through Cell 2 (31R)	99.5	98.2	92.6	98.4	103.8	102.8	101.6	90.0	107.3	92.4
Through Cell 3 (41R)	99.3	76.9	96.6	105.6	102.0	108.6	103.9	13.4	52.3	40.7
Through solution of Cell 4 slurry (D1-P)	92.9	99.9	82.6		135.1		58.8			123.6
Through first concentration cycle (CP)	92.4	91.4				91.4				
Through final concentration cycle (FP)	95.9	96.3				96.3				

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Run Number	Gamma dF							
	154	155	156	157	158	159	160	161
Through Cell 2 prod. ptt.(24P)	0.8	1.0	0.61	0.65	0.56	0.87	0.9	0.3
Through Cell 2 by-prod. ppt. (31R)	2.4	1.9	1.9	2.0	2.0	2.1	2.2	2.5
Through Cell 3 prod. ppt. (34P)	3.6	3.4	3.0	3.0	Sample	3.1	2.9	3.4
Through Cell 4 by-prod. ppt. (41R)	2.7	2.4	2.9	2.2	contam.	2.5	2.8	2.8
Through solution of Cell 4 slurry (D1-P)	4.5		4.3		4.4		3.8	
Through first conc. cycle (CP)				5.4				
Through final conc. cycle (FP)				7.9				

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DISCUSSION

Product Precipitation, Cell 4

The lanthanum fluoride product precipitation studies, conducted during runs 138 - 145, indicated that a performed precipitation (HF added prior to lanthanum) in a concentration of 0.5M HF resulted in an average product waste loss of 0.7% which was normal (0.8% average).

Beginning with run 154, therefore, the HF concentration was reduced from 1N to 0.5. The lowered concentration will reduce HF consumption and will possibly decrease corrosion in Cell 4.

Oxidation, First Concentration Cycle

Continued difficulty was met in the oxidation of the solution of the Cell 4 slurry on runs 154 - 155. It was found necessary to use an additional 5 lbs. of 10% dichromate solution and 25 lbs. of 1.5% zirconium solution, which still left 0.3% of the product in the unoxidized state.

On the recommendation of the Plant Assistance Group, the phosphoric acid concentration in the by product precipitation, Cell 5, was reduced from .1 Mol to .02 Mol on run 159, to decrease the amount of phosphoric acid entering Room D. Good oxidation results were obtained in the concentration cycle on subsequent runs. However, upon sampling the reduced solutions from tank 41 after the Molarity had been reduced in Cell 5, the analyses showed a low product content as shown in the following table.

Run Number	Percent Product
159	12.7*
160	46.7
161	39.4

*Initial active charge through Cells 5 & 6

After run 162, of the next series, the phosphoric acid concentration was restored to the original figure of .1 Mol. The product content of the reduced solution rose to normalcy.

It was found that the reduction of the phosphoric acid Molarity caused an increased bismuth concentration in the Cell 5 effluent, which, when the reduction was carried out in Cell 4, was thrown down as bismuth oxalate. Unrepresentative samples of this slurry were obtained and resulted in low analyses. However, there is little or no overall loss as shown by the 88.2% product content of the final concentration cycle.

A 6% Nitric acid wash of the product cake, Cell 4, will be begun in the next series, as suggested by the Plant Assistant Group to substitute for the lowered

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Molarity of the phosphoric acid in Cell 5 in an attempt to remove the precipitate from the concentration cycle oxidized solution.

W. J. Smith

By

WJS/ao

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M--1322

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October 21, 1944

To: Mr. J. P. Sinclair — Department: Production

From: W. Q. Smith — Department: Production

In re: Summary of Runs 227 - 235 Through Isolation

Run Number	1 - 235	227 - 235
% Yield	83.7	70.9*
% Waste	14.4	21.6
% Material Balance	98.1	92.5

*Corrected for loss in product content of recycle solutions on hand.

