HW-24898

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July 1, 1952

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To: File

From: P.E. Collins - Leader
234-5 Building - Process Assistance
Separations Technology Unit
Technical Section
by R.E. Isaacson - Engineer R.E.I.

PRODUCTION TEST 234-5 SUPPLEMENT A
PLANT PROCESS EVALUATION PRECIPITATION OF
PLUTONIUM (IV) OXALATE

[redacted]

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Production Test 234-5[1] proposed the in-line evaluation of precipitating plutonium (IV) oxalate instead of plutonium (III) oxalate, the main advantage being the elimination of the excessively corrosive HI used to adjust Pu (IV) and Pu (VI) to the Pu (III) oxidation stage. Another advantage proposed was the recycle of supernates directly to the 224 Building without processing through recovery equipment.

The Pu (IV) oxalate process was adopted on March 21, 1952: the main process steps for a 160 unit batch of plutonium nitrate solution being as follows:

Item		Approx. Time	Vol. to R.C.
1.	Transfer 500 cc product and 1500 cc 6M HNO_3 Sample Can washes to Reactor.	1/4 hr.*	
2.	Add 1000 cc 6% H_2O_2 to reactor, agitate.	1/2 hr.*	
3.	Add 2000 cc 0.8M $H_2C_2O_4$, agitate, settle decant.	1-3/4 hr.*	5 liters
4.	Add 6 liters 1.5M HNO_3-0.05M $H_2C_2O_4$, agitate, settle, decant.	3/4 hr.*	6
5.	Add 6 liters 1.5M HNO_3-0.05M $H_2C_2O_4$, agitate settle, decant.	3/4 hr.*	6
6.	Add 6 liters 1.5M HNO_3-0.05M $H_2C_2O_4$, agitate settle, decant.	3/4 hr.*	6
7.	Transfer Pu $(C_2O_4)_2$ slurry to TF.	1/2 hr.*	
8.	Transfer Pu $(C_2O_4)_2$ to fluorination boat.	1/2 hr.*	
	TOTALS (approximate)	5-3/4 hrs.*	23 liters**

* All times indicated are approximations and vary from run to run.
** The decanted supernatant solutions from two batches (46 l.) are charged directly to a Recycle Can for shipment to 224 Building.

(1) W.B. Kerr and J.M. Hay, "Production Test 234-5 Plant Process Evaluation Precipitation of Plutonium (IV) Oxalate", Secret Document No. HW-23203, (1-8-52).

[redacted]

A preliminary investigation indicates the product content of the supernatant solutions to be greater than that reported by laboratory analysis. Dissolution of solids, present as a result of either the decantation process or post-precipitation, should increase the accuracy of sampling and analyses.

This supplement proposes that 50 runs be processed to include:

1. Omission of one acid wash (Item 6 page 2).
2. Combining three batches of supernates in one Recycle Can.
3. Addition of potassium permanganate or other oxidizing reagents to dissolve solids.

Advantages expected to be derived from the above proposals are:

1. A decrease in purification time of approximately 45 minutes to 1 hour per 160 unit batch.
2. Approximately 25% decrease in recycled supernate volumes which will result in a 33% decrease in number of Recycle Cans handled.
3. More accurate determination of product content in supernates.

Purity of product will theoretically be adequate after one of the 6 liter acid washes is omitted. Impurities that are subject to removal by washing will be theoretically decreased by a factor of ca. $1.25X10^{-3}$ when using two acid washes; using 3 acid washes the factor is ca. $1.4X10^{-4}$. Recent AT analyses indicate a maximum sodium impurity of 40,000 ppm, this should be reduced to ca. 50 ppm by two acid washes. Other elements were not reported in excess of 5,000 ppm.

All responsibilities will remain as outlined in Production Test 234-5.

A B-1 Sample of all material processed during this test program will be submitted to the control laboratory for a complete spectro-chemical analysis.

The test shall be completed in one month and the write-up will be included in the final report of Production Test 234-5.

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Approvals

234-5 Process Discussion Committee

T. Prudich — Separation Section, Manufacturing Department — 7-2-52 Date

V. R. Cooper — Separations Technology Unit, Engineering Department — 7/3/52 Date

200 Areas Process Committee

R. B. Richards — Separations Technology Unit, Engineering Department — 7/9/52 Date

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Date issued 7-15-52