AEC RESEARCH AND DEVELOPMENT REPORT

SUMMARY

This nine-run series was processed according to the same procedure used on the previous series (Hanford procedure with recycling of isolation supernatants) with the exception that Ce-Zr; Ce-Zr scavengers were used in both decontamination cycles with no $(NH_4)_2SiF_6$. With the completion of these runs, sufficient plant-scale data have been obtained on the performance of scavengers and the complexing agent $(NH_4)_2SiF_6$, to lead to the conclusion that using $(NH_4)_2SiF_6$ with no scavengers in the decontamination cycles will reduce waste losses by a factor of five. Furthermore from plant data it appears that sufficient decontamination for Hanford level of operation can be obtained under these conditions. Comparison of the combinations tested in the plant is given in the discussion with data to support the above conclusions.

The flowsheet amount of $NaNO_2$ (0.1M) was used for pre-extraction treatment in this series. Extraction waste losses averaged 1.9%, slightly lower than results obtained previously with this concentration.

Decomposition of H_2O_2 in isolation recycles with $NaNO_2$ has given no trouble. The amount of $NaNO_2$ necessary has increased to about 80% of the stoichiometric amount and the temperature rise has been 10 - 12° C. over an addition period of about one hour.

The first peroxide precipitation solubilities in the series were 119, 83 and 89 mg per liter. Table on page 3 shows the variables which may affect these solubilities. The final peroxide precipitation solubilities were 16 and 53 mg per liter. For the purpose of this and subsequent reports the solubility is obtained by dividing the product content of the decanted supernatant plus washes by the volume of solution in liters at the time the precipitation is made.

See over-sheet for classification

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Individual Run Summaries

Run Numbers	227	228	229	230	231	232	233	234	235
Date Started	9-28	9-28	9-29	9-30	10-1	10-1	10-3	10-3	10-4
Age of Metal in days	62	62	60	61	61	58	60	60	61
Gamma counts per gram UNH x 10^6	1.37	1.44	1.42	1.72	1.23	1.16	1.15	1.29	0.91

Yield

Run Number	227-235	227	228	229	230	231	232	233	234	235
% Orig. prod. in extr. ppt. 24P	87.1	90.1	95.8	86.8	87.3	87.5	88.5	94.5	69.6	81.7
% Orig. prod. after 1st by-prod. ppt. 51R	100.2	98.9	104.1	116.1	95.7	101.2	98.6	102.8	92.2	93.7
% Orig. prod. in 1st prod. ppt. 54P	88.8	88.0	94.7	83.1	87.6	88.3	--	90.0	87.9	91.0
% Orig. prod. after 2nd by-prod. ppt. 41R	89.6	88.9	92.5	91.5	86.9	95.1	86.0	96.5	85.9	84.0
% Orig. prod. in 2nd prod. ppt. 44P	88.8	84.3	88.5	81.1	102.0	86.2	81.4	85.6	105.4	81.3
% Orig. (Aver.) prod. in 2nd prod. ppt. 154P	85.5	89.0	84.3	84.8	81.3	91.3	100.2	80.0	85.7	73.1
Basis after return of recycle = D8P + Ave. 11M*	200.0	100	271.7	271.7	100	218.8	218.8	100	257.6	257.6
% Prod. after LaF_3 by-prod., recycle basis: 151R	88.1	93.0	--	--	69.0	94.0	86.0	86.3	88.9	85.0
% Prod. in sol. of LaF_3 pr. slurry D1P	89.2	86.0			94.8			87.9		
% Orig. prod. in final peroxide ppt. 1FP**	70.9									

Material Balance

Run Number	227-235	227	228	229	230	231	232	233	234	235
Through 1st by-prod. ppt. 51R	105.5	105.1	108.7	121.6	101.6	106.7	104.5	107.6	97.4	97.4
Through 2nd by-prod. ppt. 41R	101.3	102.8	104.3	103.2	98.6	106.7	98.9	107.0	96.4	92.4
Through decont. cycles 44P	101.4	100.0	102.7	93.5	114.1	98.7	95.0	98.7	112.5	90.7
Through final peroxide ppt. 1FP**	92.5									

*151R and D1P for runs containing recycled product are based on the average value of the 11M plus the amount of recycled product added.

**Corrected for gain in product content of recycle solutions on hand and based on total 11M.

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Measured Losses

Run Numbers	227-235	227	228	229	230	231	232	233	234	235
Extr. ppt. effluent 23WS	1.9	3.2	1.2	1.3	1.8	1.5	2.4	1.2	2.5	1.5
1st by-product cake 71-BP-1	3.4	3.1	3.3	4.2	4.1	4.0	3.6	3.6	2.7	2.2
1st prod. ppt. effluent 53WS	2.3	4.0	2.1	2.1	1.8	2.2	3.0	1.7	1.7	1.0
2nd by-prod. cake 71-BP-2	4.0	3.6	5.7	4.0	4.0	3.8	3.9	4.0	3.7	3.8
2nd prod. ppt. effluent 43WS	1.0	1.9	1.2	0.8	0.5	0.9	0.6	0.7	0.9	1.0
3rd by-product cake 71-BP-3	0.9	1.8	1.1	0.8	0.9	0.7	0.9	0.8	0.5	0.4
LaF_3 by-product cake 71-BP-4*	1.3	2.4	0.9	1.0	1.1	1.7	1.7	1.5	1.2	1.0
LaF_3 product ppt. effluent 153WS*	1.5	2.3	0.4	1.3	0.4	1.1	2.7	0.5	2.4	1.9
1st metathesis liquor D3CW*	0.7		1.0			0.9			0.2	
1st metathesis wash D3WW*	0.4		0.5			0.5			0.3	
Total 205 Building (Basis 11M)	21.4									

Gamma dR's

Run Numbers	227-235	227	228	229	230	231	232	233	234	235
Through extr ppt. 24P	0.9	0.8	1.0	1.7	0.9	0.8	0.7	0.9	0.9	0.8
Through 1st by-pr. ppt. 51R	2.4	2.5	2.4	2.5	2.5	2.4	2.4	2.4	2.4	2.4
Through 1st prod. ppt. 54P	3.7	3.4	3.5	3.5	3.8	4.1	3.5	3.5	3.8	3.8
Through 2nd by-prod. ppt. 41R	4.4	4.2	4.7	3.9	4.6	4.4	4.7	4.3	4.2	---
Through 2nd prod. ppt. 44P	5.8	5.5	5.7	5.9	5.9	6.5	5.7	5.3	5.7	5.9
Through 2nd prod. ppt. (composite) 45P	5.8									
Through 2nd prod. ppt. D1-S	7.4		7.2			7.4			7.5	

Factors Affecting Peroxide Solubility

Run Numbers	Fe (M)	Zr (M)	Acidity (N)	H_2O_2 (%)	Settling Time	Solubility** mg/liter
227-228-229	0.0067	0.00006	0.8	9.5	9 hrs.	119
230-231-232	0.0065	Nil	0.7	8.6	20 hrs.	83
233-234-235	0.0063	Nil	0.8	8.3	15 hrs.	89

*BP-4, 153WS, D3CW and D3WW for runs contianing recycled product are based on the average value of the 11M plus the amount of recycled product added.

**Value after decanting and washing.

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With the completion of this series of runs the final demonstration of the recommended Hanford process has been made. Results confirm that the recommended process will give adequate decontamination. Waste losses, however, although they may not be considered excessive, have averaged five times as great with the Ce-Zr; Ce-Zr scavenger as with ammonium fluosilicate (no scavenger) in the cycles. It has been predicted that at Hanford concentrations the losses will be somewhat less but there seems to be no reason for doubting that they could not be lowered by the same factor of five by omitting scavengers. At Clinton the yield loss for two cycles is 9.4% greater with Ce-Zr; Ce-Zr, than with $(NH_4)_2SiF_6$ alone in both cycles. (Waste losses were 5.9% per cycle with Ce-Zr; Ce-Zr scavengers, average of 64 results, and 1.2% per cycle with $(NH_4)_2SiF_6$, average of 16 results. See table below.)

Run Nos.	Scavengers First Cycle	Scavengers Second Cycle	dF Thru 2 Cycles 45P	dF Thru Cross-Over D1-S	To Waste 1st Cycle	% Waste 2nd Cycle
194-201	Ce-Zr; Ce	Ce-Zr; Ce	5.3	6.7	3.9	3.8
202-209	Ce-Zr; Ce	$(NH_4)_2SiF_6$	5.3	7.1	4.0	.9
218-226	(Ce-Zr; Ce-Zr $(NH_4)_2SiF_6$)	$(NH_4)_2SiF_6$	5.7	7.4	4.5	1.5
186-193	Ce-Zr; Ce-Zr	Ce-Zr; Ce-Zr	5.6	6.3	6.0	6.5
210-217	Ce-Zr; Ce-Zr	Ce-Zr; Ce-Zr	5.8	7.0	6.9	4.7
227-235	Ce-Zr; Ce-Zr	Ce-Zr; Ce-Zr	5.8	7.4	5.7	5.0

No Clinton plant runs have been made with fission products, and with ammonium fluosilicate but without scavengers in both of the cycles. However, based on the results obtained to date it appears that the omission of scavengers but inclusion of ammonium fluosilicate will give decontamination factors of greater than 10^5 before the cross-over and greater than 10^7 after the cross-over. Thus the comparison of runs with Ce-Zr; Ce in the 2nd cycle vs those with $(NH_4)_2SiF_6$ alone in the 2nd cycle (Ce-Zr; Ce in the first cycle of both series) favors the complexing agent. (dF was 5.3 on both at end of 2nd cycle, 7.1 with $(NH_4)_2SiF_6$ vs. 6.7 with Ce-Zr; Ce at end of the cross-over.) A similar comparison with Ce-Zr; Ce-Zr scavengers but in this case with $(NH_4)_2SiF_6$ also in the first cycle when omitting the scavenger from the second, does not give as clear-cut a case, but still favors the complexing agent. (dF's were 5.7 and 7.4 with $(NH_4)_2SiF_6$ in 2 cycles, Ce-Zr; Ce-Zr in one vs. 5.74 and 7.2 with Ce-Zr; Ce-Zr in both cycles.) Since the improvement does not show up until after the cross-over it appears that ammonium fluosilicate is more effective in removing specific activities which are not readily removed in the cross-over than are the scavengers. Unfortunately this point cannot be checked by analyses for specific fission products since the activity after the cross-over is too low. Since all laboratory comparisons of scavengers vs. ammonium fluosilicate were only carried through the two cycles and since laboratory centrifuging tends to increase the effectiveness of scavengers, but not of ammonium fluosilicate, it is not inconsistant that laboratory results favor the scavengers.

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In comparing the decontamination results the effect of cooling time has not been considered. This is permissible since the elements which control decontamination, Cb, Zr and La, make up the same fraction of total gamma activity at 60 days (77%)* as at 30 days (73%).*

Actual measurements have been made on radiation during the product isolation steps and during the cross-over steps to check on the decontamination required at Hanford when processing material at full product levels. A factor of 10^7 at the end of the cross-over seems to be adequate, but not overly conservative. At the end of the two decontamination cycles, the greatest radiation is obtained in the vicinity of the 10N HNO_3 solution. Readings in the vicinity of this tank corrected to the case where the operator is at a distance of ten feet and is protected by 1 foot of concrete indicate that a factor of only 1.5×10^4 is required to reduce radiation to .01 mr/8 hours. Under these conditions an operator could spend 5 minutes within 1 foot of the tank when it contains a charge without exceeding his daily dose. This has a bearing on the choice of decontamination procedure since it indicates that the factor of 10^7 at the end of the cross-over should be controlling and 10^5 at the end of the 2 cycles is not a justifiable limit.

Summarizing, it is certain that waste losses can be decreased appreciably by omitting scavengers and it appears very likely that adequate decontamination (greater than 10^7 after the cross-over) can be obtained with ammonium fluosilicate alone.

W Q Smith

W. Q. Smith

WQS/mew

*From data by Lane and Brady CC 829.

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